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Application Data Sheet 37 CFR 1.76	Attorney Docket Number	02074-24012.NP.CIP
	Application Number	
Title of Invention	CMP PAD DRESSER HAVING LEVELED TIPS AND ASSOCIATED METHODS	

The application data sheet is part of the provisional or nonprovisional application for which it is being submitted. The following form contains the bibliographic data arranged in a format specified by the United States Patent and Trademark Office as outlined in 37 CFR 1.76.
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Secrecy Order 37 CFR 5.2

☐ Portions or all of the application associated with this Application Data Sheet may fall under a Secrecy Order pursuant to 37 CFR 5.2 (Paper filers only. Applications that fall under Secrecy Order may not be filed electronically.)

Inventor Information:

Inventor 1 Remove

Legal Name

Prefix	Given Name	Middle Name	Family Name	Suffix
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Residence Information (Select One) ○ US Residency ◉ Non US Residency ○ Active US Military Service

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All Inventors Must Be Listed - Additional Inventor Information blocks may be generated within this form by selecting the **Add** button. Add

Correspondence Information:

Enter either Customer Number or complete the Correspondence Information section below.
For further information see 37 CFR 1.33(a).

☐ **An Address is being provided for the correspondence Information of this application.**

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Application Information:

Title of the Invention	CMP PAD DRESSER HAVING LEVELED TIPS AND ASSOCIATED METHODS		
Attorney Docket Number	02074-24012.NP.CIP	**Small Entity Status Claimed** ☐	
Application Type	Nonprovisional		
Subject Matter	Utility		
Suggested Class (if any)		**Sub Class (if any)**	
Suggested Technology Center (if any)			
Total Number of Drawing Sheets (if any)	3	**Suggested Figure for Publication (if any)**	

Publication Information:

☐ Request Early Publication (Fee required at time of Request 37 CFR 1.219)

☐ **Request Not to Publish.** I hereby request that the attached application not be published under 35 U.S.C. 122(b) and certify that the invention disclosed in the attached application **has not and will not** be the subject of an application filed in another country, or under a multilateral international agreement, that requires publication at eighteen months after filing.

Representative Information:

Representative information should be provided for all practitioners having a power of attorney in the application. Providing this information in the Application Data Sheet does not constitute a power of attorney in the application (see 37 CFR 1.32).
Either enter Customer Number or complete the Representative Name section below. If both sections are completed the customer Number will be used for the Representative Information during processing.

Please Select One:	◉ Customer Number	○ US Patent Practitioner	○ Limited Recognition (37 CFR 11.9)
Customer Number	20551		

Domestic Benefit/National Stage Information:

This section allows for the applicant to either claim benefit under 35 U.S.C. 119(e), 120, 121, or 365(c) or indicate National Stage entry from a PCT application. Providing this information in the application data sheet constitutes the specific reference required by 35 U.S.C. 119(e) or 120, and 37 CFR 1.78.

Prior Application Status	Pending		Remove
Application Number	Continuity Type	Prior Application Number	Filing Date (YYYY-MM-DD)
	Continuation in part of	13479148	2012-05-23
Prior Application Status	Expired		Remove
Application Number	Continuity Type	Prior Application Number	Filing Date (YYYY-MM-DD)
13479148	non provisional of	61489074	2011-05-23

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Additional Domestic Benefit/National Stage Data may be generated within this form by selecting the **Add** button. Add

Foreign Priority Information:

This section allows for the applicant to claim benefit of foreign priority and to identify any prior foreign application for which priority is not claimed. Providing this information in the application data sheet constitutes the claim for priority as required by 35 U.S.C. 119(b) and 37 CFR 1.55(a).

Remove

Application Number	Country i	Filing Date (YYYY-MM-DD)	Priority Claimed
			◯ Yes ◉ No

Additional Foreign Priority Data may be generated within this form by selecting the **Add** button. Add

Authorization to Permit Access:

☒ Authorization to Permit Access to the Instant Application by the Participating Offices

If checked, the undersigned hereby grants the USPTO authority to provide the European Patent Office (EPO), the Japan Patent Office (JPO), the Korean Intellectual Property Office (KIPO), the World Intellectual Property Office (WIPO), and any other intellectual property offices in which a foreign application claiming priority to the instant patent application is filed access to the instant patent application. See 37 CFR 1.14(c) and (h). This box should not be checked if the applicant does not wish the EPO, JPO, KIPO, WIPO, or other intellectual property office in which a foreign application claiming priority to the instant patent application is filed to have access to the instant patent application.

In accordance with 37 CFR 1.14(h)(3), access will be provided to a copy of the instant patent application with respect to: 1) the instant patent application-as-filed; 2) any foreign application to which the instant patent application claims priority under 35 U.S.C. 119(a)-(d) if a copy of the foreign application that satisfies the certified copy requirement of 37 CFR 1.55 has been filed in the instant patent application; and 3) any U.S. application-as-filed from which benefit is sought in the instant patent application.

In accordance with 37 CFR 1.14(c), access may be provided to information concerning the date of filing this Authorization.

Applicant Information:

Providing assignment information in this section does not substitute for compliance with any requirement of part 3 of Title 37 of CFR to have an assignment recorded by the Office.

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Applicant 1

If the applicant is the inventor (or the remaining joint inventor or inventors under 37 CFR 1.45), this section should not be completed. The information to be provided in this section is the name and address of the legal representative who is the applicant under 37 CFR 1.43; or the name and address of the assignee, person to whom the inventor is under an obligation to assign the invention, or person who otherwise shows sufficient proprietary interest in the matter who is the applicant under 37 CFR 1.46. If the applicant is an applicant under 37 CFR 1.46 (assignee, person to whom the inventor is obligated to assign, or person who otherwise shows sufficient proprietary interest) together with one or more joint inventors, then the joint inventor or inventors who are also the applicant should be identified in this section.

Remove

○ Assignee	○ Legal Representative under 35 U.S.C. 117
○ Person to whom the inventor is obligated to assign.	○ Person who shows sufficient proprietary interest

If applicant is the legal representative, indicate the authority to file the patent application, the inventor is:

Name of the Deceased or Legally Incapacitated Inventor :

If the Assignee is an Organization check here. ☐

Prefix	**Given Name**	Middle Name	**Family Name**	Suffix

Mailing Address Information:

Address 1			
Address 2			
City		**State/Province**	
Country [i]		Postal Code	
Phone Number		Fax Number	
Email Address			

Additional Applicant Data may be generated within this form by selecting the Add button. Add

Signature:

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NOTE: This form must be signed in accordance with 37 CFR 1.33. See 37 CFR 1.4 for signature requirements and certifications

Signature	/David W. Osborne/			Date (YYYY-MM-DD)	2013-03-13
First Name	David W.	Last Name	Osborne	Registration Number	44989

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This collection of information is required by 37 CFR 1.76. The information is required to obtain or retain a benefit by the public which is to file (and by the USPTO to process) an application. Confidentiality is governed by 35 U.S.C. 122 and 37 CFR 1.14. This collection is estimated to take 23 minutes to complete, including gathering, preparing, and submitting the completed application data sheet form to the USPTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, U.S. Department of Commerce, P.O. Box 1450, Alexandria, VA 22313-1450. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. **SEND TO: Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450.**

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The Privacy Act of 1974 (P.L. 93-579) requires that you be given certain information in connection with your submission of the attached form related to a patent application or patent. Accordingly, pursuant to the requirements of the Act, please be advised that: (1) the general authority for the collection of this information is 35 U.S.C. 2(b)(2); (2) furnishing of the information solicited is voluntary; and (3) the principal purpose for which the information is used by the U.S. Patent and Trademark Office is to process and/or examine your submission related to a patent application or patent. If you do not furnish the requested information, the U.S. Patent and Trademark Office may not be able to process and/or examine your submission, which may result in termination of proceedings or abandonment of the application or expiration of the patent.

The information provided by you in this form will be subject to the following routine uses:

1. The information on this form will be treated confidentially to the extent allowed under the Freedom of Information Act (5 U.S.C. 552) and the Privacy Act (5 U.S.C. 552a). Records from this system of records may be disclosed to the Department of Justice to determine whether the Freedom of Information Act requires disclosure of these records.

2. A record from this system of records may be disclosed, as a routine use, in the course of presenting evidence to a court, magistrate, or administrative tribunal, including disclosures to opposing counsel in the course of settlement negotiations.

3. A record in this system of records may be disclosed, as a routine use, to a Member of Congress submitting a request involving an individual, to whom the record pertains, when the individual has requested assistance from the Member with respect to the subject matter of the record.

4. A record in this system of records may be disclosed, as a routine use, to a contractor of the Agency having need for the information in order to perform a contract. Recipients of information shall be required to comply with the requirements of the Privacy Act of 1974, as amended, pursuant to 5 U.S.C. 552a(m).

5. A record related to an International Application filed under the Patent Cooperation Treaty in this system of records may be disclosed, as a routine use, to the International Bureau of the World Intellectual Property Organization, pursuant to the Patent Cooperation Treaty.

6. A record in this system of records may be disclosed, as a routine use, to another federal agency for purposes of National Security review (35 U.S.C. 181) and for review pursuant to the Atomic Energy Act (42 U.S.C. 218(c)).

7. A record from this system of records may be disclosed, as a routine use, to the Administrator, General Services, or his/her designee, during an inspection of records conducted by GSA as part of that agency's responsibility to recommend improvements in records management practices and programs, under authority of 44 U.S.C. 2904 and 2906. Such disclosure shall be made in accordance with the GSA regulations governing inspection of records for this purpose, and any other relevant (i.e., GSA or Commerce) directive. Such disclosure shall not be used to make determinations about individuals.

8. A record from this system of records may be disclosed, as a routine use, to the public after either publication of the application pursuant to 35 U.S.C. 122(b) or issuance of a patent pursuant to 35 U.S.C. 151. Further, a record may be disclosed, subject to the limitations of 37 CFR 1.14, as a routine use, to the public if the record was filed in an application which became abandoned or in which the proceedings were terminated and which application is referenced by either a published application, an application open to public inspections or an issued patent.

9. A record from this system of records may be disclosed, as a routine use, to a Federal, State, or local law enforcement agency, if the USPTO becomes aware of a violation or potential violation of law or regulation.

CMP PAD DRESSER HAVING LEVELED TIPS AND ASSOCIATED METHODS

PRIORITY DATA

This application is a continuation-in-part of 13/479,148, filed on May 23, 2012, which claims the benefit of United States Provisional Patent Application Serial no. 61/489,074, filed on May 23, 2011, each of which are incorporated herein by reference.

BACKGROUND OF THE INVENTION

The semiconductor industry currently spends in excess of one billion U.S. dollars each year manufacturing silicon wafers that must exhibit very flat and smooth surfaces. Known techniques to manufacture smooth and even-surfaced silicon wafers are plentiful. The most common of these involves the process known as Chemical Mechanical Polishing (CMP) which includes the use of a polishing pad in combination with an abrasive slurry. Of central importance in all CMP processes is the attainment of high performance levels in aspects such as uniformity of polished wafer, smoothness of the IC circuitry, removal rate for productivity, longevity of consumables for CMP economics, etc.

SUMMARY OF THE INVENTION

The present disclosure provides efficient methods of conditioning a CMP pad. One such method can include pressing a CMP pad dresser against a CMP pad, where the dresser includes a monolayer of a plurality of superabrasive particles protruding from a matrix layer. The difference in protrusion distance between the highest protruding tip and the second highest protruding tip of the monolayer of superabrasive particles is less than or equal to about 10 microns and the difference in protrusion distance between the highest 10 protruding tips of the monolayer of superabrasive particles are within about 20 microns or less. The method can further include rotating the dresser against the CMP pad such that asperities are cut into the CMP pad having a maximum cutting depth of about 60 microns. In another aspect, at least 100 tips of the plurality of superabrasive particles cut asperities into the CMP pad as the dresser is rotated. In another aspect, at least 50 tips of

the plurality of superabrasive particles cut asperities into the CMP pad as the dresser is rotated. In a further aspect, at least 25 tips of the plurality of superabrasive particles cut asperities into the CMP pad as the dresser is rotated.

Furthermore, in one aspect substantially all of the asperities are cut in the CMP pad by superabrasive particles located at a peripheral location of the dresser. In one specific aspect, the peripheral location extends from an outer edge of the dresser inwardly toward a center point of the dresser for up to about 90% of the dresser radius. In another specific aspect, the peripheral location extends from an outer edge of the dresser inwardly toward a center point of the dresser for up to about 50% of the dresser radius. In yet another specific aspect, the peripheral location extends from an outer edge of the dresser inwardly toward a center point of the dresser for up to about 33% of the dresser radius. Additionally, in some aspects substantially all of the superabrasive particles are located at a peripheral location of the dresser. In another aspect, substantially all of the superabrasive particles are arranged in uniform predetermined pattern at a peripheral location of the dresser.

The method can further include interactions between the CMP pad and a workpiece being polished. For example, in one aspect the method can include rotating the CMP pad, applying a slurry to a polishing surface of the rotating CMP pad, and applying a workpiece to the polishing surface such that the workpiece is polished by the asperities in combination with the slurry. In one aspect, the workpiece is a semiconductor device. In another aspect, the semiconductor device includes one or more layers of Cu, W, an oxide layer, or a combination thereof. In yet another aspect, the semiconductor device can have a node size of less than or equal to 28 nm. In a further aspect, the semiconductor device can have a line width of less than or equal to about 30 nm. In yet a further aspect, the semiconductor device can have a wafer having a size greater than 400 mm. Furthermore, in some aspects the slurry can include an abrasive such as silica, alumina, ceria, and combinations thereof. In other apsects, the slurry can include an oxidative compound.

There has thus been outlined, rather broadly, various features of the invention so that the detailed description thereof that follows may be better understood, and so that the

present contribution to the art may be better appreciated. Other features of the present invention will become clearer from the following detailed description of the invention, taken with the accompanying claims, or may be learned by the practice of the invention.

BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 is a schematic side view of a CMP pad dresser in accordance with an embodiment of the present invention;

FIG. 2 is a flow chart diagram showing steps of a method of dressing a CMP pad in accordance with an embodiment of the present invention;

FIG. 3 is a flow chart diagram showing steps of a method of dressing a CMP pad in accordance with an embodiment of the present invention;

FIG. 4 is a schematic side view of a CMP pad dresser in accordance with an embodiment of the present invention;

FIG. 5 is a schematic side view of a CMP pad dresser in accordance with an embodiment of the present invention;

FIG. 6 is a schematic side view of a CMP pad dresser in accordance with an embodiment of the present invention; and

FIG. 7 is a schematic side view of a CMP pad dresser in accordance with an embodiment of the present invention.

It will be understood that the above figures are merely for illustrative purposes in furthering an understanding of the invention. Further, the figures are not drawn to scale, thus dimensions, particle sizes, and other aspects may, and generally are, exaggerated to make illustrations thereof clearer. Therefore, departure can be made from the specific dimensions and aspects shown in the figures in order to produce the heat spreaders of the present invention.

DETAILED DESCRIPTION

Before the present invention is disclosed and described, it is to be understood that this invention is not limited to the particular structures, method steps, or materials disclosed herein, but is extended to equivalents thereof as would be recognized by those ordinarily skilled in the relevant arts. It should also be understood that terminology employed herein is used for the purpose of describing particular embodiments only and is not intended to be limiting.

It must be noted that, as used in this specification and the appended claims, the singular forms "a," "an" and, "the" include plural referents unless the context clearly dictates otherwise. Thus, for example, reference to "a diamond particle" includes one or more of such particles and reference to "the layer" includes reference to one or more of such layers.

Definitions

In describing and claiming the present invention, the following terminology will be used in accordance with the definitions set forth below.

As used herein, the terms "conditioner" and "dresser" can be used interchangeably, and refer to a tool used to condition or dress a pad, such as a CMP pad.

As used herein, "dressing segment" refers to a dressing or conditioning element of a CMP pad dresser. Dressing segments are utilized in the present invention to carry superabrasive particles having leveled tips. Thus superabrasive particles are introduced into a CMP pad dresser by the incorporation of multiple dressing segments. It should be noted that a variety of techniques of attaching the dressing segments to the substrates, and a variety of techniques of attaching the superabrasive particles to the dressing segments, are discussed herein. It is to be understood that all of these various attachment mechanisms can be used interchangeably herein: that is, if a method of attaching a dressing segment to a substrate is discussed herein, the method of attachment discussed can also be used to attach a superabrasive particles to a dressing segment. For any particular CMP pad dresser being discussed, however, it is understood that attachment methods of the superabrasive particles to the dressing segments can differ from, or can be the same as, the method used to attach the dressing segments to the pad conditioner substrate.

As used herein, "superabrasive" may be used to refer to any crystalline, or polycrystalline material, or mixture of such materials which has a Mohr's hardness of about 8 or greater. In some aspects, the Mohr's hardness may be about 9.5 or greater. Such materials include but are not limited to diamond, polycrystalline diamond (PCD), cubic boron nitride (cBN), polycrystalline cubic boron nitride (PcBN), corundum and sapphire, as well as other superabrasive materials known to those skilled in the art. Superabrasive materials may be incorporated into the present invention in a variety of forms including particles, grits, films, layers, pieces, segments, etc. In some cases, the superabrasive materials of the present invention are in the form of polycrystalline superabrasive materials, such as PCD and PcBN materials.

As used herein, "organic material" refers to a semisolid or solid complex or mix of organic compounds. "Organic material layer" and "organic matrix" may be used interchangeably, and refer to a layer or mass of a semisolid or solid complex or mix of organic compounds, including resins, polymers, gums, etc. The organic material can be a polymer or copolymer formed from the polymerization of one or more monomers. In some cases, such organic material can be adhesive.

As used herein, the process of "brazing" is intended to refer to the creation of chemical bonds between the carbon atoms of the superabrasive particles/materials and the braze material. Further, "chemical bond" means a covalent bond, such as a carbide or boride bond, rather than mechanical or weaker inter-atom attractive forces. Thus, when "brazing" is used in connection with superabrasive particles a true chemical bond is being formed. However, when "brazing" is used in connection with metal to metal bonding the term is used in the more traditional sense of a metallurgical bond. Therefore, brazing of a superabrasive segment to a tool body does not necessarily require the presence of a carbide former.

As used herein, "particle" is as used herein in connection with diamond particles, and refers to a particulate form of diamond. Such particles may take a variety of shapes, including round, oblong, square, euhedral, etc., can be either single crystal or polycrystalline, and can have a number of mesh sizes. As is known in the art, "mesh"

refers to the number of holes per unit area as in the case of U.S. meshes. All mesh sizes referred to herein are U.S. mesh unless otherwise indicated. Further, mesh sizes are generally understood to indicate an average mesh size of a given collection of particles since each particle within a particular "mesh size" may actually vary over a small distribution of sizes.

As used herein, "sharp portion" means any narrow portion to which a crystal or particle may come, including but not limited to corners, apexes, ridges, edges, obelisks, and other protrusions. In some cases, corners and/or apexes are formed at the convergence of more than two faces of the particle or crystal, or other body, disposed in intersecting planes. In other aspects, edges and/or ridges may be formed at the convergence of two or at least two faces of the particle or crystal, or other body, disposed in intersecting planes. In some aspects, "sharp portion" can include a portion that is broken, chipped, cracked, jagged, and the like. In some aspects, degrees of sharpness can be correlated with the geometric angle formed by the particle, crystal, or other body, at the location in question. In some aspects, an angle of 90 degrees or less can be considered sharp. In other aspects, an angle of 60 degrees or less can be considered sharp. In yet other aspects, and angle of 45 degrees or less, or 30 degrees or less can be considered to be sharp.

As used herein, "peripherally located," "peripheral location" and the like, mean any particle of a dresser that is located in an area that originates at the leading edge or outer rim of a dresser and extends inwardly towards the center for up to about 90% of the radius of the dresser. In some aspects, the area may extend inwardly from about 20% to 90% of the radius. In other aspects, the area may extend in to about 50% of the radius. In yet another aspect, the area may extend in to about 33% of the radius of a dresser (i.e. 66% away from the center).

As used herein, "working superabrasive particles" are superabrasive particles that touch a CMP pad during a dressing or conditioning procedure. This touching can remove debris from the surface, it can deform the surface either elastically or plastically, or it can cut the surface to create a groove. In one specific aspect, a working superabrasive particle

can cut deeper than about 10 microns into a CMP pad during a dressing procedure.

As used herein, "non-working superabrasive particles" are superabrasive particles in a CMP pad dresser that do not significantly touch the pad sufficient to remove debris from the surface, deform the surface, cut the surface to create a groove.

As used herein, "overly-aggressive superabrasive particles" are superabrasive particles in a CMP pad dresser that aggressively dress or condition a CMP pad. In one aspect, aggressive superabrasive particles are superabrasive particles that cut deeper than about 50 microns into a CMP pad during a dressing procedure. In another aspect, aggressive superabrasive particles are superabrasive particles that remove at least 1/5 of the material from the CMP pad. In yet another aspect, aggressive superabrasive particles are superabrasive particles that remove at least 1/2 of the material from the CMP pad.

As used herein, "indicator substrate" refers to a substrate material upon which a portion of the superabrasive particles of a CMP pad dresser can be positioned and moved to make markings indicative of working superabrasive particles.

As used herein, "marking pattern" refers to a pattern on an indicator substrate created by moving superabrasive particles thereacross. The markings can be any detectable marking known, including cuts, scratches, depressions, material deposition (e.g. pigment markers, chemical markers, fluorescent markers, radioactive markers, etc.).

As used herein, "transverse" refers to a directional orientation that is cross-wise to a reference axis. In one aspect, "transverse" can include a directional orientation that is at least at a substantial right angle to the reference axis.

As used herein, "alignment orientation direction" refers to the direction of an alignment axis of the plurality of superabrasive particles. For example, a plurality of superabrasive particles aligned in a grid formation would have at least two alignment axes; an alignment axis in the column direction and an alignment axis in the row direction oriented 90° to the column direction.

As used herein, "ablate" or "ablating" refer to a process of removing a superabrasive particle from a CMP pad dresser or reducing the projection of a

superabrasive particle thus reducing the degree of contact between the superabrasive particle and the indicator substrate.

As used herein, "superabrasive segment" refers to a tool body having multiple superabrasive particles associated therewith. In some aspect, a superabrasive segment can include superabrasive polycrystalline materials as cutting elements.

As used herein, a "tool substrate" refers a portion of a pad conditioner that supports abrasive materials, and to which abrasive materials and/or superabrasive segments that carry abrasive materials may be affixed. Substrates useful in the present invention may of a variety of shapes, thicknesses, or materials that are capable of supporting abrasive materials in a manner that is sufficient to provide a pad conditioner useful for its intended purpose. Substrates may be of a solid material, a powdered material that becomes solid when processed, or a flexible material. Examples of typical substrate materials include without limitation, metals, metal alloys, ceramics, relatively hard polymers or other organic materials, glasses, and mixtures thereof. Further, the substrate may include a material that aids in attaching abrasive materials to the substrate, including, without limitation, brazing alloy material, sintering aids and the like.

As used herein, "working end" refers to an end of a particle which is oriented towards the CMP pad and during a dressing operation makes contact with the pad. Most often the working end of a particle will be distal from a substrate to which the particle is attached.

As used herein, "attitude" means the position or arrangement of a superabrasive particle in relation to a defined surface, such as a substrate to which it is attached, or a CMP pad to which it is to be applied during a work operation. For example, a superabrasive particle can have an attitude that provides a specific portion of the particle in orientation toward a CMP pad.

As used herein, "sintering" refers to the joining of two or more individual particles to form a continuous solid mass. The process of sintering involves the consolidation of particles to at least partially eliminate voids between particles.

The term "metallic" refers to both metals and metalloids. Metals include those compounds typically considered metals found within the transition metals, alkali and alkali earth metals. Examples of metals are Ag, Au, Cu, Al, and Fe. Metalloids include specifically Si, B, Ge, Sb, As, and Te. Metallic materials also include alloys or mixtures that include metallic materials. Such alloys or mixtures may further include additional additives. In the present invention, carbide formers and carbon wetting agents may be included as alloys or mixtures, but are not anticipated to be the only metallic component. Examples of such carbide formers are Sc, Y, Ti, Zr, Hf, V, Nb, Cr, Mo, Mn, Ta, W, and Tc. Examples of carbon wetting agents are Co, Ni, Mn, and Cr.

As used herein, "infiltrating" refers to a situation where a material is heated to its melting point and then flows as a liquid through the interstitial voids between particles.

As used herein, the term "substantially" refers to the complete or nearly complete extent or degree of an action, characteristic, property, state, structure, item, or result. For example, an object that is "substantially" enclosed would mean that the object is either completely enclosed or nearly completely enclosed. The exact allowable degree of deviation from absolute completeness may in some cases depend on the specific context. However, generally speaking the nearness of completion will be so as to have the same overall result as if absolute and total completion were obtained.

The use of "substantially" is equally applicable when used in a negative connotation to refer to the complete or near complete lack of an action, characteristic, property, state, structure, item, or result. For example, a composition that is "substantially free of" particles would either completely lack particles, or so nearly completely lack particles that the effect would be the same as if it completely lacked particles. In other words, a composition that is "substantially free of" an ingredient or element may still actually contain such item as long as there is no measurable effect thereof.

As used herein, the term "about" is used to provide flexibility to a numerical range endpoint by providing that a given value may be "a little above" or "a little below" the endpoint.

As used herein, a plurality of items, structural elements, compositional elements,

and/or materials may be presented in a common list for convenience. However, these lists should be construed as though each member of the list is individually identified as a separate and unique member. Thus, no individual member of such list should be construed as a de facto equivalent of any other member of the same list solely based on their presentation in a common group without indications to the contrary.

Concentrations, amounts, and other numerical data may be expressed or presented herein in a range format. It is to be understood that such a range format is used merely for convenience and brevity and thus should be interpreted flexibly to include not only the numerical values explicitly recited as the limits of the range, but also to include all the individual numerical values or sub-ranges encompassed within that range as if each numerical value and sub-range is explicitly recited. As an illustration, a numerical range of "about 1 to about 5" should be interpreted to include not only the explicitly recited values of about 1 to about 5, but also include individual values and sub-ranges within the indicated range. Thus, included in this numerical range are individual values such as 2, 3, and 4 and sub-ranges such as from 1-3, from 2-4, and from 3-5, etc., as well as 1, 2, 3, 4, and 5, individually. This same principle applies to ranges reciting only one numerical value as a minimum or a maximum. Furthermore, such an interpretation should apply regardless of the breadth of the range or the characteristics being described.

The Invention

The present disclosure generally provides CMP pad dressers, methods associated with pad conditioning (e.g., smoothing, polishing, dressing), and the CMP polishing of a work piece. Pad conditioners of the present invention can be advantageously utilized, for example, in dressing CMP pads that are used in polishing, finishing or otherwise affecting semiconductor materials. Specifically, the present disclosure concerns CMP pad dressers having superabrasive particles with substantially leveled tips. Traditional CMP pad dresser manufacturing methods, even many of those describing techniques for leveling superabrasive particle tips prior to fixation, generally contain significant variation in tip height across the surface of the dresser. Often, the superabrasive particles are affixed to

the CMP pad dresser support in a manner that disrupts any leveling that has occurred. For example, fixation techniques that utilize high heat and/or pressure can cause warping of the dresser support as the dresser cools. Thus, unless steps are taken to avoid such warpage, superabrasive particles are not maintained in their leveled state following cooling of the dresser. This can be particularly problematic with brazing techniques.

A CMP pad dresser can be used to dress or condition a CMP pad by removing dirt and debris (i.e. deglazing the pad), as well as opening up asperities in the pad surface to capture and hold chemical slurry during a polishing procedure. As has been described, due to difficulties associated with superabrasive particle leveling in many traditional manufacturing processes, only a small percentage of superabrasive particles in a typical CMP pad dresser are positioned so as to penetrate or cut into a CMP pad. As this small percentage of superabrasive particles become worn, plastic deformation of the CMP pad becomes large relative to the amount CMP of pad that is cut. Consequently, the pad becomes highly deformed and accumulated with dirt. As a result the polishing rate of the CMP pad declines, and the scratch rate of the wafer or work piece increases.

CMP pads are typically made of a relatively soft polymer, such as polyurethane. As the CMP pad is engaged by the CMP pad dresser, the polymer material is deformed first by elastic strain and then by plastic strain. Eventually, the strain energy in the deformed material exceeds the bond energy density (i.e. the hardness of the pad) and the polymer material ruptures. Thus, the function of superabrasive particles in the CMP pad dresser is to dress the CMP pad material by breaking polymeric bonds through this deformation process. It should be noted that sharp superabrasive particle tips can penetrate the CMP pad material without causing excessive deformation. As such, the sharpness of a superabrasive particle can be defined as being inverse to the deformed volume prior to rupture. In other words, the smaller the volume of deformation prior to cutting, the sharper the cutting tip. This deformation information can be used to determine the sharpness of superabrasive particles in the CMP pad dresser.

Additionally, a superabrasive particle having a tip with smaller tip radius, such as would be the case with a broken corner, can cut more cleanly through the CMP pad with

less deformation as compared to a superabrasive particle having a larger tip radius. Consequently, an irregularly shaped superabrasive particle tip can be sharper than a euhedral superabrasive corner having an obtuse angle relative to the CMP pad. This also applies to the difference between a superabrasive particle corner as compared with a superabrasive particle face.

It is thus noted that sharp superabrasive particle tips can cut CMP pad materials with less deformation and material strain. Conversely, a dull superabrasive particle may deform but not cut the CMP pad material because the strain energy does not exceed the bond energy density of the polymeric material. As the tips of such particles are worn, the contact area between the polymeric material and the particles increase. This increase in contact area results in an increase in the deformation volume of the pad. Due to the increased strain energy required for the polymeric material to rupture with such an increased deformation volume, the number of superabrasive particles cutting the polymeric material will decrease in relation to the degree of dulling during a CMP process.

CMP pad dressing can also be affected by the proportion of superabrasive particles in the CMP pad dresser that are working and the proportion that are overly-aggressively cutting. As an example, a typical CMP pad dresser can have greater than 10,000 superabrasive particles. Of these 10, 000 particles, in some cases there may only be a few working superabrasive particles that are actually able to cut the CMP pad. Additionally, out of these few working superabrasive particles, there may be a smaller proportion of overly-aggressive superabrasive particles that cut over 50% of the entire pad that is consumed during conditioning, and in some cases can remove more that 25% of the total pad material. This uneven work load distribution can cause erratic CMP performance, and can result in over consumption of the CMP pad, chipping of the overly-aggressive superabrasive particles that can scratch the wafer, unpredictable wafer removal rates, uneven wafer surface planarization, shortened CMP pad dresser life, compaction of the CMP pad with debris, and the like. Such effects can greatly lower the yield of wafers or work pieces processed under these conditions.

The present disclosure seeks to more effectively normalize the work load

distribution across the surface of the CMP pad dresser in order to more uniformly deglaze the pad and create more uniform pad asperities. The present CMP pad dressers include a layer of superabrasive particles having substantially leveled tips across the working surface of the finished CMP pad dresser. A variety of techniques can be utilized to maintain tip leveling, and any such technique is considered to be within the present scope. A few non-limiting examples of such techniques are described below.

In one aspect, as is shown in FIG. 1, a CMP pad dresser 100 includes a monolayer of a plurality of superabrasive particles 102 embedded in a matrix layer 104 such that each superabrasive particle in the monolayer protrudes from the matrix layer 104. The difference in protrusion distance 106 between the highest protruding tip 108 and the next highest protruding tip 110 of the monolayer of superabrasive particles is described as the protrusion difference. In one aspect, the protrusion difference can be less than or equal to about 50 microns. In another aspect, the protrusion difference can be less than or equal to about 20 microns. In a further aspect, the protrusion difference can be less than or equal to about 10 microns. It is noted that the term "protrusion difference" can also be used to describe the difference in protraction distance between any two or more superabrasive particles.

In another aspect, and in addition to the protrusion difference between the highest two protruding tips, the difference in protrusion distance between the highest 10 protruding tips of the monolayer of superabrasive particles are within about 30 microns or less. In yet another aspect, the difference in protrusion distance between the highest 10 protruding tips of the monolayer of superabrasive particles are within about 20 microns or less.

Additionally, in some aspects the difference in protrusion distance between the highest 1% of the protruding tips of the first monolayer of superabrasive particles can be within about 80 microns or less. In other words, for the 1% of the plurality of superabrasive particles that have the highest protruding tips, the variance in protrusion distance for that 1% is less than or equal to about 80 microns. For example, the two highest protruding superabrasive particle tips protrude to within about 10 microns or less

of each other, and in addition, the highest protruding 1% of superabrasive particle tips protrude to within about 80 microns or less of each other. In yet another aspect, the difference in protrusion distance between the highest 1% of the protruding tips of the monolayer of superabrasive particles are within about 60 microns or less. In yet another aspect, the difference in protrusion distance between the highest 1% of the protruding tips of the monolayer of superabrasive particles are within about 40 microns or less.

It is noted that the recited protrusion distances can include a distribution across the entire monolayer surface or a discrete area or segment of the monolayer. For example, the highest 1% of protruding tips can be located around the periphery of the monolayer. In one aspect, for example, substantially all of the asperities are cut in the CMP pad by superabrasive particles located at a peripheral location of the dresser. In another aspect, the peripheral location extends from an outer edge of the dresser inwardly toward a center point of the dresser for up to about 90% of the dresser radius. In yet another aspect, the peripheral location extends from an outer edge of the dresser inwardly toward a center point of the dresser for up to about 50% of the dresser radius. In a further aspect, the peripheral location extends from an outer edge of the dresser inwardly toward a center point of the dresser for up to about 33% of the dresser radius. Additionally, in some aspects, substantially all of the superabrasive particles are located at a peripheral location of the dresser. In other aspects, substantially all of the superabrasive particles are arranged in uniform predetermined pattern at a peripheral location of the dresser.

In another example, discrete regions of leveled superabrasive particle tips can be located within a larger area of superabrasive particles having a lower protrusion distance than the leveled portion. It is also contemplated that the monolayer can include multiple regions or segments of superabrasive particles that are leveled as described, within a larger area of superabrasive particles having a lower protrusion distance.

Thus, the relative leveling of the monolayer of superabrasive particles can greatly affect the morphology and distribution of asperities cut into the CMP pad by the dresser. For example, in one aspect, the monolayer is leveled such that rotating the dresser against the CMP cuts asperities having a maximum cutting depth of about 80 microns. In another

aspect, the monolayer is leveled such that rotating the dresser against the CMP cuts asperities having a maximum cutting depth of about 60 microns. In yet another aspect, the monolayer is leveled such that rotating the dresser against the CMP cuts asperities having a maximum cutting depth of about 40 microns. By forming pointed asperities, the contact pressure between the work piece and the CMP pad is increased, thus improving the polishing characteristics of the pad. As these pointed regions begin to flatten, the contact pressure begins to decrease, and slurry located therebetween is less effectively pressed against the work piece. In some aspects it can be beneficial for the contact area between the pad and the wafer to be less than or equal to about 1% of the total wafer surface area facing the pad. In other aspects, it can be beneficial for the contact area between the pad and the wafer to be less than or equal to about 0.1% of the total wafer surface area facing the pad. This small contact area is due to the wafer being perched on the contact points of the asperities. During the polishing process, the sharp tips of the asperities become worn, thus increasing the contact area and decreasing the contact pressure to a point whereby the slurry abrasives do not effectively abrade the wafer. At this point the removal rate of material from the wafer drops, and the pad needs to be redressed to resharpen the asperities.

Additionally, the load placed on the CMP pad by the dresser can affect the morphology of the spacers, such as, in some cases, the asperity depth. The load can be adjusted for a given dresser to adjust penetration depth. In one aspect, for example, the load can be from about 0.1 Kg to about 60 Kg. In other aspects, the load can be from about 2 Kg to about 20 Kg. In further aspects, the load may be from about 6 Kg to about 12 Kg. In some aspects the load may be Kg/m2. For example, in one aspect an 11.5 Kg load can allow a material removal rate from the wafer of about 200 microns per hour for Cu layers. In a related example, an 11.5 Kg load can allow a material removal rate from the wafer of about 100 microns per hour for oxides. In many cases, a lower load can be used in order to minimize the superabrasive particle damage (e.g., less than or equal to about 2 Kg).

Furthermore, leveling of the tips of the superabrasive particles in the monolayer allows differing amounts of superabrasive particles to actually cut asperities into the pad. In one aspect, for example, the tips are leveled such that at least 100 tips of the plurality of superabrasive particles cut asperities into the CMP pad as the dresser is rotated. In another aspect, the tips are leveled such that at least 50 tips of the plurality of superabrasive particles cut asperities into the CMP pad as the dresser is rotated. In a further aspect, the tips are leveled such that at least 25 tips of the plurality of superabrasive particles cut asperities into the CMP pad as the dresser is rotated.

Various methods can be utilized to measure superabrasive particle tip height to determine the difference in protrusion distance between tips. As such, any method for making such a determination is considered to be within the present scope. It should be noted that for the purposes of the present disclosure, the term "protrusion" refers to the height of a particle relative to some reference point. Techniques for such measurements can include direct measurements of the tip heights relative to a reference point such as, for example, the highest particle tip, a surface of a rigid support, the bottom surface of the matrix, etc. Measurements of particle height from the surface of the matrix material can be problematic, however, due to the irregular nature of such materials due to wicking around the superabrasive particles. In those cases whereby the matrix material is uniform, such a surface may be used to determine particle height. Additionally, a relative protrusion or height difference between two particles would be the difference in the heights between these particles measured from a common reference point. Furthermore, in some cases the superabrasive particles may lie along a slope, curvature, or some other arrangement that is not parallel to the metal support layer. In these cases, the protrusion height would be normalized against the slope, curvature, or other arrangement so that the relative protrusion height difference between particles can be measured in the absence of the slope, curvature, etc. It should be noted that superabrasive particle tip height leveling can, in some cases, be independent from the positioning or patterning of the superabrasive particles across the surface of the dresser.

One example of a direct measurement technique can include an optical scanning process to evaluate superabrasive particle tip positions. In one such process, an optical scanner can scan the surface of the CMP pad dresser to determine the height of the superabrasive particle tips relative to a fixed point. For example, the scanner can scan downward in space toward the dresser until the highest tip is located. The highest tip can then be set to the reference point, and the scanner can continue scanning in a direction toward the dresser measuring the distance from the reference point to each superabrasive particle tip across the surface of the dresser. Accordingly, the difference in protrusion distance between all of the superabrasive particles across the dresser can be directly measured.

Furthermore, measurement techniques can also include indirect measurements, such as, for example, applying the diamond monolayer to a deformable substrate that deforms relative to the protrusion distance of the particle tips. The diamond monolayer can be pressed into the deformable substrate and/or moved across the deformable substrate to form a scratch pattern therein. Tip height can thus be extrapolated from such indirect measurements.

A variety of materials are contemplated for use as superabrasive particles. Any superabrasive known that can be utilized in a CMP pad dresser should be considered to be within the present scope. Non-limiting examples of such materials include diamond materials, nitride materials, ceramics, and the like. In one aspect, the superabrasive particles include diamond materials. Such diamond materials can include natural or synthetic diamond, single crystal, polycrystalline, and the like. In another aspect, the superabrasive particles include cubic boron nitride materials. Additionally, various diamond particles sizes can be used, including mesh sizes such as 10/20, 30/40, 80/90, 90/100, 100/120, 120/140, 140/170, 170/200, 200/230, 230/270, 270/325, and 325/400.

Additionally, the plurality the superabrasive particles can be oriented so as to affect dressing performance of a CMP pad. Orienting the superabrasive particles in a specific attitude in relation to the CMP pad to be dressed creates different asperities in the pad surface, thus altering the performance of the CMP pad. Different asperities retain slurry in

different manners and thus polish a workpiece differently according to asperity depth, width, density, etc. The superabrasive particles of a CMP pad dresser can be oriented according to the desired polishing characteristics of the CMP pad. For example, if the superabrasive particles predominantly have an apex oriented towards the CMP pad, the asperities of the pad will be narrow and deep. The advantages of narrow and deep asperities are that the pad can better retain the polishing slurry, and thus the polishing rate of the wafer increases. However, the increased polishing rate may also increase the wear rate of the superabrasive particles. As such, wear rate may vary depending on the attitude of the superabrasive particles, and therefore, the orientation of each superabrasive particle may be considered when designing a device with desired performance characteristics. Generally speaking, superabrasive particle attitudes that provide higher dressing rates (i.e. deeper penetration into a pad) also wear particles out at a higher rate.

In contrast, if the superabrasive particles are oriented with a face towards the pad, the resulting asperities may polish at a lower rate. The face of the particle is generally thought to be more durable, but does not typically cut deep and narrow asperities in the pad, but rather asperities that are shallow and broad. Therefore the face portion of a particle will dress a CMP pad at a reduced rate compared to the apex portion of a particle, but the superabrasive particle will wear at a much lower rate.

The edge portions of a superabrasive particle have dressing and wear characteristics that are between those of the face and apex portions. It has been thought that if the edge portion is utilized to dress a CMP pad, the asperities are not as deep or narrow as those dressed with an apex portion, but may provide asperities having desirable intermediate characteristics. Further, the edge portion of the particle does not wear at such a high rate as that of an apex.

In one aspect, the plurality of superabrasive particles are substantially all configured in an attitude having either an apex portion oriented toward a pad to be dressed. In another aspect, the plurality of superabrasive particles are substantially all configured in an attitude having either an edge portion oriented toward a pad to be

dressed. In yet another aspect, the plurality of superabrasive particles can be configured in an attitude having a sharp portion oriented towards a pad to be dressed.

A CMP pad dresser having such substantially leveled tip arrangements can have a low scratch rate because superabrasive particles are less likely to pull out of the matrix layer due to their more uniform protrusion distribution compared to traditional dressers. Additionally, the more uniform protrusion distributions of such a dressers allows the conditioning of CMP pads in such a manner as to facilitate good polishing rates while at the same time extending the effective working life of the dresser. These benefits can be affected by, for example, uniform asperity spacing and size distribution in the CMP pad.

In one aspect, as is shown in FIG. 2, a method of conditioning a CMP pad can include 202 pressing a CMP pad dresser against a CMP pad, 204 the dresser including a monolayer of a plurality of superabrasive particles protruding from a matrix layer, wherein the difference in protrusion distance between the highest protruding tip and the second highest protruding tip of the monolayer of superabrasive particles is less than or equal to about 10 microns and the difference in protrusion distance between the highest 10 protruding tips of the monolayer of superabrasive particles are within about 20 microns or less. The method can further include 206 rotating the dresser against the CMP pad such that asperities are cut into the CMP pad having a maximum cutting depth of about 60 microns. In another aspect, as is shown in FIG. 3, the method can further include 302 rotating the CMP pad, 304 applying a slurry to a polishing surface of the rotating CMP pad, and 306 applying a workpiece to the polishing surface such that the workpiece is polished by the asperities in combination with the slurry.

In another aspect of the present disclosure, superabrasive tip leveling can be accomplished by manufacturing the CMP pad dresser in such a way that the effects of warpage are minimized. For example, in one aspect a CMP pad dresser can be made in at least a two stage process, where the first stage is performed in a manner that minimizes perturbations of the leveled tips in a process that would normally significantly move the superabrasive tips out of a leveled arrangement, such as, for example, by brazing. The

second stage of the process can further enhance the leveling of superabrasive particle tips across the CMP pad dresser.

Various exemplary configurations of CMP pad dressers having substantially leveled tips are contemplated. In one aspect, for example, a CMP pad dresser can include a first monolayer of superabrasive particles disposed on one side of a metal support layer and a second monolayer of superabrasive particles disposed on the metal support layer on an opposite side from the first monolayer. The superabrasive particles of the second monolayer are positioned to have substantially the same distribution as the superabrasive particles of the first monolayer. A rigid support is coupled to the second monolayer of superabrasive particles opposite the first monolayer to provide support to the dresser.

As is shown in FIG. 4, a first monolayer of superabrasive particles 402 is arranged on a surface of a metal support layer 404. A second monolayer of superabrasive particles 406 is arranged on the metal support layer 404 on a side that is opposite to the first monolayer. As is discussed further below, in some cases the first and second monolayers of superabrasive particles can be arranged such that corresponding superabrasive particles across the metal support layer are in substantial alignment, as is shown in FIG. 4. In other cases, the superabrasive particles of the first and second monolayers can be arranged in a substantially similar distribution, but may or may not be substantially aligned with one another across the metal support layer.

The metal support layer can be any material capable of supporting the diamond monolayer during tool formation. Such materials can include, without limitation, metal materials, metal alloy materials, braze alloy materials, ceramic materials, composites, and the like.

In one aspect, the superabrasive particles can be arranged into a predetermined pattern. Such a pattern can be a uniformly distributed pattern or a non-uniformly distributed pattern. Additionally, a variety of techniques are contemplated to facilitate the arrangement of superabrasive particles into a predetermined pattern. Predetermined is understood to mean a non-random pattern that has been determined prior to arranging the superabrasive particles. In one aspect, a predetermined pattern can also apply to a

predetermined spacing between particles. Non-limiting examples of such techniques include arrangement by a template, arrangement using spots of adhesives, arrangement on a first substrate followed by a pattern specific transfer from the first substrate to the metal support layer, and the like, including combinations thereof. The superabrasive particles from either of the monolayers can be temporarily held in position in the predetermined pattern using a variety of techniques, including, without limitation, adhesives, dimpled locations on the metal support matrix, a supporting compound such as, for example, a wax, and the like, including combinations thereof. In one specific aspect, the superabrasive particles can be temporarily coupled to the metal support layer using an adhesive that then volatilizes away and is eliminated during construction of the dresser.

In one aspect, the predetermined pattern can be a uniformly distributed grid. In another aspect, the predetermined pattern can be an annular configuration having a central region devoid of superabrasive particles. In one specific aspect, the annular configuration can include an annular ring. In another specific aspect, the annular configuration can include a portion of an annular ring. In yet another aspect, an annular configuration can include one or more concentric annular rings nested inside one another. In other aspects, the monolayer distribution can be discrete radial regions, spiral regions, and the like.

The disposition of a monolayer of superabrasive particles on each side of the metal layer thus moderates the thermal shrinkage from the brazing temperature that would otherwise buckle a distribution of superabrasive particles arranged on just one side. By applying a monolayer to each side of the metal support layer, warping forces such as thermal movement and pressure can be equalized or substantially equalized on both sides. As such, the warpage of the metal support layer can be minimized. In other words, the forces responsible for warping are applied substantially equally to each side of the metal layer and can thus at least partially cancel one another out, thus minimizing warpage that can otherwise occur. In some aspects, the superabrasive particles making up the monolayers on each side of the metal layer can have matching configurations, patterns, or orientations to one another. In this way there can be a substantially matching spatial arrangement of superabrasive particles on each side of the metal layer. In another aspect,

the configurations, patterns, or orientations can be different or varied from one another, or may be partially matching. In yet another aspect, the patterned placement of the superabrasive particles on one side of the metal support layer can be substantially aligned with the pattern of superabrasive particles on the other side of the metal layer so that particle locations match one another. In some aspects, there can be a direct correspondence between the spatial positioning of superabrasive particles on one side of the metal support layer and the spatial positioning of superabrasive particles on the other side of the metal support layer. In another aspect, the patterns of superabrasive particles may match or substantially match one another, but may be offset on opposite sides of the metal support layer so that particle locations do not match one another.

Accordingly, minimizing the warpage of the metal support layer can maintain a greater degree of leveling of superabrasive particle tips in the finished tool. When heat and/or pressure are used to make a superabrasive tool, warpage of the metal support layer can cause great variations in tip height level, even for those particles that were leveled prior to heating and/or applying pressure. In one aspect, distributing the warpage forces equally or substantially equally on both sides of a metal support layer through the arrangement of the superabrasive particles can effectively cancel forces on opposing sides and thus greatly reduce the degree of warping occurring in the metal support layer, thus also minimizing the relative height movement of the superabrasive particles relative to one another. In other aspects, CMP pad dressers can be made using processes that do not utilized high pressure and/or high temperature.

In another aspect, as is shown in FIG. 5, a first monolayer of superabrasive particles 502 is arranged on a surface of a metal support layer 504. A second monolayer of superabrasive particles 506 is arranged on the metal support layer 504 on a side that is opposite to the first monolayer. A bonding material 508 secures the first monolayer and the second monolayer to the metal support layer 504. The bonding material can be any material capable of securing the first and second monolayer to the metal support layer. In some aspect, the bonding material can be the same for securing the first monolayer and the

second monolayer, while in other aspects, the bonding material can be different for securing the first monolayer and the second monolayer.

The bonding material can be any material capable of securing superabrasive particles therein. Non-limiting examples of bonding materials include metal brazes, metal braze alloys, organic matrix materials, sintered materials, electroplated materials, and the like, including combinations thereof.

In one aspect, for example, the superabrasive particles can be brazed to the metal support layer, and thus the bonding material can be a metal braze or metal braze alloy. Metal brazing techniques are known in the art. A green braze material can be applied to the metal support layer on or around the superabrasive particles. The metal braze can be applied in any know configuration, including braze sheets, powders, pastes, sprays, and the like, including combinations thereof. Once applied to the metal support layer, the braze can be heated and melted to coat at least a portion of the metal support layer and to bond the superabrasive particles. The heating temperature can vary depending on the braze material used, but in one aspect can be from about 700°C to about 1200°C. The superabrasive particles in the first and second monolayer are thus arranged such that thermal forces exerted on the metal support layer during heating and cooling are substantially equalize in order to minimize warping.

In one non-limiting example, the superabrasive particles can be bonded to the metal support layer by brazing with a nickel-based alloy containing chromium. In another example, the brazing can include pressing the superabrasive particles with a flat ceramic material that cannot be bonded to the braze in order to level the superabrasive particle tips. Various braze alloys are contemplated, including non-limiting examples such as BNi_2, BNi_7, and the like.

Additionally, in one aspect at least one of the first and second monolayers of superabrasive particles can be coupled to the metal support layer by an electrodeposition process, and thus the bonding material can be an electrodeposited metal material. As an example of a suitable method for positioning and retaining abrasive materials prior to and during the electrodeposition process, a mold can be used that includes an insulating

material that can effectively prevent the accumulation of electrodeposited material on the molding surface. Superabrasive particles can be held on the molding surface of the mold during electrodeposition. As such, the accumulation of electrodeposited material can be prevented from occurring on the particle tips and the working surface of the pad conditioner substrate. Such techniques are described in U.S. Patent Application Serial No. 11/292,938, filed December 2, 2005, which is hereby incorporated herein by reference.

In another aspect, the superabrasive particles can be bonded to the metal support layer by sintering, and thus the bonding material can include a sintering material. For example, the bonding of the superabrasive particles to the metal support layer can include disposing a sintering compound on the metal support layer in contact with at least one of the first monolayer and the second monolayer and sintering the sintering compound to bond the at least one of the first monolayer and the second monolayer to the metal support layer. Suitable sintering methods will be readily appreciated by one of ordinary skill in the art having possession of this disclosure. Basically, a sintering compound is applied around the superabrasive particles and in contact with the metal support layer. The sintering compound can be any known sintering material that can be used to secure superabrasive particles to a substrate. Non-limiting examples of such materials can include metal and metal alloy powders, ceramic powders, and the like. One specific non-limiting example of a sintering compound is cobalt powder.

Once the sintering compound has been applied around the superabrasive particles and to the metal support substrate, heat and in some cases pressure can be applied to cause sintering to occur. In some aspects, a braze or braze alloy can be infiltrated into the sintering compound during bonding to further strengthen the bonding material matrix.

In another aspect, as is shown in FIG. 6, heat and pressure can be applied to bond the first monolayer of superabrasive particles 602 and/or the second monolayer of superabrasive particles 606 directly into the metal support layer 604. Thus, upon the application of heat and pressure, the metal support layer 604 is softened or partially melted. The superabrasive particles in one or more of the monolayers are then pressed into the metal support layer. A planar leveling surface can be used to apply pressure to

the monolayer and thus maintain the level nature of the superabrasive particle tips as the superabrasive particles are pressed into the metal support layer. Upon cooling, the distribution of superabrasive particles on either side of the metal support layer at least partially equalizes the thermal forces acting on the metal support layer, thus minimizing warping.

In yet another aspect, as is shown in FIG. 7, a CMP pad dresser can include a first monolayer of superabrasive particles 702 and a second monolayer of superabrasive particles 706 coupled to a metal support layer 704. The superabrasive particles can be coupled to the metal support layer directly or via a bonding material. The second monolayer of superabrasive particles 706 is coupled to a rigid support 708. A portion of the rigid support 708 is shown removed in FIG. 7 to expose the second monolayer. The rigid support 708 can facilitate handling and use of the CMP pad dresser. By bonding the superabrasive particles of the second monolayer to the rigid support, the first monolayer of superabrasive particles remains exposed for CMP pad conditioning operations.

The rigid support can be made from any material compatible with the abrading or dressing process. Such materials can include polymeric materials, metal materials, ceramic materials, glasses, composites, and the like. In one aspect, the rigid support can be a polymeric material and the second monolayer of superabrasive particles can be embedded therein using heat, pressure, adhesives, etc. In some aspects, the rigid support can be a non-polymeric material such as a metal layer. In such cases, the superabrasive particles can be bonded to the rigid support by adhesive attachment, soldering, brazing, electroplating, and the like. For brazing techniques, care can be taken to minimize or eliminate warpage in the metal layer during the heating and cooling process. In another aspect, one or more magnetic elements can be placed into the rigid support to attract and hold the CMP pad dresser in place, thus forming a temporary attachment. An optional locking mechanism can be utilized to further immobilize the CMP pad dresser to the rigid support during use. In some aspects the rigid support can include surface features to hold the dresser in place during rotational movement against a CMP pad. The rigid support can have approximately the same diameter as the metal support layer, a larger diameter

than the metal support layer, or, in some cases, a smaller diameter than the metal support layer.

In another aspect, a CMP pad dresser can be comprised of a plurality of dressing segments having a plurality of leveled superabrasive particles tips, where the plurality of dressing segments are held in place by a rigid support. Such a design allows a plurality of smaller dressing segments to be manufactured having precisely leveled tips. Smaller diameter metal support layers in these dressing segments can be made by processes involving heat and/or pressure with less warpage due to their smaller size. For example, a 4 inch diameter metal disc can exhibit greater warping due to brazing as compared to a 0.5 inch diameter metal disc. Thus thermal distortion and particle floating problems are decreased. A plurality of such dressing segments can then be coupled to a larger diameter rigid support by processes that do not introduce significant warpage, such as bonding in an organic material. Such dressing segments can have one or more layer(s) of superabrasive particles coupled to a support layer. In one aspect, a segment can have a single layer of superabrasive particles coupled to a metal support layer. In another aspect, a segment can have a layer of superabrasive particles coupled to each side of a metal support layer, as has been described herein. Thus, this process allows the manufacture of CMP pad dressers that can have precise tip protrusion tolerances. Additionally, in one aspect, each dressing segment can have at least three superabrasive particles that protrude to the greatest extent. If the three highest protruding superabrasive particles on every dressing segment are leveled across the entire rigid support, a CMP pad dresser is made having very precise tip leveling across the entire surface. If, for example, ten dressing segments are used to make the CMP pad dresser, then the highest protruding thirty superabrasive particles in the tool will have effectively the same protrusion distance and be substantially leveled. Various additional details regarding dressing segments are detailed in U.S. Patent Application No. 13/034,213, filed February 24, 2011, which is hereby incorporated herein by reference.

Various organic materials are contemplated for use as a rigid support and/or to be used to secure the second monolayer of superabrasive particles and/or the dressing segments to the rigid support. Examples of suitable organic matrix materials include,

without limitation, amino resins, acrylate resins, alkyd resins, polyester resins, polyamide resins, polyimide resins, polyurethane resins, phenolic resins, phenolic/latex resins, epoxy resins, isocyanate resins, isocyanurate resins, polysiloxane resins, reactive vinyl resins, polyethylene resins, polypropylene resins, polystyrene resins, phenoxy resins, perylene resins, polysulfone resins, acrylonitrile-butadiene-styrene resins, acrylic resins, polycarbonate resins, polyimide resins, and mixtures thereof. In one specific aspect, the organic material can be an epoxy resin. In another aspect, the organic material can be a polyimide resin. In yet another aspect, the organic material can be a polyurethane resin.

Additionally, so-called "reverse casting" methods can be used to accurately orient and attach the dressing segments to the rigid support. Such methods can include initially securing the plurality of dressing segments to a substrate using a "mask" material. The portions of the dressing segments protruding from the mask material can then be attached to the rigid support using the methods discussed herein, after which (or during which), the masking material can be removed.

When an organic material is utilized, methods of curing the organic material can be a variety of processes known to one skilled in the art that cause a phase transition in the organic material from at least a pliable state to at least a rigid state. Curing can occur, without limitation, by exposing the organic material to energy in the form of heat, electromagnetic radiation, such as ultraviolet, infrared, and microwave radiation, particle bombardment, such as an electron beam, organic catalysts, inorganic catalysts, or any other curing method known to one skilled in the art.

In one aspect of the present invention, the organic material can be a thermoplastic material. Thermoplastic materials can be reversibly hardened and softened by cooling and heating respectively. In another aspect, the organic material layer may be a thermosetting material. Thermosetting materials cannot be reversibly hardened and softened as with the thermoplastic materials. In other words, once curing has occurred, the process can be essentially irreversible, if desired.

As a more detailed list of what is described above, organic materials that may be useful in embodiments of the present invention include, but are not limited to: amino resins

including alkylated urea-formaldehyde resins, melamine-formaldehyde resins, and alkylated benzoguanamine-formaldehyde resins; acrylate resins including vinyl acrylates, acrylated epoxies, acrylated urethanes, acrylated polyesters, acrylated acrylics, acrylated polyethers, vinyl ethers, acrylated oils, acrylated silicons, and associated methacrylates; alkyd resins such as urethane alkyd resins; polyester resins; polyamide resins; polyimide resins; reactive urethane resins; polyurethane resins; phenolic resins such as resole and novolac resins; phenolic/latex resins; epoxy resins such as bisphenol epoxy resins; isocyanate resins; isocyanurate resins; polysiloxane resins including alkylalkoxysilane resins; reactive vinyl resins; resins marketed under the Bakelite™ trade name, including polyethylene resins, polypropylene resins, epoxy resins, phenolic resins, polystyrene resins, phenoxy resins, perylene resins, polysulfone resins, ethylene copolymer resins, acrylonitrile-butadiene-styrene (ABS) resins, acrylic resins, and vinyl resins; acrylic resins; polycarbonate resins; and mixtures and combinations thereof. In one aspect of the present invention, the organic material may be an epoxy resin. In another aspect, the organic material may be a polyimide resin. In yet another aspect, the organic material may be a polyurethane resin.

Numerous additives may be included in the organic material to facilitate its use. For example, additional crosslinking agents and fillers may be used to improve the cured characteristics of the organic material layer. Additionally, solvents may be utilized to alter the characteristics of the organic material in the uncured state. Also, a reinforcing material may be disposed within at least a portion of the solidified organic material layer. Such reinforcing material may function to increase the strength of the organic material layer, and thus further improve the retention of the individual abrasive segments. In one aspect, the reinforcing material may include ceramics, metals, or combinations thereof. Examples of ceramics include alumina, aluminum carbide, silica, silicon carbide, zirconia, zirconium carbide, and mixtures thereof.

Additionally, in one aspect a coupling agent or an organometallic compound may be coated onto the surface of a superabrasive material to facilitate the retention of the superabrasive particles in the organic material via chemical bonding. A wide variety of organic and organometallic compounds is known to those of ordinary skill in the art and

may be used. Organometallic coupling agents can form chemicals bonds between the superabrasive materials and the organic material, thus increasing the retention of the superabrasive materials therein. In this way, the organometallic coupling agent can serve as a bridge to form bonds between the organic material and the surface of the superabrasive material. In one aspect of the present invention, the organometallic coupling agent can be a titanate, zirconate, silane, or mixture thereof. The amount of organometallic coupling agent used can depend upon the coupling agent and on the surface area of the superabrasive material. Oftentimes, 0.05% to 10% by weight of the organic material layer can be sufficient.

Specific non-limiting examples of silanes suitable for use in the present invention include: 3- glycidoxypropyltrimethoxy silane (available from Dow Corning as Z-6040); γ-methacryloxy propyltrimethoxy silane (available from Union Carbide Chemicals Company as A-174); β-(3,4-epoxycyclohexyl)ethyltrimethoxy silane, γ- aminopropyltriethoxy silane, N-(β-aminoethyl)-γ-aminopropylmethyldimethoxy silane (available from Union Carbide, Shin-etsu Kagaku Kogyo K.K., etc.). Specific non-limiting examples of titanate coupling agents include: isopropyltriisostearoyl titanate, di(cumylphenylate)oxyacetate titanate, 4-aminobenzenesulfonyldodecylbenzenesulfonyl titanate, tetraoctylbis (ditridecylphosphite) titanate, isopropyltri(N-ethylamino-ethylamino) titanate (available from Kenrich Petrochemicals. Inc.), neoalkyoxy titanates such as LICA-01, LICA-09, LICA-28, LICA-44 and LICA-97 (also available from Kenrich), and the like. Specific non-limiting examples of aluminum coupling agents include acetoalkoxy aluminum diisopropylate (available from Ajinomoto K.K.), and the like. Specific non-limiting examples of zirconate coupling agents include: neoalkoxy zirconates, LZ-01, LZ-09, LZ- 12, LZ-38, LZ-44, LZ-97 (all available from Kenrich Petrochemicals, Inc.), and the like. Other known organometallic coupling agents, e.g., thiolate based compounds, can be used in the present invention and are considered within the scope of the present invention.

In other aspects of the present disclosure, superabrasive particle tips can be leveled by reducing the protrusions of particles that are outside of the desired tolerance range. Once such particles are identified, various techniques can be utilized to reduce such

protrusions. In one aspect, for example, mechanical abrasion of the dresser can reduce the protruding particles. In another aspect, a vibration tool can be used to individually break such particles. In yet another aspect, a laser such as a Nd:YAG laser can be used to disrupt such particles.

In another aspect of the present disclosure, a method of making a CMP pad dresser can include disposing a first monolayer of superabrasive particles on a metal support layer, and disposing a second monolayer of superabrasive particles on the metal support layer on a side opposite the first monolayer. The superabrasive particles of the second monolayer are positioned to have substantially the same distribution as the superabrasive particles of the first monolayer. The method can further include bonding the first monolayer of superabrasive particles and the second monolayer of superabrasive particles to the metal support layer such that symmetrical forces due to the substantially similar distribution between the first monolayer and the second monolayer precludes substantial warping of the metal support layer.

In yet another aspect, disposing at least one of the first monolayer of superabrasive particles or the second monolayer of superabrasive particles can include adhering a plurality of superabrasive particles to an adhesive transfer sheet in a predetermined pattern established by apertures in a template, and removing the template from the adhesive transfer sheet such that the plurality of superabrasive particles remain adhered to the adhesive transfer sheet in the predetermined pattern. The plurality of superabrasive particles is then transferred with the adhesive transfer sheet to the metal support layer, and the adhesive transfer sheet is removed from the plurality of superabrasive particles to form at least one of the first monolayer of superabrasive particles or the second monolayer of superabrasive particles.

In a further aspect of the present disclosure, a method of minimizing warpage of a CMP pad dresser during manufacture can include substantially equalizing warping forces on opposing sides of a metal support layer during bonding of a plurality of superabrasive particles thereto, wherein warpage of the support layer is minimized during bonding due to the equalized forces on opposing sides. In one aspect, substantially equalizing forces

includes arranging the plurality of superabrasive particles on opposing sides of the support layer such that the plurality of superabrasive particles has substantially the same distribution on either side of the support layer to substantially equalize warping forces during bonding.

In yet a further aspect of the present disclosure, a CMP pad dresser can include a plurality of superabrasive particles arranged as a working surface, where the difference in protrusion distance between the highest protruding tip and the second highest protruding tip of the first monolayer of superabrasive particles is less than or equal to about 20 microns, and where the difference in protrusion distance between the highest 1% of the protruding tips of the first monolayer of superabrasive particles, excluding the highest protruding tip, are within about 80 microns.

In another aspect, a CMP pad dresser can include a plurality of superabrasive particles arranged as a working surface, where the difference in protrusion distance between the highest protruding tip and the second highest protruding tip is less than or equal to about 10 microns, the difference in protrusion distance between the highest protruding tip and the 10th highest protruding tip is less than or equal to about 20 microns, the difference in protrusion distance between the highest protruding tip and the 100th highest protruding tip is less than or equal to about 40 microns, and the highest protruding tip has a protrusion distance of greater than or equal to about 50 microns.

It is noted that, while the above disclosure refers primarily to CMP pad dressers, other precision grinding and/or abrading tools are considered to be within the present scope. As such, the techniques and teaching disclosed herein can additionally be applied to such tools.

Additionally, it is noted that traditional CMP processing techniques, including pad dressing, has limited the critical dimension of integrated circuits. The presently disclosed CMP dressing devices and techniques can, however, allows processing of wafers having integrated circuit critical dimensions of less than or equal to 45nm, 32nm, 28nm, 22nm, or less.

Of course, it is to be understood that the above-described arrangements are only illustrative of the application of the principles of the present invention. Numerous modifications and alternative arrangements may be devised by those skilled in the art without departing from the spirit and scope of the present invention and the appended claims are intended to cover such modifications and arrangements. Thus, while the present invention has been described above with particularity and detail in connection with what is presently deemed to be the most practical and preferred embodiments of the invention, it will be apparent to those of ordinary skill in the art that numerous modifications, including, but not limited to, variations in size, materials, shape, form, function and manner of operation, assembly and use may be made without departing from the principles and concepts set forth herein.

CLAIMS

What is claimed is:

1. A method of conditioning a CMP pad, comprising:

pressing a CMP pad dresser against a CMP pad, the dresser including a monolayer of a plurality of superabrasive particles protruding from a matrix layer, wherein the difference in protrusion distance between the highest protruding tip and the second highest protruding tip of the monolayer of superabrasive particles is less than or equal to about 10 microns and the difference in protrusion distance between the highest 10 protruding tips of the monolayer of superabrasive particles are within about 20 microns or less; and

rotating the dresser against the CMP pad such that asperities are cut into the CMP pad having a maximum cutting depth of about 60 microns.

2. The method of claim 1, wherein at least 100 tips of the plurality of superabrasive particles cut asperities into the CMP pad as the dresser is rotated.

3. The method of claim 1, wherein at least 50 tips of the plurality of superabrasive particles cut asperities into the CMP pad as the dresser is rotated.

4. The method of claim 1, wherein at least 25 tips of the plurality of superabrasive particles cut asperities into the CMP pad as the dresser is rotated.

5. The method of claim 1, wherein substantially all of the asperities are cut in the CMP pad by superabrasive particles located at a peripheral location of the dresser.

6. The method of claim 5, wherein the peripheral location extends from an outer edge of the dresser inwardly toward a center point of the dresser for up to about 90% of the dresser radius.

7. The method of claim 5, wherein the peripheral location extends from an outer edge of the dresser inwardly toward a center point of the dresser for up to about 50% of the dresser radius.

8. The method of claim 5, wherein the peripheral location extends from an outer edge of the dresser inwardly toward a center point of the dresser for up to about 33% of the dresser radius.

9. The method of claim 1, wherein substantially all of the superabrasive particles are located at a peripheral location of the dresser.

10. The method of claim 1, wherein substantially all of the superabrasive particles are arranged in uniform predetermined pattern at a peripheral location of the dresser.

11. The method of claim 1, further comprising:

rotating the CMP pad;

applying a slurry to a polishing surface of the rotating CMP pad;

applying a workpiece to the polishing surface such that the workpiece is polished by the asperities in combination with the slurry.

12. The method of claim 11, wherein the workpiece is a semiconductor device,

13. The method of claim 12, wherein the semiconductor device includes one or more layers of Cu, W, an oxide layer, or a combination thereof.

14. The method of claim 12, wherein the semiconductor device has a node size of less than or equal to 28 nm.

15. The method of claim 12, wherein the semiconductor device has a line width of less than or equal to about 30 nm.

16. The method of claim 12, wherein the semiconductor device is a wafer having a size greater than 400 mm.

17. The method of claim 11, wherein the slurry includes an abrasive selected from the group consisting of silica, alumina, ceria, and combinations thereof.

18. The method of claim 11, wherein the slurry includes an oxidative compound.

19. The method of claim 17, wherein the oxidative compound is hydrogen peroxide.

20. The method of claim 11, wherein the CMP pad is polyurethane.

ABSTRACT

CMP pad dressers having leveled tips and associated methods are provided. In one aspect, for example, a method can include pressing a CMP pad dresser against a CMP pad, where the dresser includes a monolayer of a plurality of superabrasive particles protruding from a matrix layer. The difference in protrusion distance between the highest protruding tip and the second highest protruding tip of the monolayer of superabrasive particles is less than or equal to about 10 microns and the difference in protrusion distance between the highest 10 protruding tips of the monolayer of superabrasive particles are within about 20 microns or less. The method can further include rotating the dresser against the CMP pad such that asperities are cut into the CMP pad having a maximum cutting depth of about 60 microns.

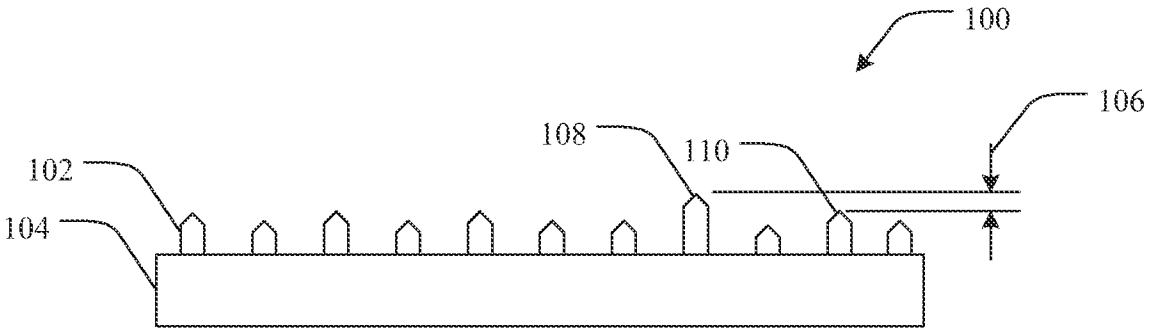

100
106
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FIG. 1

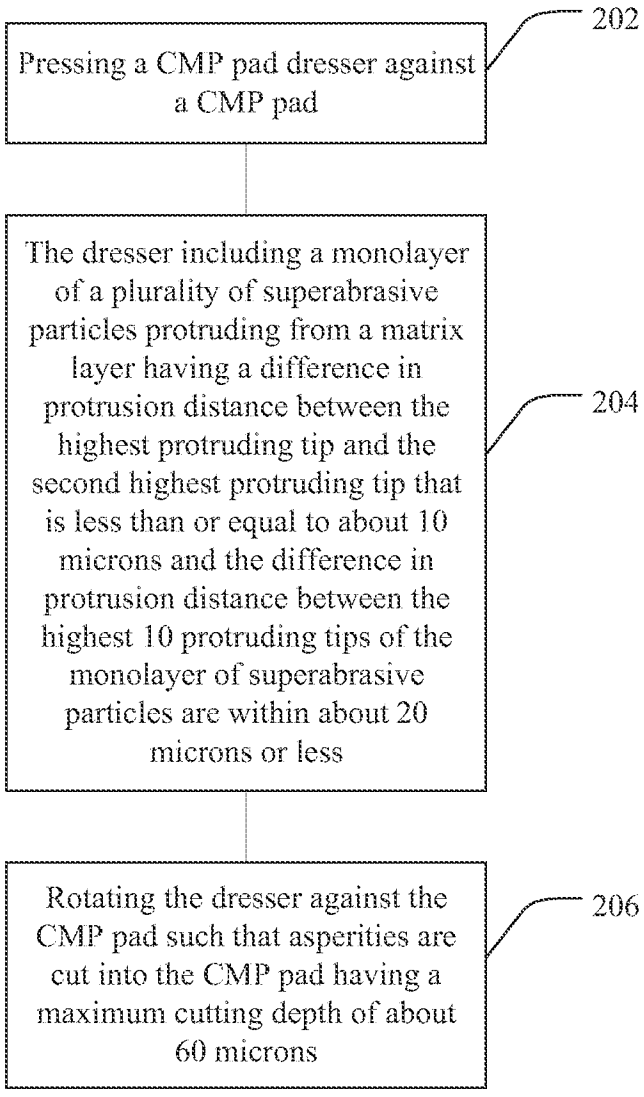

202
Pressing a CMP pad dresser against a CMP pad
204
The dresser including a monolayer of a plurality of superabrasive particles protruding from a matrix layer having a difference in protrusion distance between the highest protruding tip and the second highest protruding tip that is less than or equal to about 10 microns and the difference in protrusion distance between the highest 10 protruding tips of the monolayer of superabrasive particles are within about 20 microns or less
206
Rotating the dresser against the CMP pad such that asperities are cut into the CMP pad having a maximum cutting depth of about 60 microns

FIG. 2

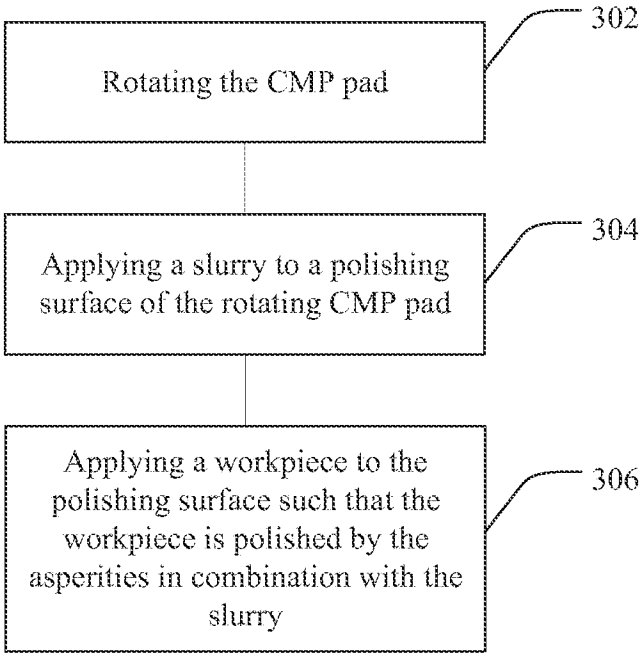


FIG. 3

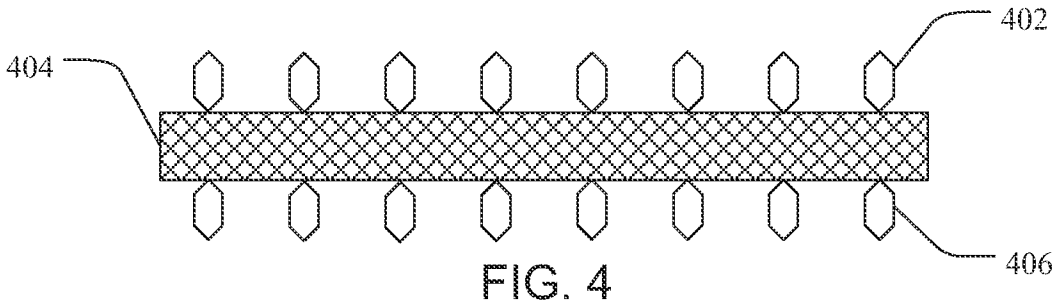


FIG. 4

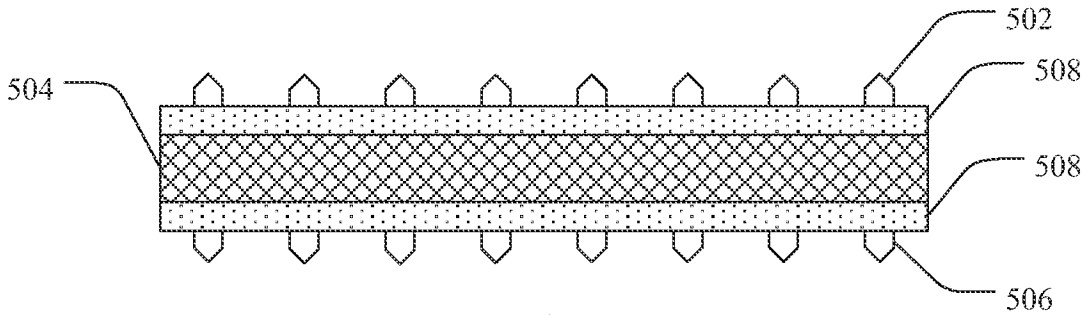


FIG. 5

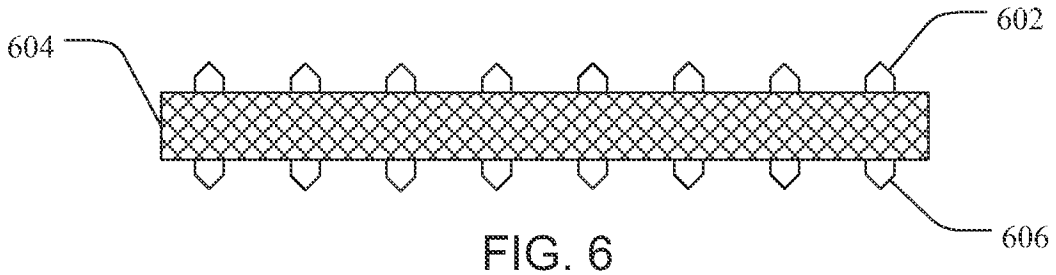


FIG. 6

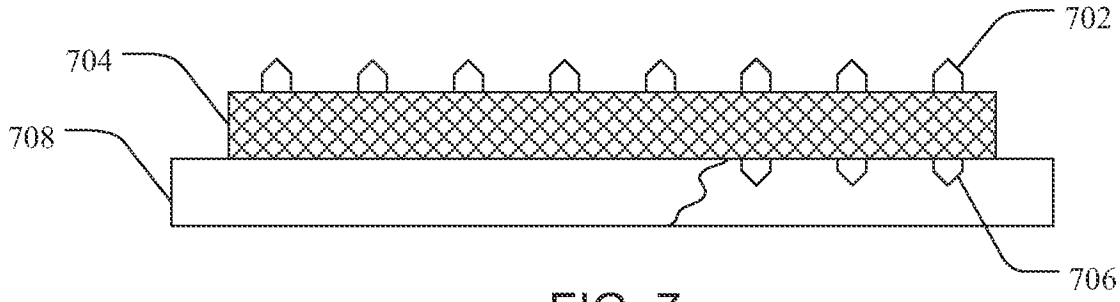


FIG. 7

Electronic Acknowledgement Receipt

EFS ID:	15214612
Application Number:	13802112
International Application Number:	
Confirmation Number:	1021
Title of Invention:	CMP PAD DRESSER HAVING LEVELED TIPS AND ASSOCIATED METHODS
First Named Inventor/Applicant Name:	Chien-Min Sung
Customer Number:	20551
Filer:	David William Osborne/Nicole Solomon
Filer Authorized By:	David William Osborne
Attorney Docket Number:	02074-24012.NP.CIP
Receipt Date:	13-MAR-2013
Filing Date:	
Time Stamp:	20:19:15
Application Type:	Utility under 35 USC 111(a)

Payment information:

Submitted with Payment	no

File Listing:

Document Number	Document Description	File Name	File Size(Bytes)/ Message Digest	Multi Part /.zip	Pages (if appl.)
1	Application Data Sheet	02074-24012NPCIPappdata.pdf	1381684 9854d9ff6d2537850b0fd75a528bfacb09818cc0	no	6
Warnings:					
Information:					

2		02074-24012NPCIPapplication.PDF	334504 9c7670a105360a69021beb51b345d1f369731f02	yes	36

Multipart Description/PDF files in .zip description		
Document Description	**Start**	**End**
Specification	1	32
Claims	33	35
Abstract	36	36

Warnings:

Information:

3	Drawings-only black and white line drawings	02074-24012NPCIPdrawings.pdf	29310 9bb44cd1c4f9a97f388b068b983206568c0857d6	no	3

Warnings:

Information:

Total Files Size (in bytes):	1745498

This Acknowledgement Receipt evidences receipt on the noted date by the USPTO of the indicated documents, characterized by the applicant, and including page counts, where applicable. It serves as evidence of receipt similar to a Post Card, as described in MPEP 503.

New Applications Under 35 U.S.C. 111

If a new application is being filed and the application includes the necessary components for a filing date (see 37 CFR 1.53(b)-(d) and MPEP 506), a Filing Receipt (37 CFR 1.54) will be issued in due course and the date shown on this Acknowledgement Receipt will establish the filing date of the application.

National Stage of an International Application under 35 U.S.C. 371

If a timely submission to enter the national stage of an international application is compliant with the conditions of 35 U.S.C. 371 and other applicable requirements a Form PCT/DO/EO/903 indicating acceptance of the application as a national stage submission under 35 U.S.C. 371 will be issued in addition to the Filing Receipt, in due course.

New International Application Filed with the USPTO as a Receiving Office

If a new international application is being filed and the international application includes the necessary components for an international filing date (see PCT Article 11 and MPEP 1810), a Notification of the International Application Number and of the International Filing Date (Form PCT/RO/105) will be issued in due course, subject to prescriptions concerning national security, and the date shown on this Acknowledgement Receipt will establish the international filing date of the application.

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APPLICATION NUMBER	FILING OR 371(C) DATE	FIRST NAMED APPLICANT	ATTY. DOCKET NO./TITLE
13/802,112	03/13/2013	Chien-Min Sung	02074-24012.NP.CIP

CONFIRMATION NO. 1021

20551
THORPE NORTH & WESTERN, LLP.
P.O. Box 1219
SANDY, UT 84091-1219

FORMALITIES LETTER

OC00000060500817

Date Mailed: 04/22/2013

NOTICE TO FILE MISSING PARTS OF NONPROVISIONAL APPLICATION

FILED UNDER 37 CFR 1.53(b)

Filing Date Granted

Items Required To Avoid Abandonment:

An application number and filing date have been accorded to this application. The item(s) indicated below, however, are missing. Applicant is given **TWO MONTHS** from the date of this Notice within which to file all required items below to avoid abandonment. Extensions of time may be obtained by filing a petition accompanied by the extension fee under the provisions of 37 CFR 1.136(a).

- The statutory basic filing fee is missing.
  *Applicant must submit $**280** to complete the basic filing fee for an undiscounted entity. If appropriate, applicant may make a written assertion of entitlement to small entity status and pay the small entity filing fee (37 CFR 1.27) or make a certification of entitlement to micro entity status and pay the micro entity filing fee (37 CFR 1.29).*

The applicant needs to satisfy supplemental fees problems indicated below.

The required item(s) identified below must be timely submitted to avoid abandonment:

- A surcharge (for late submission of the basic filing fee, search fee, examination fee or inventor's oath or declaration) as set forth in 37 CFR 1.16(f) of $ **140** for an undiscounted entity, must be submitted.

SUMMARY OF FEES DUE:

Total fee(s) required within **TWO MONTHS** from the date of this Notice is $ **1740** for an undiscounted entity
- $ **280** Statutory basic filing fee.
- $ **140** Surcharge.
- The application search fee has not been paid. Applicant must submit $ **600** to complete the search fee.
- The application examination fee has not been paid. Applicant must submit $ **720** to complete the examination fee for an undiscounted entity.

Items Required To Avoid Processing Delays:

Applicant is notified that the above-identified application contains the deficiencies noted below. No period for reply is set forth in this notice for correction of these deficiencies. However, if a deficiency relates to the inventor's oath or declaration, the applicant must file an oath or declaration in compliance with 37 CFR 1.63, or a substitute

statement in compliance with 37 CFR 1.64, executed by or with respect to each actual inventor no later than the expiration of the time period set in the "Notice of Allowability" to avoid abandonment. See 37 CFR 1.53(f).

- A properly executed inventor's oath or declaration has not been received for the following inventor(s):
  All
  Applicant may submit the inventor's oath or declaration at any time before the Notice of Allowance and Fee(s) Due, PTOL-85, is mailed.

Replies must be received in the USPTO within the set time period or must include a proper Certificate of Mailing or Transmission under 37 CFR 1.8 with a mailing or transmission date within the set time period. For more information and a suggested format, see Form PTO/SB/92 and MPEP 512.

Replies should be mailed to:

Mail Stop Missing Parts
Commissioner for Patents
P.O. Box 1450
Alexandria VA 22313-1450

Registered users of EFS-Web may alternatively submit their reply to this notice via EFS-Web.
https://sportal.uspto.gov/authenticate/AuthenticateUserLocalEPF.html

For more information about EFS-Web please call the USPTO Electronic Business Center at **1-866-217-9197** or visit our website at http://www.uspto.gov/ebc.

If you are not using EFS-Web to submit your reply, you must include a copy of this notice.

/tnegash/

Office of Data Management, Application Assistance Unit (571) 272-4000, or (571) 272-4200, or 1-888-786-0101

PATENT APPLICATION FEE DETERMINATION RECORD Substitute for Form PTO-875	Application or Docket Number 13/802,112

APPLICATION AS FILED - PART I

	(Column 1)	(Column 2)	SMALL ENTITY		OR	OTHER THAN SMALL ENTITY	
FOR	NUMBER FILED	NUMBER EXTRA	RATE($)	FEE($)		RATE($)	FEE($)
BASIC FEE (37 CFR 1.16(a), (b), or (c))	N/A	N/A	N/A			N/A	280
SEARCH FEE (37 CFR 1.16(k), (i), or (m))	N/A	N/A	N/A			N/A	600
EXAMINATION FEE (37 CFR 1.16(o), (p), or (q))	N/A	N/A	N/A			N/A	720
TOTAL CLAIMS (37 CFR 1.16(i))	20 minus 20 =	*			OR	x 80 =	0.00
INDEPENDENT CLAIMS (37 CFR 1.16(h))	1 minus 3 =	*				x 420 =	0.00
APPLICATION SIZE FEE (37 CFR 1.16(s))	If the specification and drawings exceed 100 sheets of paper, the application size fee due is $310 ($155 for small entity) for each additional 50 sheets or fraction thereof. See 35 U.S.C. 41(a)(1)(G) and 37 CFR 1.16(s).						0.00
MULTIPLE DEPENDENT CLAIM PRESENT (37 CFR 1.16(j))							0.00
* If the difference in column 1 is less than zero, enter "0" in column 2.			TOTAL			TOTAL	1600

APPLICATION AS AMENDED - PART II

		(Column 1)		(Column 2)	(Column 3)	SMALL ENTITY		OR	OTHER THAN SMALL ENTITY	
		CLAIMS REMAINING AFTER AMENDMENT		HIGHEST NUMBER PREVIOUSLY PAID FOR	PRESENT EXTRA	RATE($)	ADDITIONAL FEE($)		RATE($)	ADDITIONAL FEE($)
AMENDMENT A	Total (37 CFR 1.16(i))	*	Minus	**	=	x =		OR	x =	
	Independent (37 CFR 1.16(h))	*	Minus	***	=	x =		OR	x =	
	Application Size Fee (37 CFR 1.16(s))									
	FIRST PRESENTATION OF MULTIPLE DEPENDENT CLAIM (37 CFR 1.16(j))							OR		
						TOTAL ADD'L FEE		OR	TOTAL ADD'L FEE	

		(Column 1)		(Column 2)	(Column 3)					
		CLAIMS REMAINING AFTER AMENDMENT		HIGHEST NUMBER PREVIOUSLY PAID FOR	PRESENT EXTRA	RATE($)	ADDITIONAL FEE($)		RATE($)	ADDITIONAL FEE($)
AMENDMENT B	Total (37 CFR 1.16(i))	*	Minus	**	=	x =		OR	x =	
	Independent (37 CFR 1.16(h))	*	Minus	***	=	x =		OR	x =	
	Application Size Fee (37 CFR 1.16(s))									
	FIRST PRESENTATION OF MULTIPLE DEPENDENT CLAIM (37 CFR 1.16(j))							OR		
						TOTAL ADD'L FEE		OR	TOTAL ADD'L FEE	

* If the entry in column 1 is less than the entry in column 2, write "0" in column 3.
** If the "Highest Number Previously Paid For" IN THIS SPACE is less than 20, enter "20".
*** If the "Highest Number Previously Paid For" IN THIS SPACE is less than 3, enter "3".
The "Highest Number Previously Paid For" (Total or Independent) is the highest found in the appropriate box in column 1.

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APPLICATION NUMBER	FILING or 371(c) DATE	GRP ART UNIT	FIL FEE REC'D	ATTY.DOCKET.NO	TOT CLAIMS	IND CLAIMS
13/802,112	03/13/2013	3723	0.00	02074-24012.NP.CIP	20	1

CONFIRMATION NO. 1021

20551
THORPE NORTH & WESTERN, LLP.
P.O. Box 1219
SANDY, UT 84091-1219

FILING RECEIPT

OC000000060500816

Date Mailed: 04/22/2013

Receipt is acknowledged of this non-provisional patent application. The application will be taken up for examination in due course. Applicant will be notified as to the results of the examination. Any correspondence concerning the application must include the following identification information: the U.S. APPLICATION NUMBER, FILING DATE, NAME OF APPLICANT, and TITLE OF INVENTION. Fees transmitted by check or draft are subject to collection. Please verify the accuracy of the data presented on this receipt. **If an error is noted on this Filing Receipt, please submit a written request for a Filing Receipt Correction. Please provide a copy of this Filing Receipt with the changes noted thereon. If you received a "Notice to File Missing Parts" for this application, please submit any corrections to this Filing Receipt with your reply to the Notice. When the USPTO processes the reply to the Notice, the USPTO will generate another Filing Receipt incorporating the requested corrections**

Inventor(s)

Chien-Min Sung, Tansui, TAIWAN;

Applicant(s)

Chien-Min Sung, Tansui, TAIWAN;

Power of Attorney: None

Domestic Priority data as claimed by applicant

This application is a CIP of 13/479,148 05/23/2012
which claims benefit of 61/489,074 05/23/2011

Foreign Applications for which priority is claimed (You may be eligible to benefit from the **Patent Prosecution Highway** program at the USPTO. Please see http://www.uspto.gov for more information.) - None.
Foreign application information must be provided in an Application Data Sheet in order to constitute a claim to foreign priority. See 37 CFR 1.55 and 1.76.

Permission to Access - A proper **Authorization to Permit Access to Application by Participating Offices** (PTO/SB/39 or its equivalent) has been received by the USPTO.

If Required, Foreign Filing License Granted: 04/16/2013

The country code and number of your priority application, to be used for filing abroad under the Paris Convention, is **US 13/802,112**

Projected Publication Date: To Be Determined - pending completion of Missing Parts

Non-Publication Request: No

Early Publication Request: No

Title

CMP PAD DRESSER HAVING LEVELED TIPS AND ASSOCIATED METHODS

Preliminary Class

451

Statement under 37 CFR 1.55 or 1.78 for AIA (First Inventor to File) Transition Applications:

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Almost every country has its own patent law, and a person desiring a patent in a particular country must make an application for patent in that country in accordance with its particular laws. Since the laws of many countries differ in various respects from the patent law of the United States, applicants are advised to seek guidance from specific foreign countries to ensure that patent rights are not lost prematurely.

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Electronic Patent Application Fee Transmittal

Application Number:	13802112
Filing Date:	13-Mar-2013
Title of Invention:	CMP PAD DRESSER HAVING LEVELED TIPS AND ASSOCIATED METHODS
First Named Inventor/Applicant Name:	Chien-Min Sung
Filer:	David William Osborne/Nicole Solomon
Attorney Docket Number:	02074-24012.NP.CIP

Filed as Small Entity

Utility under 35 USC 111(a) Filing Fees

Description	Fee Code	Quantity	Amount	Sub-Total in USD($)
Basic Filing:				
Utility filing Fee (Electronic filing)	4011	1	70	70
Utility Search Fee	2111	1	300	300
Utility Examination Fee	2311	1	360	360
Pages:				
Claims:				
Miscellaneous-Filing:				
Late Filing Fee for Oath or Declaration	2051	1	70	70
Petition:				

Description	Fee Code	Quantity	Amount	Sub-Total in USD($)
Patent-Appeals-and-Interference:				
Post-Allowance-and-Post-Issuance:				
Extension-of-Time:				
Miscellaneous:				
		Total in USD ($)		**800**

Electronic Acknowledgement Receipt

EFS ID:	16136464
Application Number:	13802112
International Application Number:	
Confirmation Number:	1021
Title of Invention:	CMP PAD DRESSER HAVING LEVELED TIPS AND ASSOCIATED METHODS
First Named Inventor/Applicant Name:	Chien-Min Sung
Customer Number:	20551
Filer:	David William Osborne/Nicole Solomon
Filer Authorized By:	David William Osborne
Attorney Docket Number:	02074-24012.NP.CIP
Receipt Date:	24-JUN-2013
Filing Date:	13-MAR-2013
Time Stamp:	16:42:37
Application Type:	Utility under 35 USC 111(a)

Payment information:

Submitted with Payment	yes
Payment Type	Credit Card
Payment was successfully received in RAM	$800
RAM confirmation Number	6290
Deposit Account	
Authorized User	

File Listing:

Document Number	Document Description	File Name	File Size(Bytes)/ Message Digest	Multi Part /.zip	Pages (if appl.)

1	Fee Worksheet (SB06)	fee-info.pdf	36996 0b692961b92114dcec2f2c3308a23dcbee7561f2	no	2

Warnings:	
Information:	
Total Files Size (in bytes):	36996

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National Stage of an International Application under 35 U.S.C. 371

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New International Application Filed with the USPTO as a Receiving Office

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Sheet | | of | |

Complete if Known	
Application Number	13/802,112
Filing Date	3/13/2013
First Named Inventor	Chien-Min Sung
Art Unit	3723
Examiner Name	
Attorney Docket Number	02074-24012.NP.CIP

U.S. PATENT DOCUMENTS					
Examiner Initials*	Cite No.[1]	Document Number Number-Kind Code[2] (if known)	Publication Date MM-DD-YYYY	Name of Patentee or Applicant of Cited Document	Pages, Columns, Lines, Where Relevant Passages or Relevant Figures Appear
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INFORMATION DISCLOSURE STATEMENT BY APPLICANT

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Sheet | | of |

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Sheet | | of |

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First Named Inventor	Chien-Min Sung
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INFORMATION DISCLOSURE STATEMENT BY APPLICANT

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Sheet | | of | |

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Application Number	13/802,112
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First Named Inventor	Chien-Min Sung
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Attorney Docket Number	02074-24012.NP.CIP

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Sheet | | of | |

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First Named Inventor	Chien-Min Sung
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Attorney Docket Number	02074-24012.NP.CIP

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Sheet | | of | |

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Filing Date	3/13/2013
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Art Unit	3723
Examiner Name	
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(Use as many sheets as necessary)

Sheet | | of | |

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Art Unit	3723
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Attorney Docket Number	02074-24012.NP.CIP

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Sheet | | of |

Complete if Known	
Application Number	13/802,112
Filing Date	3/13/2013
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Attorney Docket Number	02074-24012.NP.CIP

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Sheet | | of |

Complete if Known	
Application Number	13/802,112
Filing Date	3/13/2013
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Sheet		of	

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Bibliographic data: KR20020036138 (A) — 2002-05-16

DIAMOND GRID-CHEMICAL MECHANICAL POLISH PAD DRESSER

No documents available for this priority number.

Inventor(s): LIN FRANK S; SUNG CHIEN-MIN ± (LIN FRANK S, ; SUNG CHIEN-MIN)

Applicant(s): KINIK CO ± (KINIK COMPANY)

Classification: **- international:** ***B24B1/00;*** (IPC1-7): B24B1/00
- cooperative:

Application number: KR20000066159 20001108

Priority number(s): KR20000066159 20001108

Abstract of KR20020036138 (A)

PURPOSE: A diamond grid-chemical mechanical polish pad dresser is provided to improve polishing efficiency of a CMP(Chemical Mechanical Polish) pad and prolong the service life. CONSTITUTION: A diamond grid-chemical mechanical polish pad dresser (30) constitutes plural abrasive grains(180) of desirable size attached to a plate member. The abrasive grains are extended on the plate member in predetermined height, at regular intervals. The abrasive grains are crystal grains of diamond or cubic boron nitride of a single crystal or poly-crystal type. The crystal grains are placed on a plate(40) coated with an anticorrosion layer(130). The anticorrosion layer is diamond or diamond carbon coated on the surface of a disk to prevent corrosion of brazing alloy(90) by chemical slurry used relatively to the CMP pad.

Last updated: 23.09.2013 Worldwide Database 5.8.11.4; 93p

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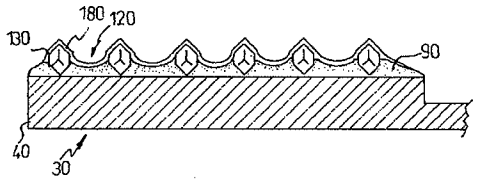
전체 청구항 수 : 총 48 항

(54) 다이아몬드 그리드 화학 기계적 연마 패드 드레서

(57) 요 약

본 발명은 돌출하는 균일하게 일정한 간격을 이룬 다수의 연마 입자들(180)을 가진 CMP 패드 드레서를 나타낸다. 상기 연마 입자들(180)은 우수한 경질 재료로, 일반적으로 다이아몬드, 다결정 다이아몬드(PCD), 입방 보론 질화물(cBN), 또는 다결정 입방 보론 질화물(PcBN)이다. 연마 입자들(180)은 부가 내식 층(130)으로 코팅된 기판(40)에 착지된다. 상기 내식층(130)은 보통 CMP 패드와 연관되어 사용된 화학 슬러리에 의해 경납 합금(90)의 부식을 방지하기 위해 디스크의 표면위로 코팅되는 다이아몬드 또는 다이아몬드형 카본이다. 화학 부식에 대한 상기 면역은 작업편을 연마하는 동안 패드를 드레싱하기 위한 CMP 패드 드레서를 허용한다. 기판(40)상의 평탄한 일정한 간격에 더하여, 연마 입자들(180)은 수직과 수평방향으로 CMP 패드의 손질 또는 드레싱을 위해 허용하는 기판(40)으로부터 떨어진 균일한 거리로 연장한다. 또한 그러한 CMP 패드 드레서를 제조하는 방법이 나타내어졌다.

대 표 도 - 도 3



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청구의 범위

청구항 1

기판 부재에 부착된 바람직한 크기 범위내에 크기를 가진 다수의 연마 입자를 구성하고,

상기 연마 입자들은 균일하게 일정한 간격을 이루고 및 기판 부재위에서 미리 결정된 높이로 연장되는 것을 특징으로 하는 화학 기계적 연마(CMP) 패드 드레서.

청구항 2

제 1 항에 있어서,

상기 연마 입자들은 단결정 또는 다결정 형태로써 다이아몬드 또는 입방 보론 질화물의 결정 입자들인 것을 특징으로 하는 화학 기계적 연마(CMP) 패드 드레서.

청구항 3

제 1 항에 있어서,

상기 크기 범위는 50 내지 250 마이크로미터인 것을 특징으로 하는 화학 기계적 연마(CMP) 패드 드레서.

청구항 4

제 1 항에 있어서,

상기 연마 입자들은 크기에 있어서 실질적으로 균일하고, 모든 연마 입자들은 서로 10%내 크기를 가지는 것을 특징으로 하는 화학 기계적 연마(CMP) 패드 드
레서.

청구항 5

제 1 항에 있어서,

상기 다수의 균일하게 일정한 간격을 이룬 연마 입자들은 미리 결정된 형태에 따라서 분포되고, 미리 결정된 거리는 두 개의 입자들사이에 유지되는 것을 특징으로 하는 화학 기계적 연마(CMP) 패드 드레서.

청구항 6

제 5 항에 있어서,

각 입자 사이의 미리 결정된 거리는 입자들의 평균 크기의 1.5 내지 10 배인 것을 특징으로 하는 화학 기계적 연마(CMP) 패드 드레서.

청구항 7

제 5 항에 있어서,

상기 미리 결정된 형태는 그리드(grid)인 것을 특징으로 하는 화학 기계적 연마(CMP) 패드 드레서.

청구항 8

제 1 항에 있어서,

기판위의 미리 결정된 높이는 모든 연마 입자들이 50 마이크로미터내로 연장하는 균일한 높이인 것을 특징으로 하는 화학 기계적 연마(CMP) 패드 드레서.

청구항 9

제 1 항에 있어서,

상기 기판위의 미리 결정된 높이는 평균 70μm 이상인 것을 특징으로 하는 화학 기계적 연마(CMP) 패드 드레서.

청구항 10

제 1 항에 있어서,

상기 연마 입자들은 자형(euhedral) 결정 형상을 가지는 것을 특징으로 하는 화학 기계적 연마(CMP) 패드 드레서.

청구항 11

제 1 항에 있어서,

상기 연마 입자들은 미리 결정된 형상을 가지는 것을 특징으로 하는 화학 기계적 연마(CMP) 패드 드레서.

청구항 12

제 1 항에 있어서,

상기 연마 입자들은 기판으로부터 떨어져 방향지어진 가장자리 또는 예리한 지점을 구비하는 것을 특징으로 하는 화학 기계적 연마(CMP) 패드 드레서.

청구항 13

제 1 항에 있어서,

상기 기판은 금속 재료로 만들어진 것을 특징으로 하는 화학 기계적 연마 (CMP) 패드 드레서.

청구항 14

제 12 항에 있어서,

상기 금속 재료는 스테인레스강인 것을 특징으로 하는 화학 기계적 연마 (CMP) 패드 드레서.

청구항 15

제 1 항에 있어서,

상기 연마 입자들은 경납 합금에 의해 기판에 부착되는 것을 특징으로 하는 화학 기계적 연마(CMP) 패드 드레서.

청구항 16

제 14 항에 있어서,

추가로 상기 경납 합금은 1 wt% 이상의 크롬 량을 가진 니켈 합금을 구성하는 것을 특징으로 하는 화학 기계적 연마(CMP) 패드 드레서.

청구항 17

제 14 항에 있어서,

상기 경납 합금은 기판의 표면위에 미리 결정된 두께이고, 각 연마 입자의 10 내지 90%가 노출되는 것을 특징으로 하는 화학 기계적 연마(CMP) 패드 드레서.

청구항 18

내식층으로 코팅되는 것을 특징으로 하는 화학 기계적 연마(CMP) 패드 드레서.

청구항 19

제 18 항에 있어서,

연마 입자들의 층은 전기 도금 니켈에 의해 기판에 결합되는 것을 특징으로 하는 화학 기계적 연마(CMP) 패드 드레서.

청구항 20

제 18 항에 있어서,

상기 내식층은 다이아몬드형 카본으로 구성되는 것을 특징으로 하는 화학 기계적 연마(CMP) 패드 드레서.

청구항 21

제 18 항에 있어서,

상기 다이아몬드형 카본은 다이아몬드 결합에 있어서 70% 이상을 함유한 것을 특징으로 하는 화학 기계적 연마(CMP) 패드 드레서.

청구항 22

제 18 항에 있어서,

상기 내식층은 3 마이크로미터 이하의 두께를 가지는 것을 특징으로 하는 화학 기계적 연마(CMP) 패드 드레서.

청구항 23

제 18 항에 있어서,

상기 다이아몬드형 카본은 95% 이상의 원자 탄소 함량을 가지는 것을 특징으로 하는 화학 기계적 연마(CMP) 패드 드레서.

청구항 24

a) 기판 부재를 제공하는 단계,

b) 상기 기판의 표면위에 다수의 연마 입자들을 균일하게 일정한 간격으로
유지하는 단계, 및

c) 각 연마 입자들이 기판 부재위에 미리 결정된 높이로 연장하도록 기판에 상기 연마 입자들을 부착하는 단계를 구성하는 것을 특징으로 하는 화학 기계적 연마(CMP) 패드 드레서 제조 방법.

청구항 25

제 24 항에 있어서,

상기 연마 입자들은 단결정 또는 다결정 형태로써 다이아몬드 또는 입방 보론 질화물의 결정 입자들인 것을 특징으로 하는 화학 기계적 연마(CMP) 패드 드레서 제조 방법.

청구항 26

제 24 항에 있어서,

단계 b) 및 c)는 추가로,

a) 연마 입자의 배치가 구멍들의 위치에 의해 제어되도록 경납 합금의 시트상에 그 안에 형성된 구멍들의 미리 결정된 형태를 가지는 형판을 놓는 단계,

b) 연마 입자들로 상기 형판의 구멍을 채우는 단계,

c) 형판 구멍내에 있지않은 연마 입자들을 제거하는 단계,

d) 상기 연마 입자들이 경납 합금으로 부분적으로 애워싸여지게 되도록 경납 합금의 시트내부 구멍에 함유된 연마 입자들을 가압하는 단계,

e) 연마 입자들이 경납 합금의 시트상에 놓인 상태로 유지하도록 형판을 제거하는 단계,

f) 기판에 연마 입자들을 함유하는 경납 합금의 시트를 부착하는 단계, 및

g) 진공로에서 제품을 경납하는 단계를 구성하는 것을 특징으로 하는 화학 기계적 연마(CMP) 패드 드레서 제조 방법.

청구항 27

제 26 항에 있어서,

상기 구멍들은 단지 하나의 연마 입자를 수용하기에 충분한 크기를 가지는 것을 특징으로 하는 화학 기계적 연마(CMP) 패드 드레서 제조 방법.

청구항 28

제 27 항에 있어서,

상기 구멍들은 미리 결정된 범위의 연마 입자들을 수용하기 위해 선택된 미리 결정된 크기를 가지는 것을 특징으로 하는 화학 기계적 연마(CMP) 패드 드레서 제조 방법.

청구항 29

제 24 항에 있어서,

상기 연마 입자들은 50 내지 250 마이크로미터의 범위인 평균 크기를 가지는 것을 특징으로 하는 화학 기계적 연마(CMP) 패드 드레서 제조 방법.

청구항 30

제 24 항에 있어서,

상기 연마 입자들은 모든 연마 입자들이 서로의 10%내 크기를 가지도록 크기에 있어서 실질적으로 균일한 것을 특징으로 하는 화학 기계적 연마(CMP) 패드 드
레서 제조 방법.

청구항 31

제 26 항에 있어서,

미리 결정된 형태의 상기 구멍들은 두 개의 입자들 사이에 미리 결정된 거리를 만들기 위해 충분한 방법내에서 일정한 간격으로 유지되는 것을 특징으로 하는 화학 기계적 연마(CMP) 패드 드레서 제조 방법.

청구항 32

제 31 항에 있어서,

상기 각 입자사이의 미리 결정된 거리는 입자들 크기의 1.5 내지 10배인 것을 특징으로 하는 화학 기계적 연마(CMP) 패드 드레서 제조 방법.

청구항 33

제 26 항에 있어서,

상기 미리 결정된 입자의 형태는 그리드인 것을 특징으로 하는 화학 기계적 연마(CMP) 패드 드레서 제조 방법.

청구항 34

제 24 항에 있어서,

기판으로부터 미리 결정된 높이는 모든 연마 입자들이 50 마이크로미터내로 연장하는 균일한 높이인 것을 특징으로 하는 화학 기계적 연마(CMP) 패드 드레서 제조 방법.

청구항 35

제 24 항에 있어서,

상기 연마 입자들은 자형 형상을 가지는 것을 특징으로 하는 화학 기계적 연마(CMP) 패드 드레서 제조 방법.

청구항 36

제 24 항에 있어서,

상기 연마 입자들은 미리 결정된 형상인 것을 특징으로 하는 화학 기계적 연마(CMP) 패드 드레서 제조 방법.

청구항 37

제 24 항에 있어서,

상기 연마 입자들은 상기 기판으로부터 떨어져 방향지어진 가장자리 또는 예리한 지점을 가지는 것을 특징으로 하는 화학 기계적 연마(CMP) 패드 드레서 제조 방법.

청구항 38

제 24 항에 있어서,

상기 기판 부재는 금속 재료로 만들어진 것을 특징으로 하는 화학 기계적 연마(CMP) 패드 드레서 제조 방법.

청구항 39

제 38 항에 있어서,

상기 금속 재료는 스테인레스강인 것을 특징으로 하는 화학 기계적 연마 (CMP) 패드 드레서 제조 방법.

청구항 40

제 26 항에 있어서,

상기 경납 합금의 시트는 유기 결합제로 서로 경납 합금 입자들을 결합하는 단계, 및

바람직한 두께의 시트로 상기 결합된 입자들을 형성하는 단계에 의해 제조되는 것을 특징으로 하는 화학 기계적 연마(CMP) 패드 드레서 제조 방법.

청구항 41

제 40 항에 있어서,

상기 시트로 경납 합금 입자들을 형성하는 단계는 압연, 압출 또는 테이프 케스팅에 의해 이루어지는 것을 특징으로 하는 화학 기계적 연마(CMP) 패드 드레서 제조 방법.

청구항 42

제 26 항에 있어서,

상기 경납 합금은 1wt% 이상의 크롬 양을 가지는 니켈 합금을 구성하는 것을 특징으로 하는 화학 기계적 연마(CMP) 패드 드레서 제조 방법.

청구항 43

제 26 항에 있어서,

상기 경납 합금의 시트는 경납 합금위의 각 연마 입자의 10 내지 90% 사이의 노출을 허용하기 위해 충분한 경납 두께를 가지는 것을 특징으로 하는 화학 기계적 연마(CMP) 패드 드레서 제조 방법.

청구항 44

제 24 항에 있어서,

추가로 내식층을 가진 상기 경납 합금과 상기 연마 입자들을 코팅하는 단계를 구성하는 것을 특징으로 하는 화학 기계적 연마(CMP) 패드 드레서 제조 방법.

청구항 45

제 44 항에 있어서,

상기 내식층은 다이아몬드, 또는 다이아몬드형 카본으로 구성되는 것을 특징으로 하는 화학 기계적 연마(CMP) 패드 드레서 제조 방법.

청구항 46

제 44 항에 있어서,

상기 내식층은 3 마이크로미터 이하의 두께를 가지는 것을 특징으로 하는 화학 기계적 연마(CMP) 패드 드레서 제조 방법.

청구항 47

제 44 항에 있어서,

상기 다이아몬드형 카본은 90% 이상의 원자 탄소 함량을 가지는 것을 특징으로 하는 화학 기계적 연마(CMP) 패드 드레서 제조 방법.

청구항 48

제 47 항에 있어서,

상기 코팅 단계는 음극 아크 방법을 사용하여 이루어지는 것을 특징으로 하는 화학 기계적 연마(CMP) 패드 드레서 제조 방법.

명세서

발명의 명칭

다이아몬드 그리드 화학 기계적 연마 패드 드레서{A DIAMOND GRID CMP PAD DRESSER}

도면의 간단한 설명

[0001] 도 1은 디스크 기판에 다이아몬드를 고정하기 위해 전기 도금 방법을 사용한 종래 기술의 CMP 패드 드레서의 측면도이고,

[0002] 도 2는 디스크 기판에 다이아몬드 입자들을 고정하기 위한 종래 경납 방법을 사용하여 만들어진 종래 기술의 CMP 패드 드레서의 측면도이고,

[0003] 도 3은 본 발명의 원리에 따라 만들어진 CMP 패드 드레서의 측면도이고,

[0004] 도 4는 본 발명의 원리에 따라 만들어진 그의 표면위에 연마 입자들을 놓기 위한 형판을 가진 경납 합금의 시트에 대한 측면도이고,

[0005] 도 5는 그의 표면위에 형판과 상기 형판의 구멍들을 채우는 연마 입자들을 가진 경납 합금 시트의 측면도로, 평평한 표면은 본 발명의 원리에 따라서 경납 합금의 시트내부로 연마 입자들을 가압하여 사용하기 위한 것을 나타내었고,

[0006] 도 6은 본 발명의 원리에 따라서 상기 내부로 가압된 연마 입자들을 가진 경납 합금 시트의 측면도이다.

발명의 상세한 설명

[0007] 본 발명은 일반적으로 CMP(화학기계적 연마) 패드(pad)를 조절하고 또는 드레싱(dressing)하기 위한 장치 및 그의 제조 방법에 관한 것이다. 특히, 본 발명은 CMP 패드를 조절하고 또는 드레싱하기 위해 다이아몬드 또는 입방 보론 질화물과 같은 우수한 경질재료를 함유한 드레싱 디스크에 관한 것이다. 더욱 특이하게, 본 발명은 화학적 침식으로부터 보호하기 위해 탄소와 같은 다이아몬드의 얇은 피막으로 코팅될 수 있는, 평탄하게 일정한 간격을 이룬 연마 입자들을 가진 드레싱 디스크에 관한 것이다.

[0008] 많은 산업체에서는 현재 어떠한 작업편을 연마하기 위해 화학기계적 평탄화 (CMP) 방법을 사용하고 있다. 특히, 제조 산업은 세라믹, 실리콘, 유리, 석영 및 금속의 웨이퍼들을 연마하기 위해 대부분이 CMP 방법에 의존하고 있다. 일반적으로 그러한 연마공정은 폴리우레탄과 같은 내구성 유기물로 만들어진 회전 패드에 대항하여 웨이퍼 부착을 수반한다. 패드에 대하여, 웨이퍼 기판을 파괴시킬 수 있는 화학물질을 함유한 화학 슬러리 및 웨이퍼 표면을 물리적으로 침식시키기 위해 작용하는 많은 양의 연마 입자들이 첨가된다. 슬러리는 회전 CMP 패드에 연속적으로 첨가되고, 및 웨이퍼상에 발휘된 이중의 화학적 및 기계적 작용들은 바람직한 방법을 통해 연마되도록 한다.

[0009] 제조된 연마 품질에 대해 특별한 중요성의 하나는, 전체 패드를 통한 연마 입자들의 분포에 있다. 패드의 최상부는 보통 폴리우레탄내에 공공과 같은 수단에 의해 입자들을 지지하고 및 패드의 최상부상에 거친 조직을 유지한다. 추가로 유연한 패드 최상부는 웨이퍼상에서 연마를 허용하기 위해 필요한 지지물을 제공한다.

[0010] 패드의 최상부의 조직을 유지하는 것에 대한 문제는 작업편, 연마 슬러리 및 드레싱 디스크로부터 나오는 연마 부스러기(debris)의 축적때문이다. 상기 축적은 패드가 슬러리의 연마 입자들을 유지할 수 없게 만드는 패드의 최상부의 "글레이징 (glazing)" 또는 경화를 일으킨다.

[0011] 따라서, 시도들이 다양한 장치들로 상기를 "빗질"하므로써 패드 최상부의 재생을 만든다. 상기 공정이 CMP 패드를 "드레싱" 또는 "조절"하는 것으로써 공지되어 왔다. 많은 형태의 장치들 및 공정들은 상기 목적을 위해 사용되었다. 그러한 장치중 하나는 그의 기판 또는 표면에 부착된 다이아몬드 입자들과 같은 매우 단단한 결정질 입자들을 가진 디스크(disk)이다.

[0012] 불행이도, 종래 방법에 의해 만들어진 그러한 다이아몬드 디스크들은 몇 가지 문제점들을 나타내었다. 첫 번째, 다이아몬드들은 디스크의 기판으로부터 박리되어졌고 및 CMP 패드 표면에 포착되었다. 상기는 연마되는 작업편에 대하여 스크래치(scratch)를 유발한다. 두 번째, 종래 디스크들은 그룹으로 덩어리지고, 또는 기판의 표면위에 평탄하지 않게 일정한 간격을 이룬 다이아몬드를 가지기 쉬워진다. 상기 평탄하지 않은 그룹은 마모 영역을 발생하는 과다하게 드레싱될 CMP 패드의 약간의 부분들을 일으키고, 한편 다른 것들은 글레이징 층을 유발하는 드레이싱 결핍이 유발된다. 어느 경우에나, 패드 연마 효율은 감소되고, 및 평탄하지 않은 연마를 일으킨다. 마지막으로, 상기 디스크들의 다이아몬드들은 디스크의 기판 표면위의 균일한 높이로 연장하지 못한다. 상기 불균일함은 또한 단지 드레서로부터 충분한 높이로 돌출하는 상기 입자들이 패드에 접촉하기 때문에 CMP 패드의 평탄하
지 않은 드레싱을 일으킨다. 패드 최상부의 평탄하지 않은 드레싱은 웨이퍼의 불균일함을 초래할 수 있다.

[0013] 디스크 기판으부터 다이아몬드의 박리는 그들이 부착되어지는 열등한 방법 때문이다. 다이아몬드들이 기판에 전기도금된 니켈에 의해 유지될 때, 강력한 결합이 아닌 다이아몬드의 기계적 체결이 있게 된다. 따라서, 상기 입자들은 그들이 요동하여 느슨하게되자마자 박리되어질 것이다. 상기 박리 공정은 전기 도금재료상에 화학 슬러리의 화학적 침식에 의해 실행된다.

[0014] 한편, 다이아몬드들이 기판위로 경납(brazing)될 때, 화학 작용력은 더욱 확고하게 다이아몬드를 지탱한다. 그러나, 화학 슬러리내 산은 입혀진 물질을 빠르게 용해시키고 및 다이아몬드를 박리시킨다. 따라서, 화학작용으로 경납의 노출을 최소화하기 위해, 연마 공정은 드레이싱이 발생하는 동안 중지되고, 및 그 후에 다시 시작되어야 한다. 상기는 다른 연마가 뒤따르고 및 그 후 드레싱은 시간을 소비하게 때문에 비효율적이다.

[0015] 전술된 관점에서, CMP 패드의 평탄한 손질(grooming)을 제공하는 CMP 패드 드레서가 바람직할 수 있다. 또한, 평탄한 깊이로 CMP 패드를 손질하는 CMP 패드 드레서가 매우 바람직할 수 있다. 추가로, 다이아몬드 입자 박리에 덜 민감한 CMP 패드 드레서가 매우 바람직할 수 있다. 마지막으로, 화학 슬러리의 산 침식에 저항하고, 및 연속적으로 CMP 패드를 드레싱할 수 있고, 심지어 산 슬러리에서 연마가 수행되어지는 CMP 패드 드레서가 극히 바람직할 수 있다.

[0016] 본 발명의 목적은 평탄하게 드레스 또는 조절을 위한 연마 입자들을 사용할 수 있는 CMP 패드 드레서, CMP 패드를 제공하는 것이 본 발명의 목적이다.

[0017] 본 발명의 다른 목적은 연마 입자 박리에 덜 민감한 CMP 패드 드레서를 제공하기 위한 것이다.

[0018] 본 발명의 또 다른 목적은 CMP 패드를 일정하게 드레싱할 수 있고, 심지어는 패드가 산 슬러리내에서 연마 작용으로 체결되도록 내식성이 있는 CMP 패드 드레서를 제공하기 위한 것이다.

[0019] 또한 본 발명의 목적은 연마되어진 웨이퍼를 오염시키는 디스크로부터 성분들의 용해를 방지하는 화학 베리어를 제공하기 위한 것이다.

[0020] 추가로 본 발명의 목적은 평탄하게 CMP 패드를 드레싱하고 또는 조절하는 방법을 제공하기 위한 것이다.

[0021] 또한 본 발명의 목적은 패드가 산 슬러지에 담궈질 때 연마 입자 박리에 대하여 CMP 패드 드레서의 민감성을 감소시키는 방법을 제공하기 위한 것이다.

[0022] 상기 목적들 및 특별하게 인용되지 않은 다른 것들은 많은 평평하게 일정한 간격을 이룬 기판에 부착된 연마 입자들을 가지는 CMP 패드 드레서의 특별하게 설명된 실시예를 통해 이루어졌다. 일반적으로, 상기 입자들은 단결정 또는 다결정 형태에서, 다이아몬드, 또는 입방형 보론 질화물(cBN)과 같은 매우 단단한 기판중의 하나이다.

[0023] 본 발명의 CMP 패드 드레서를 형성하는 하나의 방법에서, 브레이징 파우다 와 유기 결합제가 첫 번째 반죽(dough)을 형성하기 위해 완전히 혼합된다. 그 후
상기 반죽은 경납 합금의 유연한 시트를 형성하기 위해 두 개의 롤러 사이를 통해 압연된다. 그 후 연마 입자들은 다수의 평평하게 일정한 간격을 이룬 구멍들을 포함하는 형판의 사용에 의해 경납 합금의 시트상에 평평하게 놓여진다. 상기 형판의 구멍들은 하나의 연마 입자 또는 "그리트(grit)"의 크기보다 크지만, 두 개의 크기보다 작다. 모든 구멍들이 연마 입자들로 채워질 때, 어느 과도한 연마 입자들은 제거되고, 및 연마 입자들은 강판과 같은 일반적으로 평평한 표면을 사용하므로써 그 안에 그들이 묻히도록 경납 합금 시트내부로 가압된다. 그 후 상기 형판은 제거되고, 및 연마 입자를 함유한 경납 합금은 아크릴 아교를 가진 기판에 부착된다. 마지막으로, 전체 조립체는 경납 공정을 완성하고 및 확고하게 기판에 연마 입자들을 고정시키기 위해 진공로에서 경납된다.

[0024] 다른 방편으로, 연마 입자들은 상기에 기술된 것 처럼 형판을 사용하여 아크릴 아교를 가진 기판에 부착될 수 있다. 다음, 경납 합금 입자들은 연마 입자들과 기판위로 나타내어진다. 마지막으로, 전체 조립체는 경납 공정을 완성하고 및 확고하게 기판에 연마 입자들을 부착하기 위해 진공 경납로에서 가열된다.

[0025] 제어된 방법에서 연마 입자들을 위치시키기 위해 형판을 사용하므로써, 어떠한 바람직한 형태의 배치는 이루어질 수 있다. 상기 패턴은 거의 어떠한 상상할 수 있는 패턴일 수 있지만, 그러나 대부분이 상당히 기판위에 연마 입자들을 평평하게 일정한 간격을 유지시킬 수 있는 능력을 제공한다. 또한, 균일한 크기인 구멍들을 가진 형판을 사용하므로써, 각 연마 입자의 균일한 크기가 확보된다. 마지막으로, 기판 내부로 연마 입자들을 가압하기 위한 평평한 표면의 사용은 기판 표면위로 돌
출하는 연마 입자들의 균일한 높이를 만든다. 상기 균일한 높이의 연마 입자들은 균일한 깊이로 CMP 패드의 드레싱 또는 홈파기(plowing)를 확보한다. 추가로, 기판을 가로지른 연마 입자들의 균일한 분포는 그의 표면을 가로지른 패드의 균일한 드레싱을 위해 허용한다.

[0026] 기판에 연마 입자들을 부착한 후, 부가적인 내식 재료의 얇은 코팅이 CMP 패드 드레이서에 적용될 수 있다. 그러한 코팅은 효과적으로 CMP 패드 드레서의 표면을 밀봉한다. 그러한 밀봉제는 연마 입자 및 경납을 보호하고, 또는 다른 정착제는 연마 슬러리, 특히 산을 함유한 상기 슬러리들의 화학물질로부터 화학 침식에 대한 그들의 민감성을 감소시킨다. CMP 패드 드레서의 표면이 화학 분해로 덜 민감하게 되는 것 처럼, 또한 연마 입자 박리에 대한 그의 민감성은 줄어든다. 따라서, CMP 패드 드레서는 기판에 대하여 연마 입자들을 결합하는 작용제가 화학 박리로부터 보호되기 때문에 연마 작용동안에도 CMP 패드를 연속적으로 드레서할 수 있다.

[0027] 본 발명의 상기 및 다른 목적, 형태 및 장점들이 첨부된 도면들과 관련하여 나태내어진 다음의 상세한 설명으로부터 더욱 명백히 나타내어질 것이다.

[0028] 현재 CMP 패드 드레서 및 수반된 사용 방법 및 제조가 나타내어지고 및 기술되어지기전, 상기는 본 발명이 여기에서 나타내어진 특별한 공정 단계 및 재료에 제한을 두지 않았지만, 관계 기술에서 보통 기술된 것들에 의해 인식되어진 것 처럼 그의 대등한 연장으로 이해되어질 것이다. 또한 상기는 여기에서 사용된 용어는 단지 특별한 실시를 기술하기 위한 목적으로 사용되었고 및 제한되길 의도되지 않
은 것으로 이해되어져야 한다.

[0029] 상기 상기 명세서 및 첨부된 청구범위에서 사용된 것 처럼, 단일 형태들 "a", "and", "the"는 문맥이 분명하게 다른 것으로 규정되지 않는 한 복수의 지지 대상을 포함하는 것으로 주목되어야 한다. 따라서, 예를 들면, "연마 입자" 또는 "그리트"로 인용은 하나 이상의 그러한 연마 입자들 또는 그리트들에 대한 인용을 포함한다.

[0030] 본 발명을 기술하고 및 청구함에 있어서, 다음의 용어는 아래 설명된 정의에 따라서 사용될 것이다.

[0031] 여기에서 사용된 것으로, "연마 입자", 또는 "그리트", 또는 유사한 말들은 어떤 우수한 단단한 결정질, 또는 다결정 물질, 또는 물질의 혼합물을 의미하고 및 포함하지만 다이아몬드, 다결정 다이아몬드(PCD), 입방 보론 질화물, 및 다결정 입방 보론 질화물(PCBN)로 제한되지 않는다. 추가로, "연마 입자", "그리트", "다이아몬드", "다결정 다이아몬드(PCD)", "입방 보론 질화물" 및 "다결정 입방 보론 질화물(PCBN)"의 용어들은 서로 교환하여 사용될 수 있다.

[0032] 여기에서 사용된 것으로써, "기판"은 연마 입자들이 부착될 수 있는 표면을 가진 CMP 드레서의 기부(基部)를 의미한다. 상기 기부는 어떠한 형상, 두께 또는 재료일 수 있고 및 포함하지만, 그러나 금속, 합금, 세라믹 및 그의 혼합물로 제한되지 않는다.

[0033] 여기에서 사용된 것으로써, "자형(euhedral)"은 고유 형태를 가짐을 의미하고, 또는 성장된 결정학적면을 가진 변경되지 않은 자연 형상을 가짐을 의미한다.

[0034] 여기에서 사용된 것으로써, "끝이 뾰족한(sharp point)"은 결정으로 되는 어떠한 협소한 정점을 의미하고, 포함하지만 모서리들, 융기들, 오벨리스크(obelisk)들, 및 다른 돌출부들로 제한되지 않는다.

[0035] 여기에서 사용된 것으로써, "금속"은 어떠한 금속의 형태, 금속 합금 또는 그의 혼합물을 의미하고 및 특별하게 포함하지만 강, 철, 및 스테인레스강으로 제한되지 않는다.

[0036] 발명자는 CMP 패드를 조절 또는 드레싱의 효율성 및 품질을 개선하기 위한 장치를 발견하였다. 장치를 사용하고 및 제조하기 위한 방법이 여기에 포함되었다. CMP 패드를 조절하고 드레싱하기 위한 장치를 사용하므로써, 디스크 수명 및 패드 수명이 연장될 뿐만 아니라 패드가 사용될 수 있는 동일성이 연장되었고, 따라서 작업이 완성되는 처리량이 향상되었다. 또한, 연마된 웨이퍼의 균일성 및 결함율이 개선되었다.

[0037] 도 1을 참고로, 거기에는 다수의 다이아몬드 입자들(50), 전기 도금된 기판 (40)을 구비한 CMP 패드 드레서가 나타내어져있다. 전기 도금 재료(60)는 일반적으로 산 용액으로부터 석출된 니켈이다. 그러한 전기 도금 방법은 고가이고 및 시간이 소모될 뿐만 아니라, 공정에 의해 발생된 폐기물 때문에 환경적으로 위험하다.

[0038] 전기 도금된 CMP 패드 드레서(10)는 도 1에 나타낸 것 처럼 나타내어진 많은 단점을 가지고 있다. 첫 번째, 전기 도금 재료(60)는 다이아몬드 입자(50)로 어떠한 화학 결합을 형성할 수 없다. 따라서, 단지 약한 가공력은 기판(60)위로 다이아몬드 입자들(50)을 유지한다. 그러한 가공력은 패드 드레서가 CMP 패드에 대항하여
문질러질 때 공간(70)과 같은 전기 도금 재료(60)내에 공공들을 남겨 전기 도금 재료(60)로부터 쉽게 느슨해지는 다이아몬드 입자들(50)에 작용하는 큰 마찰력에 의해 빠르게 극복된다. 그러한 공공들은 슬러리로부터 화학 제품 및 연마 입자들 뿐만 아니라 작업편의 연마된 잔유물로 빠르게 채워지게된다. 그러나, 그러한 증착된 잔유물은 경화되고 및 떨어질 때, 종종 미세 스크래치를 발생시키고, 연마된 웨이퍼의 생산량을 감소시킨다.

[0039] 전기 도금 재료(60)에 의해 생산된 가공력이 기판(40)상에서 다이아몬드 입자들(50)을 지지하고, 다이아몬드 입자(50)를 노출하는 유일한 수단이고, 상기 전기도금 재료가 최소로 유지되어야 한다. 따라서, 전기 도금 재료(60)와 CMP 패드사이의 접촉은 피할 수 없다. 그러한 접촉은 전기 도금 재료를 마모시키고 및 다이아몬드 입자(50)들의 방출을 촉진시킨다.

[0040] 또한, 전기 도금 재료(60)는 볼록한 부분(80)과 같은 장소에서 다이아몬드 (50)위로 성장하는 경향이 있다. 상기 과도한 성장은 이미 낮은 노출 및 다이아몬드 입자(50)들의 빽빽한 간격에 더하여, 만약 불가능하지 않다면, 어려운 CMP 패드 내부로 다이아몬드 입자(50)의 상당한 관통을 만든다. 그러한 관통없이, 드레싱 공정은 심하게 방해를 받는다.

[0041] 도 2를 참고로, 거기에는 경납 재료(90) 및 종래 경납 기술을 사용하여 기판(40)에 경납된 다이아몬드 입자(50)들을 가지고, 기판(40)을 가진 종래 기술의 CMP 드레서 패드를 나타내었다. 경납 재료들은 일반적으로 탄화물 형성자와 혼합된 금속 합금을 구성한다. 그러한 탄화물 형성자들은 차례로 기판과 결합하여, 재료
(90)를 경납하기 위해 화학적 결합으로 다이아몬드 입자(50)들을 허용한다. 상기 결합 장치는 그리트 부착 강도를 상당히 증가시키지만, 약간의 바람직하지 않은 부작용에 의해 수반되어진다.

[0042] 경납 재료(90)는 다이아몬드 입자(50)들을 완전히 덮는 것으로부터 유지하기 위해서 최소로 유지되어야 한다. 따라서, 다이아몬드 입자(50)들은 단지 경납 재료 (90)의 얇은 코팅으로 감싸진다. 상기 문제는 일반적인 경납 재료들이 기계적으로 약하다는 사실에 의해 혼합된다. 상기 기계적 약함은 경납 재료 그 자체가 분리된 다이아몬드 입자로 잘리워질 수 있기 때문에 다이아몬드 입자(50)들과 경납 재료 (90)사이의 화학 결합의 강도를 상쇄시킨다.

[0043] 경납 재료(90)를 가진 부가적인 문제는, 상기에 언급된 전기 도금 니켈과 같이 연마 슬러리에 의해 화학 침식에 매우 민감하다는 것이다. 그러한 화학 침식은 경납 재료(90)를 약하게 하는 것과 같이 다이아몬드 입자(50)들의 분리에 기여한다. 따라서, 화학 슬러리에 CMP 패드 드레서(20)의 노출을 감소시키기 위해서, 작업편의 연마가 중지되어야 하고, 및 화학 슬러리는 패드 드레서(20)가 적용되기전 패드를 떠나는 것을 허용한다. 그러한 연마 공정에서 중지는 마무리 제품을 제조하기 위해 요구된 시간을 증가시키고, 및 따라서 비효율적이다.

[0044] 종래 경납에 대한 다른 결점은 용융 금속 합금의 표면 장력이 기판(40)에 적용될 때 "덩어리"로 연마 입자(50)들을 발생시키는 경향이 있다는 것이다. 그러한 덩어리는 100으로 설명되었고, 틈(110)을 남긴다. 전체 효과는 비효율적이 손질을 만드는 다이아몬드 입자(20)들의 고르지 않은 분포를 남긴다. 상기 비효율성은 틈
(110) 때문이고, 조절되지 않고 남아 있는 CMP 패드의 영역을 발생시킨다.

[0045] 상기 고르지 않은 조절은 작업편의 마모 영역이 적당하게 조절된 영역보다 덜 효과적으로 연마되기 때문에 고르지 않은 연마를 받는 전체 결과로 다른 것 보다 빠르게 마모되는 CMP 패드의 영역을 발생시킨다.

[0046] 연마 입자들의 덩어리를 만드는 다른 효과는 경납 재료(90)내에 작은 무더기 (mound)의 형성이다. 작은 무더기 형성은 약간의 다이아몬드 입자들을 다른 연마 입자들의 것보다 큰 기판(40)위로 높게 상승시킨다. 따라서, 높아진 돌출 입자들은 CMP 패드 내부로 깊게 관통하고, 그들은 어떠한 손질 효과에 의해 연마 입자들이 돌출하는 것을 더 적게 방지할 것이다. 상기는 또한 비효율성 및 부적합한 조절을 발생시킨다.

[0047] 종래 기술의 CMP 패드 드레서와 대조적으로, 본 발명은 CMP 패드의 고른 드레싱을 허용한다. 도 3을 참고로, 거기에는 본 발명의 원리에 따라서 만들어진 CMP 패드 드레서를 나타낸다. 상기 CMP 패드 드레서는 경납 재료(90)로 기판(40)에 부착된 다수의 연마 입자(180)를 가진다. 연마 입자(180)들은 어떠한 우수한 경질 재료일 수 있다. 바람직한 재료들은 다이아몬드로 제한되지 않은 다결정질 다이아몬드(PCD), 입방 보론 질화물(CBN) 및 다결정 입방 보론 질화물(PCBN)을 포함한다.

[0048] 또한 도 3에 나타내어진 것 처럼, 내식 층(130)이다. 상기 내식 층은 연마 입자(180)들이 아래에 기술된 방법을 통해 기판(40)에 부착되어진 후 CMP 패드 드레서의 표면위에 형성된다. 내식 층(130)은 다이아몬드, 또는 다이아몬드형 카본과 같은 다른 우수한 경질 재료이다. 바람직한 실시예에서, 내식 층(130)은 비다이아
몬드 카본의 메트릭스내에 약 70% 이상의 다이아몬드로 구성되었다. 내식 층(130)은 임의의 두께일 수 있으나, 일반적으로 0.5 내지 3μm의 범위이다. 바람직한 실시예에서, 내식 층(130)은 약 1의 두께를 가진다. 그러한 얇은 내식 층(130)은 물리 증기 증착(PVD)방법에 의해 제조될 수 있다. 흑연 음극을 가진 음극 아크의 사용과 같은 PVD 방법은 기술내에서 공지되었고 및 내식 층(130)을 제조하기 위해 사용될 수 있다.

[0049] 내식 층(130)에 의해 제공된 장점은 효과적으로 가공 표면을 "밀봉"하는 것이고, 및 또한 화학 침식에 취약할 수 있는 CMP 패드 드레서의 다른 바람직한 표면들을 밀봉할 수 있다. 밀봉제로써, 내식 층(130)은 CMP 패드내에 유지된 연마 화학 슬러리에 의해 화학 침식으로부터 경납 재료(90)를 보호한다. 상기 보호는 패드가 작업편을 연마할 때에도 CMP 패드 드레서(30)가 연속적으로 CMP 패드를 드레싱하는 것을 허용하고, 및 종래 기술의 CMP 패드 드레서의 수명을 연장하기 위해 사용된 제조 중지를 제거한다. CMP 패드의 연속적이고 및 고른 드레싱은 더 많은 제품 생산을 허용하고, 및 CMP 패드의 수명 및 효과를 연장한다.

[0050] 기판(40)에 연마 입자(180)들을 부착하는 하나의 방법이 도 4-6에 나타내어졌다. 첫 번째, 구멍(150)을 가진 형판(140)은 경납 합금(90)의 시트위에 놓인다. 형판의 사용은 바람직한 형태로 구멍들을 가진 형판을 디자인하므로써 제어될 연마 입자(180)들의 배치를 허용한다. 연마 입자 배치를 위한 형태들은 CMP 패드 드레서가 사용될 특별하게 필요한 조건들을 맞추기 위한 기술에서 보통 기술된 하나에 의해 선택될 수 있다.

[0051] 본 발명의 하나의 양상에서, 구멍들의 분포는 경납 합금(90)에 의해 결합된 연마 그리트(180)들 사이에 바람직한 양의 공간을 만들기 위해 미리 결정된 구멍들 사이에 공간을 가진 그리드(grid) 형태이다. 바람직한 실시예에서, 상기 그리트는 각 그리트 크기의 약 1.5 내지 약 10배의 거리로 고르게 일정한 간격을 이룬다.

[0052] 형판(140)이 경납 합금 시트(90)에 놓인 후, 구멍(150)들은 연마 입자 (180)들로 채워진다. 구멍(150)들은 단지 하나의 연마 입자가 각 구멍에 채워지도록 바람직한 크기를 가진다.

어떠한 크기의 연마 입자, 또는 그리트는 수용할 수 있지만, 그러나 본 발명의 양상에서, 입자들 크기는 직경으로 약 100 내지 350 마이크로미터이다.

[0053] 본 발명의 다른 양상에서, 형판내에 구멍들의 크기는 크기에서 변하거나 또는 실질적으로 균일한 크기인 연마 입자들의 형태를 얻기 위해서 요구되어질 수 있다. 바람직한 실시예에서, 형판의 구멍들은 서로의 크기에서 50 마이크로미터내에 있는 그리트를 단지 선택하기 위해 충분하다. 상기 그리트 크기의 균일성은 각 연마 입자의 가공 하중이 고르게 분포되는 것 처럼, CMP 패드 손질의 균일성에 기인한다. 차례로, 고른 가공 하중 분포는 개개의 연마 입자들상에 응력을 감소시키고, 및 CMP 패드 드레서(30)의 효과적인 수명을 연장한다.

[0054] 형판(150)의 구멍들이 그리트(180)로 채워진 후, 어떤 과도한 연마 입자들은 제거되고, 및 평평한 표면(160)이 연마 입자(180)에 적용된다. 평평한 표면(160)은 경납 합금 시트(90)내부로 내려오는 연마 입자(180)들을 밀어낼 수 있는 것과 같은 강하고 단단한 재료로 이루어져야 한다. 그러한 재료들은 일반적으로 포함되지만,
강, 철, 그의 합금등에 제한되지 않는다.

[0055] 연마 입자(180)들은 도 6에서 경납 합금 시트(90)내에 포함되는 것으로 나타내어졌다. 표면(160)이 평평하기 때문에, 연마 입자(180)들은 기판(40)으로부터 균일한 거리로 떨어져 연장할 것이다. 상기 거리는 형판(140)의 두께에 의해 결정될 것이고, 및 바람직한 실시예에서, 각 연마 입자는 상기 거리의 50 마이크로미터내로 연장할 것이다.

[0056] 도 4-6에서 나타내어진 것과 같이 연마 입자(180)들은 둥글다. 그러나, 도 3에서 그들은 뾰족하였다. 본 발명의 범위는 자형을 포함한 어떠한 형상, 또는 자연적으로 형상을 이룬 입자들의 연마 입자들을 포함한다. 그러나, 바람직한 실시예에서, 연마 입자(180)들은 기판(40)으로부터 떨어진 방향으로 연장하는 뾰족한 포인트 또는 가장자리를 가진다.

[0057] 연마 입자(180)들이 경납 합금 시트(90)내에 포함되어진 후, 상기 시트는 도 3에 나타내어진 것 처럼, 기판(40)에 부착된다. 사용된 경납 합금은 기술내에서 공지된 어떠한 경납 재료일 수 있지만, 그러나 바람직하게 중량 %로 2% 이상의 크롬을 함유한 니켈 합금이다.

[0058] 연마 입자(180)들이 경납 합금 시트(90)내에 포함되었기 때문에, 액체 경납 합금의 표면 장력은 입자 덩어리를 발생시키기에는 불충분하다. 또한, 경납의 두께는 훨씬 낮은 수준으로 발생하고 및 "무더기"가 형성되지 않는다. 반대로, 경납은 상당한 지지물과 슬러리 틈을 제공하는 각 연마 입자사이의 오목한 표면을 형성한다. 마지막으로, 바람직한 실시예에서, 경납 합금 시트(90)의 두께는 경납 재료
(90)의 외부 표면위로 돌출하기 위한 각 연마 입자의 약 10 내지 약 90%를 허용하기 위해 선택되어진다.

[0059] 경납 합금 시트(90)내에 연마 입자(180)를 포함하기 위한 방법의 결과로써, 고른 공간(120)이 만들어진다. 또한, 연마 그리트(180)들은 CMP 패드에 적용될 때를 의미하는 기판(40)위의 거리 또는 균일한 높이로 연장하고, 그들은 패드 섬유내부 균일한 깊이로 돌출할 것이다. 상기 고른 공간 및 균일한 돌출은 CMP가 고르게 드레싱되고 손질되는 것을 발생시키는 것으로, CMP 패드의 연마 효율성을 증가시키고 및 그의 사용 수명을 연장시킨다.

[0060] 본 발명의 더 많은 이해를 위해서, 실시예들이 아래에 제공되어졌다. 이러한 실시예들은 본 발명의 범위의 제한내에서 사용됨을 의미하지는 않는다.

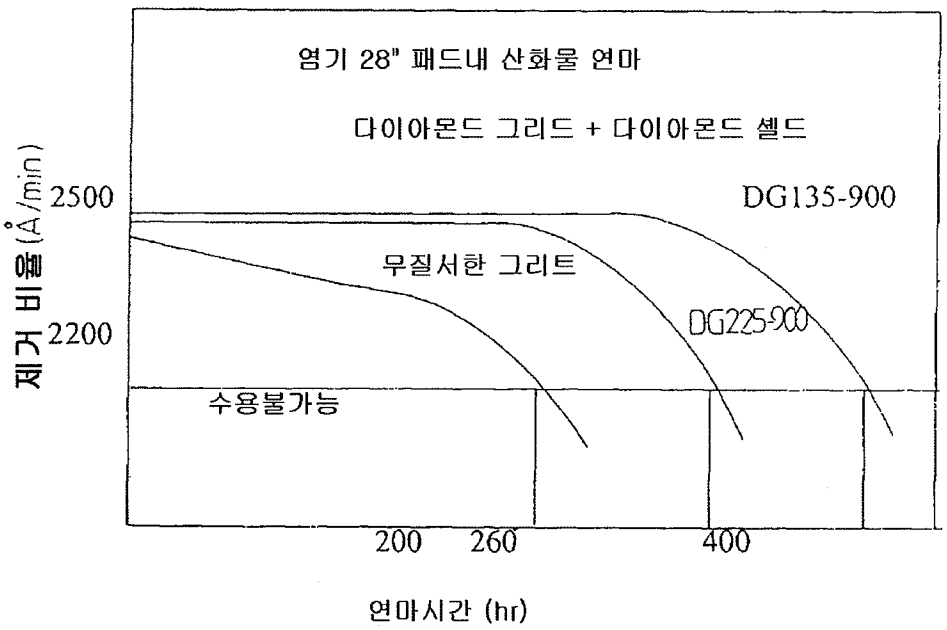
[0061] 실시예 1

[0062] 두 개의 CMP 패드 드레서 디스크들이 다음과 같이 제조되었다. 경납 합금 시트는 두개의 롤러사이에서 금속 파우다와 유기 결합체의 혼합물로 만들어진다. 135 및 225 마이크로미터의 평균 크기를 가진 제네럴 일렉트릭사에 의해 제조된 MBS970의 다이아몬드 그리트들은 경납 합금 시트내부로 형판의 도움에 의해 포함된다. 사용된 형판은 각 다이아몬드 그리트사이의 900 마이크로미터의 거리로 그리드 패턴 내부로 다이아몬드 그리트를 형성하였다.

[0063] 경납 합금 시트내부에 다이아몬드 그리트 입자들의 배치 후, 상기 시트는 아크릴 아교를 사용하여 금속 기판에 부착된다. 그 후 상기 조립체는 1000℃의 온도
로 진공로에서 경납된다. 얻어진 제품들은 약 100 밀리미터의 직경 및 약 6.5 밀리미터의 두께를 가진 두 개의 평평한 디스크이다.

[0064] 그 후 상기 디스크들은 무질서한 형상으로 배치된 다이아몬드 입자들의 양을 5배 이상 가진 디스크에 대항하여 시험되어졌다. 상기 디스크들은 스트라우스바우 (STRAUSBOUGH) 기계상에 장착된 28 인치 CMP 패드를 드레싱하기 위해 사용되었다. 상기 패드는 알칼리성 슬러리내에 8 인치 실리콘 웨이퍼를 연마하기 위해 사용되었다. 시험 결과가 아래 표 1에 나타내어졌다. DG 135-900은 900μm의 거리로 분리된 135 마이크로미터 입자들을 가진 디스크이고, 및 DG 225는 900μm의 거리로 분리된 225 마이크로미터 입자들을 가진 디스크이다.

[0065]



[0066] 나타내어진 것 처럼, 균일한 입자 배치를 가진 두 개의 디스크들은 무질서하게 배치된 다이아몬드를 가진 디스크를 상당히 능가하였다. 또한, 135 마이크로미
터 입자들을 가진 디스크는 거의 무질서한 입자 디스크의 수행력의 두배였다.

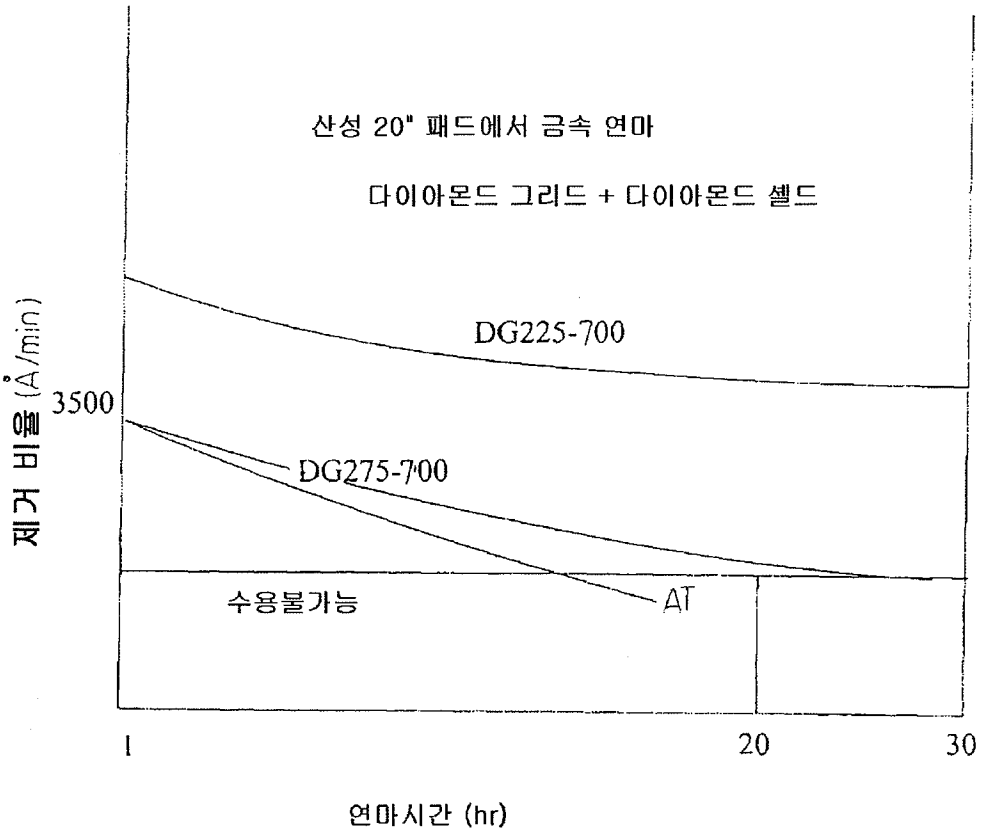
[0067] 실시예 2

[0068] 두 개의 부가 다이아몬드 디스크들은 실시예 1의 방법에 의해 제조되었다. 그러나, 225 마이크로미터 및 275 마이크로미터 크기의 다이아몬드가 사용되었다. 또한, 각 디스크는 경납 합금을 보화하기 위해 1 마이크로미터 두께 다이아몬드형 탄소 코팅으로 코팅되었다. 상기 다이아몬드형 탄소 피막은 음극 아크 방법에 의해 증착되었다.

[0069] 그 후 상기 디스크들은 8 인치 실리콘 웨이퍼를 연마하기 위한 적용된 재료 기계(Mirra)상에 장착된 CMP 패드를 드레싱하므로써 종래 다이아몬드 디스크와 비교되었다. 상기 패드는 3.0 pH의 산성 슬러리에 담궈졌다. 상기 드레싱은 연마가 발생되는 동안 동시에 수행되었다.

결과가 아래 표 2에 나타내어졌다. DG 275-700은 고르게 간격을 유지한 크기에 있어서 275 마이크로미터의 700μm 그리트를 함유한 디스크이다. DG 225-700은 고르게 간격을 유지한 크기에 있어서 225 마이크로미터의 700μm 그리트를 함유한 디스크이고, 및 AT는 보호 코팅없이 무질서하게 분포된 그리트를 함유하는 종래 다이아몬드 디스크이다.

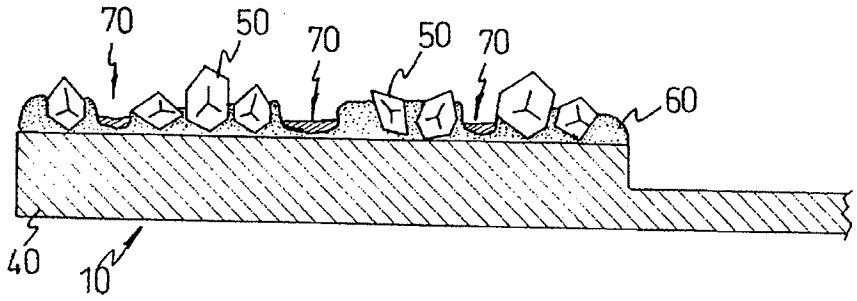
[0070]

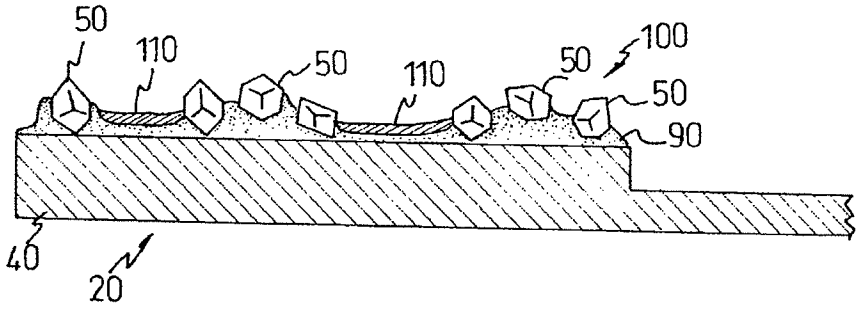


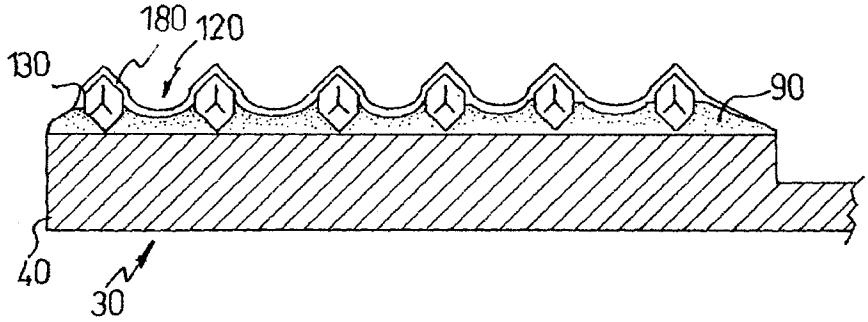
[0071] 표 2로부터 볼 수 있는 것 처럼, 종래 제조된 다이아몬드 디스크는 연마된 웨이퍼의 제거 비율을 유지할 수 없다. 또한, 금속 결합은 단지 1.5 시간동안 연마 슬러리의 산성 분위기에서 유지된다. 그 후, 다이아몬드는 떨어지기 시작하고 및 고가의 웨이퍼상에 많은 스크래치를 발생시킨다. 그러나, 본 발명의 디스크들은 산성 분위기에서 30 시간이상동안 유지되었다. 그러한 수명의 범위는 더 나은 CMP 패드 드레싱 결과를 얻을 수 있고, 및 종래 기술보다 상당히 개선된 웨이퍼 제조의 전체 비용 및 생산량을 구성한다. 물론, 상기는 상기 기술된 실시예가 본 발명의 원리의 적용으로 단지 설명되는 것으로 이해되어졌다.

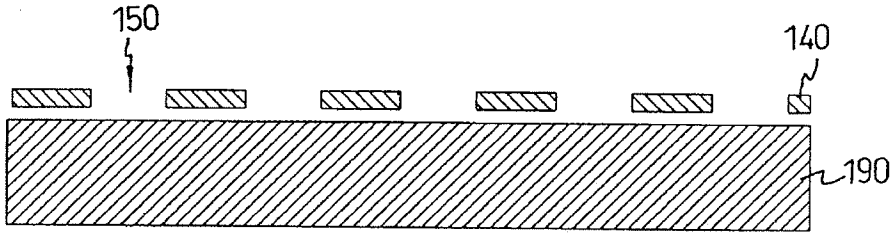
[0072] 많은 변경 및 다른 장치들이 본 발명의 범위 및 범주로부터 벗어남 없이 본 기술내에서 기술된 것들에 의해 발명되어졌고 및 첨부된 청구범위는 그러한 변경 및 장치들을 보호하기 위해 의도되어졌다. 따라서, 본 발명이 본 발명의 가장 실질적이고 및 바람직한 실시예로 간주되는 것과 관련하여 특별하고 및 상세하게 상기에 기술되어졌지만, 상기는 크기의 다양함, 재료, 형상, 형태, 기능, 작동 방법, 조립체, 및 용도에 제한을 두지 않고 포함된 많은 변경이 여기에서 설명된 개념 및 원리로부터 벗어남 없이 만들어질 수 있는 기술내에서 보통 기술의 것들로 명백해질 것이다.

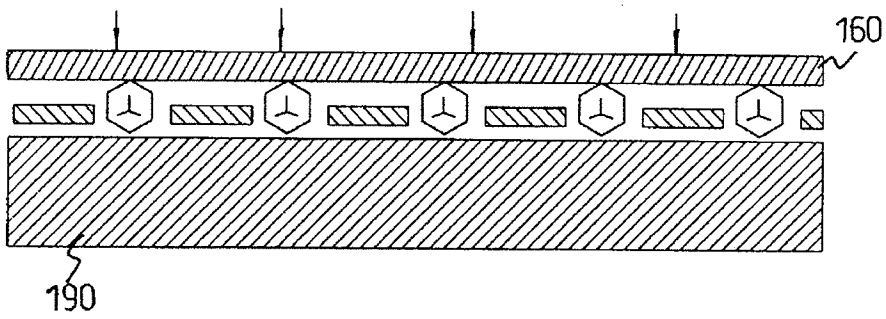
[0073] 상기한 바와 같이, 본 발명은 경납 합금 시트내에 연마 입자를 포함하기 위한 방법의 결과로써, 고른 공간이 만들어지고, 연마 그리트들은 CMP 패드에 적용될 때를 의미하는 기판 위의 거리 또는 균일한 높이로 연장하고, 그들은 패드 섬유내부 균일한 깊이로 돌출함에 따라, 상기 고른 공간 및 균일한 돌출로 CMP가 고르게 드레싱되고 손질되어 CMP 패드의 연마 효율성을 증가키고 및 그의 사용 수명을 연장시킨다.

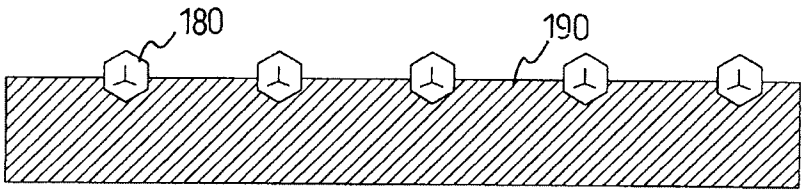

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CONTOURED CMP PAD DRESSER AND ASSOCIATED METHODS

No documents available for this priority number.

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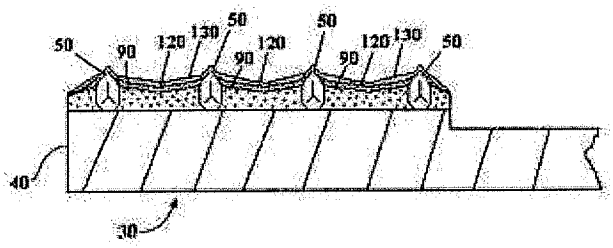
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Abstract of KR20070063569 (A)

CMP pad dressers with increased pad dressing work loads on centrally located abrasive particles (310) during dressing of a CMP pad, and methods associated therewith are disclosed and described. The increase in work loads on centrally located particles (310) improves pad dressing performance and also extends the service life of the pad dresser.



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(54) 윤곽지어진 CMP 패드 드레서 및 관련 방법들

(57) 요약

CMP 패드의 드레싱 동안 중심에 위치한 연마 입자들(310)에 패드 드레싱 작업 부하를 증가시킨 CMP 패드 드레서, 및 이와 관계된 방법이 개시되고 설명된다. 중심에 위치한 입자들(310)에 대한 작업 부하의 증가는 패드 드레싱 성능을 개선시키고 또한 패드 드레서의 사용 수명을 연장시킨다.

대표도

도 10

특허청구의 범위

청구항 1.

둘레에 위치한 입자들의 CMP 패드 내부로의 침투를 감소시키고 중심에 위치한 입자들의 CMP 패드 내부로의 침투를 증가시키는 패턴으로 초연마 입자들을 배열하는 것을 포함하는,

CMP 패드를 드레서로 드레싱하는 동안 CMP 패드 드레서에서 중심에 위치한 초연마 입자들에 작업 부하를 증가시키는 방법.

청구항 2.

제 1항에 있어서, 상기 초연마 입자들의 패턴은 둘레에 위치한 입자들의 작업 말단으로부터 중심에 위치한 입자들의 작업 말단까지 상향의 기울기를 제공함을 특징으로 하는, CMP 패드 드레서에서 중심에 위치한 초연마 입자들에 작업 부하를 증가시키는 방법.

청구항 3.

제 2항에 있어서, 상기 기울기는 둘레에 위치한 입자들에서 중심에 위치한 입자들까지 드레서의 작업 표면 위의 입자 높이를 증가시킴으로써 제공됨을 특징으로 하는, CMP 패드 드레서에서 중심에 위치한 초연마 입자들에 작업 부하를 증가시키는 방법.

청구항 4.

제 2항에 있어서, 상기 기울기는 패드 속도(velocity) 및 가요성을 측정하여 결정됨을 특징으로 하는, CMP 패드 드레서에서 중심에 위치한 초연마 입자들에 작업 부하를 증가시키는 방법.

청구항 5.

제 2항에 있어서, 상기 기울기는 0.1% 내지 0.5% 임을 특징으로 하는, CMP 패드 드레서에서 중심에 위치한 초연마 입자들에 작업 부하를 증가시키는 방법.

청구항 6.

제 5항에 있어서, 상기 기울기는 0.2%임을 특징으로 하는, CMP 패드 드레서에서 중심에 위치한 초연마 입자들에 작업 부하를 증가시키는 방법.

청구항 7.

제 1항에 있어서, 상기 초연마 입자들의 패턴은 중심에 위치한 입자들의 밀도 보다 더 높은, 둘레에 위치한 입자들의 밀도를 제공함을 특징으로 하는, CMP 패드 드레서에서 중심에 위치한 초연마 입자들에 작업 부하를 증가시키는 방법.

청구항 8.

제 7항에 있어서, 상기 둘레에 위치한 입자들의 밀도는 중심에 위치한 입자들의 밀도보다 5배 이상 더 큼을 특징으로 하는, CMP 패드 드레서에서 중심에 위치한 초연마 입자들에 작업 부하를 증가시키는 방법.

청구항 9.

제 7항에 있어서, 상기 둘레에 위치한 입자들의 밀도는 중심에 위치한 입자들의 밀도보다 2배 이상 더 큼을 특징으로 하는, CMP 패드 드레서에서 중심에 위치한 초연마 입자들에 작업 부하를 증가시키는 방법.

청구항 10.

제 7항에 있어서, 상기 둘레에 위치한 입자들의 밀도는 중심에 위치한 입자들의 밀도보다 1.25배 이상 더 큼을 특징으로 하는, CMP 패드 드레서에서 중심에 위치한 초연마 입자들에 작업 부하를 증가시키는 방법.

청구항 11.

제 7항에 있어서, 중심에 그리고 둘레에 위치한 입자들 사이의 초연마 입자들은 중심에 위치한 입자들 및 둘레에 위치한 입자들의 밀도 사이의 밀도로 배치됨을 특징으로 하는, CMP 패드 드레서에서 중심에 위치한 초연마 입자들에 작업 부하를 증가시키는 방법.

청구항 12.

제 7항에 있어서, 상기 초연마 입자들의 패턴은 둘레에 위치한 입자들에서는 높고 중심에 위치한 입자들에서는 낮은, 실질적으로 연속적인 밀도 구배를 제공함을 특징으로 하는, CMP 패드 드레서에서 중심에 위치한 초연마 입자들에 작업 부하를 증가시키는 방법.

청구항 13.

제 1항에 있어서, 상기 초연마 입자들의 패턴은 둘레에 위치한 입자들의 애티튜드에 의하여 제공되는 침투보다 CMP 패드 내부로 더 큰 입자 침투를 유발하는 애티튜드를 가지는 중심에 위치한 입자들을 제공함을 특징으로 하는, CMP 패드 드레서에서 중심에 위치한 초연마 입자들에 작업 부하를 증가시키는 방법.

청구항 14.

제 13항에 있어서, 상기 중심에 위치한 입자들의 애티튜드는 이들의 작업 말단에서 꼭지점(apex)이며, 둘레에 위치한 입자들의 애티튜드는 이들의 작업 말단에서 에지 또는 면임을 특징으로 하는, CMP 패드 드레서에서 중심에 위치한 초연마 입자들에 작업 부하를 증가시키는 방법.

청구항 15.

제 13항에 있어서, 상기 중심에 위치한 입자들의 애티튜드는 이들의 작업 말단에서 에지이며, 둘레에 위치한 입자들의 애티튜드는 이들의 작업 말단에서 면임을 특징으로 하는, CMP 패드 드레서에서 중심에 위치한 초연마 입자들에 작업 부하를 증가시키는 방법.

청구항 16.

제 13항에 있어서, 상기 중심에 위치한 입자들의 애티튜드는 이들의 작업 말단에서 꼭지점(apex)이며, 둘레에 위치한 입자들의 애티튜드는 이들의 작업 말단에서 면이고, 사이에 위치한 입자들은 이들의 작업 말단에서 에지의 애티튜드를 가짐을 특징으로 하는, CMP 패드 드레서에서 중심에 위치한 초연마 입자들에 작업 부하를 증가시키는 방법.

청구항 17.

제 1항, 2항, 7항 또는 13항 중 어느 한 항에 있어서, 상기 중심에 위치한 입자들의 작업 부하는 상기 둘레에 위치한 입자들의 작업 부하의 적어도 30% 까지 이내까지 증가됨을 특징으로 하는, CMP 패드 드레서에서 중심에 위치한 초연마 입자들에 작업 부하를 증가시키는 방법.

청구항 18.

제 17항에 있어서, 상기 중심에 위치한 입자들의 작업 부하는 둘레에 위치한 입자들의 작업 부하의 적어도 10% 이내까지 증가됨을 특징으로 하는, CMP 패드 드레서에서 중심에 위치한 초연마 입자들에 작업 부하를 증가시키는 방법.

청구항 19.

제 17항에 있어서, 상기 중심에 위치한 입자들의 작업 부하는 둘레에 위치한 입자들의 작업 부하와 실질적으로 동일하게 증가됨을 특징으로 하는, CMP 패드 드레서에서 중심에 위치한 초연마 입자들에 작업 부하를 증가시키는 방법.

청구항 20.

제 17항에 있어서, 모든 입자들의 작업 부하는 실질적으로 동일함을 특징으로 하는, CMP 패드 드레서에서 중심에 위치한 초연마 입자들에 작업 부하를 증가시키는 방법.

청구항 21.

제 2항 또는 13항에 있어서, 상기 초연마 입자들은 각각 개별적으로 예정된 패턴에 따라 기판 상의 특정 위치에 배치됨을 특징으로 하는, CMP 패드 드레서에서 중심에 위치한 초연마 입자들에 작업 부하를 증가시키는 방법.

청구항 22.

제 21항에 있어서, 상기 패턴은 실질적으로 균일한 격자임을 특징으로 하는, CMP 패드 드레서에서 중심에 위치한 초연마 입자들에 작업 부하를 증가시키는 방법.

청구항 23.

제 1항, 2항, 7항 또는 13항 중 어느 한 항에 있어서, 상기 초연마 입자들은 다이아몬드, 다결정질 다이아몬드 (PCD), 입방정 질화 붕소 (cBN), 및 다결정질 입방정 질화 붕소 (PCBN)로 구성된 그룹에서 선택됨을 특징으로 하는, CMP 패드 드레서에서 중심에 위치한 초연마 입자들에 작업 부하를 증가시키는 방법.

청구항 24.

제 23항에 있어서, 상기 초연마 입자들은 다이아몬드임을 특징으로 하는, CMP 패드 드레서에서 중심에 위치한 초연마 입자들에 작업 부하를 증가시키는 방법.

청구항 25.

제 1항, 2항, 7항 또는 13항 주 어느 한 항에 있어서, 초연마 입자들이 결합되는 기판을 제공하는 단계를 더욱 포함함을 특징으로 하는, CMP 패드 드레서에서 중심에 위치한 초연마 입자들에 작업 부하를 증가시키는 방법.

청구항 26.

제 25항에 있어서, 상기 초연마 입자들은 브레이징, 소결, 또는 전기도금에 의하여 기판에 결합됨을 특징으로 하는, CMP 패드 드레서에서 중심에 위치한 초연마 입자들에 작업 부하를 증가시키는 방법.

청구항 27.

제 1항, 2항, 7항 또는 13항 중 어느 한 항에 있어서, 상기 초연마 입자들은 실질적으로 균일한 형상을 가짐을 특징으로 하는, CMP 패드 드레서에서 중심에 위치한 초연마 입자들에 작업 부하를 증가시키는 방법.

청구항 28.

제 27항에 있어서, 상기 균일한 형상은 자형임을 특징으로 하는, CMP 패드 드레서에서 중심에 위치한 초연마 입자들에 작업 부하를 증가시키는 방법.

청구항 29.

제 27항에 있어서, 상기 균일한 형상은 팔면체임을 특징으로 하는, CMP 패드 드레서에서 중심에 위치한 초연마 입자들에 작업 부하를 증가시키는 방법.

청구항 30.

제 25항에 있어서, 상기 기판은 가요성의 금속 또는 세라믹 물질로 제조됨을 특징으로 하는, CMP 패드 드레서에서 중심에 위치한 초연마 입자들에 작업 부하를 증가시키는 방법.

청구항 31.

제 30항에 있어서, 상기 금속 물질은 스테인레스 강임을 특징으로 하는, CMP 패드 드레서에서 중심에 위치한 초연마 입자들에 작업 부하를 증가시키는 방법.

청구항 32.

다음을 포함하는 CMP 패드 드레서:

기판; 및

상기 기판에 부착된 복수의 초연마 입자들, 여기서 상기 초연마 입자들은 둘레에 위치한 입자들의 작업 말단으로부터 중심에 위치한 입자들의 작업 말단까지 상향의 기울기를 제공하는 예정된 패턴으로 배열됨.

청구항 33.

다음을 포함하는 CMP 패드 드레서:

기판; 및

상기 기판에 부착된 복수의 초연마 입자들, 여기서 상기 초연마 입자들은 중심에 위치한 입자들의 밀도 보다 더 높은, 둘레에 위치한 입자들의 밀도를 제공하는 예정된 패턴으로 배열됨.

청구항 34.

다음을 포함하는 CMP 패드 드레서:

기판; 및

상기 기판에 부착된 복수의 초연마 입자들, 여기서 상기 초연마 입자들은 중심에 위치한 입자들에, 둘레에 위치한 입자들의 애티튜드에 의하여 제공되는 침투보다, CMP 패드 내부로 더 큰 입자 침투를 유발하는 애티튜드를 제공하는 예정된 패턴으로 배열됨.

청구항 35.

제 32항에 있어서, 상기 기울기는 둘레에 위치한 입자들로부터 중심에 위치한 입자들까지 드레서의 작업 표면 위의 입자 높이를 증가시킴으로써 제공됨을 특징으로 하는, CMP 패드 드레서.

청구항 36.

제 32항에 있어서, 상기 기울기는 패드 속도(velocity) 및 가요성의 측정치로서 결정됨을 특징으로 하는, CMP 패드 드레서.

청구항 37.

제 32항에 있어서, 상기 기울기는 0.1% 내지 0.5%임을 특징으로 하는, CMP 패드 드레서.

청구항 38.

제 32항에 있어서, 상기 기울기는 0.2%임을 특징으로 하는, CMP 패드 드레서.

청구항 39.

제 33항에 있어서, 상기 둘레에 위치한 입자들의 밀도는 중심에 위치한 입자들의 밀도 보다 5배 이상임을 특징으로 하는, CMP 패드 드레서.

청구항 40.

제 33항에 있어서, 둘레에 위치한 입자들의 밀도는 중심에 위치한 입자들의 밀도 보다 2배 이상 더 큼을 특징으로 하는, CMP 패드 드레서.

청구항 41.

제 33항에 있어서, 둘레에 위치한 입자들의 밀도는 중심에 위치한 입자들의 밀도 보다 1.25배 이상 더 큼을 특징으로 하는, CMP 패드 드레서.

청구항 42.

제 33항에 있어서, 중심에 위치한 입자들과 둘레에 위치한 입자들 사이의 초연마 입자들은 중심에 위치한 입자들의 밀도와 둘레에 위치한 입자들의 밀도 사이의 밀도로 배치됨을 특징으로 하는, CMP 패드 드레서.

청구항 43.

제 33항에 있어서, 상기 초연마 입자들의 패턴은 둘레에 위치한 입자들에서는 높고 중심에 위치한 입자들에서는 낮은, 연속적 밀도 구배를 제공함을 특징으로 하는, CMP 패드 드레서.

청구항 44.

제 34항에 있어서, 중심에 위치한 입자들의 애티튜드는 이들의 작업 말단에서 꼭지점(apex)이고, 둘레에 위치한 입자들의 애티튜드는 이들의 작업 말단에서 에지 또는 면임을 특징으로 하는, CMP 패드 드레서.

청구항 45.

제 34항에 있어서, 중심에 위치한 입자들의 애티튜드는 이들의 작업 말단에서 에지이고, 둘레에 위치한 입자들의 애티튜드는 이들의 작업 말단에서 면임을 특징으로 하는, CMP 패드 드레서.

청구항 46.

제 34항에 있어서, 중심에 위치한 입자들의 애티튜드는 이들의 작업 말단에서 꼭지점(apex)이고, 둘레에 위치한 입자들의 애티튜드는 이들의 작업 말단에서 면이며, 이들 사이에 위치한 입자들은 이들의 작업 말단에서 에지의 애티튜드를 가짐을 특징으로 하는, CMP 패드 드레서.

청구항 47.

제 32항, 33항, 또는 34항 중 어느 한 항에 있어서, 상기 중심에 위치한 입자들의 작업 부하는 둘레에 위치한 입자들의 작업 부하의 적어도 30% 이내까지 증가됨을 특징으로 하는, CMP 패드 드레서.

청구항 48.

제 47항에 있어서, 상기 중심에 위치한 입자들의 작업 부하는 둘레에 위치한 입자들의 작업 부하의 적어도 10% 이내까지 증가됨을 특징으로 하는, CMP 패드 드레서.

청구항 49.

제 47항에 있어서, 상기 중심에 위치한 입자들의 작업 부하는 둘레에 위치한 입자들의 작업 부하와 실질적으로 같아지도록 증가됨을 특징으로 하는, CMP 패드 드레서.

청구항 50.

제 47항에 있어서, 모든 입자들의 작업 부하는 실질적으로 동일함을 특징으로 하는, CMP 패드 드레서.

청구항 51.

제 32항 또는 34항에 있어서, 초연마 입자들은 각각 개별적으로 예정된 패턴에 따라 특정 위치에 배치됨을 특징으로 하는, CMP 패드 드레서.

청구항 52.

제 51항에 있어서, 상기 패턴은 실질적으로 균일한 격자임을 특징으로 하는, CMP 패드 드레서.

청구항 53.

제 32항, 33항, 또는 34항 중 어느 한 항에 있어서, 상기 초연마 입자들은 다이아몬드, 다결정질 다이아몬드 (PCD), 입방정 질화 붕소 (cBN), 및 다결정질 입방정 질화 붕소 (PCBN)로 구성되는 그룹에서 선택됨을 특징으로 하는, CMP 패드 드레서.

청구항 54.

제 53항에 있어서, 상기 초연마 입자들은 다이아몬드임을 특징으로 하는, CMP 패드 드레서.

청구항 55.

제32항, 33항, 또는 34항 중 어느 한 항에 있어서, 상기 초연마 입자들은 브레이징, 소결, 또는 전기도금에 의해 기판에 부착됨을 특징으로 하는, CMP 패드 드레서.

청구항 56.

제 32항, 33항, 또는 34항 중 어느 한 항에 있어서, 상기 초연마 입자들은 실질적으로 균일한 형상을 가짐을 특징으로 하는, CMP 패드 드레서.

청구항 57.

제 56항에 있어서, 상기 균일한 형상은 자형임을 특징으로 하는, CMP 패드 드레서.

Ex. 1003, Page 99

청구항 58.

제 56항에 있어서, 상기 균일한 형상은 팔면체임을 특징으로 하는, CMP 패드 드레서.

청구항 59.

제 56항에 있어서, 상기 균일한 형상은 입방정 팔각임을 특징으로 하는, CMP 패드 드레서.

청구항 60.

제 32항, 33항 또는 34항 중 어느 한 항에 있어서, 상기 기판은 가요성의 금속 또는 세라믹 물질로 제조됨을 특징으로 하는, CMP 패드 드레서.

청구항 61.

제 60항에 있어서, 상기 금속 물질은 스테인레스 강임을 특징으로 하는, CMP 패드 드레서.

청구항 62.

다음 단계들을 포함하는, 제 32항 내지 34항 중 어느 한 항의 CMP 패드 드레서를 제조하는 방법:

기판을 제공하는 단계; 및

둘레에 위치한 입자들의 CMP 패드 내부로의 침투는 감소시키고 중심에 위치한 입자들의 CMP 패드 내부로의 침투는 증가시키는 패턴으로 기판에 복수의 초연마 입자들을 부착하는 단계.

명세서

기술분야

발명의 분야

본원 발명은 일반적으로 화학적 기계적 연마 (CMP) 패드를 드레싱 또는 컨디셔닝 하기 위한 장치 및 방법에 관계한다. 따라서, 본원 발명은 화학 및 물질 과학 분야에 관계된다.

배경기술

발명의 배경

많은 산업들은 특정 피가공물을 연마하기 위해 화학적 기계적 가공(CMP)을 사용하고 있다. 특히, 컴퓨터 제조 산업은 세라믹, 실리콘, 유리, 석영 및 이들의 금속들의 웨이퍼들을 연마하기 위하여 CMP 공정에 크게 의존하기 시작했다. 이러한 연마 공정들은 일반적으로 폴리우레탄과 같은 내구성 유기 물질로 제조된 회전식 패드에 대면하는 웨이퍼 사용을 수반한다. 패드에, 웨이퍼 물질을 손상시킬 수 있는 화학물질을 함유하는 화학물질 슬러리 및 웨이퍼 표면을 물리적으로 부식시키기 위하여 작용하는 양의 연마 입자들이 첨가된다. 슬러리는 회전하는 CMP 패드에 연속적으로 첨가되며, 웨이퍼에 가해지는 이원 화학물질 및 기계적 힘은 웨이퍼가 원하는 방식으로 연마되게 한다.

구현되는 연마의 품질에 특히 중요한 것은 패드 전체에 두루 퍼져있는 연마 입자들의 분포이다. 패드의 상부는 통상적으로 섬유, 또는 소형 공극과 같은 메커니즘으로 입자들을 지지하는데, 섬유 또는 소형 공극과 같은 메커니즘들은 패드의 회전 운동에 의해 발생되는 원심력으로 인해 패드가 이탈하는 것을 방지하기에 충분한 마찰력을 제공한다. 그러므로, 패드 상부를 가능한 한 가요성인 상태로유지하는 것, 그리고 섬유들을 가능한 한 직립으로 유지하는 것, 또는 새로운 연마 입자들을 수용할 수 있는 여분의 개구 및 공극들이 존재하도록 확보하는 것이 중요하다.

패드의 상부를 유지하는 것에 관한 문제는 피가공물, 연마 슬러리, 및 드레싱 디스크에서 생긴 연마 부스러기의 축적에 의해 야기된다. 이러한 축적은 패드 상부의 "글레이징" 또는 경화를 야기하며, 섬유질들을 헝클어 버리므로, 패드가 슬러리의 연마 입자들을 덜 지지할 수 있도록 만들고, 패드의 전체적인 연마 성능을 현저히 감소시킨다. 또한, 많은 패드들에 있어서, 슬러리를 지지하기 위하여 사용되는 공극들은 막히게 되고, 패드의 연마 표면의 전체적인 조도는 저하되어 편평해진다. 그러므로, 다양한 장치를 사용하여 패드 상부를 "코밍(combing)" 또는 "컷팅(cutting)"하여 재생시키기 위한 시도들이 이루어졌다. 이러한 공정은 CMP 패드를 "드레싱" 또는 "컨디셔닝"하는 것으로 공지되었다. 이러한 목적을 위하여 많은 유형의 장치들 및 공정들이 사용되었다. 이러한 하나의 장치는 표면에 부착된 다이아몬드 입자들과 같은 복수의 초경질 결정질 입자들을 구비한 디스크 또는 이들의 기판이다.

불행하게도, 전통적인 방법에 의하여 제조된 이러한 연마 디스크들은 여러 가지 문제점들을 보인다. 첫째, 연마 입자들은 디스크의 기판으로부터 제거되어 CMP 패드 섬유질에 붙잡힐 수 있다. 이는 연마되는 피가공물의 긁힘 및 파손을 초래한다. 둘째, 선행 기술의 제조 방법들은 기판의 표면위에 고르지 않은 간격의 그룹으로 밀집되는 연마 입자들을 가지는 디스크들을 생산하기 쉽다. 입자들 사이에 생성된 균일하지 않은 간격은 CMP 패드의 일부분을 과다하게 드레싱되게 하여 마모흔들을 생성하고, 그밖의 다른 부분들을 덜 드레싱하여, 광택있는 층들을 생성한다. 셋째, 이러한 디스크들의 연마 입자들은 패드를 균일한 깊이로 침투하도록 배열되지 않는다. 이러한 비-균일성은 CMP 패드의 고르지 않은 드레싱을 추가적으로 생성한다. 마지막으로, CMP 패드가 유연한 정도에 따라, 드레서의 처음 리딩 에지 앞부분에서는 드레서에 의하여 생성되는 하향력으로 인하여 볼록해지거나 둥글어지는 경향이 있다. 이러한 볼록함은 패드가 드레서의 잔여 부분아래서 지나갈 때, 패드의 함몰이 일어나게 할 수 있으며, 순차적으로 남아있는 연마 입자들, 특히 패드 드레서의 중심에 위치한 입자들이 패드 전체에 걸쳐 덜 깊게 침투하게 또는 심지어는 침투하지 않게 할 수 있다. 드레서 입자들에 대한 이러한 고르지 않은 작업 부하는 패드를 고르지않게 드레싱 되도록 할 수 있으며, 또한 드레서를 고르지 않게 마모되게 하거나 조기에 마모되어 닳도록 할 수 있다.

또한 현재의 CMP 패드 드레서가 가지는 또다른 결점은 패드 컨디셔너의 사용 수명 감소이다. CMP 패드 컨디셔너의 사용 효과 및 효율성은 작업하는 연마 입자들의 수 및 각 입자가 수행하는 작업의 양에 의해 결정된다. 상기 지적한 바와 같이, 패드 컨디셔너의 사용 수명은 초연마 입자들에 대한 작업 부하의 고르지 않은 분포에 의해 감소될 수 있다. 가요성 CMP 패드가 드레서의 압력하에서 함몰할 때, 패드 컨디셔너의 리딩 에지 결정들에 과다한 마모가 발생할 수 있는데, 이는 이러한 결정들이 대부분의 작업 부하를 견디게 되기 때문이다. 또한, 중심에 위치한 연마 입자들은 동일한 작업 부하를 수용하지 못하게 된다. 이러한 작업 부하의 불일치는 리딩 에지 입자들에 대한 마모 속도를 증가시키고, 중심에 위치한 입자들의 소모 이전에 드레서를 쓸모없게 만들 수 있다.

입자 보유와 관련하여, 두가지 요인들이 연마 입자들로 하여금 선행 기술의 패드 드레서 디스크로부터 제거되기 쉽게 한다. 첫째, 제거는 종종 연마 입자들이 부착되었던 열등한 방법으로 인하여 발생한다. 전기도금된 니켈 또는 그밖의 다른 기저에 놓인 물질들에 의해서만 기판에 지지된 연마 입자들은 약한 기계적 힘에 의하여만 확보되며, 어떤 형태의 화학적 결합에 의하여도 확보되지 않는다. 그러므로, 이러한 입자들은 마찰력과 같은 강한 기계적 힘에 노출될 때 용이하게 제거되게 된다. 더욱이, 입자 제거는 화학물질 슬러리에 의하여 제공되는 전기도금 물질에 대한 화학적 공격에 의해 촉진된다.

대조적으로, 연마 입자들이 기판 위에 브레이징 될 때, 화학적 결합은 입자들을 더욱 견고하게 지지한다. 그러나, 화학물질 슬러리의 산은 브레이즈-입자 결합들을 급속하게 약하게 하며, 패드 드레싱의 마찰력 하에서 연마 입자들을 제거하게 할 수 있다. 그러므로, 화학물질들에 대한 브레이즈의 노출을 최소화하고 패드 드레서의 가용 수명을 연장하기 위하여, 연마 공정들은 드레싱이 발생하는 동안 정지되어야 한다. 결과적으로 연마 및 그 이후 드레싱을 교대시키는 연쇄는 시간을 낭비시키며, 비효율적이다.

브레이징 공정 동안 패드 드레서 작업 표면의 뒤틀림 또한 종종 연마 입자들을 제거되게 한다. 브레이징 공정 동안 패드 드레서는 상당한 고온에 노출되어야 한다. 이러한 극도의 열에 대한 노출은 패드 드레서의 작업 표면을 뒤틀리게 할 수 있으므로, 패드 드레서 작업 표면의 매끄러움 및 평면성을 손상시킨다. 결과적으로, 작업 표면의 브레이즈 부분은 거칠게 되며,

Ex. 1003, Page 101

높고 낮은 지점들을 가지게 될 것이다. 이러한 지점들은 바람직하지 않은데, 왜냐하면 이들은 브레이즈를 조각조각 떨어지기 시작하도록 만들 수 있으며, 피가공물의 연마된 표면에 미세-긁힘을 만들 수 있다. 또한, 이러한 울퉁불퉁함은 드레서의 또다른 가공, 및 연마 입자 보유력과 관련하여 문제점을 유발할 수 있다.

전술한 관점에서, 최대의 효율성 및 수명을 가지며 최적의 드레싱 결과를 구현하기 위하여 제작되고 구성된 CMP 패드 드레서가 계속하여 요구되고 있다.

발명의 요약

따라서, 한 양태에서, 본원 발명은 CMP 패드를 드레싱 하는 동안 CMP 패드 드레서에서 중심에 위치한 초연마 입자들에 대한 작업 부하를 증가시키기 위한 방법 및 CMP 패드 드레서 구조물을 제공한다. 이러한 한 방법에서, 각각이 기판 부재에 연결되고 예정된 패턴에 따라 특정 위치에 지지된 복수의 초연마 입자들을 가지는 CMP 패드 드레서가 제공된다. 초연마 입자들은 둘레에 위치한 입자들의 CMP 패드 내부로의 침투는 감소시키고 중심에 위치한 입자들의 CMP 패드 내부로의 침투는 증가시키는 패턴으로 배열될 수 있으므로, 중심에 위치한 초연마 입자들에 대하여 가해지는 작업 부하를 최적화 시킬 수 있다. 일반적으로, 입자들은 단일 결정 또는 다결정질 형태의 다이아몬드, 또는 입방정 질화 붕소 (cBN)와 같은 초경질 물질들이다.

본원 발명의 한 구체예에서, 중심에 위치한 초연마 입자들에 대한 작업 부하를 증가시키는 방법은 둘레에 위치한 입자들의 작업 말단으로부터 중심에 위치한 입자들의 작업 말단까지 상향인 기울기를 제공하는 패턴으로 배열된 초연마 입자들을 보유하는 기판을 가지는 CMP 패드 드레서의 이용을 포함한다. 또한, 이용되는 기울기의 정확한 각도는 중심에 위치한 입자들이 부담하는 작업 부하를 제어하도록 형성될 수 있다. 이러한 기울기는 다양한 방식으로 생성될 수 있다. 예를 들면, 한 양태에서, 기울기는 실질적으로 편평한 기판 위에 또는 내부에 초연마 입자들을 배치함으로써 생성될 수 있는데, 여기서 기판의 작업 표면위의 초연마 입자들의 높이는 둘레에 위치한 입자들로부터 중심에 위치한 입자들을 향하여 증가한다. 몇몇 경우에, 바람직한 기울기 각도는 패드 속도(velocity) 및 패드 가요성의 측정으로서 결정될 수 있다.

본원 발명의 또다른 바람직한 구체예에서, 중심에 위치한 입자들에 대한 작업 부하를 증가시키는 방법은 중심에 위치한 입자들보다 더 높은 밀도로 둘레에 위치한 초연마 입자들을 배치하는 패턴으로 기판에 결합된 복수의 초연마 입자들을 가지는 CMP 패드 드레서를 제공하는 것을 포함할 수 있다. 더 높은 밀도로 밀집된 입자들은 서로 더 먼 간격으로 밀집된 입자들만큼 깊게 패드 내부로 침투할 수 없음이 발견되었다. 그러므로, 기판 상의 입자들의 밀도를 변화시킴으로써 작업 부하는 한 부위에서 다른 부위로 이전될 수 있다.

또한 본원 발명의 또다른 구체예에서, 중심에 위치한 입자들에 대한 작업 부하를 증가시키는 방법은 중심에 위치한 입자들을 둘레에 위치한 입자들의 애티튜드(attitude)에 의해 제공되는 침투보다 CMP 패드 내부로 더 높은 입자 침투를 유발시키는 애티튜드로 배향시킴으로써 구현될 수 있다. 한 양태에서, 중심에 위치한 입자들의 애티튜드는 이들의 작업 말단에 꼭지점(apex)을 제공할 수 있으며, 둘레에 위치한 입자들의 애티튜드는 이들의 작업 말단에 면 또는 에지를 제공할 수 있다. 또다른 양태에서, 중심에 위치한 입자들의 애티튜드는 이들의 작업 말단에 에지를 제공할 수 있으며, 둘레에 위치한 입자들의 애티튜드는 이들의 작업 말단에 면을 제공할 수 있다. 또한 또다른 양태에서, 중심에 위치한 입자들의 애티튜드가 이들의 작업 말단에 꼭지점(apex)을 제공할 때, 둘레에 위치한 입자들의 애티튜드는 이들의 작업 말단에서 면을 제공할 수 있으며, 둘레에 위치한 입자들과 중심에 위치한 입자들 사이에 위치한 입자들의 애티튜드는 이들의 작업 말단에 에지를 제공할 수 있다. 상기 언급된 사용 방법 이외에도, 본원 발명은 또한 중심에 위치한 초연마 입자들에서 증가된 작업 부하를 보여주는 CMP 패드 드레서를 제공하는 방법을 포함한다. 일반적으로 말하면, 이러한 방법은 다음 단계들을 포함한다: 1) 기판을 제공하는 단계; 및 2) 둘레에 위치한 입자들의 CMP 패드 내부로의 침투는 감소시키고 중심에 위치한 입자들의 CMP 패드 내부로의 침투는 증가시키는 패턴으로 복수의 초연마 입자들을 기판 위에 부착하는 단계.

상기 설명된 방법을 사용하여, 상당한 이점들을 보이는 CMP 패드 드레서를 제조할 수 있다. 예를 들면, CMP 패드 드레서의 작업 표면은 외부 또는 이들의 "리딩 에지"를 지나치게 접촉하기 보다는 드레서의 중심 이하의 부분에서 CMP 패드의 접촉을 증가시키기 위하여 배열될 수 있다. 이러한 중심 접촉의 증가는 드레서의 둘레부로부터 드레서의 중심부로 작업 부하의 일부를 이전시키므로, 드레서의 사용 수명을 길게하고 드레서로 하여금 패드를 더욱 효과적으로 절삭 및 그루밍(grooming)할 수 있게 한다. 상기 배열을 결합시킨 CMP 패드 드레서는 본원 발명에 포함되며, 상기 언급된 방법들을 뒷받침하기 위하여 이루어진 특정 구조를 가지는 CMP 패드 드레서들도 포함한다.

본원 발명의 상기 언급된 특징들 및 이점들은 첨부된 도면 및 다음의 상세한 설명을 참고하면 더욱 자명해질 것이다.

Ex. 1003, Page 102

발명의 상세한 설명

발명의 상세한 설명

본원의 CMP 패드 드레서 및 수반되는 사용 및 제조 방법을 개시하고 설명하기에 앞서, 본원 발명은 본원에 개시된 특정 공정 단계들 및 물질들에 제한되는 것이 아니라, 관련 분야의 통상의 당업자가 인식하게 되는 이들의 균등물에까지 확장됨을 이해하여야 한다. 또한 본원에서 사용되는 용어는 단지 특정 구체예들을 설명하기 위하여 사용되는 것이며 제한하기 위하여 사용되되는 것이 아님을 이해하여야 한다.

본원 명세서 및 첨부된 청구항에서 사용되는, 단수형들 "하나" 및, "그것"은 명확하게 다른 언급이 없으면 복수형을 포함함을 알아두어야 한다. 그러므로, 예를 들면, 하나의 "연마 입자" 또는 "그릿"이라는 언급은 하나 또는 그 이상의 연마 입자들 또는 그릿들에 대한 언급을 포함한다.

정의

본원 발명을 설명하고 청구함에 있어서, 아래 설명된 정의에 따라 다음의 용어가 사용될 것이다.

본원에서 사용되는, "연마 입자" 또는 "그릿" 또는 유사한 용어들은 초경질의 결정질 또는 다결정질 물질, 또는 이들 물질들의 혼합물을 의미하며, 다이아몬드, 다결정질 다이아몬드 (PCD), 입방정 질화 붕소, 및 다결정질 입방정 질화 붕소 (PCBN)를 포함하나, 이에 제한되는 것은 아니다. 또한, "연마 입자", "그릿", "다이아몬드", "다결정질 다이아몬드 (PCD)", "입방정 질화 붕소" 및 "다결정질 입방정 질화 붕소(PCBN)" 의 용어들은 호환적으로 사용될 수 있다.

본원에서 사용되는, "기판"은 연마 입자들을 지지하는, 그리고 연마 입자들이 고정될 수 있는, CMP 드레서의 일부를 의미한다. 본원 발명에서 유용한 기판들은 의도한 목적에 유용한 공구를 제공하기에 충분한 방식으로 연마 입자들을 지지할 수 있는, 임의의 형상, 두께 또는 물질일 수 있다. 기판들은 고체 물질, 가공시 고체가 되는 분말 물질, 또는 가요성 물질일 수 있다. 전형적인 기판 물질들의 예에는 금속, 금속 합금, 세라믹, 및 이들의 혼합물을 포함하나, 이에 제한되는 것은 아니다. 또한 기판은 브레이징 합금 물질을 포함할 수 있다.

본원에서 사용되는, "작업 표면"은 작업하는 동안 위를 향하는, 또는 CMP 패드와 접촉하게 되는 CMP 패드 드레서의 표면을 의미한다.

본원에서 사용되는, "리딩 에지"는 CMP 패드가 움직이는 방향에 기초하여, 또는 패드가 움직이는 방향에 기초하여, 또는 이들 모두에 기초하여 전두 에지인 CMP 패드 드레서의 에지를 의미한다. 특히, 몇몇 양태에서, 리딩 에지는 특정하게 드레서의 에지 부위를 포함할 뿐만 아니라, 또한 실제 에지로부터 약간 안쪽으로 연장하는 드레서의 일부들을 포함할 수 있는 것으로 생각된다. 한 양태에서, 리딩 에지는 CMP 패드 드레서의 외부 에지를 따라 위치될 수 있다. 또다른 양태에서, CMP 패드 드레서는 CMP 패드 드레서 작업 표면의 중심 또는 내부 부분 위에 적어도 하나의 효과적인 리딩 에지를 제공하는 연마 입자들의 패턴으로 형성될 수 있다. 다시 말하면, 드레서의 중심 또는 내부 부분은 드레서의 외부 에지 위에 리딩 에지의 효과와 유사한 기능적 효과를 제공하도록 형성될 수 있다.

본원에서 사용되는, "날카로운 부분"은 결정이 올 수 있는 협소한 부분을 의미하며, 코너, 융기, 에지, 오벨리스크, 및 그밖의 다른 돌출부들을 포함하나, 이에 제한되는 것은 아니다.

본원에서 사용되는, "중심에 위치한 입자"는 통상의 드레싱 환경하에서 둘레에 위치한 입자에 비하여 감소된 작업 부하를 수용하는 드레서의 입자를 의미한다. 몇몇 양태에서, "중심" 또는 "중심에 위치한"이란 드레서의 중심 지점에서 시작하여 드레서 에지를 향해 바깥쪽으로 드레서 직경의 최대 약 90%까지 연장하는 영역을 의미한다. 몇몇 양태에서, 상기 영역은 반경의 약 20% 내지 약 90%까지 바깥쪽으로 연장할 수 있다. 그밖의 다른 양태에서, 상기 영역은 반경의 약 50%까지 연장할 수 있다. 또한 또다른 양태에서, 상기 영역은 드레서 반경의 약 33%까지 연장할 수 있다.

본원에서 사용되는, "둘레에 위치한"이란 통상적인 드레싱 환경하에서 중심에 위치한 입자들에 비하여 과다한 작업 부하를 수용하는 드레서의 입자를 의미한다. 몇몇 양태에서, "둘레의" 또는 "주변의" 또는 "둘레에 위치한"이란 드레서의 리딩 에지 또는 외부 가장자리에서 시작하여 드레서 직경의 최대 약 90%까지 중심을 향하여 안쪽으로 연장하는 영역을 의미할 수

있다. 몇몇 양태에서, 상기 영역은 반경의 약 20% 내지 90%까지 안쪽으로 연장할 수 있다. 그밖의 다른 양태에서, 상기 영역은 반경의 약 50%까지 연장할 수 있다. 또한 또다른 양태에서, 상기 영역은 드레서 직경의 약 33%까지 연장할 수 있다 (즉, 중심으로부터 66% 떨어짐).

본원에서 사용되는, "작업 부하"란 드레서를 사용하는 동안 드레서에 있는 입자들에 가해지는 작업량 또는 힘의 양을 의미한다.

본원에서 사용되는, "작업 말단"이란 CMP 패드를 향하여 배향되고 드레싱 작업을 하는 동안 패드와 접촉하는 입자의 말단을 의미한다. 거의 대부분 입자의 작업 말단은 입자가 부착되는 기판으로부터 말단이 될 것이다.

본원에서 사용되는, "비정질 브레이즈"란 비-결정질 구조를 가지는 균질의 브레이즈 조성물을 의미한다. 이러한 합금은 가열시 부조화 용융하는 공융상을 실질적으로 전혀 함유하지 않는다. 비록 간결한 합금 조성물을 확보하기 어렵지만, 본원에서 사용되는 비정질 브레이징 합금은 좁은 온도 범위에 걸쳐 실질적으로 조화용융 거동을 보여야 한다.

본원에서 사용되는, "합금"이란 제 2 물질과 금속의 고체 또는 액체 혼합물을 의미하며, 상기 제 2 물질은 탄소와 같은 비-금속, 금속, 또는 금속의 성질을 향상 또는 개선시키는 합금이 될 수 있다.

본원에서 사용되는, "금속 브레이징 합금," "브레이징 합금," "브레이즈 합금", "브레이즈 재료" 및 "브레이즈"는 호환적으로 사용될 수 있으며, 초연마 입자들, 및 매트릭스 지지 재료 또는 기판을 서로 실질적으로 결합시키기 위하여 화학적으로 결합할 수 있는 금속 합금을 의미한다. 본원에 개시된 특정 브레이즈 합금 성분들 및 조성물은 이와 관계되어 개시된 특정 구체예에 제한되는 것이 아니며, 본원에 개시된 본원 발명의 어떠한 구체예에서도 사용될 수 있다.

본원에서 사용되는, "브레이징" 공정은 초연마 입자들의 탄소 원자들과 브레이즈 재료 사이의 화학적 결합 생성을 의미하는 것으로 간주된다. 또한, "화학적 결합"이란 기계적 인력 또는 그보다 약한 원자 내부의 인력이라기 보다는, 탄화물 또는 보라이드 결합과 같은 공유 결합을 의미한다. 그러므로, "브레이징"이 초연마 입자들과 관련하여 사용되는 경우, 진정한 화학적 결합이 형성되는 것이다. 그러나, "브레이징"이 금속 대 금속 결합과 관련하여 사용되는 경우, 이 용어는 더욱 전형적인 야금학적 결합의 의미로 사용된다. 그러므로, 공구 몸체에 대한 초연마 세그먼트의 브레이징은 탄화물 형성제의 존재를 요하지 않는다.

본원에서 사용되는, "초연마 입자들" 및 "초연마 그릿들"은 호환적으로 사용될 수 있으며, 천연 또는 인공 다이아몬드, 초경질 결정질, 또는 다결정질 물질, 또는 물질들의 혼합물의 입자들을 의미하며, 다이아몬드, 다결정질 다이아몬드 (PCD), 입방정 질화 붕소 (CBN), 및 다결정질 입방정 질화 붕소 (PCBN)를 포함하나, 이에 제한되는 것은 아니다. 또한, "연마 입자", "그릿", "다이아몬드", "PCD", "CBN" 및 "PCBN"이란 용어들은 호환적으로 사용될 수 있다.

브레이징 공정과 관련하여 본원에서 사용되는, "직접적으로"란 결합 매질로서 단일 브레이징 금속 또는 합금을 사용하여 초연마 입자들 및 동일한 재료 사이에 화학적 결합의 형성을 의미하는 것으로 간주된다.

본원에서 사용되는, "돌기"란 다양한 표면 분석의 특성들에 의하여 평가된 표면의 거칠음을 의미한다. 다양한 측정들은 피크 높이 또는 그 위의 돌출, 및 밸리의 깊이 또는 그 내부에 함몰하는 함몰부와 같은 표면 돌기의 지표로서 사용될 수 있다. 더욱이, 돌기의 측정치에는 주어진 표면 영역 내부의 피크 또는 밸리의 수 (즉, 피크 또는 밸리 밀도), 및 피크들 또는 밸리들 사이의 거리가 포함될 수 있다.

본원에서 사용되는, "세라믹"이란 비-금속 물질, 때때로 금속 물질과 함께 연소함에 의해 제조될 수 있는, 상당히 내열성이며 내식성인, 경질의, 때때로 결정질의 재료를 의미한다. 세라믹으로 고려되는 수많은 산화물, 질화물, 및 탄화물 재료들이 당해 분야에 잘 알려져 있으며, 산화 알루미늄, 산화 알루미늄, 산화 규소, 질화 붕소, 질화 규소, 및 탄화 규소, 탄화 텅스텐, 등이 포함되나, 이에 제한되는 것은 아니다.

본원에서 사용되는, "금속성"이란 임의의 타입의 금속, 금속 합금, 또는 이들의 혼합물을 의미하며, 구체적으로는 강(스틸), 철, 및 스테인레스 강이 포함되나, 이에 제한되는 것은 아니다.

본원에서 사용되는, "격자"는 복수의 사각형을 형성하는 선들의 패턴을 의미한다.

거리 및 크기와 관련하여 본원에서 사용되는, "균일한"이란 전체 약 75 마이크로미터 미만으로 상이한 크기를 의미한다.

본원에서 사용되는, "Ra"란 피크 및 이웃하는 밸리 사이의 높이차로 결정되는 표면 조도의 측정치를 의미한다. 또한, "Rmax"는 표면 위에서 가장 높은 피크와 표면에서 가장 낮은 밸리 간의 높이차로 결정되는 표면 조도의 측정치이다.

콘센트레이션, 양, 및 그밖의 다른 수치 데이타는 본원에서 범위 형식으로 표현되거나 나타내어질 수 있다. 이러한 범위 형식은 단순히 편의 및 간결함을 위해 사용되는 것이므로, 범위의 한계로 명확히 언급되는 수치 값들 뿐만 아니라, 그 범위 내에 포함되는 모든 개개의 수치 값들 또는 하위-범위들도 마치 각각의 수치 값 및 하위-범위가 명확히 언급된 것처럼 포함되는 것으로 해석되어야 함을 이해하여야 한다.

설명하자면, "약 1 마이크로미터 내지 약 5 마이크로미터"의 수치 범위는 약 1 마이크로미터 내지 약 5 마이크로미터 중 명확히 언급된 수치들 뿐만 아니라, 지시된 범위에 속하는 개별적인 수치들 및 하위-범위들도 포함되는 것으로 해석되어야 한다. 그러므로, 상기 수치 범위에는 2, 3, 및 4와 같은 개개의 수치들 및 1-3, 2-4, 및 3-5, 등과 같은 하위-범위까지도 포함된다. 이와 동일한 원리가 단 하나의 수치 값을 언급하는 범위들에도 적용된다. 더욱이, 이러한 해석은 설명되는 범위의 폭 및 특성들에 관계없이 적용되어야 한다.

발명

출원인은 CMP 패드 컨디셔닝 또는 드레싱의 효율성과 품질을 개선하기 위한 장치 및 방법들을 발견하였다. CMP 패드를 컨디셔닝 또는 드레싱 하기 위한 장치를 사용함으로써, 패드 수명이 연장될 뿐만 아니라, 또한 패드가 사용될 수 있는 항구성, 및 그리하여 장치가 구현하는 장치의 작업 속도가 개선된다.

이제 도 1을 참고하면, 선행 기술의 CMP 패드 드레서(10)이 도시되는데, 이것은 기판 (40)에 전기도금된 복수의 연마 입자들(50)을 가진다. 전기도금 재료(60)는 일반적으로 산 용액으로부터 침전된 니켈이다.

기판에 연마 입자들 (50)을 부착하기 위하여 전기도금 재료(60)만을 사용하는 CMP 패드 드레서는 도 1에 도시된 바와 같이 자명한 많은 결점들을 가진다. 첫째, 전기도금 재료는 연마 입자들과 화학적 결합을 형성할 수 없다. 그러므로, 단지 약한 기계적 힘들이 기판(40) 상에서 연마 입자들을 지지한다. 패드 드레서가 CMP 패드를 대면하여 회전되는 경우, 이러한 기계적 힘들은 연마 입자들에 작용하는 마찰력에 의해 신속하게 극복된다. 결과적으로 연마 입자들은 전기도금 재료로부터 쉽게 헐거워져서, 전기도금 재료에 공간(70)과 같은 공극들을 남긴다. 이러한 공극들은 피가공물에서 연마되어 나온 잔여물 뿐만 아니라, 슬러리로부터 화학물질 및 연마 입자들로 신속히 채워진다. 이러한 물질들은 전기도금 재료를 화학적으로 공격하며 더욱 약화시킨다.

전기도금 재료(60)에 의해 생성된 기계적 힘은 기판(40) 위에 연마 입자들(50)을 지지시키는 단 하나의 수단이기 때문에, 연마 입자들 above the 전기도금 재료 위의 연마 입자들의 노출은 최소로 유지되어야 한다. 그럼에도 불구하고, 전기도금 재료와 CMP 패드 사이의 접촉은 필연적이다. 이러한 접촉은 전기도금 재료를 마모시키며, 연마 입자들의 방출을 더욱 촉진시킨다. 추가적으로, 제조하는 동안, 전기도금 재료는 볼록한 부분(80)과 같은 공간에서 연마 입자들 주위에 쌓이기 쉽다. 불가능한 것이 아니라면, 이미 낮은 노출 및 연마 입자들의 빽빽한 공간 이외에도 이러한 볼록한 부분들은 연마 입자들의 CMP 패드 섬유질 내부로의 상당한 침투를 어렵게 한다. 이러한 침투가 없다면, 드레싱 공정의 효과는 불리하다.

이제 도 2를 보면, 브레이징 재료(90) 및 전통적인 진공로 브레이징 기술을 사용하여 기판에 브레이즈된 연마 입자들(50)을 가지는 기판(40)을 구비한 선행 기술의 CMP 패드 드레서가 도시된다. 브레이징 재료(90)은 일반적으로 탄화물 형성제와 혼합된 금속 합금을 포함한다. 이러한 탄화물 형성제는 연마 입자들을 브레이징 재료에 화학적으로 결합할 수 있게 하여 순차적으로 기판과 결합할 수 있게 한다. 이러한 결합 배열은 CMP 드레서의 전체 강도를 현저히 증가시키지만, 몇가지 바람직하지 않은 부작용을 수반한다.

브레이징 재료(90)는 연마 입자들(50)을 완전히 커버하지 않도록 하기 위하여 최소로 유지되어야 한다. 그러므로, 연마 입자들은 브레이징 재료의 얇은 코팅만으로 감싸진다. 이러한 문제는 전형적인 브레이징 재료들이 기계적으로 너무 약하다는 사실에 의해 사화된다. 이러한 기계적 결함은 연마 입자들과 브레이징 재료 사이에 생성되는 화학 결합의 강도를 상쇄시킨다. 사실상, 연마 입자들과 브레이징 재료 사이의 화학 결합들은 떨어져 나간 연마 입자들과 함께 브레이징 재료 자체가 종종 전단되어 나가기에 충분히 강하다.

브레이징 재료(90)는 또한 연마 슬러리에 의한 화학적 공격에 매우 민감하다. 이는 연마 입자들(50)의 탈착에 기여하는데, 왜냐하면 연마 입자들의 탈착은 브레이징 재료를 더욱 약화시키기 때문이다. 화학물질 슬러리에 대한 CMP 패드 드레서

(20)의 노출을 감소시키기 위하여, 피가공물의 연마는 중단되어야 하며, 화학물질 슬러리는 패드 드레서에 도포되기 전에 패드에서 제거되게 하여야 한다. 연마 공정에서 이러한 중단은 패드가 사용될수 있는 항구성을 현저히 감소시키고, 완제품을 제조하는데 필요한 시간을 증가시키므로, 비효율적이다.

연마 입자들(50)을 전통적인 브레이징만을 사용하여 기판(40)에 결합시키는 것에 대한 또다른 결점은 용융 금속 합금이 기판에 사용될 경우 연마 입자들의 "밀집"을 유발하기 쉽다는 것이다. 이러한 밀집은 (100)에 도시되는데, 의도하지 않은 공백(110)을 남긴다. 전체적인 효과는 연마 입자들의 비-균일한 분포이며, 이것은 그루밍(grooming)을 비효율적이게 한다. 또한, 공백들은 패드의 고르지않은 컨디셔닝을 유발하며, 이는 궁극적으로 CMP 패드의 특정 영역을 그밖의 다른 영역보다 더 빠르게 마모시키며, 전체적으로는, 마모된 영역들이 적절히 컨디셔닝된 영역 보다 덜 효과적으로 연마되기 때문에 피가공물이 고르지 않은 연마를 수용하게 되는 결과를 가져온다.

연마 입자들의 밀집은 브레이징 재료(90)에 둑을 형성함으로써 또다른 손실을 만든다. 둑 형성은 일부 연마 입자들을 기판(40) 위의 높이까지 상승하여 형성되며, 이것은 그밖의 다른 연마 입자들의 높이보다 더 높다. 그러므로, 가장 높이 돌출한 연마 입자들은 CMP 패드의 섬유질 내부로 매우 깊이 침투할 수 있으며, 그보다 덜 돌출한 연마 입자들이 CMP 패드를 접촉하지 못하게 하거나 유용한 그루밍 효과를 가지지 못하게 할 것이다.

선행 기술의 CMP 패드 드레서와 대조적으로, 본원 발명은 CMP 패드의 고른 드레싱을 가능하게 한다. 이제 도 3을 보면, 본원 발명의 원리에 따라 제조된 CMP 패드 드레서(30)이 도시되어 있다. CMP 패드 드레서는 브레이징 재료(90)을 가진 기판(40)에 결합된 복수의 연마 입자들(50)을 가진다.

연마 입자들(50)은 다양한 초경질 재료로 이루어질 수 있다. 이러한 재료의 예에는 다이아몬드, 다결정질 다이아몬드(PCD), 입방정 질화 붕소 (CBN) 및 다결정질 입방정 질화 붕소 (PCBN)가 포함되나, 이에 제한되는 것은 아니다.

추가적으로 도 3에는 최종 브레이징 공정 후에 처리되는 오버레이 재료층(120)이 도시된다. 상기 언급한 바와 같이, 오버레이는 브레이징 합금의 작업 표면보다 실질적으로 더 매끄러운 작업 표면을 제공한다. 이러한 매끄러움 및 평면성은 수많은 이점들을 제공하는데, 이 이점들에는 브레이즈를 조각조각 떨어뜨리는 것으로부터 미세-긁힘의 발생 감소 및 내식층이 포함되는 경우 내식층과 더 우수한 결합이 포함된다. 한 양태에서, 오버레이 재료의 작업 표면은 약 1 마이크로미터 미만의 Ra 값을 가질수 있다.

수많은 적절한 오버레이 재료가 사용될 수 있다. 그러나, 한 양태에서, 오버레이 재료들에는 주석, 니켈, 텅스텐, 코발트, 크롬, 및 지르코늄 니켈 합금과 같은 이들의 합금이 포함되나, 이에 제한되는 것은 아니다. 오버레이 재료는 다양한 방법들에 의해 처리될 수 있다. 오버레이 재료를 처리하는 방법들의 예에는 전기도금 및 물리적 증기 증착 (PVD)이 포함된다. 오버레이 재료층은 특수한 결과를 달성하기 위하여 필요한 두께일 수 있으나, 본원 발명의 한 양태에서, 오버레이 재료층은 약 0.1 내지 50 마이크로미터 두께의 두께를 가질 수 있다. 또다른 양태에서, 오버레이의 두께는 약 0.1 내지 약 5 마이크로미터일 수 있다.

또한 도 3에는 내식성 층(130)이 도시되어 있다. 선택적인 내식성 층은 연마 입자들(50)이 기판(40)에 고정된 후 CMP 패드 드레서의 표면 위에 형성된다. 한 양태에서, 내식성 층은 다이아몬드-형 탄소 (DLC), 또는 비정질 다이아몬드와 같은 초연마 재료일 수 있다. 한 구체예에서, 내식성 층은 약 80% 이상의 원자 탄소 함량을 가진다. 추가적으로, 내식성 층은 특수한 결과를 달성하기 위하여 필요한 다양한 두께를 가질 수 있으나, 일반적으로 그 두께는 0.5 내지 5 마이크로미터 범위이다. 한 양태에서, 내식성 층은 3 마이크로미터 미만의 두께를 가진다. 이러한 얇은 내식성 층은 CMP 패드 드레서의 작업 표면이 CMP 패드를 드레싱하는 연마 입자들의 능력을 감소시키지 않고 보호되는 것을 보장한다. 내식성 층은 일반적으로 물리적 증기 증착(PVD)법을 사용하여 제조된다. 흑연 양극과 함께 양극 아크를 사용하는 것과 같은 PVD 법은 일반적으로 당해 분야에 공지이다.

내식성 층(130)에 의해 제공되는 하나의 이점은 내식성 층이 작업 표면을 효과적으로 "밀폐"한다는 점이며, 또한 화학적 공격에 약할 수 있는, CMP 패드 드레서(30)의 그밖의 다른 표면들을 밀폐할 수 있다는 점이다. 밀폐제로서, 내식성 층은 CMP 패드 내부에 지지된 연마성 화학물질 슬러리에 의한 화학적 공격으로부터 브레이징 재료(90)를 보호한다. 이러한 보호는 CMP 패드 드레서로 하여금 즉석에서 CMP 패드를 드레싱할 수 있게 하며, 선행 기술의 CMP 패드 드레서의 가용 수명을 연장시키기 위하여 사용되는 제조 정지를 제거할 수 있게 한다. CMP 패드의 연속적이고 고른 드레싱은 더욱 많은 제조 작업 산출량을 가능하게 하며, CMP 패드의 수명 및 효율성을 연장시킨다.

Ex. 1003, Page 106

내식성 층(130)이 본원 발명의 몇몇 구체예에서 사용될 수 있으나, 오버레이 재료(120)는 그 자체로써 그리고 자연히 상당한 내식성을 가진다는 점이 두드러진다. 오버레이 재료만이 사용되고, 내식성 층이 사용되지 않는 경우, 이와 같은 많은 제조 이점들을 상당한 정도로 얻을 수 있다.

연마 입자들(180)을 기판에 고정시키는 한 가지 방법이 도 4-6에 도시된다. 먼저, 개구들(150)을 가지는 형판(140)이 브레이징 합금(190) 쉬트 위에 배치된다. 본원 발명의 한 양태에서, 브레이징 합금 쉬트는 연속적 비정질 브레이징 합금의 압연 쉬트일 수 있다. 또다른 양태에서, 브레이징 합금 쉬트는 결합제 재료와 함께 지지되는 브레이징 합금 분말일 수 있다. 또다른 양태에서, 브레이징 합금 분말에는 그밖의 다른 금속성 분말들이 포함될 수 있으며, 이러한 그밖의 다른 분말들은 브레이징 쉬트에서 재료의 대부분을 구성할 수 있다. 또한 또다른 양태에서, 쉬트는 기판으로서 기능하기에 충분할 수 있다. 형판의 사용은 개구들을 가지는 형판을 원하는 패턴으로 디자인함으로써 특정 위치에 각 연마 입자를 제어하여 배치할 수 있게 한다.

형판(140)이 브레이징 합금 쉬트(190) 위에 배치된 후, 개구들(150)은 연마 입자들(180)로 채워진다. 개구들은 오직 하나의 연마 입자 만이 각 개구에 채워지도록 예정된 크기를 가진다. 어떠한 크기의 연마 입자 또는 그릿이라도 사용할 수 있으나, 본원 발명의 한 양태에서, 입자 크기는 약 100 내지 약 350 마이크로미터 직경일 수 있다.

본원 발명의 또다른 양태에서, 형판에 있는 개구들의 크기는 균일한 크기 범위에 속하는 크기를 가지는 연마 입자들의 패턴을 얻기 위하여 주문제작될 수 있다. 한 구체예에서, 형판의 개구들은 50 마이크로미터 이하의 변화량을 가지는 크기 범위에 속하는 그릿들만을 선택하기에 충분하다. 이러한 그릿 크기의 균일성은 CMP 패드 그루밍의 균일성에 기여하는데, 이는 각각의 연마 입자의 작업 부하가 고르게 분포되기 때문이다. 순차적으로, 고른 작업 부하 분포는 개별적인 연마 입자들에 대한 응력(stress)을 감소시키고, CMP 패드 드레서의 유효 수명을 연장시킨다.

형판(150)의 개구들이 그릿들(180)로 모두 채워진 후, 과량의 연마 입자들이 제거되고, 편평한 표면(160)이 연마 입자들에 처리된다. 편평한 표면(160)은 브레이징 합금 쉬트(190) 내부로 연마 입자들을 내리누를 수 있도록 극히 강하고 단단한 재료여야 한다. 이러한 재료들에는 전형적으로 스틸, 철, 이들의 합금 등이 포함되나, 이에 제한되는 것은 아니다.

연마 입자들(180)은 도 6에서 브레이징 합금 쉬트(190)에 묻혀 있는 것으로 도시된다. 표면(160)이 편평하기 때문에, 연마 입자들은 기판으로부터, 예정된 균일한 높이까지 확장될 것이다. 이러한 균일한 높이는 T형판(140)의 두께에 의해 결정될 것이며, 바람직한 구체예에서, 각각의 연마 입자는 상기 거리의 50 마이크로미터 이내까지 확장할 것이다. 이와 같이, 각각의 연마 입자는 CMP 패드 상에서 실질적으로 동일한 깊이로 그루밍된다. 그러나, 어떠한 응용예에서는, 그릿 높이가 균일한 것이 바람직하지 않을 수도 있음을 알아두어야 한다. 이와 같이, 다양한 높이의 그릿 패턴들은 그러한 디자인을 제공하기 위해 형판(140) 및 표면(160)을 다양한 높이로 형성함으로써 제공될 수 있음을 당해 분야의 통상의 당업자들은 알고 있을 것이다. 예를 들면, 한 양태에서, 표면(160)은 둘레에 위치한 입자들을 중심에 위치한 입자들보다 더 아래로 가압하기 위하여 오목한 형상을 가질 수도 있다. 알 수 있는 바와 같이, 이러한 오목한 형상은, 둘레의 입자들의 작업 말단에서의 낮은 지점에서 시작하여 중심에 위치한 입자들의 작업 말단에서의 높은 지점까지 기울어지는, 연마 입자들을 위한 기울기를 제공할 것이다.

도 4-6에 도시된 연마 입자들(180)은 둥글다. 그러나, 도 3에서, 이들은 뾰족하다. 자형, 팔면체, 입방정 팔면체, 또는 천연적으로 형성된 입자들을 포함하여, 어떠한 형상의 연마 입자들이라도 본원 발명의 범위에 포함된다. 그러나, 한 구체예에서, 연마 입자들은 기판(40)으로부터 한 방향으로 뻗어나가는 뾰족한 지점 또는 꼭지점(apex)을 보유한 예정된 형상을 가진다.

다른 구체예에서, 연마 입자들(180)을 브레이징 합금 쉬트(190) 내부로 가압하기 보다는, 연마 입자들은 브레이징 합금 쉬트 표면 위에 접착제를 도포하여 형판에서와 같은 위치에 고정될 수 있다. 이러한 방식에서, 입자들은 형판이 제거되고 열처리되는 동안 제 위치에 고정된 채로 남는다. 또한 본원 발명의 또다른 구체예에서, 형판(140)은 그 위에 얇은 접착제를 보유한 전달 쉬트(도시되지 않음) 위에 놓일 수도 있다. 이러한 경우, 입자들은 상기 설명한 형판 절차를 사용하여 전달 쉬트에 부착되게 된다. 이후 형판은 제거되고, 전달 쉬트는 쉬트를 대면하는 연마 입자들과 함께 브레이징 쉬트(190) 위에 놓인다. 브레이징 쉬트위에 도포된 상기 접착제 층은 전달 쉬트 위의 접착제보다 훨씬 더 강한 접착제이다. 그러므로, 연마 입자들은 형판에 의해 지정된 패턴으로 브레이징 합금의 쉬트로 전달된다.

연마 입자들(180)이 브레이징 합금 쉬트(190) 내부에 적어도 부분적으로 묻히거나, 쉬트에 부착된 후, 쉬트는 도 3에 도시된 바와 같이 기판에 고정된다. 대안적으로 몇몇 구체예에서, 브레이징 합금 쉬트는 기판에 먼저 고정될 수 있으며, 연마 입자들은 본원에 설명된 형판 절차를 사용하여 후속적으로 브레이징 합금 쉬트에 추가된다. 사용되는 브레이징 합금은 당

해 분야에 공지된 어떠한 브레이징 재료일 수도 있으나, 한 양태에서, 2 중량% 이상의 크롬 함량을 가지는 니켈 합금일 수 있다. 이러한 조성의 브레이징 합금은 그 자체로 그리고 자연적으로 거의 초경질이 될 것이며, 연마제를 함유하는 슬러리로부터의 화학적 공격에 덜 민감할 것이다. 그러므로, 내식성 층(130), 및 오버레이 재료(120)는 선택적이다.

연마 입자들(50)이 브레이징 합금 쉬트(90) 안에 또는 위에 단단히 지지되기 때문에, 액체 브레이징 합금의 표면 장력은 도 2에서 도시된 바와 같은 입자 밀집을 유발하기에 불충분하다. 게다가, 브레이즈 농축은 훨씬 적은 정도로 발생하며, "둑(mound)"이 거의 또는 전혀 형성되지 않는다. 그보다, 브레이즈는 각 연마 입자 사이에 약간 오목한 표면을 형성하는데, 이는 추가적인 구조적 지지를 제공한다. 한 구체예에서, 브레이징 합금 쉬트(90)의 두께는 각 연마 입자의 적어도 약 10 내지 90%를 브레이징 재료(90)의 외부 또는 작업 표면 위로 돌출될 수 있도록 예정된다. 또다른 양태에서, 오버레이 재료(120)가 사용될 때, 연마 입자들은 각 연마 입자의 적어도 약 10 내지 약 90%가 오버레이 재료(120)의 외부 또는 작업 표면 위로 돌출될 수 있도록 선택되거나 배치될 수 있다.

가공하는 동안 연마 입자들(50)을 고정된 위치에 유지시키는 방법의 결과로서, 연마 입자들 사이에 고른 간격들이 형성될 수 있다. 게다가, 연마 그릿들은 기판(40) 위의 균일한 높이 또는 거리까지 확장할 수 있는데, 이는 CMP 패드에 적용될 때, 연마 그릿들은 패드 섬유질 내의 균일한 깊이까지 돌출하게 되는 것을 의미한다. 고른 간격 및 균일한 돌출은 CMP가 고르게 드레싱 또는 그루밍될 수 있게 하며, 이는 순차적으로 CMP 패드의 연마 효율성을 증가시키고, CMP 패드의 가용 수명을 연장시킨다. 연마 입자들을 브레이징 합금에 묻거나 부착시키는 상기 설명한 방법 이외에도, 당업자들은 연마 입자들을 기판에 고정시킨 후, 브레이즈를 그 위에 배치하는 것과 같은 적절한 대안 절차들을 알고 있을 것이다. 이러한 경우, 입자들은 상기 언급된 형판 방법을 사용하여 기판 위에 배치될 수 있으며, 아교, 또는 그밖의 다른 결합제에 의하여 제 위치에 지지될 수 있다. 이후 브레이즈 재료는 헹궈지거나, 연마 입자들 주위의 기판 위에 배치되고, 오버레이 재료가 첨가된다.

본원 발명은 상기 설명한 방법을 사용하여 생성될 수 있는 연마 입자 배치를 위한 널리 다양한 패턴들을 포함하지만, 본원 발명의 한 양태는 CMP 패드 드레서가 사용되는 특수한 필요성 및 조건들을 더욱 적절하게 충족시키는 특수한 예정 패턴의 인식물이다. 상기 패턴들을 구현하기 위하여, 각각의 그릿은 패턴 디자인에 따라 특수 위치에 배치되고 지지된다. 이러한 패턴들은 특수한 CMP 패드 드레싱 결과를 달성하기 위해 실제로 유용하며, 알 수 있는 바와 같이 특수한 그루밍 결과를 달성하기 위하여 다양해질 수 있다.

예를 들면, 많은 공지된 패드의 그루밍 결과는 그릿들을 특정한 배열로 배치함으로써 개선될 수 있다. 특히, CMP 패드가 가요성이기 때문에, 드레서에 의해 발생되는 하향 압력은 주어진 방향으로 움직이는 드레서의 리딩 에지와 패드가 접촉될 때 패드를 상승하게 또는 올려지게 한다. 상승하는 작용은 연마 입자들과 더 최대의 접촉을 가능하게 하기 때문에 드레서의 리딩에지에서 패드의 드레싱을 개선시킬 수 있지만, 이는 또한 드레서의 리딩 에지 아래 이미 통과된 패드 부분에서는 침적 작용을 유발할 수 있다. 심지어 어떠한 침적도 전혀 발생하지 않는다 하더라도, 패드는 일단 드레서 아래에서 다시 한 번 상승할 수 있게 되지 않기 때문에, 일반적으로 리딩 에지 후방에 있는 드레서 잔여 부분의 드레싱 작용은 리딩 에지의 드레싱 작용보다 덜 효과적이다 (즉, 연마 입자들의 첫번째 열은 드레서, 또는 회전하는 CMP 패드, 또는 모두의 방향성 거동에 의해 지정된 바에 따라 패드와 직면됨). 이와 같이, 드레싱 작업 부담의 대부분이 드레서의 리딩 에지에 있는 연마 입자들에 배치되며, 고르지 않은 입자 마모가 발생한다.

각 입자의 침투 깊이는 주로 두 개의 요인, 즉, 다른 입자들로부터의 분리 거리 및 돌출 높이에 의해 제어된다. 성긴 간격의 입자들은 빽빽하게 밀집된 입자들보다 더욱 공격적으로 드레싱 할 것이다. 그러므로, 본원 발명의 한 양태에서, 연마 입자들의 패턴은 내부 또는 중심 위치(즉, 리딩 에지에 후속하는 위치)에서 드레서 아래에 있는 동안 CMP 패드를 상승하게 하고, 그리하여 입자들이 리딩 에지의 연마 입자들에 후속하는 연마 입자들에 의해 드레싱되도록 배열될 수 있다. 실제로, 이러한 배열은 드레서의 작업 표면을 따라 리딩 에지의 다양성을 제공한다. 다시 말하면, 둘레에 있는 입자들은 중심에 위치한 입자들의 밀도 보다 더 큰 밀도를 가진다. 둘레의 입자들의 밀도는 중심 입자들의 밀도보다 적어도 약 1.25, 2, 또는 5배 더 클 수 있다. 또한 밀도는 둘레의 입자들에서 높고 중심 입자들에서 낮은 구배가 될 수 있다. 이러한 방식에서, 다양한 밀도들은 CMP 패드가 패드 드레서의 중심 부분 아래에 있는 동안 CMP 패드를 상승하게 하고, 드레싱 효율을 증가시킨다. 알게 되는 바와 같이, 다양한 입자 배열 또는 패턴들은 상기 작용들을 구현하는데 필요한 연마 입자들의 간격을 제공할 수 있으며 특수하게 필요로하는 드레싱 결과를 달성하기 위하여 사용될 수 있다.

도 7에서 실시예로서 설명되는 바와 같이, 본원 발명의 한 양태에서, 연마 입자들은 패드 드레서(30)의 리딩 에지(200)를 따라서만 배치된 연마 입자들을 가지기 위하여 배열될 수 있다. 이제 도 8을 보면, 본원 발명의 또다른 양태에서, 연마 입자들은 중심(210)에서 보다 리딩 에지(200)을 따라 더 고도로 밀집되도록 (즉 더 큰 밀도를 가지도록) 배열될 수 있다. 대조적으로, 본원 발명의 또다른 양태에서, 연마 입자들은 리딩 에지를 따라서 보다는 중심에 더 많이 밀집되도록 배열될 수 있다 (도시되지 않음). 또한 또다른 양태에서, 입자들은 둘레에 있는 입자들 보다는 패드 드레서의 중심 부분에서 더 많은 밀집도로 배열되어 분포될 수 있다. 또한, 중심 및 둘레 부분들 사이에 위치한 입자들은 중심 부분과 둘레 부분의 밀도 사이

의 밀도를 가지도록 배열된다. 이제 도 9를 보면, 본원 발명의 또다른 양태에서, 연마 입자들은 전술한 패드 상승을 가능하게 하기에 충분한, 각 입자들 사이의 간격으로 균일하게 분포되도록 배열될 수 있다. 한 양태에서, 균일하게 분포된 입자들은 격자를 형성할 수 있으며, 각 개별적인 그릿 크기의 약 1.5 내지 약 10배의 거리로 고르게 간격지어 질 수 있다. 당업자가 알게 되는 바와 같이, 연마 입자들은 또한 콘센트레이션에 있어서 리딩 에지로부터 CMP 패드 드레서의 중심을 향하여 증가하거나 감소하는 다양한 콘센트레이션 구배로 배열될 수 있다 (도시되지 않음).

또다른 구체예에서, 본원 발명은 드레서를 구비한 CMP 패드의 드레싱을 하는 동안 CMP 패드 드레서에서 중심에 위치한 초연마 입자들에 대한 작업 부하를 증가시키는 방법을 제공한다. 상기 방법은 둘레에 위치한 입자들의 CMP 패드 내부로의 침투는 감소시키고 중심에 위치한 입자들의 CMP 패드 드레서 내부로의 침투는 증가시키는 패턴으로 초연마 입자들을 배열한다. 몇몇 양태에서, 초연마 입자들은 예정된 패턴에 따라 CMP 패드 기판위의 특수한 위치에 각각 개별적으로 배치된다. 중심에 위치한 입자들에 대한 작업 부하는 둘레에 위치한 입자들의 작업 부하의 적어도 약 10% 내지 약 30% 이내까지 증가될 수 있다. 또한 작업 부하는 둘레의 입자들 또는 모든 입자들의 작업 부하와 실질적으로 동일할 수 있다.

중심에 위치한 입자들에 대한 작업 부하를 증가시키는 것은 여러가지 방식으로 구현될 수 있다. 예를 들면, 초연마 입자들은, 도 10에 도시된 바와 같이, 둘레의 입자들의 작업 말단으로부터 중심 입자들의 작업 말단을 향하여 상향의 기울기를 제공하는 패턴으로 배열될 수 있다. 작업 부하를 증가시키는 또다른 방법은, 상기 설명한 바와 같이, 중심에 위치한 입자들의 밀도보다 더 높은, 둘레에 위치한 입자들의 밀도를 제공하는 패턴으로 초연마 입자들을 고정시키는 것이다. 마지막으로, 도 11에 도시된 바와 같이, 상기 패턴은 둘레에 위치한 입자들의 애티튜드에 의해 제공되는 침투보다 CMP 패드 내부로 더 큰 입자 침투를 유발하는 애티튜드를 중심에 위치한 입자들에 제공하도록 배열될 수 있다.

도 10을 참고하면, 본원 발명은 둘레의 초연마 입자들(320)로부터 중심에 위치한 입자들(310)을 향하여 상향의 기울기(305)를 가지는 초연마 입자들에 결합된 기판(300)을 제공함으로써 중심에 위치한 초연마 입자들에 대한 작업 부하를 증가시키는 CMP 패드 드레서를 제공한다. 상향의 기울기는 둘레에 위치한 입자들로부터 중심에 위치한 입자들 쪽으로 입자 높이를 증가시킴으로써 생성될 수 있다. 결과적으로, 상향의 기울기는 중심의 입자들과 CMP 패드에 더욱 큰 접촉을 제공함으로써 작업 부하를 둘레의 입자들로부터 중심의 입자들로 전달한다. 증가된 접촉은 CMP 패드의 드레싱 및 패드 컨디셔너의 총 마모를 개선시킨다. 기울기는 패드 속도(velocity) 및 패드 가요성의 측정으로서 결정된다. 일반적으로, 패드는 패드가 리딩 에지 입자들과 접촉될 때 함몰하는 변형가능한 매질이다. 통상적으로, 패드의 함몰은 패드의 가요성 및 패드의 회전속도에 따라 증대될 것이다. 본원 발명의 바람직한 구체예에서, 기울기는 약 0.1% 내지 0.5%이며, 바람직하게는 기울기는 0.2%이다.

대안으로서, 기울기는 기판의 배열을 변형시킴으로써 얻어질 수 있다. 도 10에 도시된 바와 같이, CMP 패드 드레서의 기판은 실질적으로 편평하나, 몇몇 양태에서, 기판은 회전하는 CMP 패드의 함몰에 맞게 변형하기 위해 윤곽지어질 수 있다. 이러한 윤곽은 연마 입자들(310 및 320)의 작업 말단에 필요한 기울기를 제공할 수 있다. 이러한 경우, 기판 작업 표면 위의 입자들의 높이는 실질적으로 균일할 것이다. 기판은 통상적으로 금속성, 세라믹, 또는 가요성 재료로 제조된다. 한 구체예에서, 기판은 스테인레스 강일 수 있다. 몇몇 양태에서, 기판은 가공시에 고체로 되는 분말화된 재료일 수 있다. 또다른 양태에서, 분말은 크롬과 같은, 약 2 중량% 이상의 양의 탄화물 형성 원소와 조합된 니켈과 같은 금속의 브레이징 합금을 포함할 수 있다. 몇몇 양태에서, 기판은 본질적으로 브레이징 재료로 구성될 수 있다.

도 11은 CMP 패드를 드레싱하는 동안 CMP 패드 드레서의 중심에 위치한 입자들에 대한 작업 부하를 증가시키는 CMP 패드 드레서의 도시이다. 연마 입자들은 입자들의 애티튜드에 따라 상이한 속도로 마모한다. 일반적으로, 꼭지점(apex)의 애티튜드는 CMP 패드 내부로 더 많은 침투를 제공할 것이며 그밖의 다른 애티튜드 보다 더욱 공격적으로 그루밍할 것이다. 면의 애티튜드를 가지는 입자는 최소량의 침투를 제공하며, 패드를 드레싱함에 있어서 가장 적은 공격성을 띤다. 애티튜드로서 에지를 가지는 입자는 중간 정도의 그루밍 및 침투 특성을 제공한다. 도 11을 참고하면, 기판(400)은 복수의 초연마 입자들(410, 420, 430)을 예정된 패턴으로 수용한다. 패턴은 패드 드레서의 둘레에 위치한 입자들의 애티튜드에 의해 제공되는 침투보다 더 높은 입자 침투를 CMP 패드 내부로 유발하는 애티튜드를 중심에 위치한 입자들에 제공하도록 배열된다. 중심에 위치한 초연마 입자들(410)은 입자들의 작업 말단(405)에 꼭지점(apex)을 제공하는 애티튜드로 배향된다. 이러한 입자들은 패드를 더욱 공격적으로 다듬으며, 그밖의 다른 입자들에 의해 제공되는 애티튜드보다 더 큰 정도의 침투를 가진다. 둘레의 입자들(420)은 이들의 작업 말단에서 에지 또는 면(430)을 제공하는 애티튜드로 배향될 수 있다. 특히, 도 11의 구체예에서 도시된 바와 같이, 중심에 위치한 입자들은 이들의 작업 말단(405)에서 꼭지점(apex)을 제공하는 애티튜드로 배향되고, 둘레의 입자들의 애티튜드(430)는 이들의 작업 말단(405)에서 면이며, 이들 사이의 입자들(420)은 이들의 작업 말단에서 에지를 제공하는 애티튜드로 배향될 수 있다. 그러나, 그밖의 다른 전형적인 구체예에서, 둘레의 입자들과 중심의 입자들 사이에 위치한 입자들은 다른 유형 중 하나와 동일할 것이다. 하나의 추가 구체예에서 (도시되지 않음), 중심에 위치한 입자들은 이들의 작업 말단에 에지를 제공하는 애티튜드로 배향될 수 있으며, 둘레에 위치한 입자들은 이들의 작업 말단에 면을 제공하는 애티튜드로 배향될 수 있다.

Ex. 1003, Page 109

상기 언급한 방법 및 장치들 이외에도, 본원 발명은 본원에 설명된 CMP 패드 드레서의 제조 방법을 제공한다. 한 양태에서, 이러한 방법은 기판을 제공하는 단계 및 둘레에 위치한 입자들의 CMP 패드 내부로의 침투를 감소시키고 중심에 위치한 입자들의 CMP 패드 내부로의 침투를 증가시키는 패턴으로 초연마 입자들을 기판에 부착하는 단계를 포함한다.

산업상 이용 가능성

수많은 변형 및 수정이 본원 발명의 원리 및 범위에서 벗어나지 않고 당업자에 의하여 고안될 수 잇으며, 첨부된 청구항들은 이러한 변형 및 배열을 뒷받침하는 것으로 간주된다. 그러므로, 가장 실용적이고 바람직한 본원 발명의 구체예로 생각되는 것과 관련하여 본원 발명을 특수하고 상세하게 상기와 같이 설명하였으나, 본원에 설명된 원리 및 개념에서 벗어나지 않고 크기, 재료, 형상, 형태, 기능, 작업 방식, 조합, 및 용도의 변화(이에 제한되는 것은 아님)를 포함한, 수많은 변형이 이루어질 수 있음이 당업자에게는 자명할 것이다.

도면의 간단한 설명

도면의 간단한 설명

도 1은 본원 발명의 한 구체예에 따른 디스크 기판에 연마 입자들을 고정시키기 위하여 전기도금법을 사용하는 선행 기술의 CMP 패드 드레서의 측면도이다.

도 2는 디스크 기판에 연마 입자들을 고정시키기 위하여 전통적인 브레이징법을 사용하여 제조된 선행 기술의 CMP 패드 드레서의 측면도이다.

도 3은 본원 발명의 한 구체예에 따라 제조된 CMP 패드 드레서의 측면도이다.

도 4는 본원 발명의 한 구체예에 따라, 브레이징 합금 쉬트의 표면위에 연마 입자들을 배치하기 위한 형판을 구비한 브레이징 합금 쉬트의 측면도이다.

도 5는 브레이징 합금 쉬트의 표면에 형판을 구비한 브레이징 합금 쉬트의 측면도이며, 연마 입자들이 형판의 개구들을 채우고 있다. 본원 발명의 한 구체예에 따라 편평한 표면은 브레이징 합금 쉬트 내부로 연마 입자들을 가압하는데 사용하기 위하여 도시되어 있다.

도 6은 본원 발명의 한 구체예에 따라 브레이징 합금 쉬트 내부로 가압된 연마 입자들을 가지는 브레이징 합금 쉬트의 측면도이다.

도 7은 본원 발명의 한 구체예에 따라, 실질적으로 오직 드레서의 리딩 에지를 따라서 연마 입자들이 존재하도록 기판에 결합된 연마 입자들을 가지는 CMP 패드 드레서의 작업 표면의 평면도이다.

도 8은 본원 발명의 한 구체예에 따라, 중심에서 보다 리딩 에지에서 더 많은 입자들이 존재하도록 기판에 결합된 연마 입자들을 가지는 CMP 패드 드레서의 작업 표면의 평면도이다.

도 9는 본원 발명의 한 구체예에 따라, 입자들이 두루 균일하게 분포되도록 기판에 결합된 연마 입자들을 가지는 CMP 패드 드레서의 작업 표면의 평면도이다.

도 10은 본원 발명의 한 구체예에 따라 제조된 CMP 패드 드레서의 측면도이다.

도 11은 본원 발명의 한 구체예에 따라 제조된 CMP 패드 드레서의 측면도이다.

도면

도면1

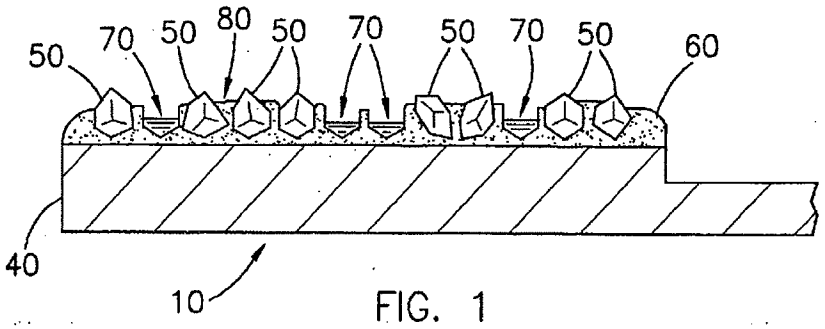


FIG. 1
(PRIOR ART)

도면2

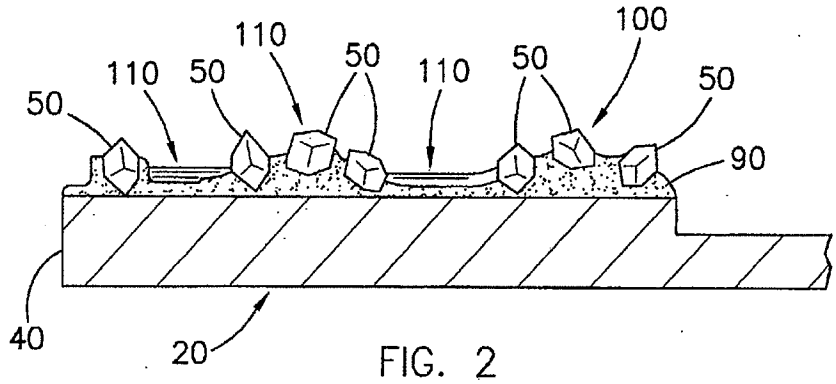


FIG. 2
(PRIOR ART)

도면3

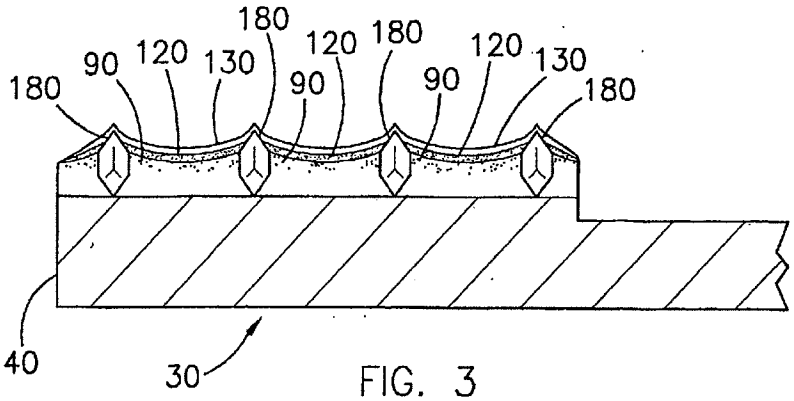
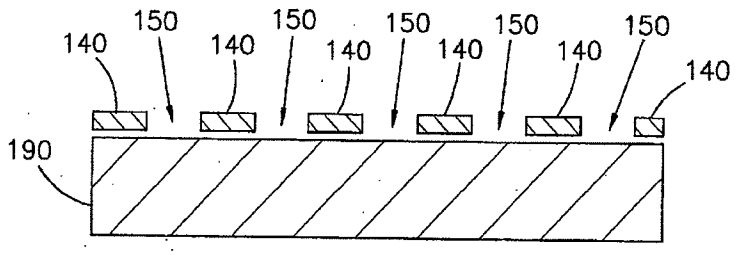
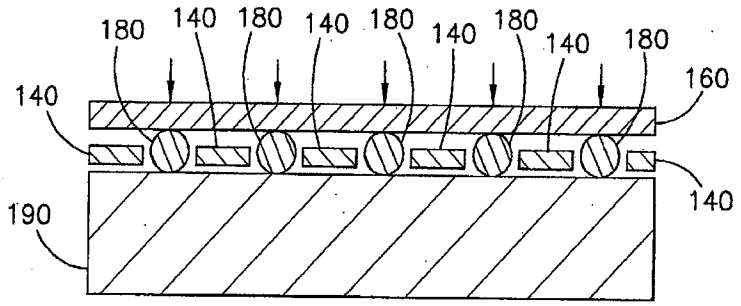


FIG. 3

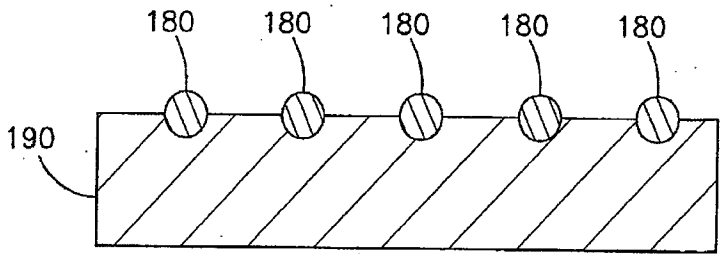
도면4



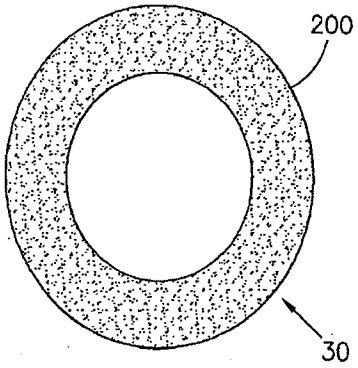
도면5



도면6



도면7



Ex. 1003, Page 112

도면8

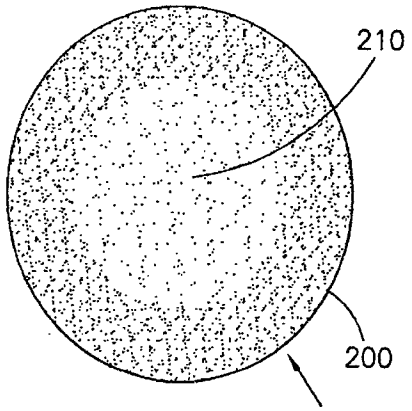


FIG. 8

도면9

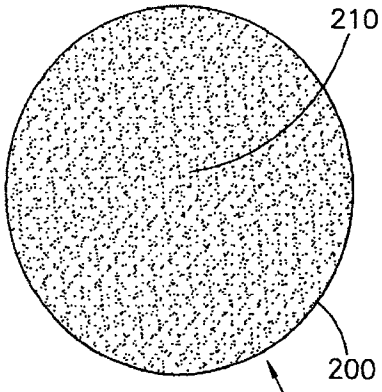


FIG. 9

도면10

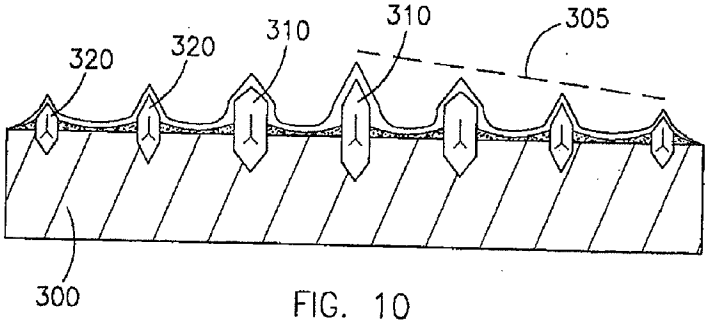
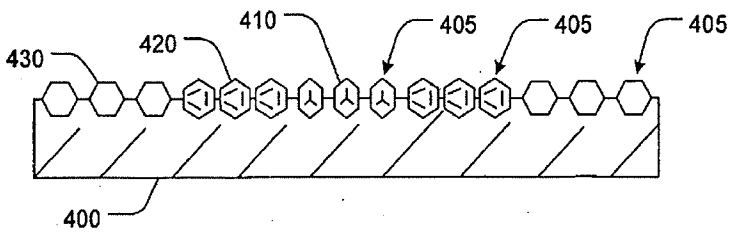


FIG. 10

도면11



PATENT COOPERATION TREATY

From the INTERNATIONAL SEARCHING AUTHORITY

To:
OSBORNE DAVID W.

P.O. BOX 1219 SANDY UT 84091-1219 USA

RECEIVED
SEP - 3 2013
THORPE NORTH & WESTERN

PCT

NOTIFICATION OF TRANSMITTAL OF THE INTERNATIONAL SEARCH REPORT AND THE WRITTEN OPINION OF THE INTERNATIONAL SEARCHING AUTHORITY, OR THE DECLARATION

(PCT Rule 44.1)

Date of mailing *(day/month/year)*	27 August 2013 (27.08.2013)
Applicant's or agent's file reference	**FOR FURTHER ACTION** See paragraphs 1 and 4 below
02074 - 24012NPCIPPC T	
International application No. **PCT/US2013/042538**	International filing date *(day/month/year)* **23 May 2013 (23.05.2013)**
Applicant **SUNG CHIEN-MIN**	

1. ☒ The applicant is hereby notified that the international search report and the written opinion of the International Searching Authority have been established and are transmitted herewith.

   Filing of amendments and statement under Article 19:

   The applicant is entitled, if he so wishes, to amend the claims of the international application (see Rule 46):

   When? The time limit for filing such amendments is normally two months from the date of transmittal of the international search report.

   Where? Directly to the International Bureau of WIPO, 34 chemin des Colombettes 1211 Geneva 20, Switzerland, Facsimile No.: +41 22 338 82 70

   For more detailed instructions, see *PCT Applicant's Guide*, International Phase, paragraphs 9.004 . 9.011.

2. ☐ The applicant is hereby notified that no international search report will be established and that the declaration under Article 17(2)(a) to that effect and the written opinion of the International Searching Authority are transmitted herewith.

3. ☐ **With regard to any protest** against payment of (an) additional fee(s) under Rule 40.2, the applicant is notified that:

   ☐ the protest together with the decision thereon has been transmitted to the International Bureau together with any request to forward the texts of both the protest and the decision thereon to the designated Offices.

   ☐ no decision has been made yet on the protest; the applicant will be notified as soon as a decision is made.

4. **Reminders**

The applicant may submit comments on an informal basis on the written opinion of the International Searching Authority to the International Bureau. The International Bureau will send a copy of such comments to all designated Offices unless an international preliminary examination report has been or is to be established. Following the expiration of 30 months from the priority date, these comments will also be made available to the public.

Shortly after the expiration of **18 months** from the priority date, the international application will be published by the International Bureau. If the applicant wishes to avoid or postpone publication, a notice of withdrawal of the international application, or of the priority claim, must reach the International Bureau before the completion of the technical preparations for international publication (Rules 90bis.1 and 90bis.3).

Within **19 months** from the priority date, but only in respect of some designated Offices, a demand for international preliminary examination must be filed if the applicant wishes to postpone the entry into the national phase **until 30 months** from the priority date (in some Offices even later); otherwise, the applicant must, **within 20 months** from the priority date, perform the prescribed acts for entry into the national phase before those designated Offices.

In respect of other designated Offices, the time limit of **30 months** (or later) will apply even if no demand is filed within 19months.

For details about the applicable time limits, Office by Office, see www.wipo.int/pct/en/texts/time_limits.html and the PCT Applicant's Guide, National Chapters.

Name and mailing address of the ISA/KR Korean Intellectual Property Office 189 Cheongsa-ro, Seo-gu, Daejeon Metropolitan City, 302-701, Republic of Korea	Authorized officer COMMISSIONER
Facsimile No. 82-42-472-7140	Telephone No. 82-42-481-8755

Form PCT/ISA/220 (July 2010)

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" " .CIP3.CON.ACN2	IDS	11-27-13	
" T5100.CIP6	IDS	11-27-13	
" " " 7	IDS	11-27-13	
" " " 8	IDS	11-27-13	
" " " 9			

INITIAL

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	Modify Abstract	9-27-13	
	Article 19	10-27-13	
	Chpt II Demand	3-23-14	
24012-NP	IDS	11-27-13	
" .CIP	IDS	11-27-13	

INITIAL 9/9

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	Description	Due Date	Initials
32979.NP.CIP.CON	IDS	11-27-13	
" " CIP2	IDS	11-27-13	
" " .CON	IDS	11-27-13	
3356-32820.NP2	IDS	11-27-13	
00802-T5110.CIP10	IDS	11-27-13	

INITIAL

* Attention

Copies of the documents cited in the international search report can be searched in the following Korean Intellectual Property Office English website for three months from the date of mailing of the international search report.

http://www.kipo.go.kr/en/ => PCT Services => PCT Services

ID : PCT international application number
PW : **6GGTL9PA**

Inquiries related to PCT International Search Report or Written Opinion prepared by KIPO as an International Searching Authority can be answered not only by KIPO but also through IPKC (Intellectual Property Korea Center), located in Vienna, VA, which functions as a PCT Help Desk for PCT applicants.

Homepage: http://www.ipkcenter.com

Email: ipkc@ipkcenter.com

DOCKETED SEP - 6 2014

PATENT COOPERATION TREATY

PCT

INTERNATIONAL SEARCH REPORT

(PCT Article 18 and Rules 43 and 44)

Applicant's or agent's file reference 24012NPCIPPC	**FOR FURTHER ACTION**	see Form PCT/ISA/220 as well as, where applicable, item 5 below.
International application No. **PCT/US2013/042538**	International filing date *(day/month/year)* **23 May 2013 (23.05.2013)**	(Earliest) Priority Date *(day/month/year)* 23 May 2012 (23.05.2012)
Applicant **SUNG CHIEN-MIN**		

This International search report has been prepared by this International Searching Authority and is transmitted to the applicant according to Article 18. A copy is being transmitted to the International Bureau.

This international search report consists of a total of 5 sheets.

☐ It is also accompanied by a copy of each prior art document cited in this report.

1. **Basis of the report**
   a. With regard to the **language**, the international search was carried out on the basis of:
      ☒ the international application in the language in which it was filed
      ☐ a translation of the international application into _____, which is the language of a translation furnished for the purposes of international search (Rules 12.3(a) and 23.1(b))
   b. ☐ This international search report has been established taking into account the **rectification of an obvious mistake** authorized by or notified to this Authority under Rule 91 (Rule 43.6*bis*(a)).
   c. ☐ With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, see Box No. I.
2. ☐ **Certain claims were found unsearchable** (See Box No. II)
3. ☐ **Unity of invention is lacking** (See Box No. III)
4. With regard to the **title,**
   ☒ the text is approved as submitted by the applicant.
   ☐ the text has been established by this Authority to read as follows:
5. With regard to the **abstract,**
   ☒ the text is approved as submitted by the applicant.
   ☐ the text has been established, according to Rule 38.2, by this Authority as it appears in Box No. IV. The applicant may, within one month from the date of mailing of this international search report, submit comments to this Authority.
6. With regard to the drawings,
   a. the figure of the **drawings** to be published with the abstract is Figure No. 1
      ☒ as suggested by the applicant.
      ☐ as selected by this Authority, because the applicant failed to suggest a figure.
      ☐ as selected by this Authority, because this figure better characterizes the invention.
   b. ☐ none of the figure is to be published with the abstract.

INTERNATIONAL SEARCH REPORT

International application No.
PCT/US2013/042538

A. CLASSIFICATION OF SUBJECT MATTER

H01L 21/304(2006.01)i

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
H01L 21/304; B24B 53/02; B24B 53/017; B05D 3/02; C09K 3/14; B24B 53/12; B24B 1/00

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched
Korean utility models and applications for utility models
Japanese utility models and applications for utility models

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)
eKOMPASS(KIPO internal) & Keywords: CMP, dresser, pad, protrusion, distance

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 2010-0248595 A1 (CHARLES DINH-NGOC et al.) 30 September 2010 See abstract; paragraphs [0065], [0075]-[0076], [0167] and figures 2A, 24B, 24D.	1-20
A	KR 10-2007-0063569 A (CHIEN-MIN SUNG) 19 June 2007 See abstract; pages 2-4, 6-8 and figures 5-6, 10.	1-20
A	WO 2012-040374 A2 (RITEDIA CORPORATION et al.) 29 March 2012 See abstract; page 9, line 31 - page 17, line 2 and figures 1-4.	1-20
A	KR 10-2002-0036138 A (KINIK COMPANY) 16 May 2002 See abstract; pages 3-5 and figures 1-6.	1-20
A	US 2007-0266639 A1 (CHIEN-MIN SUNG) 22 November 2007 See abstract; paragraphs [0054]-[0061] and figures 1-7.	1-20

☐ Further documents are listed in the continuation of Box C.

☒ See patent family annex.

* Special categories of cited documents:
"A" document defining the general state of the art which is not considered to be of particular relevance
"E" earlier application or patent but published on or after the international filing date
"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of citation or other special reason (as specified)
"O" document referring to an oral disclosure, use, exhibition or other means
"P" document published prior to the international filing date but later than the priority date claimed
"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents,such combination being obvious to a person skilled in the art
"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
27 August 2013 (27.08.2013)	**27 August 2013 (27.08.2013)**

Name and mailing address of the ISA/KR	Authorized officer
Korean Intellectual Property Office 189 Cheongsa-ro, Seo-gu, Daejeon Metropolitan City, 302-701, Republic of Korea Facsimile No. +82-42-472-7140	CHOI Sang Won Telephone No. +82-42-481-8291

Form PCT/ISA/210 (second sheet) (July 2009)

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No.
PCT/US2013/042538

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
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		WO 2007-136864 A3	31/01/2008

Electronic Acknowledgement Receipt

EFS ID:	17045818
Application Number:	13802112
International Application Number:	
Confirmation Number:	1021
Title of Invention:	CMP PAD DRESSER HAVING LEVELED TIPS AND ASSOCIATED METHODS
First Named Inventor/Applicant Name:	Chien-Min Sung
Customer Number:	20551
Filer:	David William Osborne/Eileen Fuessel
Filer Authorized By:	David William Osborne
Attorney Docket Number:	02074-24012.NP.CIP
Receipt Date:	04-OCT-2013
Filing Date:	13-MAR-2013
Time Stamp:	16:25:08
Application Type:	Utility under 35 USC 111(a)

Payment information:

Submitted with Payment	no

File Listing:

Document Number	Document Description	File Name	File Size(Bytes)/ Message Digest	Multi Part /.zip	Pages (if appl.)
1	Transmittal Letter	trans.PDF	79676 4b789f559c827832ee8d8bbac0235d689e00d8d9	no	4
Warnings:					
Information:					

2	Information Disclosure Statement (IDS) Form (SB08)	a.PDF	567956 0098ba7a13feae38cfe9299837b815f38a25fe26	no	8
Warnings:					
Information:					
This is not an USPTO supplied IDS fillable form					
3	Information Disclosure Statement (IDS) Form (SB08)	b.PDF	96797 1e9579c22c1f71438a69b624db15e63b83bcfb87	no	2
Warnings:					
Information:					
This is not an USPTO supplied IDS fillable form					
4	Foreign Reference	kr138.pdf	1099615 839367cfa03ad84f74e9abd48d79f30434980d5d	no	23
Warnings:					
Information:					
5	Foreign Reference	kr569.pdf	1373578 e2e093d38bfbf5d187e5223b20afa6ac42a8208f	no	23
Warnings:					
Information:					
6	Non Patent Literature	oa786.pdf	1863560 1698ca128843fa4ffe95f2520fd19eb4db6ade8d	no	38
Warnings:					
Information:					
7	Non Patent Literature	oa917.pdf	1011364 2dc5df18b14fd1c3b069dec0c18418165290d868	no	24
Warnings:					
Information:					
8	Non Patent Literature	oa198.pdf	1216275 9334c1e12aa5fa5fb70b000a11b123086bc6a739	no	25
Warnings:					
Information:					
9	Non Patent Literature	Searchreport538.pdf	360731 e26c0cdcfa73a5833a96852f843eb2cbe6c7f93d	no	7
Warnings:					
Information:					

10	Non Patent Literature	ntc786.pdf	492014 073a66691618ef39ee45a7e29afd8994b1843181	no	7

Warnings:	
Information:	
Total Files Size (in bytes):	8161566

This Acknowledgement Receipt evidences receipt on the noted date by the USPTO of the indicated documents, characterized by the applicant, and including page counts, where applicable. It serves as evidence of receipt similar to a Post Card, as described in MPEP 503.

New Applications Under 35 U.S.C. 111

If a new application is being filed and the application includes the necessary components for a filing date (see 37 CFR 1.53(b)-(d) and MPEP 506), a Filing Receipt (37 CFR 1.54) will be issued in due course and the date shown on this Acknowledgement Receipt will establish the filing date of the application.

National Stage of an International Application under 35 U.S.C. 371

If a timely submission to enter the national stage of an international application is compliant with the conditions of 35 U.S.C. 371 and other applicable requirements a Form PCT/DO/EO/903 indicating acceptance of the application as a national stage submission under 35 U.S.C. 371 will be issued in addition to the Filing Receipt, in due course.

New International Application Filed with the USPTO as a Receiving Office

If a new international application is being filed and the international application includes the necessary components for an international filing date (see PCT Article 11 and MPEP 1810), a Notification of the International Application Number and of the International Filing Date (Form PCT/RO/105) will be issued in due course, subject to prescriptions concerning national security, and the date shown on this Acknowledgement Receipt will establish the international filing date of the application.

IN THE UNITED STATES PATENT & TRADEMARK OFFICE

ART UNIT: 3723 EXAMINER: FIRST NAMED INVENTOR: Chien-Min Sung SERIAL NO.: 13/802,112 FILED: 3/13/2013 CONF. NO.: 1021 FOR: CMP PAD DRESSER HAVING LEVELED TIPS AND ASSOCIATED METHODS DOCKET NO.: 02074-24012.NP.CIP	**CERTIFICATE OF MAILING UNDER 37 C.F.R. § 1.8** DATE OF DEPOSIT: October 4, 2013 I hereby certify that this paper or fee (along with any paper or fee referred to as being attached or enclosed) is being submitted on the date indicated above via: ☒ EFS Web ☐ facsimile to _____ ☐ the United States Postal Service with sufficient postage as first class mail addressed to: Mail Stop _____, Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450. /David W. Osborne/ David W. Osborne

INFORMATION DISCLOSURE STATEMENT UNDER 37 C.F.R. §§ 1.56, 1.97 and 1.98

Mail Stop Amendment
Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

Dear Sir/Madam:

Pursuant to the duty of disclosure under 37 C.F.R. §§ 1.56, 1.97 and 1.98, the Applicant requests consideration of this Information Disclosure Statement, which is being submitted in accordance with:

☒ 37 C.F.R. § 1.97(b)(1) or (3), within three months of the filing date of the application, or before a first office action on the merits, whichever occurs last;

☐ 37 C.F.R. § 1.97(c), after a first office action on the merits, but before a Final Office Action or a Notice of Allowance, whichever occurs first, and is accompanied by **EITHER** a statement in accordance with 37 C.F.R. § 1.97(e), **OR** the fee set forth in 37 C.F.R. § 1.17(p); or

☐ 37 C.F.R. § 1.97(d), after a Final Office Action or Notice of Allowance, whichever occurs first, but on or before payment of the issue fee, and is accompanied by **BOTH** a statement in

accordance with 37 C.F.R. § 1.97(e), **AND** the fee set forth in 37 C.F.R. § 1.17(p).

SUBMISSIONS

Please find enclosed, pursuant to 37 C.F.R. §§ 1.97 and 1.98:

☒ Form **PTO/SB/08A** and/or **PTO/SB/08B** listing the references submitted for consideration, which contains a list of all patents, publications and all other items that have come to the attention of one or more of the individuals designated in 37 C.F.R. § 1.56(c).

☒ Legible copies of the listed non-patent documents, unpublished U.S. applications and foreign documents or their relevant portions are included.

☐ All English translations of each non-English reference, if any, within the possession, custody, control or availability of anyone designated in 37 C.F.R. § 1.56(c) (see 37 C.F.R. §1.98 are included.

☐ For all listed references that are not either in the English language, or accompanied by a translation into English, a concise explanation of relevance as required under 37 C.F.R. § 1.98(a)(3) is enclosed attached to each.

Copies of references listed on the enclosed PTO/SB/08A and/or PTO/SB/08B are **not** enclosed:

☒ because, under 37 C.F.R. § 1.98.(d), they were previously cited by or submitted to the Office in application number 13/479,148, which is relied upon for an earlier filing date under 37 C.F.R. § 1.20.

☐ because they are substantially cumulative of an enclosed reference.

STATEMENT IN ACCORDANCE WITH 37 C.F.R. § 1.97(e)

☐ I hereby certify that each item contained in the information disclosure statement was first cited in a communication from a foreign patent office in a counterpart foreign application not more than three months prior to the filing of the information disclosure statement.

☐ I hereby certify that no item contained in the information disclosure statement was cited in a communication from a foreign patent office in a counterpart foreign application, and, to the knowledge of the person signing the certification after making reasonable inquiry, no item of information contained in the information disclosure statement was known to any individual designated in §1.56(c) more than three months prior to the filing of the information disclosure statement.

REMARKS

While no representation is made that any of these references may be "prior art" within the meaning of that term in accordance with 37 C.F.R. §§ 102 or 103, the enclosed list of references is disclosed so as to comply with the duty of disclosure set forth in 37 C.F.R. § 1.56.

Moreover, while no representation is made that a specific search of office files or patent office records has been conducted or that no better art exists, the undersigned attorney of record believes that the references listed, together with any other references which may have been previously submitted or listed, are the closest to the claimed invention (taken in its entirety) of which the undersigned is presently aware, and no art which is closer to the claimed invention (taken in its entirety) has been knowingly withheld.

☐ Check No. ____ in the amount of $ ____(amount in § 1.17(p)) constituting the submission fee set forth in 37 C.F.R. § 1.17(p) is enclosed.

In the event that 37 C.F.R. § 1.97(c) applies and the Examiner is not satisfied that the Statement meets the requirements of 37 C.F.R. § 1.97(e), or in any other event remediable by a fee, please credit any over payment or charge any additional fees to Deposit Account No. 20-0100 of the undersigned.

Dated this 4th day of October, 2013.

Respectfully submitted,

/David W. Osborne/

David W. Osborne
Attorney for Applicant
Registration No. 44989

THORPE NORTH & WESTERN, LLP
Customer No. 20,551
P.O. Box 1219
Sandy, Utah 84091-1219
Telephone: (801) 566-6633

PATENT APPLICATION FEE DETERMINATION RECORD

Substitute for Form PTO-875

Application or Docket Number: 13/802,112

APPLICATION AS FILED - PART I

FOR	(Column 1) NUMBER FILED	(Column 2) NUMBER EXTRA	SMALL ENTITY RATE($)	SMALL ENTITY FEE($)	OR	OTHER THAN SMALL ENTITY RATE($)	OTHER THAN SMALL ENTITY FEE($)
BASIC FEE (37 CFR 1.16(a), (b), or (c))	N/A	N/A	N/A	70		N/A	
SEARCH FEE (37 CFR 1.16(k), (i), or (m))	N/A	N/A	N/A	300		N/A	
EXAMINATION FEE (37 CFR 1.16(o), (p), or (q))	N/A	N/A	N/A	360		N/A	
TOTAL CLAIMS (37 CFR 1.16(i))	20 minus 20 =	*	x 40 =	0.00	OR		
INDEPENDENT CLAIMS (37 CFR 1.16(h))	1 minus 3 =	*	x 210 =	0.00			
APPLICATION SIZE FEE (37 CFR 1.16(s))	If the specification and drawings exceed 100 sheets of paper, the application size fee due is $310 ($155 for small entity) for each additional 50 sheets or fraction thereof. See 35 U.S.C. 41(a)(1)(G) and 37 CFR 1.16(s).			0.00			
MULTIPLE DEPENDENT CLAIM PRESENT (37 CFR 1.16(j))				0.00			
* If the difference in column 1 is less than zero, enter "0" in column 2.			TOTAL	730		TOTAL	

APPLICATION AS AMENDED - PART II

		(Column 1) CLAIMS REMAINING AFTER AMENDMENT		(Column 2) HIGHEST NUMBER PREVIOUSLY PAID FOR	(Column 3) PRESENT EXTRA	SMALL ENTITY RATE($)	SMALL ENTITY ADDITIONAL FEE($)	OR	OTHER THAN SMALL ENTITY RATE($)	OTHER THAN SMALL ENTITY ADDITIONAL FEE($)
AMENDMENT A	Total (37 CFR 1.16(i))	*	Minus	**	=	x =		OR	x =	
	Independent (37 CFR 1.16(h))	*	Minus	***	=	x =		OR	x =	
	Application Size Fee (37 CFR 1.16(s))									
	FIRST PRESENTATION OF MULTIPLE DEPENDENT CLAIM (37 CFR 1.16(j))							OR		
						TOTAL ADD'L FEE		OR	TOTAL ADD'L FEE	

		(Column 1) CLAIMS REMAINING AFTER AMENDMENT		(Column 2) HIGHEST NUMBER PREVIOUSLY PAID FOR	(Column 3) PRESENT EXTRA	SMALL ENTITY RATE($)	SMALL ENTITY ADDITIONAL FEE($)	OR	OTHER THAN SMALL ENTITY RATE($)	OTHER THAN SMALL ENTITY ADDITIONAL FEE($)
AMENDMENT B	Total (37 CFR 1.16(i))	*	Minus	**	=	x =		OR	x =	
	Independent (37 CFR 1.16(h))	*	Minus	***	=	x =		OR	x =	
	Application Size Fee (37 CFR 1.16(s))									
	FIRST PRESENTATION OF MULTIPLE DEPENDENT CLAIM (37 CFR 1.16(j))							OR		
						TOTAL ADD'L FEE		OR	TOTAL ADD'L FEE	

* If the entry in column 1 is less than the entry in column 2, write "0" in column 3.
** If the "Highest Number Previously Paid For" IN THIS SPACE is less than 20, enter "20".
*** If the "Highest Number Previously Paid For" IN THIS SPACE is less than 3, enter "3".
The "Highest Number Previously Paid For" (Total or Independent) is the highest found in the appropriate box in column 1.

UNITED STATES PATENT AND TRADEMARK OFFICE

UNITED STATES DEPARTMENT OF COMMERCE
United States Patent and Trademark Office
Address: COMMISSIONER FOR PATENTS
P.O. Box 1450
Alexandria, Virginia 22313-1450
www.uspto.gov

APPLICATION NUMBER	FILING or 371(c) DATE	GRP ART UNIT	FIL FEE REC'D	ATTY.DOCKET.NO	TOT CLAIMS	IND CLAIMS
13/802,112	03/13/2013	3723	800	02074-24012.NP.CIP	20	1

CONFIRMATION NO. 1021

UPDATED FILING RECEIPT

20551
THORPE NORTH & WESTERN, LLP.
P.O. Box 1219
SANDY, UT 84091-1219

OC000000065764345

Date Mailed: 01/03/2014

Receipt is acknowledged of this non-provisional patent application. The application will be taken up for examination in due course. Applicant will be notified as to the results of the examination. Any correspondence concerning the application must include the following identification information: the U.S. APPLICATION NUMBER, FILING DATE, NAME OF APPLICANT, and TITLE OF INVENTION. Fees transmitted by check or draft are subject to collection. Please verify the accuracy of the data presented on this receipt. **If an error is noted on this Filing Receipt, please submit a written request for a Filing Receipt Correction. Please provide a copy of this Filing Receipt with the changes noted thereon. If you received a "Notice to File Missing Parts" for this application, please submit any corrections to this Filing Receipt with your reply to the Notice. When the USPTO processes the reply to the Notice, the USPTO will generate another Filing Receipt incorporating the requested corrections**

Inventor(s)

Chien-Min Sung, Tansui, TAIWAN;

Applicant(s)

Chien-Min Sung, Tansui, TAIWAN;

Power of Attorney: None

Domestic Priority data as claimed by applicant

This application is a CIP of 13/479,148 05/23/2012
which claims benefit of 61/489,074 05/23/2011

Foreign Applications for which priority is claimed (You may be eligible to benefit from the **Patent Prosecution Highway** program at the USPTO. Please see http://www.uspto.gov for more information.) - None.
Foreign application information must be provided in an Application Data Sheet in order to constitute a claim to foreign priority. See 37 CFR 1.55 and 1.76.

Permission to Access - A proper **Authorization to Permit Access to Application by Participating Offices** (PTO/SB/39 or its equivalent) has been received by the USPTO.

If Required, Foreign Filing License Granted: 04/16/2013
The country code and number of your priority application, to be used for filing abroad under the Paris Convention, is **US 13/802,112**
Projected Publication Date: 04/10/2014
Non-Publication Request: No
Early Publication Request: No
** **SMALL ENTITY** **

Title

CMP PAD DRESSER HAVING LEVELED TIPS AND ASSOCIATED METHODS

Preliminary Class

451

Statement under 37 CFR 1.55 or 1.78 for AIA (First Inventor to File) Transition Applications: No

PROTECTING YOUR INVENTION OUTSIDE THE UNITED STATES

Since the rights granted by a U.S. patent extend only throughout the territory of the United States and have no effect in a foreign country, an inventor who wishes patent protection in another country must apply for a patent in a specific country or in regional patent offices. Applicants may wish to consider the filing of an international application under the Patent Cooperation Treaty (PCT). An international (PCT) application generally has the same effect as a regular national patent application in each PCT-member country. The PCT process **simplifies** the filing of patent applications on the same invention in member countries, but **does not result** in a grant of "an international patent" and does not eliminate the need of applicants to file additional documents and fees in countries where patent protection is desired.

Almost every country has its own patent law, and a person desiring a patent in a particular country must make an application for patent in that country in accordance with its particular laws. Since the laws of many countries differ in various respects from the patent law of the United States, applicants are advised to seek guidance from specific foreign countries to ensure that patent rights are not lost prematurely.

Applicants also are advised that in the case of inventions made in the United States, the Director of the USPTO must issue a license before applicants can apply for a patent in a foreign country. The filing of a U.S. patent application serves as a request for a foreign filing license. The application's filing receipt contains further information and guidance as to the status of applicant's license for foreign filing.

Applicants may wish to consult the USPTO booklet, "General Information Concerning Patents" (specifically, the section entitled "Treaties and Foreign Patents") for more information on timeframes and deadlines for filing foreign patent applications. The guide is available either by contacting the USPTO Contact Center at 800-786-9199, or it can be viewed on the USPTO website at http://www.uspto.gov/web/offices/pac/doc/general/index.html.

For information on preventing theft of your intellectual property (patents, trademarks and copyrights), you may wish to consult the U.S. Government website, http://www.stopfakes.gov. Part of a Department of Commerce initiative, this website includes self-help "toolkits" giving innovators guidance on how to protect intellectual property in specific countries such as China, Korea and Mexico. For questions regarding patent enforcement issues, applicants may call the U.S. Government hotline at 1-866-999-HALT (1-866-999-4258).

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Title 37, Code of Federal Regulations, 5.11 & 5.15

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NOT GRANTED

No license under 35 U.S.C. 184 has been granted at this time, if the phrase "IF REQUIRED, FOREIGN FILING LICENSE GRANTED" DOES NOT appear on this form. Applicant may still petition for a license under 37 CFR 5.12, if a license is desired before the expiration of 6 months from the filing date of the application. If 6 months has lapsed from the filing date of this application and the licensee has not received any indication of a secrecy order under 35 U.S.C. 181, the licensee may foreign file the application pursuant to 37 CFR 5.15(b).

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United States Patent and Trademark Office
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Alexandria, Virginia 22313-1450
www.uspto.gov

APPLICATION NUMBER	FILING OR 371(C) DATE	FIRST NAMED APPLICANT	ATTY. DOCKET NO./TITLE
13/802,112	03/13/2013	Chien-Min Sung	02074-24012.NP.CIP

CONFIRMATION NO. 1021

20551
THORPE NORTH & WESTERN, LLP.
P.O. Box 1219
SANDY, UT 84091-1219

NOTICE

OC000000065764346

Date Mailed: 01/03/2014

INFORMATIONAL NOTICE TO APPLICANT

Applicant is notified that the above-identified application contains the deficiencies noted below. No period for reply is set forth in this notice for correction of these deficiencies. However, if a deficiency relates to the inventor's oath or declaration, the applicant must file an oath or declaration in compliance with 37 CFR 1.63, or a substitute statement in compliance with 37 CFR 1.64, executed by or with respect to each actual inventor no later than the expiration of the time period set in the "Notice of Allowability" to avoid abandonment. See 37 CFR 1.53(f).

The item(s) indicated below are also required and should be submitted with any reply to this notice to avoid further processing delays.

- A properly executed inventor's oath or declaration has not been received for the following inventor(s):
  All

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APPLICATION NUMBER	FILING OR 371(C) DATE	FIRST NAMED APPLICANT	ATTY. DOCKET NO./TITLE
13/802,112	03/13/2013	Chien-Min Sung	02074-24012.NP.CIP

CONFIRMATION NO. 1021

20551
THORPE NORTH & WESTERN, LLP.
P.O. Box 1219
SANDY, UT 84091-1219

PUBLICATION NOTICE

OC000000067700986

Title:CMP PAD DRESSER HAVING LEVELED TIPS AND ASSOCIATED METHODS

Publication No.US-2014-0099868-A1
Publication Date:04/10/2014

NOTICE OF PUBLICATION OF APPLICATION

The above-identified application will be electronically published as a patent application publication pursuant to 37 CFR 1.211, et seq. The patent application publication number and publication date are set forth above.

The publication may be accessed through the USPTO's publically available Searchable Databases via the Internet at www.uspto.gov. The direct link to access the publication is currently http://www.uspto.gov/patft/.

The publication process established by the Office does not provide for mailing a copy of the publication to applicant. A copy of the publication may be obtained from the Office upon payment of the appropriate fee set forth in 37 CFR 1.19(a)(1). Orders for copies of patent application publications are handled by the USPTO's Office of Public Records. The Office of Public Records can be reached by telephone at (703) 308-9726 or (800) 972-6382, by facsimile at (703) 305-8759, by mail addressed to the United States Patent and Trademark Office, Office of Public Records, Alexandria, VA 22313-1450 or via the Internet.

In addition, information on the status of the application, including the mailing date of Office actions and the dates of receipt of correspondence filed in the Office, may also be accessed via the Internet through the Patent Electronic Business Center at www.uspto.gov using the public side of the Patent Application Information and Retrieval (PAIR) system. The direct link to access this status information is currently http://pair.uspto.gov/. Prior to publication, such status information is confidential and may only be obtained by applicant using the private side of PAIR.

Further assistance in electronically accessing the publication, or about PAIR, is available by calling the Patent Electronic Business Center at 1-866-217-9197.

Office of Data Managment, Application Assistance Unit (571) 272-4000, or (571) 272-4200, or 1-888-786-0101

PTO/SB/08B (08-03)
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INFORMATION DISCLOSURE STATEMENT BY APPLICANT

(Use as many sheets as necessary)

Sheet | | of |

Complete if Known	
Application Number	13/802,112
Filing Date	3/13/2013
First Named Inventor	Chien-Min Sung
Art Unit	3723
Examiner Name	
Attorney Docket Number	02074-24012.NP.CIP

NON PATENT LITERATURE DOCUMENTS

Examiner Initials*	Cite No.[1]	Include name of author (in CAPITAL LETTERS), title of the article (when appropriate), title of the item (book, magazine, journal, serial, symposium, catalog, etc.), date, page(s), volume-issue number(s), publisher, city and/or country where published.	T[2]
		US Application 13/239,189; filed 09/21/2011; Chien-Min Sung; Notice of Allowance dated 05/03/2013	
		US Application 13/113,779; filed 05/23/2011; Chien-Min Sung; office action dated 11/12/2013	
		US Application 13/153,176; filed 06/03/2011; Chien-Min Sung; office action dated 12/06/2013	
		US Application 13239,198; filing date 09/21/2011; Chien-Min Sung; Notice of Allowance mailed 03/04/2014.	

Examiner Signature		Date Considered	

*Examiner: Initial if reference considered, whether or not citation is in conformance with MPEP 609. Draw line through citation if not in conformance and not considered. Include copy of this form with next communication to applicant.

[1]Applicant's unique citation designation number (optional). [2]Applicant is to place a check mark here if English language Translation is attached. This collection of information is required by 37 CFR 1.98. The information is required to obtain or retain a benefit by the public which is to file (and by the USPTO to process) an application. Confidentially is governed by 35 U.S.C. 122 and 37 CFR 1.14. This collection is estimated to take 2 hours to complete, including gathering, preparing, and submitting the completed application form to the USPTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, P.O. Box 1450, Alexandria, VA 22313-1450. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. **Send to: Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450.**

If you need assistance in completing the form, call 1-800-PTO-9199 (1-800-786-9199) and select option 2.

Electronic Acknowledgement Receipt

EFS ID:	19359222
Application Number:	13802112
International Application Number:	
Confirmation Number:	1021
Title of Invention:	CMP PAD DRESSER HAVING LEVELED TIPS AND ASSOCIATED METHODS
First Named Inventor/Applicant Name:	Chien-Min Sung
Customer Number:	20551
Filer:	David William Osborne/Warren Archibald
Filer Authorized By:	David William Osborne
Attorney Docket Number:	02074-24012.NP.CIP
Receipt Date:	19-JUN-2014
Filing Date:	13-MAR-2013
Time Stamp:	19:41:12
Application Type:	Utility under 35 USC 111(a)

Payment information:

Submitted with Payment	no

File Listing:

Document Number	Document Description	File Name	File Size(Bytes)/ Message Digest	Multi Part /.zip	Pages (if appl.)
1	Transmittal Letter	tranmsittal.PDF	79774 ce7576249ac2e1f2cb7f06113718e16403b87bdf	no	4

Warnings:	
Information:	

2	Information Disclosure Statement (IDS) Form (SB08)	b.PDF	78788 7a9f66f830f88cdaaffc8f2f98d91365ef9e99a1	no	1
Warnings:					
Information:					
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3	Non Patent Literature	NOAMay32013.pdf	591291 22ad0686c8da045d20d7c6d2579130cae5eced9a	no	13
Warnings:					
Information:					
4	Non Patent Literature	OANov122013.pdf	1186987 693879edc2d83addf94cbc81c1bb5dc2e280433c	no	27
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5	Non Patent Literature	OADec62013.pdf	1242313 acad8a7be72218a81c457c21c7be440ee95d482b	no	27
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6	Non Patent Literature	NOAMarch42014.pdf	464133 9c21aac063762b977df19e4dce3df97746d571df	no	7
Warnings:					
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		Total Files Size (in bytes):	3643286		

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New Applications Under 35 U.S.C. 111

If a new application is being filed and the application includes the necessary components for a filing date (see 37 CFR 1.53(b)-(d) and MPEP 506), a Filing Receipt (37 CFR 1.54) will be issued in due course and the date shown on this Acknowledgement Receipt will establish the filing date of the application.

National Stage of an International Application under 35 U.S.C. 371

If a timely submission to enter the national stage of an international application is compliant with the conditions of 35 U.S.C. 371 and other applicable requirements a Form PCT/DO/EO/903 indicating acceptance of the application as a national stage submission under 35 U.S.C. 371 will be issued in addition to the Filing Receipt, in due course.

New International Application Filed with the USPTO as a Receiving Office

If a new international application is being filed and the international application includes the necessary components for an international filing date (see PCT Article 11 and MPEP 1810), a Notification of the International Application Number and of the International Filing Date (Form PCT/RO/105) will be issued in due course, subject to prescriptions concerning national security, and the date shown on this Acknowledgement Receipt will establish the international filing date of the application.

IN THE UNITED STATES PATENT & TRADEMARK OFFICE

ART UNIT: 3723 EXAMINER: FIRST NAMED INVENTOR: Chien-Min Sung SERIAL NO.: 13/802,112 FILED: 3/13/2013 CONF. NO.: 1021 FOR: CMP PAD DRESSER HAVING LEVELED TIPS AND ASSOCIATED METHODS DOCKET NO.: 02074-24012.NP.CIP	**CERTIFICATE OF MAILING UNDER 37 C.F.R. § 1.8** DATE OF DEPOSIT: June 19, 2014 I hereby certify that this paper or fee (along with any paper or fee referred to as being attached or enclosed) is being submitted on the date indicated above via: ☒ EFS Web ☐ facsimile to _____ ☐ the United States Postal Service with sufficient postage as first class mail addressed to: Mail Stop _____, Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450. /Warren Archibald/ Warren Archibald

SUPPLEMENTAL INFORMATION DISCLOSURE STATEMENT UNDER 37 C.F.R. §§ 1.56, 1.97 and 1.98

Mail Stop Amendment
Commissioner for Patents
P.O. Box 1450
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Dear Sir/Madam:

Pursuant to the duty of disclosure under 37 C.F.R. §§ 1.56, 1.97 and 1.98, the Applicant requests consideration of this Information Disclosure Statement, which is being submitted in accordance with:

☒ 37 C.F.R. § 1.97(b)(1) or (3), within three months of the filing date of the application, or before a first office action on the merits, whichever occurs last;

☐ 37 C.F.R. § 1.97(c), after a first office action on the merits, but before a Final Office Action or a Notice of Allowance, whichever occurs first, and is accompanied by **EITHER** a statement in accordance with 37 C.F.R. § 1.97(e), **OR** the fee set forth in 37 C.F.R. § 1.17(p); or

☐ 37 C.F.R. § 1.97(d), after a Final Office Action or Notice of Allowance, whichever occurs first, but on or before payment of the issue fee, and is accompanied by **BOTH** a statement in

accordance with 37 C.F.R. § 1.97(e), **AND** the fee set forth in 37 C.F.R. § 1.17(p).

SUBMISSIONS

Please find enclosed, pursuant to 37 C.F.R. §§ 1.97 and 1.98:

☒ Form **PTO/SB/08A** and/or **PTO/SB/08B** listing the references submitted for consideration, which contains a list of all patents, publications and all other items that have come to the attention of one or more of the individuals designated in 37 C.F.R. § 1.56(c).

☒ Legible copies of the listed non-patent documents, unpublished U.S. applications and foreign documents or their relevant portions are included.

☒ All English translations of each non-English reference, if any, within the possession, custody, control or availability of anyone designated in 37 C.F.R. § 1.56(c) (see 37 C.F.R. §1.98 are included.

☐ For all listed references that are not either in the English language, or accompanied by a translation into English, a concise explanation of relevance as required under 37 C.F.R. § 1.98(a)(3) is enclosed attached to each.

Copies of references listed on the enclosed PTO/SB/08A and/or PTO/SB/08B are **not** enclosed:

☐ because, under 37 C.F.R. § 1.98.(d), they were previously cited by or submitted to the Office in application number _____, which is relied upon for an earlier filing date under 37 C.F.R. § 1.20.

☐ because they are substantially cumulative of an enclosed reference.

STATEMENT IN ACCORDANCE WITH 37 C.F.R. § 1.97(e)

☐ I hereby certify that each item contained in the information disclosure statement was first cited in a communication from a foreign patent office in a counterpart foreign application not more than three months prior to the filing of the information disclosure statement.

☐ I hereby certify that no item contained in the information disclosure statement was cited in a communication from a foreign patent office in a counterpart foreign application, and, to the knowledge of the person signing the certification after making reasonable inquiry, no item of information contained in the information disclosure statement was known to any individual designated in §1.56(c) more than three months prior to the filing of the information disclosure statement.

REMARKS

While no representation is made that any of these references may be "prior art" within the meaning of that term in accordance with 37 C.F.R. §§ 102 or 103, the enclosed list of references is disclosed so as to comply with the duty of disclosure set forth in 37 C.F.R. § 1.56.

Moreover, while no representation is made that a specific search of office files or patent office records has been conducted or that no better art exists, the undersigned attorney of record believes that the references listed, together with any other references which may have been previously submitted or listed, are the closest to the claimed invention (taken in its entirety) of which the undersigned is presently aware, and no art which is closer to the claimed invention (taken in its entirety) has been knowingly withheld.

☐ Check No. ____ in the amount of $ ____(amount in § 1.17(p)) constituting the submission fee set forth in 37 C.F.R. § 1.17(p) is enclosed.

In the event that 37 C.F.R. § 1.97(c) applies and the Examiner is not satisfied that the Statement meets the requirements of 37 C.F.R. § 1.97(e), or in any other event remediable by a fee, please credit any over payment or charge any additional fees to Deposit Account No. 20-0100 of the undersigned.

Dated this 19th day of June, 2014.

Respectfully submitted,

David W. Osborne/

David W. Osborne
Attorney for Applicant
Registration No. 44989

THORPE NORTH & WESTERN, LLP
Customer No. 20,551
P.O. Box 1219
Sandy, Utah 84091-1219
Telephone: (801) 566-6633

PTO/SB/08B (08-03)
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INFORMATION DISCLOSURE STATEMENT BY APPLICANT

(Use as many sheets as necessary)

Sheet | | of |

Complete if Known	
Application Number	13/802,112
Filing Date	3/13/2013
First Named Inventor	Chien-Min Sung
Art Unit	3723
Examiner Name	
Attorney Docket Number	02074-24012.NP.CIP

NON PATENT LITERATURE DOCUMENTS

Examiner Initials*	Cite No.[1]	Include name of author (in CAPITAL LETTERS), title of the article (when appropriate), title of the item (book, magazine, journal, serial, symposium, catalog, etc.), date, page(s), volume-issue number(s), publisher, city and/or country where published.	T[2]
		US Application 13/153,176; filing date 06/03/2011; Chien-Min Sung; office action dated 12/06/2013	
		US Application 09/447,620; filed 11/22/1999; Chien-Min Sung	
		US Application 08/832,852; filed 04/04/1997; Chien-Min Sung	
		PCT Application PCT/US2011/052627; filing date 09/21/2011; chien-Min Sung; International Search Report mailed 05/11/2012	
		US Application 13/362,917; filed 01/31/2012; Chien-Min Sung; office action dated 06/14/2012	
		US Application 12/168, 110; filed 07/05/2008; Chien-Min Sung; office action dated 05/14/2012	
		US Application 12/059,422; filed 03/30/2008; Chien-Min Sung; office action dated 08/22/2012	
		US Appication 12/255, 823; filed 10/21/2008; Chien-Min Sung; notice of allowance dated 09/21/2012	
		US Application 12/168, 110; filed 07/05/2008; Chien-Min Sung; notice of allowance dated 09/28/2012	
		US Appication 13/239, 189; filed 09/21/2011; Chien-Min Sung; office action dated 12/21/2012	
		US Application 12/255, 823; filed 10/22/2008; Chien-Min Sung; notice of allowance dated 12/26/2013	
		PCT/US2012/039199; filed 05/23/2012; Chien-Min Sung; International Search Report dated 12/18/2012	
		US Application 13/362, 917; filed 01/13/2012; Chien-Min Sung; office action dated 12/31/2012	
		US Application 12/168, 100; filed 07/05/2008; Chien-Min Sung; notice of allowance dated 01/18/2013	
		US Application 12/059, 422; filed 03/31/20078; Chien-Min Sung; office action dated 01/24/2013	
		US Application 13/153, 176; filing date 06/03/2011; Chien-Min Sung; office action dated 07/01/2014	
		US Application 13/113, 779; filing ddate 05/23/2011; Chien-Min Sung; office action dated 07/02/2014.	

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INFORMATION DISCLOSURE STATEMENT BY APPLICANT

(Use as many sheets as necessary)

Sheet		of	

Complete if Known	
Application Number	13/802,112
Filing Date	3/13/2013
First Named Inventor	Chien-Min Sung
Art Unit	3723
Examiner Name	
Attorney Docket Number	02074-24012.NP.CIP

Examiner Signature		Date Considered	

*Examiner: Initial if reference considered, whether or not citation is in conformance with MPEP 609. Draw line through citation if not in conformance and not considered. Include copy of this form with next communication to applicant.

[1]Applicant's unique citation designation number (optional). [2]Applicant is to place a check mark here if English language Translation is attached. This collection of information is required by 37 CFR 1.98. The information is required to obtain or retain a benefit by the public which is to file (and by the USPTO to process) an application. Confidentially is governed by 35 U.S.C. 122 and 37 CFR 1.14. This collection is estimated to take 2 hours to complete, including gathering, preparing, and submitting the completed application form to the USPTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, P.O. Box 1450, Alexandria, VA 22313-1450. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. **Send to: Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450.**

If you need assistance in completing the form, call 1-800-PTO-9199 (1-800-786-9199) and select option 2.

Espacenet

Bibliographic data: CN1351922 (A) — 2002-06-05

Reparing and milling device for chemical-mechanical polishing soft pad and its producing method

Inventor(s): LIN XINZHENG [CN]; SONG JIANMIN [CN] ± (XINZHENG LIN, ; JIANMIN SONG, ; XINZHENG,SONG LIN)

Applicant(s): CHINA EMERG WHEEL ENTPR CO LTD [CN] ± (CHINA EMERG WHEEL ENTERPRISE CO., LTD)

Classification: **- international:** ***B24B53/12;*** (IPC1-7): B24B53/12
- cooperative:

Application number: CN2000128301 20001107

Priority number (s): CN2000128301 20001107

Also published as: CN100344410 (C)

Abstract of CN1351922 (A)

The present invention is one kind of chemical and mechanical polishing soft pad trimmer with great amount of uniformly spaced diamonds or isometric boron nitride grains. The granular abrasive is brazed to a substrate and coated with an anticorrosive layer containing diamond or diamond-like graphite to prevent the corrosion of chemical grinding paste to the brazed alloy. The structure makes it possible to trim the soft pad while using the soft pad to polish. In addition, the abrasive grains protruding beyond the substrate in the same height makes the soft pad trimmed homogeneously in both vertical and horizontal directions.

[19]中华人民共和国国家知识产权局

[51]Int. Cl[7]
B24B 53/12

[12]发明专利申请公开说明书

[21]申请号 00128301.4

[43]公开日 2002年6月5日

[11]公开号 CN 1351922A

[22]申请日 2000.11.7 [21]申请号 00128301.4

[71]申请人 中国砂轮企业股份有限公司
地址 中国台湾
[72]发明人 林心正 宋健民

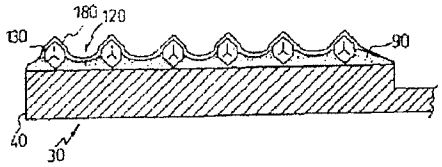
[74]专利代理机构 上海专利商标事务所
代理人 张民华

权利要求书4页 说明书8页 附图页数4页

[54]发明名称 化学－机械抛光软垫的修磨器及其制造方法

[57]摘要

一种化学—机械抛光软垫的修磨器及一种制造这种软垫修磨器的方法，该修磨器表面有大量均匀间隔的磨料颗粒，颗粒质地为金刚石或立方氮化硼等。磨料颗粒钎焊于基底，再覆以一层含有金刚石或仿金刚石石墨的防腐层，以防钎焊料合金受化学研磨膏的腐蚀。这种措施使修磨器得以在软垫进行抛光工作的同时修磨软垫。此外，磨料颗粒还基本等高地突出基底，使垂直、水平两方向均得以均匀地梳理或修磨软垫。



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权 利 要 求 书

1. 一种化学一机械抛光软垫的修磨器，包括尺寸在所需范围内且固接于基底的大量磨料颗粒，其特征在于：磨料颗粒在基底上固接成相互均匀间隔，并以一种预定的高度突出基底。

2. 根据权利要求 1 的化学一机械抛光软垫的修磨器，其特征在于：其中磨料颗料为金刚石或立方氮化硼晶体颗粒，单晶或多晶状态不论。

3. 根据权利要求 1 的化学一机械抛光软垫的修磨器，其特征在于：其中该尺寸范围为介于 50 至 250 微米之间。

4. 根据权利要求 1 的化学一机械抛光软垫的修磨器，其特征在于：其中磨料颗粒尺寸大抵均匀，各颗粒的尺寸差在 10%之内。

5. 根据权利要求 1 的化学一机械抛光软垫的修磨器，其特征在于：其中均匀间隔的磨料颗粒依照一种预定图形进行分布，使任何两粒颗粒间保持预定的距离。

6. 根据权利要求 5 的化学一机械抛光软垫的修磨器，其特征在于：其中各颗粒间预定距离为颗粒尺寸的 1.5 至 10 倍。

7. 根据权利要求 5 的化学一机械抛光软垫的修磨器，其特征在于：其中该预定图形为一栅格。

8. 根据权利要求 1 的化学一机械抛光软垫的修磨器，其特征在于：其中该预定高度为一均匀高度，对此所有磨料颗粒的突出偏差在 50 微米之内。

9. 根据权利要求 1 的化学一机械抛光软垫的修磨器，其特征在于：其中磨料颗粒具有自形的形状。

10. 根据权利要求 1 的化学一机械抛光软垫的修磨器，其特征在于：其中磨料颗粒具有预定的形状。

11. 根据权利要求 1 的化学一机械抛光软垫的修磨器，其特征在于：其中磨料颗料具有背向基底的尖点。

12. 根据权利要求 1 的化学一机械抛光软垫的修磨器，其特征在于：其中基底由金属材料制成。

13. 根据权利要求 12 的化学一机械抛光软垫的修磨器，其特征在于：其中金属材料为不锈钢。

14. 根据权利要求 1 的化学一机械抛光软垫的修磨器，其特征在于：其中磨

料颗粒藉助于钎焊料合金固定于基底上。

15. 根据权利要求 14 的化学一机械抛光软垫的修磨器，其特征在于：其中该钎焊料合金包括一种铬含量至少约为 2%重量百分比的镍合金。

16. 根据权利要求 14 的化学一机械抛光软垫的修磨器，其特征在于：其中该钎焊料合金以一种预定的厚度覆于基底的表面，并使各磨料颗料约有 10—90%露出。

17. 根据权利要求 1 的化学一机械抛光软垫的修磨器，其特征在于：其中该软垫修磨器覆以一防腐层。

18. 根据权利要求 17 的化学一机械抛光软垫的修磨器，其特征在于：其中该防腐层含有金刚石的石墨。

19. 根据权利要求 17 的化学一机械抛光软垫的修磨器，其特征在于：其中该防腐层含有仿金刚石的石墨。

20. 根据权利要求 17 的化学一机械抛光软垫的修磨器，其特征在于：其中防腐层的厚度约小于 3 微米。

21. 根据权利要求 18 的化学一机械抛光软垫的修磨器，其特征在于：其中仿金刚石的石墨的原子碳含量至少约为 95%。

22. 一种制造化学一机械抛光软垫修磨器的方法，其诸步骤包括 a ）提供一基底，其特征在于：该法的步骤还包括：

b ）于基底的某一表面均匀地间隔大量磨料颗粒，以及

c ）将磨料颗料固定于基底成，使各磨料颗粒以一种预定的高度突出基底。

23. 根据权利要求 22 的化学一机械抛光软垫修磨器的制造方法，其特征在于：其中磨料颗粒为金刚石。

24. 根据权利要求 22 所述的化学一机械抛光软垫修磨器的制造方法，其特征在于：其中磨料颗粒为立方氮化硼质地的晶体颗粒，单晶或多晶不论。

25. 根据权利要求 22 所述的化学一机械抛光软垫修磨器的制造方法，其特征在于：其中步骤 b ）与 c ）尚包括下列步骤：

a ）将一形成有预定孔图形的模板置于一钎焊料合金片上，使磨料颗粒的放置可受孔位置的控制，

b ）于模板的各孔内充填磨料颗粒，

c ）去除任何未进入模板孔内的磨料颗粒，

d ）将滞留于模板孔内的磨料颗粒压入钎焊料合金片中，使磨料颗粒部份埋

置于钎焊料合金内，

e）除去模板，使磨料颗粒留在钎焊料合金片上，以及

f）使带磨料颗粒的钎焊料合金片固接于基底。

26. 根据权利要求 25 的化学一机械抛光软垫修磨器的制造方法，其特征在于：其中各孔的尺寸为仅足以容置一粒磨料颗粒。

27. 根据权利要求 26 的化学一机械抛光软垫修磨器的制造方法，其特征在于：其中各孔具有预定的尺寸，可容纳预定尺寸的磨料颗粒。

28. 根据权利要求 22 的化学一机械抛光软垫修磨器的制造方法，其特征在于：其中该等磨粒颗的尺寸各在大约 50 至 250 微米的范围内。

29. 根据权利要求 22 的化学一机械抛光软垫修磨器的制造方法，其特征在于：其中磨料颗粒的尺寸大致均匀，使所有磨料颗粒的尺寸差在 10%之内。

30. 根据权利要求 25 的化学一机械抛光软垫修磨器的制造方法，其特征在于：其中成预定图形的各孔之间间隔成足以在任何两粒颗粒间产生预定的距离。

31. 根据权利要求 30 的化学一机械抛光软垫修磨器的制造方法，其特征在于：其中各颗粒间该预定的距离为颗粒尺寸的 1.5 至 10 倍。

32. 根据权利要求 25 的化学一机械抛光软垫修磨器的制造方法，其特征在于：其中该预定图形为一栅格。

33. 根据权利要求 22 的化学一机械抛光软垫修磨器的制造方法，其特征在于：其中该高出基底的预定高度为一均匀高度，对此所有磨料颗粒的突出偏差在 50 微米之内。

34. 根据权利要求 22 的化学一机械抛光软垫修磨器的制造方法，其特征在于：其中磨料颗粒具有自形的形状。

35. 根据权利要求 22 的化学一机械抛光软垫修磨器的制造方法，其特征在于：其中磨料颗粒具有一种预定的形状。

36. 根据权利要求 22 的化学一机械抛光软垫修磨器的制造方法，其特征在于：其中各磨料颗料具有一背向基底的尖点。

37. 根据权利要求 22 的化学一机械抛光软垫修磨器的制造方法，其特征在于：其中基底由金属材料制成。

38. 根据权利要求 37 的化学一机械抛光软垫修磨器的制造方法，其特征在于：其中该金属材料为不锈钢。

39. 根据权利要求 25 的化学一机械抛光软垫修磨器的制造方法，其特征在于：

其中钎焊料合金片的制造步骤为：以一种有机结合剂将钎焊料合金颗粒结合在一起，并使这些相结合的颗粒形成一片厚度符合需要的片体。

40. 根据权利要求 39 的化学一机械抛光软垫修磨器的制造方法，其特征在于：其中使钎焊料合金颗粒形成片体的步骤为经滚轧法进行。

41. 根据权利要求 39 的化学一机械抛光软垫修磨器的制造方法，其特征在于：其中使钎焊料合金颗粒形成片体的步骤为经挤压法进行。

42. 根据权利要求 39 的化学一机械抛光软垫修磨器的制造方法，其特征在于：其中使钎焊料合金颗粒形成片体的步骤为经带条铸造法进行。

43. 根据权利要求 25 的化学一机械抛光软垫修磨器的制造方法，其特征在于：其中钎焊料合金包括一种铬含量至少约为 2%重量百分比的镍合金。

44. 根据权利要求 25 的化学一机械抛光软垫修磨器的制造方法，其特征在于：其中钎焊料合金片具有隆起的钎焊厚度，足以让各磨料颗粒约 10—90%露出钎焊料合金。

45. 根据权利要求 22 的化学一机械抛光软垫修磨器的制造方法，其特征在于：其中尚括包括一步骤：即对磨料颗粒以及钎焊料合金覆以一防腐层。

46. 根据权利要求 45 的化学一机械抛光软垫修磨器的制造方法，其特征在于：其中该防腐层含有金刚石的石墨。

47. 根据权利要求 45 的化学一机械抛光软垫修磨器的制造方法，其特征在于：其中该防腐层含有仿钻石的石墨。

48. 根据权利要求 45 的化学一机械抛光软垫修磨器的制造方法，其特征在于：其中防腐层的厚度小于约 3 微米。

49. 根据权利要求 45 的化学一机械抛光软垫修磨器的制造方法，其特征在于：其中仿金刚石的石墨的原子碳含量至少约为 95%。

50. 根据权利要求 49 的化学一机械抛光软垫修磨器的制造方法，其特征在于：其中该覆层步骤是使用阴极电弧法进行。

Ex. 1003, Page 148

说　明　书

化学一机械抛光软垫的修磨器及其制造方法

本发明涉及一种修磨或调理化学一机械抛光软垫的修磨器及其方法，特别是一种含有超硬材料（如金刚石或立方氮化硼）、供修磨或调理化学一机械抛光软垫的修磨盘。

许多任务业今使用化学一机械方法抛光某些工件。尤其是计算机制造业已充分依靠化学一机械抛光法来抛光陶、硅、玻璃、石英以及金属质的芯片。这种抛光方法通常需要将芯片压于一由耐用的有机物质，如聚亚胺脂制成的旋转软垫上。并对该软垫添加一种研磨膏，内含可切削芯片物质的化学品，以及一定量的磨料颗粒，其作用是以物理方法刻蚀芯片表面。研磨膏持继地加于旋转中的软垫上，而得以有双重的化学兼机械力施加于芯片上，使之以所需的方式抛光。

对所产生的抛光品质尤为重要的，乃是软垫各处磨料颗粒的分布状况。软垫的顶部通常藉空穴之类卡持颗粒。挠性的软垫顶部可提供所需的支承，让磨料得以作用于芯片。

对软垫顶部进行保养，此问题归因于来自工件、研磨膏、以及修磨盘的抛光污腻的堆积。这种堆积会造成软垫顶部的发亮或硬化，使软垫不太能吸附研磨膏中的磨料颗粒。

因此，人们已作出诸多尝试，以各种装置“梳理”软垫顶部来修复，此法现已称之为“修磨”或“调理”化学一机械抛光软垫。为此，已使用多种装置及方法。这些装置中的一种为一圆盘，其表面或基底上固定大量的超硬晶体颗粒，例如固接于其某一表面或基底上的金刚石颗粒。

但是，这种由传统方法制成的金刚石盘表现出种种问题。首先，金刚石会自该盘的基底上脱落并沾附在化学一机械抛光软垫表面。其次，此种旧式生产方法有可能使生产出的圆盘上金刚石成多组簇集，即非均匀地散布于基底的表面。这种非均匀的成组现象，使该软垫的某些部位被过度修磨，造成磨损区域，而其它部位修磨不足，造成光亮层。另一种情况是，软垫的抛光效率降低，并出现抛光不匀现象。最后，这些圆盘的金刚石不能以一种均匀的高度突出圆盘表面。这种非均匀性又造成对该软垫的不均匀修磨，因为仅是那些足够突出的颗粒方可接触

Ex. 1003, Page 149

软垫。

金刚石自圆盘基底脱落，是因为它们被固接的方法不当。当金刚石是由电镀的镍加以固定于基底上时，无结合力惟有对金刚石的机械锁紧作用。因此一旦摇松，这些颗粒即会脱落。此种脱落现象又因化学品，即化学研磨膏中的酸对电镀材料的化学侵蚀而加剧。

另一方面，当金刚石是钎焊于基底上时，化学力会更牢固地固定金刚石。然而，化学研磨膏的酸性会快速溶化钎焊料而使金刚石脱落。因此，为使钎焊料曝露于化学品的程度降至最小，抛光过程需时时暂停，期间进行修磨，而后方可再度开始抛光。这种依次交替地抛光与修磨，浪费大量时间，而且极度低效。

鉴于上述，故需要一种可对化学一机械抛光软垫提供调理作用的软垫修磨器。此外，非常需要一种可将化学一机械抛光软垫调理至均匀深度的软垫修磨器。再者，很需要一种不易使金刚石颗料脱落的修磨器。最后，极需要一种可防化学研磨膏的酸性侵蚀，而且甚至可在以酸性研磨膏进行抛光的同时，连续修磨化学一机械抛光软垫的软垫修磨器。

本发明的目的是提供一种使用磨料颗粒来均匀修磨化学一机械抛光软垫的软垫修磨器。

本发明的另一目的是提供一种不易让磨料颗粒脱落的软垫修磨器。

本发明的又一目的是提供一种防腐的软垫修磨器，使之可以恒定地修磨化学一机械抛光软垫，纵然该软垫正于酸性研磨膏中进行研磨时亦然。

本发明的再一目的提供一种化学保持层，以防修磨盘元素溶解而污染研磨中的芯片。

本发明的另一目的是提供一种均匀修磨化学一机械抛光软垫的方法。

本发明的又一目的是提供一种方法，以降低软垫修磨器的磨料颗粒脱落的可能性，纵然当该软垫浸润酸性研磨膏时亦然。

按照本发明的一个方面，本发明的技术方案在于提供一种化学一机械抛光软垫的修磨器，其包括尺寸在所需范围内且固接于基底的大量磨料颗粒。其特点在于：磨料颗粒在基底上固接成相互均匀间隔，并以一种预定的高度突出基底。

而且，上述的修磨器上还可覆以一防腐层。

按照本发明的另一方面，本发明的技术方案在于提供一种制造上述修磨器的方法，其诸步骤包括：

a）提供一基底；b）于基底的某一表面均匀地间隔大量磨料颗粒；以及，

Ex. 1003, Page 150

c）将磨料颗料固定于基底成，使各磨料颗粒以一种预定的高度突出基底。

而且，上述方法还可包括一步骤：即对磨料颗粒以及钎焊料合金覆以一防腐层。

本发明与已有技术相比优点和积极效果非常明显。由以上的技术方案可知，本发明的软垫修磨器可均匀地修磨抛光软垫，而且磨料颗粒不易脱，特别是可以在软垫进行抛光的同时，对软垫进行修磨，从而大大提升软垫的工作效率，并延长其使用寿命。

以下结合附图进一步说明本发明的具体结构特征及目的。其中，诸附图为：

图 1 是一种现有的软垫修磨器的侧视图，该修磨器使用电镀法将金刚石固定于盘状基底上。

图 2 是另一种现有的软垫修磨器的侧视图，该修磨器经使用传统钎焊法将金刚石颗粒固定于盘状基底上而制成。

图 3 是本发明的软垫修磨器的侧视图。

图 4 是本发明的钎焊料合金片，以及将磨料颗粒置于其表面的模板的侧视图。

图 5 是钎焊料合金片表面覆以模板，且磨料颗粒充填该模板的各孔的侧视图。图中显示依照本发明的原理，有一平面将磨料颗料压入钎焊料合金片。

图 6 是钎焊料合金片中已依据本发明的原理压入磨料颗粒的侧视图。

图 7 表示了作为本发明实例一制作的两个软垫修磨盘对一个 28 英寸的化学一机械抛光软垫进行修磨的情况。

图 8 表示了作为本发明实例二制作的两个金刚石修磨盘修磨一个用于研磨 8 英寸硅芯片的化学一机械抛光软垫的情况。

请注意，在说明书及权利要求书中，下列技术术语使用时将依照以下提出的定义。如文中所使用的“磨料颗粒”或“砂粒”，或者相似的词，表示任何超硬质的晶体或多晶体物质，或者这些物质的混合物，而且包括（但不限于）金刚石、多晶体金刚石、立方氮化硼、以及多晶体立方氮化硼。再者，“磨料颗粒”、“砂粒”、“金刚石”、“多晶体金刚石”、“立方氮化硼”、以及“多晶体立方氮化硼”等各词可互换使用。如文中所使用的“基底”，表示修磨器中表面可固定磨料颗粒的基础部份。该基础部份可为任何形状、厚度、或者材料，而且包括（但不限于）金属、合金、陶瓷及其混合物。如文中所使用的“自形”，表示自然形状，即未被人为变更的天然形状。如文中所使用的“尖锐点”，表示晶体中可能出现的任何尖狭顶峰，而且包括（但不限于）角、棱、尖峰以及其它突起。如文中所使用的“金属”，表

Ex. 1003, Page 151

示任何类型的金属、金属合金、或者它们的混合物，尤其包括（但不限于）钢、铁、以及不锈钢。

现参阅图 1，图中显示一现有的软垫修磨器 10，该修磨器中有大量金刚石颗粒 50 电镀于一基底 40。电镀材料 60 通常为经酸液沉淀而出的镍。这种电镀法不仅成本高且费时，而且亦因该法所产生的残留物质而对环境有害。

由图 1 可看出，电镀而成的软垫修磨 10 有许多缺点。首先，电镀材料 60 不能与金刚石颗粒 50 形成任何化学结合。因此，仅有微弱的机械力将金刚石颗粒 50 固定于基底 60 上。当该软垫修磨器压在化学一机械抛光软垫上旋转时，这种机械力会被作用于金刚石颗粒 50 上的较大摩擦力快速克服，使之易于自电镀材料 60 松落，于电镀材料 60 上留下孔穴，例如空隙 70。此种孔穴会被快速充填以工件上磨下的残留物，以及来自研磨膏的化学品及磨粒颗粒，为此进一步削电镀材料 60。由于电镀材料 60 所产生的机械力是唯一将金刚石颗粒 50 固定于基底 40 上的手段，使金刚石颗粒 50 露出电镀材料的高度必须保持最小。因此，电镀材料 60 与化学一机械抛光软垫的接触不可避免。这种接触会磨损电镀材料，而易使金刚石颗粒 50 脱落。此外，电镀材料 60 易在各金刚石 50 四周的凹部 80 等处起泡。这些气泡，加之过低的露出高度以及金刚石颗粒 50 间的紧密间距，即使能也难以使金刚石颗粒 50 深深戳入软垫纤维。无此种戳入，则对修磨过程大为有碍。

现参见图 2，图中显示现有的软垫修磨器 20，其基底 40 有金刚石颗粒 50 用钎焊材料 190 及传统的钎焊技术钎焊于基底 40。钎焊材料通常含有一种混以碳化物形成元素的合金。这种碳化物形成元素可让金刚石颗粒 50 得以以化学方式结合于钎焊料，后者则进而结合于基底 40。这种结合方式可大为增加修磨器 20 的强度，但会伴随某些副作用。钎焊材料 90 必须保持最低之量，以免完全覆盖金刚石颗粒 50。因此，金刚石颗粒 50 仅被包以薄薄一层钎焊材料 90。此问题偏遇典型的钎焊材料机械性能非常弱。这种机械性能的弱抵销了金刚石颗粒 50 与钎焊材料间的化学结合强度，因为钎焊材料本身会随金刚石颗粒的脱落而被割断。

另一个与钎焊材料 90 有关的问题是，其非常易受研磨膏的化学腐蚀。这种化学腐蚀会促成金刚石颗粒 50 的脱落，因为其使原已机械性能弱的钎焊材料 90 变得更弱。因此，为减少软垫修磨器 20 接触化学研磨膏的时间，软垫修磨器 20 应用前，对工件的抛光必须暂停，而让化学研磨膏得离软垫而去。这种抛光过中的暂停使精加工产品的时间大为增加，因而低效。

传统钎焊法的另一缺点为，当加诸于基底 40 时，融熔状金属合金的表面张

Ex. 1003, Page 152

力易使磨料颗粒聚集成簇状。此种簇状标以 100，间隔为 110。其总体表现为金刚石颗粒 20 的非均匀分布，为此使调理效率较低。此低效率应当归因于间隔 110，因为其使化学一机械抛光软垫的部份区域依然保持为未调理前的状态。这种非均匀的调理使软垫的一部份区域磨损较他处快，总体结果为工件接受一种非均匀的抛光，因为已磨损的区域抛光效率低于经正确调理的区或。

磨料颗粒的簇状所产生的另一效应，是会于钎焊材料 90 中形成大量隆起。隆起的形成会提升某些磨料颗粒相对基底 40 的高度，使之高于其它磨料颗粒。因此，最高凸的磨料颗粒会深深戳入软垫的纤维，妨碍不甚高凸的磨料颗粒发挥任何梳理作用。此举亦造成调理效果的低效与不合理。

与现有的软垫修磨器相反，本发明得以均匀地修磨化学一机械抛光软垫。

现参见图 3，图中显示一以本发明原理制成的软垫修磨器。该软垫修磨器有大量磨料颗粒 180 以钎焊材料 90 固定于一基底 40。磨料颗粒 180 可为任何超硬质的材料。较佳的材料包括（但不限于）金刚石、多晶体金刚石、立方氮化硼以及多晶体立方氮化硼。

同时示于图 3 的，为一防腐层 130。此防腐层形成于软垫修磨器表面之前，磨料颗料先以下述方法固定于基底 40 上。防腐层 130 为另一种超硬质材料，如金刚石、仿金刚石的石墨、或者立方氮化硼，而且是可以匹配磨料颗粒 180 的材料。在一较佳实施例中，防腐层 130 约含有至少为 90－95%的金刚石，或者仿金刚石的石墨。防腐层 130 可为任何厚度，但通常在 0.5 至 5μm的范围内。在一较佳实施例中，防腐层 130 的厚度约为 1 至 3μm。这种防腐薄层 130 可采用物理气相淀积法（PVD）产生。物理汽相淀积法，如使用石墨电弧及石墨阴极的，已为业界所知，而且可用以产生防腐层 130。

由防腐层 130 产生的优点是可有效密封工件表面，而且也可密封化学一机械抛光软垫上易受化学侵蚀的其它所需表面。作为密封层，防腐层 130 可保护钎焊材料 90，使之免受沾于该软垫上的化学研磨膏的侵蚀。这种保护作用甚至于软垫正研磨工件之时，让软垫修磨器 30 得以连续修磨该软垫，从而可消除过去用以延长现有软垫修磨器寿命的生产停顿。对化学一机械抛光软垫的连续与均匀修磨，使得可有较高的产量，并且可延长该软垫的寿命与效率。

图 4 至 6 显示一种将磨料颗粒 180 固定于基底 40 上的方法。首先，将具有大量孔 150 的模板 140 置于钎焊料合金片 190 上。使用该模板可让磨料颗粒 180 的放置得以通过将模板的各孔设计成所需的分布图形而加以控制。用于磨料颗粒

Ex. 1003, Page 153

放置的分布图形，可由熟悉该项技术的普通技术人员加以选定，以满足该软垫修磨器的特定使用要求。

作为本发明的一个方面，这些孔的分布将呈一种栅格图形，且孔距预定为可使钎焊料合金片 190 上的磨料砂粒 180 间产生所需的间隔。在一较佳实施例中，该等砂粒均匀间隔，间距为各砂粒尺寸的 1.5 至 10 倍。当模板 140 置于钎焊料合金片 190 后，各孔 150 即被充填以磨料颗粒 180。孔 150 有预定的尺寸，使各孔中仅可容纳一粒磨料颗粒。任何尺寸的磨料颗粒或砂粒均可使用，但作为本发明的一个方面，颗粒尺寸为直径约 50 至 250 微米之间。

作为本发明的另一方面，模板中各孔的尺寸也可定制，以使所获得分布图形为令各磨料颗粒尺寸不一，或者尺寸大抵均一。在一较佳实施例中，该模板的各孔为足以仅选择相互尺寸差在 10%微米内的砂粒。这种砂粒尺寸的均匀性有助于对化学一机械抛光软垫梳理的均匀性，因为各磨粒颗粒的工作负荷均匀分布。进而，该均匀的工作负荷分布可减小各磨料颗粒的应力，而延长软垫修磨器 30 的有效寿命。当模板 150 的各孔被充填以砂粒 180 后，将任何多余的磨料颗粒除去，并将平面 160 加诸于磨料颗粒 180。平面 160 必须是质地为极强固的刚性材料，因为其必须能将磨料颗粒 180 下压至钎焊料合金片 190 内。典型材料包括（但不限于）钢、铁，以及它们的合金等。

图 6 显示磨料颗粒 180 埋置于钎焊料合金片 190 中。由于表面 160 为平面状，故磨料颗料 180 以一种均匀的距离突出基底 40。这种距离取决于模板 140 的厚度，并且在较佳实施例中，各磨料颗粒将突出成使此距离在 50 微米之内。

图 4 至 6 中所示的磨料颗粒为圆形，而图 3 中的为尖形。本发明的范围包罗任何形状的磨料颗粒，包括自形即天然形状的颗粒。不过在较佳实施例中，磨粒颗粒 180 有一背向基底 40 的尖点。

当磨料颗粒 180 埋置于钎焊料合金片 190 中后，即可将该片固定于基底 40，如图 3 所示。所使用的钎焊料合金可为业内所知的任何钎焊材料，但最好为镍合金，且铬含量大于 2%重量百分比。此种成份的钎焊料合金自身质地超硬，而且不易受内含磨料的研磨膏的化学侵蚀。因此，当使用一种超硬的钎焊材料时，防腐层 130 为最佳。

由于磨料颗料 180 埋置于钎焊料合金片 190 中，故液态钎焊料合金的表面张力不足以使颗粒簇集。此外，钎焊料厚度以远为薄的程度出现，且无隆起形成。再者，钎焊料于各磨料颗粒间形成一种凹面，为此提供强大支承。最后，在较佳

Ex. 1003, Page 154

实施例中，钎焊料合金片 190 的厚度选定成可让各磨料颗粒有大约 10 至 90%突出钎焊材料 190 的外表面。

由于本法先将磨料颗粒埋置于钎焊料合金片 190 内之故，遂形成均匀的间隔。此外，磨料颗粒 180 可突出基底 40 均匀的高度或距离，此举意味着当应用于化学一机械抛光软垫时，它们将突入软垫纤维内同一深度。均匀的间隔及匀一的突入使软垫得以被均匀地修磨或梳理，进而提升该软垫的研磨效率并延长其使用寿命。

为便于更好地理解本发明，以下将提供两实例。不过这些实例绝非意味着用作限制本发明的范围。

〔实例 1〕

以下制作两个软垫修磨盘。即通过滚轧金属粉末混合物以及两轧辊间的有机结合剂，预制一钎焊料合金片。再藉助于一模板，将通用电子公司(General Electric Company)生产、平均尺寸为 135 及 225 微米的 MBS970 号金刚石砂粒埋入该钎焊料合金片。所使用的模板使金刚石砂粒形成一种令各金刚石砂粒距离为 900 微米的栅格图形。当金刚石砂粒置入钎焊料合金片后，采用丙烯酸凝胶将该片胶合于一金属基底。所形成的组件随后于真空炉内 1000° 的温度下进行钎焊。所产生的产品为两个直径约 100 毫米、厚度约 6.5 毫米的平盘。再将两盘与一个以随机方式放置两倍金刚石颗粒量的修磨盘进行对比试验。具体为修磨一个 28 英寸的化学一机械抛光软垫（该软垫以碱性研磨膏研磨 8 英寸的硅芯片）。试验结合显示在图 7 中。表中 DG135－900 是颗粒为 135 微米的盘，而 DG225 是颗粒为 225 微米的盘。

由图 7 可看出，颗粒均匀放置的两盘均大大优于金刚石随机放置的盘。此外，颗粒为 135 微的盘，性能几乎高达该随机颗粒盘的两倍。

〔实例 2〕

以实例 1 的方法再制两金刚石修磨盘。不过，金刚石尺寸采用 225 微米及 275 微米。此外，各盘覆以 1 微米厚的仿金刚石的石墨层，以保护钎焊料合金。仿金刚石的石墨膜以阴极电弧法加以沉积。这些盘然后与一传统的金刚石修磨盘进行比较，具体方法是修磨一个用于研磨 8 英寸硅芯片的化学一机械抛光软垫。该软垫浸润 P H值为 3.0 的酸性研磨膏。修磨过程于研磨进行的同时就地完成。其结果显示在图 8 内。表中 DG275－900 是含有尺寸为 275 微米且均匀间隔的砂粒的盘。DG225－900 是含有尺寸为 225 微米且均匀间隔的砂粒的盘，而 A T 是该传统式金刚石盘。

Ex. 1003, Page 155

由图 8 可看出，以传统方式生产出的金刚石盘，于研磨膏的酸性环境中不能保持 1 小时以上。然而，本发明的两盘可保持 30 小时以上。这种寿命期使得有大为良好的修磨效果，并且对现有技术构成重大改进。当然，应理解上述实施例仅是对应用本发明原理的例示性说明。

熟悉这项技术的人员自可在不偏离本发明范围的条件下，设计出大量的变形形态及变更形态，本案附录的权利要求意在涵盖这种变形形态能及变更形态。因此，尽管以上本发明已结合目前看最实用且最佳的本发明实施例，以特列情形加以详细描述，然对熟悉该项技术的普通人士而言，可在不偏离本文中所提出的原理及概念的条件下作出大量变形形态，包括（但不限于）在尺寸、材料、形状、形式、功能、操作方式、组合以及使用方面的变更。

说 明 书 附 图

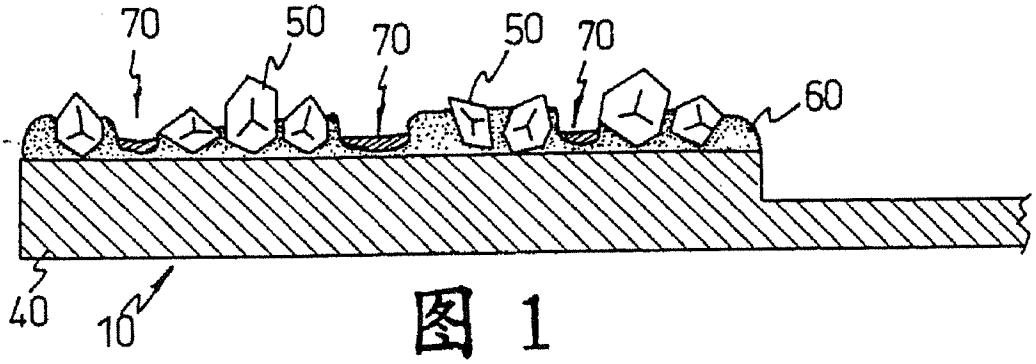


图 1

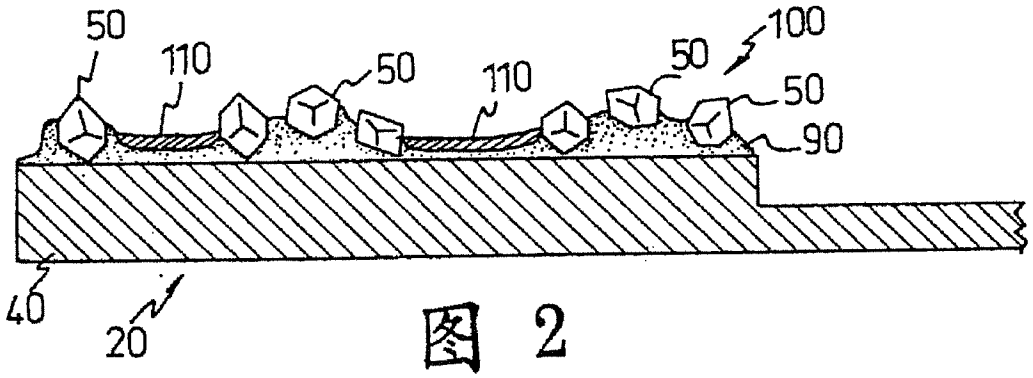


图 2

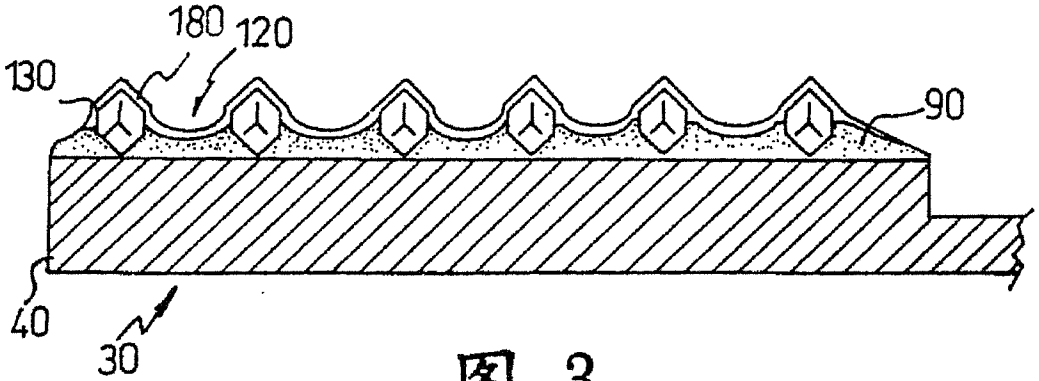


图 3

Ex. 1003, Page 157

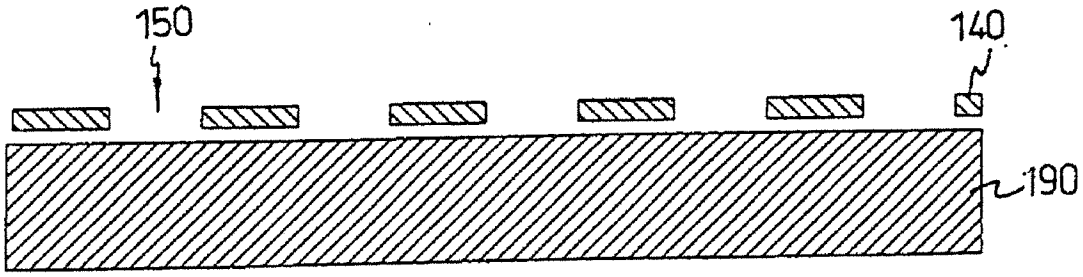


图 4

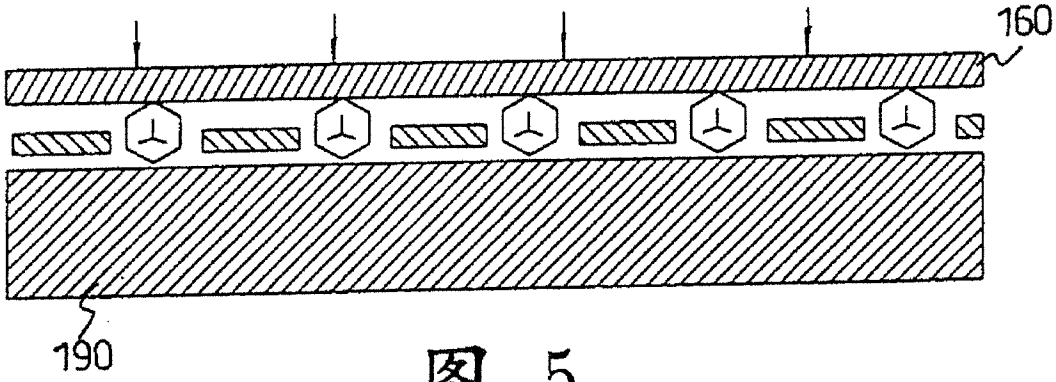


图 5

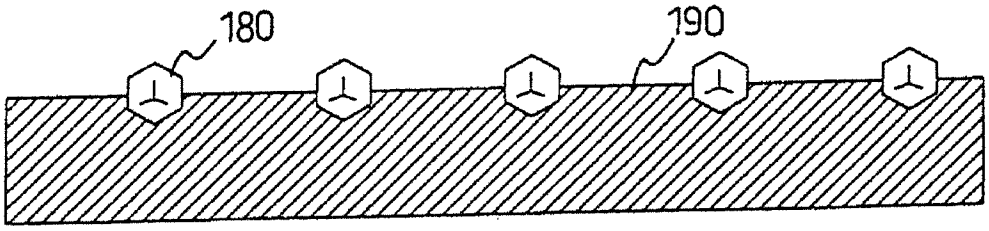


图 6

Ex. 1003, Page 158

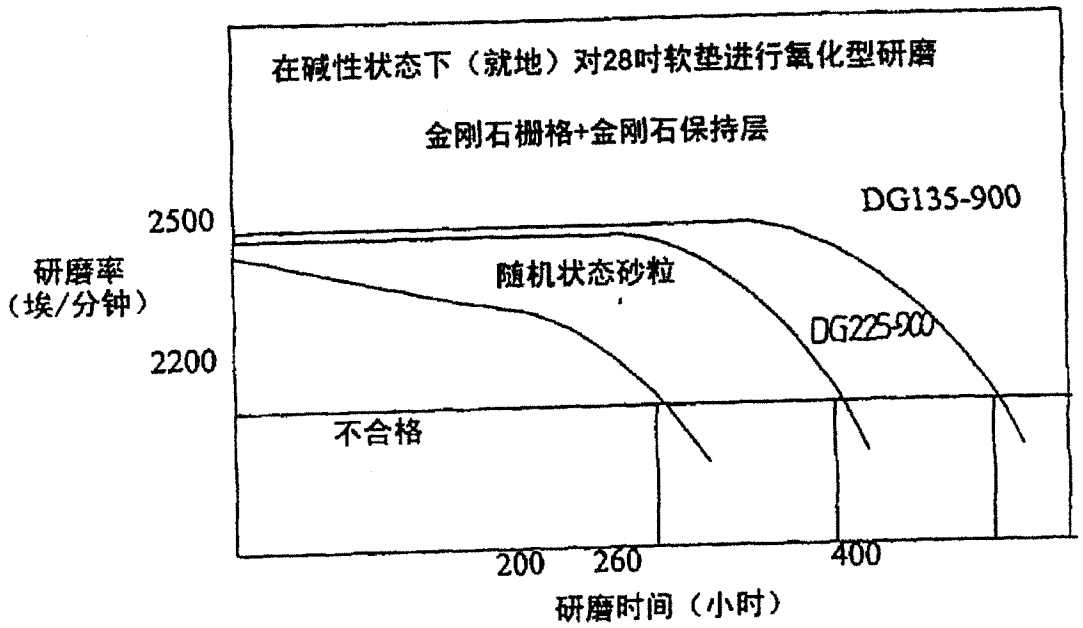


图　7

Ex. 1003, Page 159

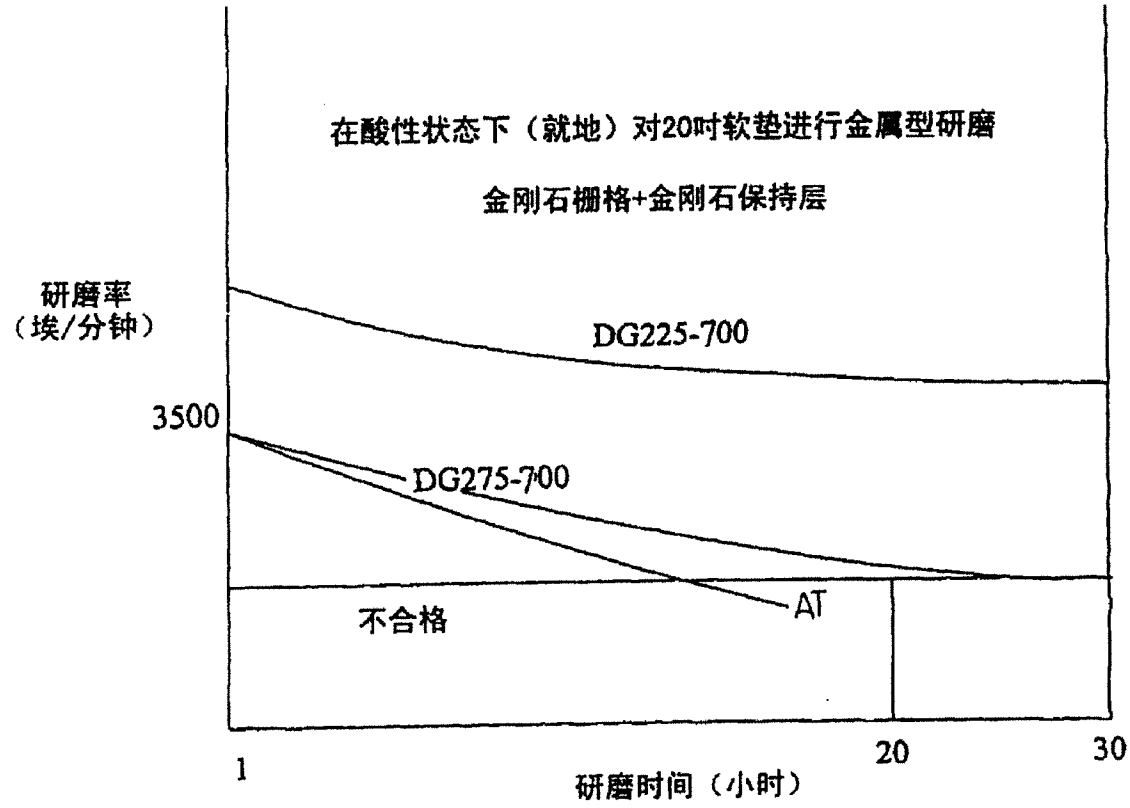

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图 8

CN1494984

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Sander for polishing cloth and polishing cloth sanding method using said sander

CN 1494984 A

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ABSTRACT translated from Chinese

The invention provides a polishing cloth with grinding and the use of the sander polishing method for polishing cloth, the polishing cloth with the polishing device to adjust the grinding surface of the state, even in the abrasive polishing surface of the tip shape of the state of generating individual differences can also cause a uniform polishing cloth surface, but also the polishing cloth surface given adaptation workpiece appropriate polishing performance, the grinding device is configured, the metal base (2) of the circumferential edge portion with polished surface (4) a polishing cloth with a sander (1), the grinding surface (4), respectively, are juxtaposed alternately different from each other abrasive particle consisting of a first and second particle group (5), (6), in the metal substrate (2), set the abrasive grains can be adjusted to each of these groups (5,6) maximum particle size of the abrasive grains contained in the front end plane (S1), (S2) between the height difference (δ) of the adjusting mechanism (7).

DESCRIPTION translated from Chinese

Polishing cloth with a sander and use the sander polishing cloth polishing method

Technology

The present invention relates to a chemical mechanical polishing (Chemical MechanicalPolishing; hereinafter referred to as CMP) in the distribution network during the polishing cloth or foreign material removing clogging on the occasion of use, and the polishing cloth with polishing cloths with its grinding method.

BACKGROUND

In general, a silicon wafer substrate for manufacturing a semiconductor integrated circuit such as a silicon wafer substrate, or the process of micro-electronic circuits, for removing exist irregularities on the surface of the substrate or the purpose of crystal defects by CMP processing. In this CMP processing, a predetermined load while the wafer substrate is attached to the polishing apparatus to suppress the fixed disk constituted by the foamed polyurethane polishing cloth on the side of the slurry supplied to the polishing liquid known that both the wafer and the polishing cloth rotating polishing.

As the slurry in CMP, with iron oxide, barium oxide, cerium oxide, aluminum oxide, colloidal silica polishing particles suspended in potassium hydroxide, dilute hydrochloric acid, dilute nitric acid, aqueous hydrogen peroxide, ferric nitrate, slurry of the polishing solution etc., these are based on the polishing rate and the wafer or a wafer substrate to be processed on the type of object to said selected appropriately.

This CMP processing, in order to use the same or more than the polishing cloth to polish the wafer repeatedly, with an increase in the number of CMP processing, the removed workpiece polishing debris or aggregated fine particles into the polishing cloth holes and cause clogging, the wafer polishing rate decreases. Therefore, it is necessary to regularly or periodically be removed after distribution network clogging the surface of the polishing cloth, polishing cloth reproduction surface roughness of the surface to restore the polishing

CLAIMS (10) translated from Chinese

1 A polishing cloth with sanders, is rotatable on the surface of the metal substrate is formed from a polished surface polished with the polishing cloth, characterized in that the grinding surface in the circumferential direction of the juxtaposed multiple a particle group, in the metal substrate, for all or part of the particle group, setting can be adjusted by a plurality of abrasive grains are formed in the distal end of said polishing surface reference plane height difference adjustment mechanism.

(2) as claimed in claim 1, wherein the polishing with a polishing cloth, characterized in that the adjusting mechanism of a particle group, are fixed to the composition and the metal base of the adjusting mechanism are formed separately on a base, along The polished surface of the metal substrate of the particle group in the peripheral portion juxtaposed annularly.

3 as claimed in claim 1, wherein the polishing with a polishing cloth, characterized in that the abrasive grains are formed from the group of the polished surface and the peripheral edge of the annular disk shape in parallel with the periphery of the grinding surface at an angle The shape of the helical segments, small round shape is selected among one or both of the flat shape.

4 as claimed in claim 3, wherein the polishing with a polishing cloth, characterized in that the particle group are formed from the planar shape is selected among the two kinds of planar shape, the plane shape of two different particle group In the polished surface in the circumferential direction alternately juxtaposed.

5 as claimed in claim 1 or 2, wherein the polishing with a polishing cloth, characterized in that the juxtaposed surfaces of the polishing abrasive grains group is a group of the same size of each abrasive particle group is formed, or, by different abrasive particle size particle group is formed to form two.

As claimed in claim 5, wherein the polishing with a polishing cloth, characterized in that the juxtaposed surfaces of abrasive grains in the

speed of operation is known as grinding, used in this operation is referred to as polished cloth with sander tool.

Because the diamond abrasive grains in the polishing cloth in polishing is an excellent material, so that the use of diamond abrasive grains of the polishing cloth with sanders, nickel-plated stainless steel by electrodeposition of diamond abrasive grains is generally useful method of the. In addition to metal brazing material made with the aid of the brazed diamond grit on stainless steel (for example, see JP-A No. Hei 10-012579), by sintering the diamond abrasive material and the holding and fixing the reaction sintering method (such as , see JP-A No. 2001-179638). Also for the polishing cloth to obtain a stable ability to remove the surface layer of the object, also proposed are arranged at equal intervals in a CMP polishing cloth polishing abrasive grains (for example, see JP-A No. 2000-141204, Laid-Open Publication No. 2002-127017) .

However, in this prior art the polishing cloth with sanders, since in its structure, can not be avoided due to the tip shape of the abrasive grinding surface in a manner to produce individual differences, so even with the same polishing cloth sanders is difficult to cause a homogeneous surface polishing cloth, in addition, the surface state of the polishing cloth is necessary to be adjusted according to the workpiece, for example, formed on the surface of the silicon wafer wafer interlayer oxide film, the surface condition by polishing a rough cloth state, caused by mechanical polishing cloth to remove the polishing processing speed enhancement factor, in addition, the Cu wiring, for the presence of the surface of the polishing cloth polishing liquid (slurry) of the factors that caused by chemical reactions caused by mechanical polishing cloth ratio more factors removal processing purposes, it is necessary to maintain a certain degree of surface roughness of the polishing cloth, therefore, on the polishing surface of the polishing cloth with the polishing cloth is sanders, it is necessary to prepare the required number of, grinding state of the surface of the object to be processed adaptation, there is such a problem costs.

SUMMARY OF THE INVENTION

The present invention solves the technical problem, is to provide a polishing cloth can be adjusted by means of a grinding surface of the grinding device in the state, even if the shape of the abrasive polishing surface of the front end of the state of generating individual differences, the grinding surface can also be resulting in a uniform adjustment of the surface of the polishing cloth, or can be imparted to the surface of the polishing cloth to adapt appropriate workpiece polishing performance of the polishing and the polishing cloth with the polishing cloth with its grinding method.

To solve the above problems, the present invention is polished with the polishing cloth to be rotatable in the metal substrate is formed on a surface made of polished surface polished with the polishing cloth, characterized in that the grinding surface in the circumferential direction, a plurality of parallel particle group, in the metal substrate for all or a portion of a particle group, can be adjusted by a plurality of abrasive grains are formed of the front plane of the polished surface of the step difference adjusting mechanism.

If the polishing cloth with the polishing device, since the adjusting mechanism by means of a plurality of abrasive grains can be adjusted in each of the above groups plane height difference, even in the abrasive polishing surface of the tip shape of the state of individual differences arising may also adjust the particle group of the plurality of difference in height of the plane, polished to adjust the surface state of the polishing cloth surface resulting in uniform, or the surface of the polishing cloth can be polished surface polishing cloth, giving adapt workpiece appropriate polishing performance. In this case, the high particle group plane mainly used for polishing the surface of the abrasion cloth, base particle group is mainly used for low surface roughness of the polishing cloth adjusted.

In the polishing cloth with the polishing vessel, it can be taken as a group of the adjusting mechanism of abrasive particles, are fixed to the metal substrate with said composition formed separately on a base of the adjustment mechanism, along the polished surface of the metal substrate The peripheral edge portion of the particle group are juxtaposed annularly.

If the above particle group respectively from the periphery of the grinding surface of the annular plate shape in parallel with the periphery of the grinding surface of the angled shape of the helical segments, small round shape is selected among one or both of the plane formed in the shape, it is best removed by the polishing cloth of a polishing slurry of the cutting chips or agglutination easily discharged to the outside.

In the grain group, respectively, a planar shape is formed by selecting from the two planar shape of the occasion, the plane shape of two different grinding surface of the particle group in the circumferential direction are alternately arranged side by side is appropriate.

polishing base, the abrasive particle size different from each other formed by a first and a second particle group particle group to form, these first and second particle group in the polished surface of the particle group in the circumferential direction alternately juxtaposed.

As claimed in claim 6, wherein the polishing with a polishing cloth, characterized in that the first particle group of the same particle size of the abrasive grains or both formed.

As claimed in claim 1 or 2, wherein the polishing cloth with the polishing, characterized in that each particle group, each of the abrasive particles in the polishing surface of said two-dimensionally arranged with regularity, mutually adjacent The minimum abrasive particles form a regular triangle or a parallelogram lattice arranged.

9 A polishing cloth for polishing method using a claims 1 to 8 as described in any one of the polishing cloth with a sander polishing cloth, characterized in that by means of the adjusting mechanism, the abrasive grains adjacent to each other plane of said group setting of a certain height difference between, grinding the polishing cloth.

10 A polishing cloth for polishing method using a claimed in claims 6 to 8 wherein any one of the polishing cloth with a sander polishing cloth, characterized in that, by means of the adjusting means, the polished surface of the first 1 particle group in a height adjustment of the reference plane than the second particle group in a certain amount of height above the reference plane, polishing the polishing cloth.

In addition, the polished surface of juxtaposed groups of the abrasive grains may be made of each of the same size of abrasive grains formed by the same group, or by a different abrasive particle size particle group is formed to form two .

Moreover, in parallel provided on the polished surface of the particle group, the abrasive particle size different from each other are formed a first group and a second abrasive grain group to form the occasion, the first particle group and the the second particle group in the circumferential direction of the grinding surface juxtaposed alternately be appropriate. In this case, the abrasive particle size may be from the same or two different grain size of abrasive grains to form the first group.

Here, in the above group of abrasive, abrasive particles in the polishing surface of said two-dimensionally arranged with regularity, the abrasive particles adjacent to each other resulting in the smallest lattice triangle or a parallelogram if arranged, can to further improve the stability and uniformity of polishing.

Further, the above problem can be by the adjusting mechanism, adjacent to each other between the reference surface of said particle group setting of a certain height difference, grinding polishing cloth of the polishing cloth for polishing method to solve.

In the polishing cloth for polishing method, a plurality of abrasive grains by the first group and the second particle group group to form the occasion, by means of the adjusting means, the polished surface of the first particle group basis Height adjustment of the surface than the second particle group in a certain amount of height above the reference plane, polishing the polishing cloth is suitable.

If such a polishing cloth with the present invention and a polishing cloth with its grinding polishing method, since the adjustment mechanism can be adjusted by means of the particle group in the front end by the abrasive grains are formed in said reference surface Polished surface height difference, even abrasive surface of the tip shape of the state of polished produce individual differences can also cause a uniform polishing cloth surface, and that the surface of the polishing cloth can also be given a suitable adaptation of the workpiece polished performance.

BRIEF DESCRIPTION

Figure 1A ~ 1D are said polishing cloth of the present invention with a polishing abrasive cluster configuration The different shapes of the first embodiment of a perspective diagram.

Figure 2A ~ 2D is a diagram of the present invention sander polishing abrasive cloth with the configuration of cluster shape different from the first perspective view of a second embodiment.

1A in Figure 3 is a cross-sectional view of line III-III.

2A in Figure 4 is a cross-sectional view of IV-IV.

Figure 5 is shows the first abrasive grains in a group 5 a schematic view of the arrangement state.

Figure 3 Figure 6 is a partially enlarged view.

Specific embodiments

Figure 1A ~ 1D shows the polishing cloth of the present invention the particle group with sanders shaped configuration different from the first embodiment. 1 is polished by the polishing cloth with the front side at its center with a circular concave portion 2a, and in its periphery surface of the metal substrate 3 is formed on the polished surface 4 made of an annular disk-shaped metal substrate 2 is constituted, In the polished surface 4, respectively, a plurality of annularly arranged in parallel along the circumferential direction of the separate first and second particle group 5,6. In other words, along the metal substrate surface 3 of the metal substrate 2 is the periphery a plurality of annularly arranged side by side forming the polishing abrasive surface groups 5,6 4.

Moreover, as shown in Figure 3, the metal substrate 1 is provided with a regulating mechanism 7, so that each particle group in the 5 and 6 to contain the maximum grain size of the front end of the plane as the reference surface S1, S2, the can be adjusted on the polished surface 4, 5 and 6 of the plurality of abrasive grains of each group plane S1, S2 each level difference between δ.

This is not necessarily required in the metal base 2 is provided the concave portion 2a.

Specifically, the adjusting mechanism 7 by the base station 7a, 7b and gaskets adjusting screw 7d composition, wherein the abutment surface 7a of the fixed abrasive group 5 or 6, and down slidably fitted into the opening in the metal base 2 the surface 3 of the concave portion 7e; adjusting screw 7b is provided in the base 7a of the back, screwed from the metal substrate 2 in the recessed portion 7e of the bottom surface of the metal substrate 2 on the back side of the through screw hole 7c; When adjusting the fixed the grains of the base 7a of the group at the 5,6 position, the height of the reference plane, the spacer clip 7d in the back surface of the base 7a of the recess between the bottom 7b. Moreover, the adjusting mechanism are fixed to the base 7 7a plurality of abrasive grains on the surface of the metal cluster 5,6 substrate 2 along the peripheral edge portion annularly juxtaposed, forming the polished surface 4.

Therefore, in order to adjust the reference surface of the particle group in 5,6 S1, S2 of the height position of the spacer having a desired thickness into the metal base 7d concave portion 7e 2, through the means of the adjusting screw clamp 7b 7e on the bottom surface of the recess and the back surface of the base 7a between the respective particle group can be adjusted at the datum 5,6 S1, S2 of the metal substrate surface 3 of the height position, i.e. a plurality of abrasive grains group 5, 6 of the plane S1, S2 each level difference between δ, polished surface 4 can be adjusted state. Furthermore, in the present embodiment, the means of adjustment mechanism 7, the plane S1, S2 of the metal substrate surface 3 of the height can be within the range of 0 ~ 300μm regulation.

Among the grain group, base particle group is mainly used for high abrasion polishing cloth, base particle group is mainly used for low surface roughness of the polishing cloth adjusted. That is, since by applying the grinding load is concentrated on the whole high abrasive effect on the plane base, the load applied per unit area increases, so that they wear mainly used for polishing cloth, on the other hand, lower plane The particle group to some extent the burden of the load, the high plane of said polishing abrasive cloth group causes reduced wear rate. Furthermore, the mutual height difference between the reference plane δ becomes great occasions, as a particle group in the low plane burden load completely, so that they do not play a polishing cloth to the effect of reducing the wear rate. This group of a plane is also low polishing abrasive cloth cutting chips function effectively discharged.

In addition, polishing with the polishing cloth 1 is, the plurality of abrasive grains each different size from the group of 5,6 abrasive grains formed by the first and the second group 5 group 6 to form abrasive grains, these two grinding 5,6 swarm metal substrate 2 along the peripheral edge portion of the circumferential direction are alternately arranged side by side polished surface 4 is formed.

Specifically, the first particle group 5, shown in Figure 5, Figure 6, the size of the size of two grains 50, 51 are formed, the abrasive grains 50, 51, shown in Figure 5, 7a, the base surface of the polished surface 4 of the aforementioned two-dimensionally on a regular manner equally arranged adjacent to each other resulting in the smallest abrasive particles each triangle or a parallelogram lattice rows. Moreover a large interval between the abrasive particles 50 smaller than the interval between the abrasive particles 51 to be wide.

By thus granularity of the size of two grains are arranged in a regular manner equally possible to further improve the stability and uniformity of polishing.

On the other hand, in the second particle group 6, although it can be in particle size of the first abrasive grains group 5 different or more of the same particle size abrasive grit size in both forms to form any kind, but preferably in conjunction with the first particle group 5 Similarly, the abrasive are two-dimensionally arranged in a regular manner equally.

Here, the group forming the abrasive grit 5,6, diamond abrasive grains can be used for example, the abrasive particle size, in general, it is best defined in JIS B 4130 # 325 / # 400 ~ # 60 / # 80 range.

Further, forming the first abrasive grains group 5 is not necessarily required particle size mentioned above is the size of two grains 50 and 51, it can be the same size of abrasive particles (see Fig 2A ~ 2D embodiment of FIG.). Further, the adjustment mechanism 7 is not necessarily required in the first and second abrasive grains group 5 group 6 of the two prescription settings, or only part of the particle group, i.e., the first or the second particle group 5 mill one group of six tablets set. In this case, adjusting mechanism 7 is not set particle group, the base station 7a fixed to the metal substrate directly to the peripheral portion 2.

Thus, if each particle group with grains of different size, or different sizes of the abrasive grit group manipulation mixing, large size of the abrasive wear is mainly used for the polishing cloth, the small size of the abrasive mainly used for polishing cloth surface roughness adjusted by the adjustment of their size, can be a surface roughness of the polishing cloth ended for a particular CMP of the object state.

Further, as shown in Fig 1A ~ Figure 1D, the polishing cloth with sanders 1, the first and second abrasive grains group 5 group 6 of the planar shape of the shape from the small circle, as with the metal base 2 grinding the peripheral surface 4 is parallel to the peripheral edge of the annular disk shape, and the metal substrate 2 is polished surface of the peripheral edge 4 of the peripheral edge of the angled shape of helical segments by selecting one or two. Figure 1A shows a group of two grains are small round shape of the case 5, 6, Figure 1B shows the first group 5 is a small round shape of the abrasive grains and the second abrasive sheet group 6 is a spiral shaped case, Figure 1C 5,6 shows two groups are abrasive annular disk shape of the occasion, Figure 1D shows two groups 5,6 are abrasive spiral slice shape of the occasion. By such a planar shape is formed as a particle group 5,6 3 along the surface of the metal substrate is a metal substrate 2 are arranged in the peripheral edge portion of the annular grinding surface 4 is formed, because the grinding and polishing cloth to remove cutting chips or aggregation easy to sanders slurry discharged outside, in addition you can adjust its discharge performance.

Furthermore, the particle group 5,6, as shown in Figure 6, the abrasive material 52 to maintain the resilient holding is formed by means of an adhesive agent 8 such as the holding of the holding of the abrasive material 52 is fixed to the adjustment mechanism 7 in abutment surface 7a of the particle group is fixed to the base 5, 6, 7a.

Further, as the retaining material 52, although in the case of abrasive grains of diamond, the diamond abrasive grains can be used and reaction sintering of silicon and / or silicon alloy, but as long as is appropriate for the holding of the holding abrasive material is not particularly limited, by the General solder plating of nickel or the engagement means is also possible to maintain.

Next, by the polishing cloth on a polishing device 1 is polished polishing cloth will be described by means of the adjusting mechanism 7, the interaction of adjacent first and second abrasive grains group 5 group 6 of the plane S1 , S2, the setting of the metal substrate surface 3 of a certain height difference between the δ, grinding the polishing cloth. In other words, by means of the adjusting mechanism 7 is adjusted to the first reference surface S1 group 5 abrasive than one group 6 of the second reference surface S2 abrasive grains of the metal substrate surface 3 of the height of the vertical direction of a higher amount δ, by appropriately adjusting the polished surface 4 of the state of the polishing cloth can be polished surface of the workpiece to accommodate desired state.

If like polishing cloth with the polishing device 1, since by adjusting the plane S1, S2 of the level difference δ, the polished surface 4 can be adjusted to the state can play the desired polishing performance, not even in the polished surface 4 individual differences generated in a state, it can cause a uniform polishing cloth surface, and can be imparted to the surface of the polishing cloth to adapt appropriate workpiece polishing performance.

Figure 2A ~ 2D illustrate the present invention with a sander polishing abrasive cloth cluster shape different from the first configuration of Embodiment 2. In the present embodiment, the polishing with a polishing cloth 10, the plurality of abrasive grains group by only 5 of the first particle group composed of grains by the same group of 5 of these metal substrate 2 along the periphery of the ring juxtaposed shaped to form a polished surface 4.

In addition, the particle group 5 planar shape, as in the first embodiment, same as the case may be from the small round shape, as with the polished surface 4 of the periphery of parallel annular disk shape, and the polished surface 4 of the peripheral edge into a certain angle choosing the shape of a spiral slice. Figure 2A shows a group 5 are all small round grain shape of the occasion, Figure 2B shows the shape of the particle group and the screw 5 is a small round sheet shape having the planar shape of the two different abrasive grains in the polishing surface 4 group 5 alternately in the circumferential direction of the case of juxtaposed, Fig 2C shows particle group in the shape of the annular plate 5 are all occasions, Figure 2D shows the particle group in the shape of helical segments 5 are all occasions.

On adjusting mechanism 7 includes other structures, as in the first embodiment together, so description is omitted here to avoid repetition.

Further, in the above-mentioned polishing cloth 10 is polished with a polishing cloth, as shown in Figure 4, by means of adjustment mechanism 7 adjacent to each other in a group 5 of the abrasive grains set between plane S1 of the metal substrate surface 3 of a certain The height difference δ and sanding.

If like polishing cloth with the polishing device 10, then polished with the polishing cloth 1 is also used because it can properly adjust the particle group in the adjacent plane 5 of the height difference δ between S1 and the state of the polished surface 4 Adjust to the desired state, so even if the polishing surface 4 not only the state of individual differences generated, it can cause a uniform polishing cloth surface, and can be imparted to the surface of the polishing cloth to adapt appropriate workpiece polishing performance.

Furthermore, the adjusting mechanism 7 itself a configuration, not limited to the constitution described above with reference to FIG, may be used within the scope of the fine particle group in the plane vertical to adjust the various institutions.

The following detail together with comparative examples illustrate the present invention and a polishing cloth with grinding sander polishing using the polishing cloth of the embodiment of the method. However, the present invention is not limited in any way to these examples.

[First Embodiment] As the first abrasive grain group 5, respectively, with the equivalent particle size # 120 / # 140 is in the range of 150 ~ 170μm diamond abrasive grains 50, and the equivalent size # 325 / # 400 in the range of 55 ~ 65μm diamond abrasive grains 51, 50, 51 through the holding these grains reaction sintering material 52, 50, 51 to obtain abrasive grain held by the holding material 52 is a sintered body.

At this time, the two abrasive grains 50 and 51 are arranged in the surface are arranged, including particle size # 120 / # 140 abrasive grains 50 of the front of the plane, containing the grain size # 325 / # 400 abrasive grains 51 steps of the plane of the front end 40 ~ 60μm, and the abrasive grains 50 and 51 adjacent to each other resulting in a minimum grid of equilateral triangles, size # 120 / # 140 abrasive grains 50 are spaced at equal intervals of 2.0mm, size # 325 / # 400 abrasive grains 51 is spaced intervals of 0.4mm.

On the other hand, as the second group 6 of the abrasive grains, with a grain size equivalent to # 60 / # 80 is in the range of 250 ~ 320μm diamond abrasive grains, these are arranged at equal intervals of 0.8mm.

Moreover, in the thus obtained sintered body made by machining, processed into a predetermined size and shape, forming the first and second abrasive grains group 5 group 6, the particle group in these rings were used 5,6 epoxy resin bonded to the adjustment mechanism 7 in φ100mm diameter stainless steel SUS316L of the base 7a. Furthermore, in the present embodiment, the above first and second abrasive grains group 5 group 6 is taken as the planar shape as shown in FIG 1D polished surface 4 and the peripheral edge of the angled shape of the helical segments.

The polishing cloth made of such a sander to 19.6kPa pressure to 100rpm rotational pressure in the polyurethane foam polishing cloth, with that polishing cloth 80rpm same direction while rotating each of the supply side of the wet silica containing 25ml polishing slurry (卉 yeah Robotics system SS-25) while grinding polishing cloth.

In this case, the adjusting mechanism 7 by means of the above 4 on the polished surface of said first abrasive grains group 5 end of the second reference surface S1 and the abrasive grains group 6 S2 plane end surface of the metal substrate 3, adjusted to a height difference δ three levels 15,30,60 μm, measured speed grinding (polishing cloth wear rate) and the surface state of the polishing cloth (polishing cloth surface roughness), the measurement of the wafer with the polishing cloth polishing rate These measurement results are shown in Table 1.

Furthermore, in this embodiment, n is the number of samples taken to the number 20 is calculated in each sample measured in the measurement values of each of the above average (Ave) are shown in Table 1. And σn-1 in Table 1 represents the standard deviation of each measurement. In addition, the polishing rate of the wafer was measured using wafer ADE Corporation flatness measuring device (ultragage 9800), based on the measurement results before and after polishing calculating the average polishing rate.

Table 1

Image not available. View PDF

Shown in table 1 of this, if the present invention is a polishing cloth with sanders, can be adjusted in a plurality of abrasive particle group of the particle group of the particle size or height difference between the reference plane δ, the polished surface adjustment into the desired state, in particular, to ascertain the particle group by the above difference in height between the reference surface speed big difference δ wear. Thus, with a polishing cloth with sander can perform stable under various conditions of grinding polishing cloth.

Also, if the measured value of the above embodiment, the description of the following measured value is compared to the comparative example to improve the overall performance, and because the standard deviation of various measured values are very small, the resulting discrete small polishing cloth can be used for polishing cloth polishing sander gives stable performance.

[Comparative Example 1] corresponds with the size # 80 / # 100 is in the range of 210 ~ 250μm diamond abrasive grains with abrasive grains spaced intervals of 0.25mm arranged in such state, the use made of the nickel plating is fixed a prior art CMP polishing cloth sanders, as in the first embodiment under the same conditions grinding polyurethane foam polishing cloth. The grinding results are shown in Table 2.

[Comparative Example 2] corresponds with the size # 120 / # 140 is in the range of 150 ~ 170μm diamond abrasive grains, the grains are arranged in the surface of the sintered body, arranged adjacent to each other, a first result of the smallest grains lattice is a regular triangle, the side of the abrasive grains to become 2.1mm equally spaced intervals, using the composition of the abrasive grains group planar shape, as shown in Figure 2C, and polishing the surface parallel to the peripheral edge of the annular disk the shape of the polishing cloth with sanders, as in the first embodiment under the same conditions grinding polyurethane foam polishing cloth. Results grinding it together with the first comparative example are shown in Table 2.

[Comparative Example 3] with the first embodiment of the first particle group in the same, respectively, with equal size # 120 / # 140 is in the range of 150 ~ 170μm diamond abrasive grains, and grain size equivalent to # 325 / # 400 The range is 55 ~ 65μm diamond abrasive grains. Moreover, these abrasive particles, abrasive particles are arranged in the surface of the sintered body containing Granularity arranged 120 / # 140 abrasive front of the plane, containing the grain size # 325 / # 400 in the plane of the front end abrasive steps a 40 ~ 60μm, and the abrasive grains adjacent to each other resulting in a minimum grid of equilateral triangles, size # 120 / # 140 abrasive interval of intervals of 2.0mm, size # 325 / # 400 grit intervals of 0.4 mm in intervals. Moreover, with the arrangement of abrasive grains having such a group is formed as shown in Figure 2C and polished surface parallel to the periphery of annular disk shape, polished surface of the metal substrate along the periphery of which are arranged annularly The polishing cloth with a grinding device, as in the first embodiment under the same conditions grinding polyurethane foam polishing cloth. Results grinding it together with the first and second comparative examples are shown in Table 2.

Table 2

Image not available. View PDF

REFERENCED BY

Citing Patent	Filing date	Publication date	Applicant	Title
US8414362	Mar 2, 2010	Apr 9, 2013	Chien-Min Sung	Methods of bonding superabrasive particles in an organic matrix

CLASSIFICATIONS

International Classification	B24B53/02, B24B53/017, B24D13/14, B24D7/06, H01L21/304, B24B53/12, B24D3/10
Cooperative Classification	B24D7/06, B24B53/017
European Classification	B24B53/017, B24D7/06

LEGAL EVENTS

Date	Code	Event	Description
Nov 14, 2012	C17	Cessation of patent right	
Sep 13, 2006	C14	Granted	
Jul 14, 2004	C10	Request of examination as to substance	
May 12, 2004	C06	Publication	

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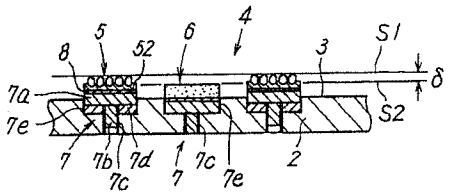
代理人 韩登营

权利要求书 2 页 说明书 12 页 附图 4 页

[54] 发明名称 抛光布用打磨器和使用该打磨器的抛光布的打磨方法

[57] 摘要

本发明提供一种抛光布用打磨器和使用该打磨器的抛光布的打磨方法，使可调整抛光布用打磨器中的打磨面的状态，即使在磨粒的前端形状等打磨面的状态中产生个体差异，也可以造成均一的抛光布表面，而且能够对抛光布表面赋予适应被加工物的适当的抛光性能，上述打磨器构成为，在金属基体(2)的周缘部上备有打磨面(4)的抛光布用打磨器(1)中，在该打磨面(4)上分别交互地并列设置由彼此不同的粒度的磨粒组成的第 1、第 2 磨粒群(5)、(6)，在上述金属基体(2)上，设置能够任意调节这些各磨粒群(5、6)中包含的粒度最大的磨粒的前端的基准面(S1)、(S2)间的高低差(δ)的调节机构(7)。



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1. 一种抛光布用打磨器，是在能够旋转的金属基体的表面上形成打磨面而成的抛光布用打磨器，其特征在于，

在上述打磨面上，在其圆周方向上并列设置多个磨粒群，

在上述金属基体上，对全部或一部分磨粒群，设置能够调节由多个磨粒的前端分别形成的基准面的上述打磨面上的高低差的调节机构。

2. 如权利要求 1 中所述的抛光布用打磨器，其特征在于，有上述调节机构的磨粒群，分别固定于构成与上述金属基体分开形成的上述调节机构的基台上，沿着上述打磨面中的金属基体的周缘部各磨粒群环状地并列设置。

3. 如权利要求 1 中所述的抛光布用打磨器，其特征在于，上述磨粒群分别形成为从与上述打磨面的周缘平行的环形片形状、与上述打磨面的周缘成一定的角度的螺旋片形状、小圆形状之中选择的一种或两种的平面形状。

4. 如权利要求 3 中所述的抛光布用打磨器，其特征在于，上述磨粒群分别形成为从上述平面形状之中选择的两种平面形状，

这些平面形状不同的两种磨粒群在打磨面的圆周方向上交互地并列设置。

5. 如权利要求 1 或 2 中所述的抛光布用打磨器，其特征在于，并列设置在打磨面上的各磨粒群是由彼此同一粒度的磨粒所形成的磨粒群，或者，由不同的粒度的磨粒所形成的两种磨粒群来构成。

6. 如权利要求 5 中所述的抛光布用打磨器，其特征在于，并列设置在打磨面上的磨粒群，由彼此不同的粒度的磨粒所形成的第 1 磨粒群和第 2 磨粒群来构成，

这些第 1 磨粒群和第 2 磨粒群在打磨面的圆周方向上交互地并列设置。

7. 如权利要求 6 中所述的抛光布用打磨器，其特征在于，上述

Ex. 1003, Page 169

第 1 磨粒群由同一粒度的磨粒或者两种粒度的磨粒来形成。

8. 如权利要求 1 或 2 中所述的抛光布用打磨器，其特征在于，在各磨粒群中，各磨粒颗粒在前述打磨面上二维地具有规律性地排列，相互邻接的磨粒颗粒形成的最小格子为正三角形或者平行四边形地排列。

9. 一种抛光布的打磨方法，是用权利要求 1～8 中所述的任何一项抛光布用打磨器打磨抛光布的方法，其特征在于，

借助上述调节机构，在相互邻接的磨粒群的上述基准面间设置一定的高低差，打磨上述抛光布。

10. 一种抛光布的打磨方法，是用权利要求 6～8 中所述的任何一项抛光布用打磨器打磨抛光布的方法，其特征在于，

借助上述调节机构，上述打磨面中的第 1 磨粒群的基准面的高度调节成比第 2 磨粒群的基准面的高度高出一定量，打磨上述抛光布。

Ex. 1003, Page 170

抛光布用打磨器和使用该打磨器的抛光布的打磨方法

技术领域

本发明涉及在机械的化学抛光（Chemical Mechanical Polishing；以下简称 CMP）中在进行抛光布的布网孔堵塞或异物去除之际使用的抛光布用打磨器和用它的抛光布的打磨方法。

背景技术

一般来说在硅晶片基板上制造半导体硅晶片基板或集成电路等微细电子电路的过程中，出于去除存在于基板表面上的凹凸或结晶缺陷的目的用 CMP 加工。在此一 CMP 加工中，一边以规定的载荷把晶片基板压住粘贴于抛光装置的固定盘的由泡沫聚氨酯构成的抛光布上，一边供给称为浆液的抛光液，使晶片与抛光布两方旋转而进行抛光。

作为上述 CMP 加工中的浆液，用使氧化铁、碳酸钡、氧化铈、氧化铝、胶态二氧化硅等的抛光颗粒悬浮于氢氧化钾、稀盐酸、稀硝酸、过氧化氢水溶液、硝酸铁等抛光液中的浆液，这些是根据抛光速度和晶片或晶片基板上的前述被加工物的种类适当选择。

此一 CMP 加工，为了用同一抛光布多张或者多次重复地抛光晶片，随着 CMP 加工的次数的增大，所去除的被加工物的碎屑或凝集的抛光颗粒进入抛光布的微细的孔而引起堵塞，晶片的抛光速度降低。因此有必要经常或者定期地进行去除布网孔堵塞后的抛光布的表层，再生抛光布表面的面粗糙性而恢复抛光速度的被称为打磨的作业，在此一作业中使用被称为抛光布用打磨器的工具。

因为金刚石磨粒在抛光布的打磨中是优良的材料，故研究了利用金刚石磨粒的抛光布用打磨器，通过镀镍在不锈钢上电沉积金刚石磨粒的方法一般被实用化了。除此以外提出了借助金属钎料把金刚

Ex. 1003, Page 171

石磨粒钎焊于不锈钢上的方法（例如，参照特开平 10－012579 号公报），通过烧结使金刚石磨粒与保持料反应烧结而固定的方法（例如，参照特开 2001－179638 号公报）。此外出于得到稳定的抛光布的表层去除能力的目的，还提出了均等间隔地排列磨粒的 CMP 抛光布打磨器(例如,参照特开 2000－141204 号公报、特开 2002－127017 号公报）。

可是，在上述这种现有技术中的抛光布用打磨器中，因为在其结构上，无法避免因磨粒的前端形状等在打磨面的方式上产生个体差异，故即使使用同一抛光布用打磨器也难以造成均一的抛光布表面，此外，抛光布的表面状态有必要根据被加工物进行调整，例如，对在硅晶片表面形成层间氧化膜的晶片，通过抛光布的表面状态成为粗糙状态，抛光布引起的机械去除加工的因素增强而抛光速度提高，此外，对于 Cu 配线，出于使存在于抛光布表面的抛光液（浆液）的化学反应引起的因素比抛光布引起的机械去除加工的因素更强的目的，有必要维持某种程度的一定的抛光布的表面粗糙度，因此，关于打磨该抛光布的表面的抛光布用打磨器也是，有必要准备需要数量的、打磨面的状态适应上述被加工物的，存在着成本提高这样的问题。

发明内容

本发明将要解决的技术课题,在于提供一种通过能够调整抛光布用打磨器中的打磨面的状态，即使在磨粒的前端形状等打磨面的状态中产生个体差异，也可以通过该打磨面的调整造成均一的抛光布表面，或者，能够对抛光布表面赋予适应被加工物的适当的抛光性能的抛光布用打磨器和用它的抛光布的打磨方法。

为了解决上述课题,本发明的抛光布用打磨器是在能够旋转的金属基体的表面上形成打磨面而成的抛光布用打磨器，其特征在于，在上述打磨面上，在其圆周方向上并列设置多个磨粒群，在上述金属基体上设有对全部或一部分磨粒群，能够调节由多个磨粒的前端

Ex. 1003, Page 172

分别形成的基准面的上述打磨面中的高低差的调节机构。

如果用上述抛光布用打磨器，则由于借助上述调节机构可以任意调节多个磨粒群中的上述各基准面的高低差，所以即使在磨粒的前端形状等打磨面的状态中产生个体差异也可以通过调节上述多个磨粒群中的基准面的高低差，调整打磨面的状态，造成均一的抛光布表面，或者，可以对抛光布表面即抛光布的抛光面，赋予适应被加工物的适当的抛光性能。在此一场合，基准面高的磨粒群主要用于抛光布表面的磨耗，基准面低的磨粒群主要用于抛光布的表面粗糙度的调整。

在上述抛光布用打磨器中，也可以取为有上述调节机构的磨粒群，分别固定于与上述金属基体分开形成的构成上述调节机构的基台上，沿着上述打磨面中的金属基体的周缘部各磨粒群环状地并列设置。

如果上述磨粒群分别以从与上述打磨面的周缘平行的环状片形状、与上述打磨面的周缘成一定的角度的螺旋片形状、小圆形状之中选择的一种或两种的平面形状形成，则最好由打磨所去除的抛光布的切削屑或凝集的浆液容易向外部排出。

在上述磨粒群分别形成为从上述平面形状之中选择的两种平面形状的场合，这些平面形状不同的两种磨粒群在打磨面的圆周方向上交互地并列设置是适当的。

此外，并列设置在打磨面上的各磨粒群也可以由彼此同一粒度的磨粒所形成的同一的磨粒群，或者，由不同的粒度的磨粒所形成的两种磨粒群来构成。

而且，在并列设置在上述打磨面上的磨粒群，由彼此不同的粒度的磨粒所形成的第 1 磨粒群和第 2 磨粒群来构成的场合，把这些第 1 磨粒群和第 2 磨粒群在打磨面的圆周方向上交互地并列设置是适当的。在此一场合，可以由同一粒度的磨粒或者不同的两种粒度的磨粒来形成上述第 1 磨粒群。

这里，虽然在上述各磨粒群中，磨粒颗粒在前述打磨面上二维地

Ex. 1003, Page 173

具有规律性地排列，相互邻接的磨粒颗粒造成的最小格子如果成为正三角形或者平行四边形地排列，则可以进一步提高打磨的稳定性和均匀性。

进而，上述课题可以通过靠上述调节机构，在相互邻接的磨粒群的上述基准面间设置一定的高低差，打磨上述抛光布的抛光布的打磨方法来解决。

在上述抛光布的打磨方法中，在多个磨粒群由上述第 1 磨粒群和第 2 磨粒群来构成的场合，借助上述调节机构，上述打磨面中的第 1 磨粒群的基准面的高度调节成比第 2 磨粒群的基准面的高度高出一定量，打磨上述抛光布是合适的。

如果用这种本发明的抛光布用打磨器和用它的抛光布的打磨方法，则由于可以借助调节机构任意调节在各磨粒群中由磨粒的前端分别形成的磨粒基准面的上述打磨面上的高低差，所以即使磨粒的前端形状等打磨面的状态中产生个体差异，也可以造成均一的抛光布表面，而且，对抛光布表面也可以赋予适应被加工物的适当的抛光性能。

附图说明

图 1A～图 1D 是表示使本发明的抛光布用打磨器的磨粒群的配置形状不同的第 1 实施方式的立体图。

图 2A～图 2D 是表示使本发明的抛光布用打磨器的磨粒群的配置形状不同的第 2 实施方式的立体图。

图 3 是图 1A 中的 III－III 剖视图。

图 4 是图 2A 中的 IV－IV 剖视图。

图 5 是表示第 1 磨粒群 5 中的磨粒的排列状态的示意图。

图 6 是图 3 中的局部放大图。

具体实施方式

图 1A～图 1D 示出本发明的抛光布用打磨器的磨粒群的配置形状

Ex. 1003, Page 174

不同的第 1 实施方式。抛光布用打磨器 1 由在其表侧的中心有圆形的凹部 2a，和在其周围的金属基体表面 3 上形成环状的打磨面 4 而成的圆盘状的金属基体 2 来构成，在上述打磨面 4 上，分别环状地并列设置多个沿着其圆周方向独立的第 1 和第 2 磨粒群 5、6。换句话说，沿着金属基体表面 3 即上述金属基体 2 的周缘部环状地并列设置多个磨粒群 5、6 形成上述打磨面 4。

而且，如图 3 中所示，在上述金属基体 1 上设有调节机构 7，以便在各磨粒群 5、6 中以包含粒度最大的磨粒的前端的平面为基准面 S1、S2 时，可以任意调节在打磨面 4 上，上述多个磨粒群 5、6 中的各基准面 S1、S2 间相互的高低差 δ。

这里，没有必要一定在上述金属基体 2 上设置上述凹部 2a。

具体地说，上述调节机构 7 由基台 7a、调节用螺栓 7b 和垫片 7d 构成，其中，基台 7a 的表面上固定磨粒群 5 或 6，上下滑动自如地嵌入开口于上述金属基体 2 的表面 3 的凹部 7e; 调节用螺栓 7b 设在该基台 7a 的背面，螺纹配合于从上述金属基体 2 的凹部 7e 的底面向该金属基体 2 的背面侧贯通的螺纹孔 7c; 当调节固定于上述基台 7a 的磨粒群 5、6 处的基准面的高度位置时，垫片 7d 夹在上述基台 7a 的背面与上述凹部 7b 的底面之间。而且，分别固定于上述调节机构 7 的基台 7a 的表面上的多个磨粒群 5、6 沿着上述金属基体 2 的周缘部环状地并列设置，形成上述打磨面 4。

因而，为要调节上述磨粒群 5、6 的基准面 S1、S2 的高度位置把具有所需厚度的垫片 7d 放入上述金属基体 2 的凹部 7e 内，通过借助上述调节用螺栓 7b 夹持于该凹部 7e 的底面与上述基台 7a 的背面之间，可以调节上述各磨粒群 5、6 处的基准面 S1、S2 对金属基体表面 3 的高度位置，即多个磨粒群 5、6 处的各基准面 S1、S2 间相互的高低差 δ，可以调整打磨面 4 的状态。再者，在本实施方式中，借助调节机构 7，上述基准面 S1、S2 对金属基体表面 3 的高度可以在 0～300 μm 的范围内调节。

上述磨粒群当中，基准面高的磨粒群主要用于抛光布的磨耗，基

Ex. 1003, Page 175

准面低的磨粒群主要用于抛光布的表面粗糙度的调整。即，因为通过加在打磨器总体上的载荷集中地作用于基准面高的磨粒群，每单位面积的负载载荷增大，故它们主要用于抛光布的磨耗，另一方面，基准面低的磨粒群负担载荷到某种程度，使上述基准面高的磨粒群引起的抛光布的磨耗速度降低。再者，在基准面间相互的高低差δ变得极大的场合，因为成为基准面低的磨粒群完全不负担载荷，故成为它们不发挥使抛光布的磨耗速度降低的效果。此一基准面低的磨粒群还在抛光布的切削屑的排出中有效地起作用。

此外，在抛光布用打磨器 1 中，上述多个磨粒群 5、6 由彼此粒度不同的磨粒所形成的第 1 磨粒群 5 与第 2 磨粒群 6 来构成，这两种磨粒群 5、6 沿着上述金属基体 2 的周缘部在其圆周方向上交互地并列设置形成打磨面 4。

具体地说，上述第 1 磨粒群 5，如图 5、图 6 中所示，由大小两种粒度的磨粒 50、51 来形成，这些磨粒 50、51，如图 5 中所示，在基台 7a 的表面上即前述打磨面 4 上二维地有规则性地均等地排列，相互邻接的磨粒颗粒彼此造成的最小格子成为正三角形或者平行四边形地排列着。再者大的磨粒颗粒 50 彼此的间隔比小的磨粒颗粒 51 彼此的间隔要宽。

通过像这样把大小两种粒度的磨粒有规则性地均等地排列，可以进一步提高打磨的稳定性和均匀性。

另一方面，在上述第 2 磨粒群 6 中，虽然也可以以粒度与上述第 1 磨粒群 5 的磨粒不同的同一粒度的磨粒或者多种粒度的磨粒这两种形式中的任何一种来形成，但是最好是与上述第 1 磨粒群 5 同样，磨粒二维地有规则性地均等地排列。

这里，作为形成上述磨粒群 5、6 的磨粒，可以用例如金刚石磨粒，该磨粒的粒度，一般来说，最好是 JIS B 4130 中规定的#325／#400～#60／#80 的范围。

此外，形成上述第 1 磨粒群 5 的磨粒，没有必要一定如上所述是大小两种粒度的磨粒 50、51，也可以是同一粒度的磨粒（参照图 2A～

Ex. 1003, Page 176

图 2D 的实施方式）。此外，上述调节机构 7 没有必要一定在第 1 磨粒群 5 和第 2 磨粒群 6 的两方中设置，也可以仅在磨粒群的一部分，即第 1 磨粒群 5 或第 2 磨粒群 6 的一方中设置。在此一场合，未设置调节机构 7 的磨粒群，基台 7a 直接固定于金属基体 2 的周缘部。

这样一来，如果每个磨粒群用不同粒度的磨粒，或者在各磨粒群中使不同粒度的磨粒混合，大粒度的磨粒主要用于抛光布的磨耗，小粒度的磨粒主要用于抛光布的表面粗糙度的调整，通过它们的粒度的调整，可以把抛光布的表面粗糙度弄成适合特定对象物的 CMP 加工的状态。

进而，如图 1A～图 1D 中所示，在抛光布用打磨器 1 中，上述第 1 磨粒群 5 和第 2 磨粒群 6 的平面形状可以从小圆形状、成为与上述金属基体 2 的周缘即打磨面 4 的周缘平行的环形片形状、与上述金属基体 2 的周缘即打磨面 4 的周缘成一定角度的螺旋片形状之中选择一种或两种。图 1A 示出两个磨粒群 5、6 都是小圆形状的场合，图 1B 示出第 1 磨粒群 5 是小圆形状而第 2 磨粒群 6 是螺旋片形状的场合，图 1C 示出两个磨粒群 5、6 都是环形片形状的场合，图 1D 示出两个磨粒群 5、6 都是螺旋片形状的场合。通过把形成为这种平面形状的磨粒群 5、6 沿着金属基体表面 3 即金属基体 2 的周缘部排列成环状，形成打磨面 4，因打磨而去除的抛光布的切削屑或凝集的浆液容易向打磨器外部排出，此外可以调整其排出性能。

再者，上述磨粒群 5、6，如图 3、图 6 中所示，磨粒靠保持料 52 保持而形成，通过借助粘接剂 8 等把保持该磨粒的保持料 52 固定于上述调节机构 7 中的基台 7a 的表面，该磨粒群 5、6 固定于上述基台 7a。

此外，作为上述保持料 52，虽然在磨粒为金刚石的场合，可以利用和金刚石磨粒反应烧结的硅和／或硅合金，但是只要是适合于磨粒的保持的保持料没有特别限制，靠一般的电镀镍或借助钎料的接合来保持也是可能的。

接下来就对由上述抛光布用打磨器 1 进行的抛光布的打磨方法进行说明，借助上述调节机构 7，在交互邻接的第 1 磨粒群 5 和第 2

Ex. 1003, Page 177

磨粒群 6 的各基准面 S1、S2 间，设定对金属基体表面 3 的一定的高低差 δ，打磨上述抛光布。换句话说，借助上述调节机构 7，调节成上述第 1 磨粒群 5 的基准面 S1 一方比上述第 2 磨粒群 6 的基准面 S2 上述金属基体表面 3 的垂直方向的高度高出一定量 δ，通过适当调整上述打磨面 4 的状态，可以把上述抛光布表面打磨成适应被加工物的想要的状态。

像这样如果用上述抛光布用打磨器 1，则因为通过调节基准面 S1、S2 的高低差 δ，可以把打磨面 4 的状态调节成可以发挥想要的抛光性能，故不仅即使在打磨面 4 的状态中产生个体差异，也可以造成均一的抛光布表面，而且可对抛光布表面赋予适应被加工物的适当的抛光性能。

图 2A～图 2D 示出本发明的抛光布用打磨器的磨粒群的配置形状不同的第 2 实施方式。在本实施方式的抛光布用打磨器 10 中，多个磨粒群仅由上述第 1 磨粒群 5 组成，通过把这些同一的磨粒群 5 沿着金属基体 2 的周缘部并列设置成环状而形成打磨面 4。

此外，该磨粒群 5 的平面形状，与上述第 1 实施方式的场合同样，可以从小圆形状、成为与上述打磨面 4 的周缘平行的环形片形状、与上述打磨面 4 的周缘成一定角度的螺旋片形状之中选择。图 2A 示出磨粒群 5 全都是小圆形状的场合，图 2B 示出磨粒群 5 为小圆形状和螺旋片形状，具有这两种不同的平面形状的磨粒群 5 在打磨面 4 的圆周方向上交互地并列设置的场合，图 2C 示出磨粒群 5 全都是环形片形状的场合，图 2D 示出磨粒群 5 全都是螺旋片形状的场合。

关于包含调节机构 7 的其他构成，因为与上述第 1 实施方式共同，所以为了避免重复这里省略说明。

而且，在用上述抛光布用打磨器 10 打磨抛光布时，如图 4 中所示，借助调节机构 7 在相互邻接的上述磨粒群 5 的基准面 S1 间设定对金属基体表面 3 的一定的高低差 δ 并进行打磨。

像这样如果用上述抛光布用打磨器 10，则与上述抛光布用打磨器 1 同样，因为可以适当调节上述邻接的磨粒群 5 的基准面 S1 间的

Ex. 1003, Page 178

高低差δ而把打磨面 4 的状态调整成想要的状态，故不仅即使在打磨面 4 的状态中产生个体差异，也可以造成均一的抛光布表面，而且可对抛光布表面赋予适应被加工物的适当的抛光性能。

再者，上述调节机构 7 本身的构成，不限于参照图说明的上述构成，可以采用在微细的范围内可以上下调整磨粒群的基准面的各种机构。

以下与比较例一起具体地示出本发明的抛光布用打磨器和使用该打磨器的抛光布的打磨方法的实施例。但是，本发明不受这些实施例的任何限定。

〔第 1 实施例〕

作为第 1 磨粒群 5 的磨粒，分别用相当于粒度#120／#140 的处于 150～170 μm 的范围的金刚石磨粒 50，与相当于粒度#325／#400 的处于 55～65 μm 的范围的金刚石磨粒 51，通过把这些磨粒 50、51 与保持料 52 反应烧结，得到磨粒 50、51 被保持于保持料 52 的烧结体。

此时，把上述两种磨粒 50、51 在磨粒排列面上排列成，包含粒度#120／#140 的磨粒 50 的前端的平面，与包含粒度#325／#400 的磨粒 51 的前端的平面的级差为 40～60 μm，并且邻接的磨粒 50、51 彼此造成的最小格子为正三角形，粒度#120／#140 的磨粒 50 的间隔为 2.0 mm 的等间隔，粒度#325／#400 的磨粒 51 的间隔为 0.4 mm 的等间隔。

另一方面，作为第 2 磨粒群 6 的磨粒，用相当于粒度#60／#80 的处于 250～320 μm 范围的金刚石磨粒，把这些排列成 0.8 mm 的等间隔。

而且，在把这样做所得到的烧结体通过机械加工，加工成规定的尺寸·形状，形成第 1 磨粒群 5 和第 2 磨粒群 6 后，把这些磨粒群 5、6 分别用环氧树脂粘接于调节机构 7 中的 SUS316L 不锈钢制的直径φ100 mm 的基台 7a 上。再者，在本实施例中，把上述第 1 磨粒群 5 和第 2 磨粒群 6 的平面形状取为图 1D 中所示的与打磨面 4 的周缘成

Ex. 1003, Page 179

一定角度的螺旋片形状。

把这样做成的抛光布用打磨器以 19.6kPa 的压力压在以 100 rpm 旋转的泡沫聚氨酯制抛光布上，与该抛光布同一方向以 80 rpm 旋转，同时一边供给每分 25 ml 包含湿二氧化硅的抛光浆液（キャボット制 SS－25）一边进行抛光布的研磨。

此时，借助上述调节机构 7 把前述打磨面 4 上的前述第 1 磨粒群 5 的磨粒前端基准面 S1 与前述第 2 磨粒群 6 的磨粒前端基准面 S2 对金属基体表面 3 的高低差 δ 调节成 15、30、60μm 三个水准，测定打磨速度（抛光布的磨耗速度）与抛光布的表面状态（抛光布的表面粗糙度）后，测定用该抛光布的晶片的抛光速度，这些测定结果示于表 1。

再者，在本实施例中，n 数即取样数取为 20，算出在各试样中所测定的上述各测定值的平均值（Ave）示于表 1。而且表 1 中 σ_{n-1} 表示各测定结果的标准偏差。此外，在晶片的抛光速度的测定中用 ADE 公司制的晶片平坦度测定器（ultragage 9800），根据抛光前后的测定结果算出平均抛光速度。

表 1

			实施例		
磨粒间隔	第 1 磨粒群	#120／#140	2.0 mm		
		#320／#400	0.4 mm		
	第 2 磨粒群	#60／#80	0.8 mm		
磨粒基准面的高低差 δ（μm）			15	30	60
抛光布的磨耗速度（μm／Hour）		Ave.	69.2	108	122
		σn－1	1.33	1.25	1.78
抛光布的表面粗糙度 Ra（μm）		Ave.	4.12	4.05	4.19
		σn－1	0.12	0.15	0.14
晶片抛光速度（nm／min）		Ave.	126	129	121
		σn－1	6.7	7.2	5.6
划痕发生率（%）			0.00	0.00	0.00

Ex. 1003, Page 180

如此一表 1 中所示，如果用本发明的抛光布用打磨器，则可以调节多个磨粒群中的磨粒的粒度或各磨粒群基准面间的高低差 δ，把打磨面调整成想要的状态，特别是，弄清了通过上述磨粒群的基准面的高低差 δ 磨耗速度有很大差别。因此，借助一个抛光布用打磨器可以在种种条件下进行稳定的抛光布的打磨。

此外，如果用上述实施例的测定值，则与以下说明的比较例的测定值相比总体上性能提高，而且因为各种测定值的标准偏差非常小，所以所得到的抛光布中离散极小，可以给抛光布用打磨器赋予稳定的抛光性能。

〔第 1 比较例〕

用把粒度相当于#80／#100 的处于 210～250 μm 的范围的金刚石磨粒以磨粒间隔 0.25 mm 的等间隔地排列的状态，使用由电镀镍固定而成的现有技术中的 CMP 抛光布打磨器，在与第 1 实施例同一条件下研磨泡沫聚氨酯制抛光布。其研磨结果示于表 2。

〔第 2 比较例〕

用把粒度相当于#120／#140 的处于 150～170μm 的范围的金刚石磨粒，在烧结体的磨粒排列面上，排列成邻接的第 1 磨粒彼此造成的最小格子为正三角形，其一边的磨粒间隔成为 2.1 mm 的等间隔，使用由该磨粒组成的磨粒群的平面形状为，如图 2C 中所示的，与打磨面的周缘平行的环形片形状的抛光布用打磨器，在与第 1 实施例同一条件下研磨泡沫聚氨酯制抛光布。其研磨结果与第 1 比较例一起示于表 2。

〔第 3 比较例〕

与第 1 实施例的第 1 磨粒群同样，分别用粒度相当于#120／#140 的处于 150～170 μm 的范围的金刚石磨粒，和粒度相当于#325／#400 的处于 55～65 μm 的范围的金刚石磨粒。而且，把这些磨粒，在烧结体的磨粒排列面上排列成包含粒度#120／#140 的磨粒的前端的平面，与包含粒度#325／#400 的磨粒的前端的平面的级差成为

Ex. 1003, Page 181

40～60 μm，并且邻接的磨粒彼此造成的最小格子为正三角形，粒度#120／#140 的磨粒的间隔为 2.0 mm 的等间隔，粒度#325／#400 的磨粒的间隔为 0.4 mm 的等间隔。而且，用具有这种磨粒排列的磨粒群，形成为图 2C 中所示的成为与打磨面的周缘平行的环形片形状，在打磨面中沿着金属基体的周缘部环状地排列而成的抛光布用打磨器，在与第 1 实施例同一条件下研磨泡沫聚氨酯制抛光布。其研磨结果与第 1、2 比较例一起示于表 2。

表 2

		第 1 比较例	第 2 比较例	第 3 比较例
金刚石粒度		#80／#100	#120／#140	①#120／#140 ②#325／#400
磨粒间隔		0.25 mm	2.1 mm	①2.1 mm ②0.4 mm
磨粒保持方法		电镀	烧结	
抛光布的磨耗速度（μm／Hour）	Ave.	61.6	189	138
	σn－1	7.84	14.6	2.43
抛光布的表面粗糙度 Ra（μm）	Ave.	3.01	3.32	3.45
	σn－1	0.36	0.36	0.13
晶片抛光速度（nm／min）	Ave.	105	98.3	120
	σn－1	35.2	25.3	9.6
划痕发生率（%）		0.52	0.00	0.00

Ex. 1003, Page 182

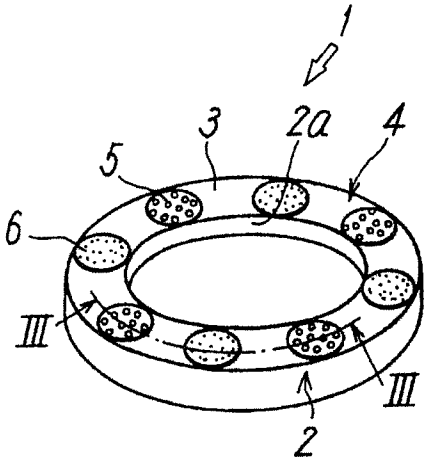


图 1A

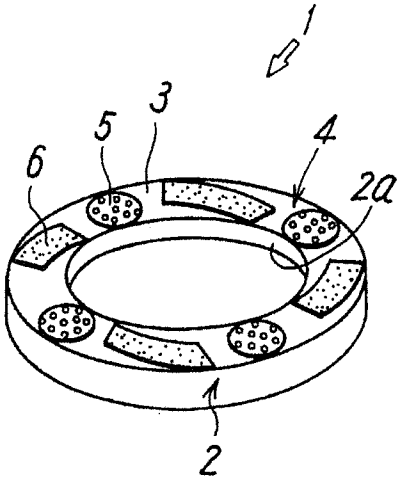


图 1B

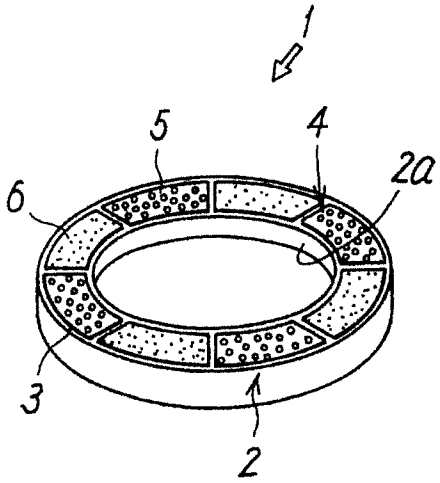


图 1C

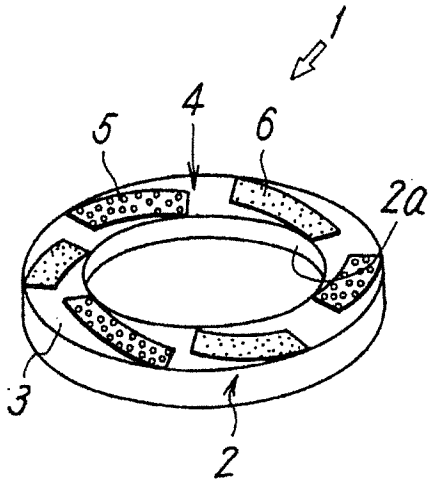


图 1D

Ex. 1003, Page 183

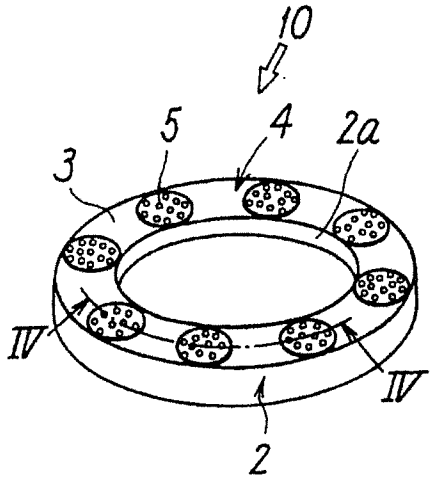


图 2A

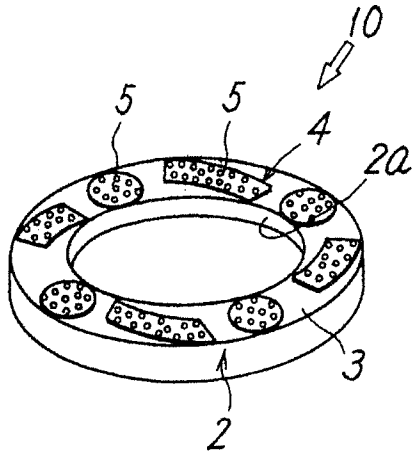


图 2B

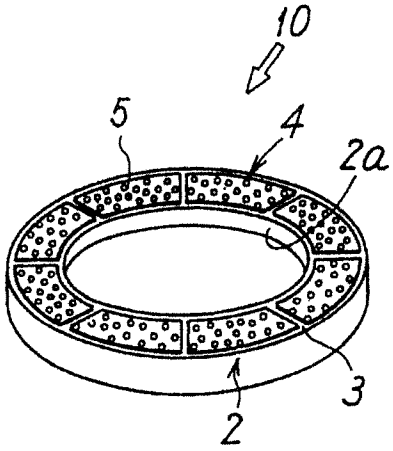


图 2C

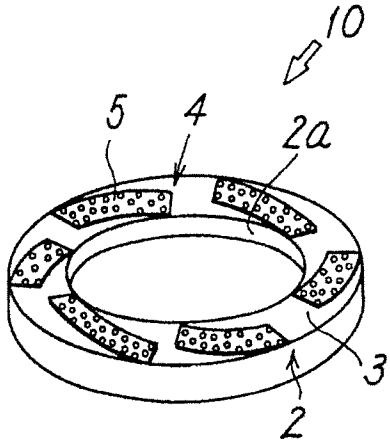


图 2D

Ex. 1003, Page 184

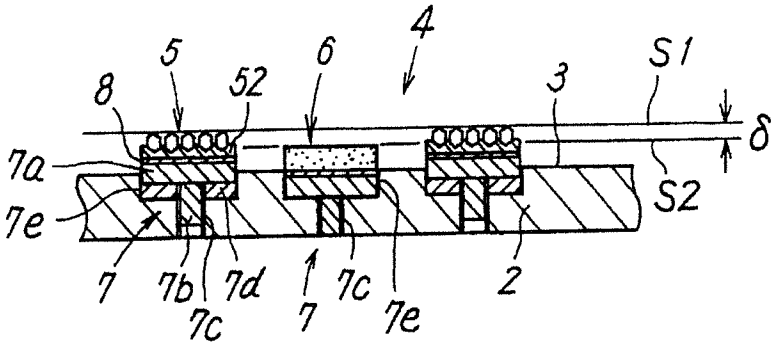


图 3

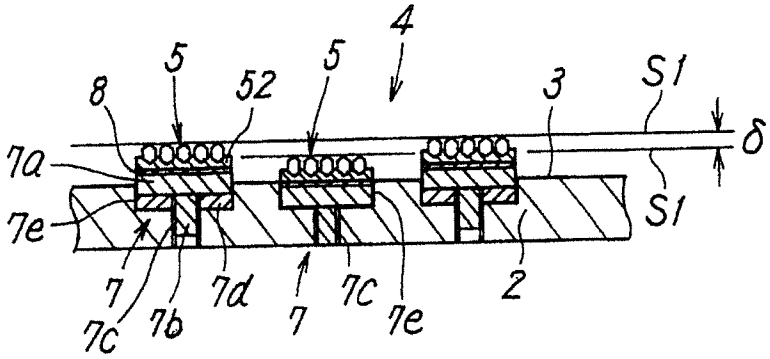


图 4

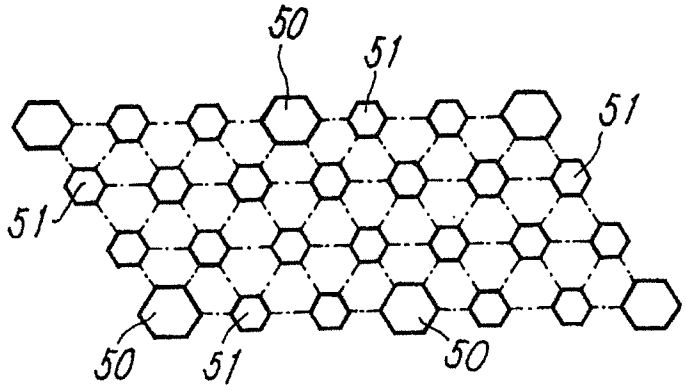


图 5

Ex. 1003, Page 185

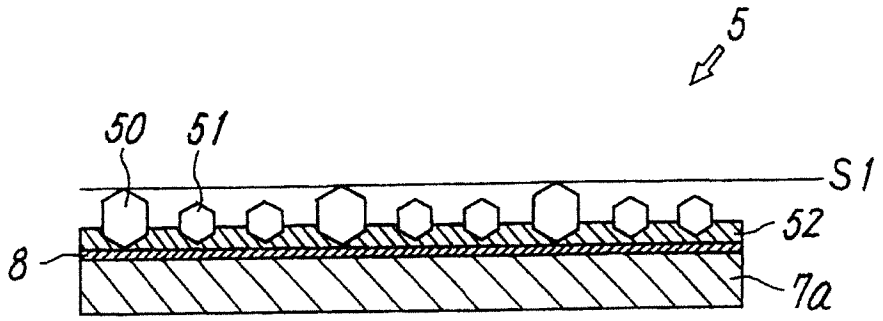


图 6

Ex. 1003, Page 186

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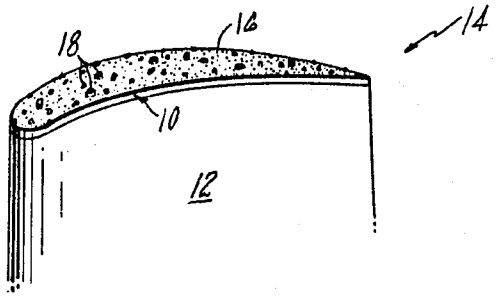
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(54) **Method for depositing a layer of abrasive material on a substrate.**

(57) Techniques are described for the formation of an abrasive surface layer (14) on an article (12). The surface layer (14) is characterized by a single layer of particles (18) which are evenly spaced apart, in a high density arrangement, within a metallic matrix (16). In the deposition of the particles (18) on the article (12), a vacuum is drawn through a perforated plate (42), wherein the location of each perforation (44) corresponds to the desired location of one particle (18) on the article surface (11). The vacuum holds one particle (18) over each perforation (44) in the plate (42), then the plate (42) is positioned over the article (12) and the vacuum level adjusted so that the particles (18) fall onto the article (12). The matrix (16) is then deposited by plasma arc spraying a superalloy powder. The invention is particularly suited to the formation of an abrasive layer on the tip surface of a blade used in a gas turbine engine.

FIG. 1



EP 0 238 434 A2

Description

Method for Depositing a Layer of Abrasive Material on a Substrate

Cross Reference

This invention is related to the copending and commonly assigned application "Abrasive Surface Coating Process for Superalloys", U.S. Serial No. 624,446, to Eaton et al, filed June 25, 1984.

Technical Field

The present invention relates to a method for depositing particulate material on a substrate. More specifically, it relates to a method for depositing a surface layer containing spaced apart abrasive particulate on the tip surface of a turbine engine component.

Background

Gas turbine engines and other turbomachines have rows of rotating blades contained within a generally cylindrical base. As the blades rotate, their tips move in close proximity to the case. To maximize engine operating efficiency, the leakage of the gas or other working fluid around the blade tips should be minimized. As has been known for some time, this may be achieved by blade and sealing systems in which the blade tips rub against a seal attached to the interior of the engine case. Generally, the blade tip is made to be harder and more abrasive than the seal; thus, the blade tips will cut into the seal during those portions of the engine operating cycle when they come into contact with each other.

One type of blade tip is described in U.S. Patent No. 4,249,913 to Johnson et al, entitled "Alumina Coated Silicon Carbide Abrasive", of common ownership herewith. In the Johnson et al invention, silicon carbide abrasive particles of 0.20-0.76 mm average diameter are coated with a metal oxide such as alumina and incorporated by powder metal techniques in nickel or cobalt base matrix alloys. A powder metal compact containing up to about 45 volume percent particulate may be made which is then bonded to the tip of the blade. The resulting abrasive blade tip is particularly well suited for rubbing metal as well as ceramic airseals; the latter type of seals have found wide use in modern gas turbine engines.

As described in greater detail in the copending and commonly assigned application "Abrasive Surface Coating Process for Superalloys" to Eaton et al, U.S. Serial No. 624,446, which is incorporated by reference, improved techniques for the fabrication of blade tips, relative to the type of methods described in Johnson et al, are desired. Specifically, the blade tip should be as thin as possible, yet still provide the required abrasive characteristics. The quantity of abrasive silicon carbide should be minimized, due to its high expense.

Components having thin layers of abrasive particles which are randomly distributed in a matrix material are known. For example, coated abrasives made from alumina, silica and silicon carbide are common products, as are metal bonded diamond and cubic boron nitride grinding tools. Such tools are often made by electrodeposition techniques. In U.S. Patent No. 4,227,703, such techniques are used to deposit an abrasive layer on a turbine blade tip. However, the limited composition of electrodeposited matrix alloys limits the usefulness of this method. Sprayed deposits containing metal and ceramic abrasives are also well known. See, e.g., commonly assigned U.S. Patent No. 4,386,112. However, such processes for spraying abrasive and matrix metal particles are inherently inefficient in that only a fraction of the sprayed material actually hits and adheres to the surface. These difficulties are especially significant in light of the relatively small size, e.g., about 6 by 50 mm, and curved shape of a typical turbine blade tip.

When an abrasive layer is provided on a superalloy turbine blade tip, its method of application must be metallurgically compatible with obtaining or maintaining the desired properties of the superalloy substrate. Since turbine blade alloys reflect a highly refined metallurgical art, there are limits on the techniques used with abrasive layer fabrication. Also, the abrasive layer is not a structural material and its weight imposes stresses on the blade substrate during use, i.e., when the blade rotates at high speed. Thus it is highly desirable that the minimum thickness abrasive layer be applied. Since blades are finished to length tolerances of 0.05 mm or less, this means that both the preparation of the substrate and the application of the abrasive layer must be carried out with high precision. All these considerations place severe restraints on the kinds of materials and processing techniques which are useful.

Disclosure of the Invention

An object of the invention is a method for providing a single layer of spaced apart particles on the surface of an article. Another object of the invention is to deposit such particles in a high concentration and uniform distribution on a turbine blade tip. Yet another object of the invention is to provide an abrasive layer having high temperature operating capability on the tip surface of a turbine blade.

According to the invention, a method for depositing a single layer of particles in a desired pattern on the surface of an article comprises the steps of (a) drawing a vacuum through a transfer tool having a plurality of apertures arranged in said desired pattern, the size of each aperture being such that the vacuum holds only one particle in overlying relation to each aperture; (b) positioning the tool and the particles held thereto over the article surface; and (c) decreasing the vacuum level such that the particles fall onto the surface, their position on the surface substantially corresponding with the position of the apertures. Preferably, a layer of adhesive is present on the surface so that when the

Ex. 1003, Page 188

particles drop onto it, they are adhesively attached to the surface. Most preferably, the particles have a metal cladding thereon, and the article is then heated to volatilize the adhesive and sinter bond the clad particles to the surface.

The invention is particularly useful in the fabrication of an abrasive layer on the tip surface of a rotor blade used in a gas turbine engine. For desired operating characteristics, the particle density per unit area of blade tip surface must be maximized, while at the same time the interparticle contact must be minimized. Also, the particles must be securely bonded to the blade tip to withstand the stresses of engine operation. To fabricate such an abrasive layer on a blade tip, after the metal clad particles are sinter bonded to the tip surface, a matrix alloy material is deposited on the surface so as to cover the particles and to fill in the spaces between the particles. The particulate bearing matrix is then simultaneously heated and pressed to eliminate any voids in the matrix material and securely bond the matrix to the substrate and, by interdiffusion, to the cladding on each particle. The abrasive layer is then machined to a relatively flat surface, and then part of the matrix is chemically removed to cause portions of the particles to project into space when blades having such an abrasive layer are installed in an engine, these exposed particles will rub the seal during engine operation, and minimize the leakage of working fluids around the blade tips, thus improving engine operating efficiency.

The abrasive particles are particularly sized with respect to the matrix layer thickness. Control over the particle sizing and aspect ratio are necessary to insure that a portion of most of the particles projects into space, while at the same time, all of the particles are securely bonded to the blade tip surface. Particles sized between Nos. 35-40 U.S. Sieve Series (0.42-0.50 mm nominal openings) are used when the matrix layer thickness is about 0.38 mm. Most preferably, the particles are uniformly spaced apart in a regular pattern on the blade tip surface. Depending on the expected interaction between the abrasive layer and the seal during engine operation, the particle density may be varied between about 35 to 110 particles per cm^2. This relatively close spacing necessitates using particles with aspect ratios less than 1.9 to 1, so that less than about 15% of the particles will contact one another after they are bonded to the tip surface.

The abrasive layer produced according to the invention is economical in the use of materials and has good durability when interacting with ceramic seals.

The foregoing and other subjects, features and advantages of the present invention will become more apparent from the following description of preferred embodiments and accompanying drawings.

Brief Description of the Drawings

Figure 1 generally shows the radially outer portion of a typical gas turbine blade having an abrasive layer made according to the invention;

Figure 2 shows in side view the appearance of a prior art abrasive layer;

Figure 3 shows in side view the appearance of an abrasive layer produced according to the teachings of the present invention;

Figures 4-7 show simplified, schematic views of the method by which abrasive particles are placed on the surface of a turbine blade tip according to the present invention;

Figure 8 shows a perspective view of Figure 6;

Figure 9 shows in side view the appearance of the abrasive particles enveloped in a matrix material;

and

Figure 10 shows the abrasive layer machined to a flat surface.

Best Mode for Carrying Out the Invention

In an example of the practice of the invention, and referring to Figures 1 and 3, an abrasive layer 10 is formed on the tip surface 11 of the airfoil portion 12 of a gas turbine blade 14. The blade 14 is preferably made of a nickel base superalloy (such as the single crystal alloy of U.S. Pat. No. 4,209,348), while the abrasive layer 10 is comprised of a nickel base superalloy matrix 16 and alumina coated silicon carbide particles 18. As will be evident, other materials may be used in the practice of the invention.

The abrasive layer 10 is subjected to very high stresses during engine operation, and therefore it is important that the layer 10 have a certain configuration and properties to perform its function. In particular, the particles 18 must be disposed on the tip surface 11 in a certain manner to obtain optimum performance.

In a prior art abrasive layer 20 for a turbine blade 21 shown in Figure 2, there are randomly disposed abrasive particles 22 within a matrix metal 24. A braze type bond joint 26, such as described in U.S. Patent No. 3,678,570, holds the layer 20 on the blade tip 32. In such a prior art abrasive layer 20, there sometimes tends to be a brick of particles 22 at the blade leading and trailing edges, 34, 36, respectively. Some of the particles 22 touch each other, which results in the inefficient use thereof.

Referring to Figs. 1 and 3, the abrasive layer 10 made according to the invention is characterized by a single layer of abrasive particles 18 surrounded by matrix material 16. Use of a single layer of abrasives 18 minimizes the mass of the entire layer 10, thus reducing the centripetal force on the blade 14 as it rotates during engine operation. The matrix metal 16 has a thickness W less than the overall thickness T of the particles 18. As a result, a portion of each particle 18 projects into space, thereby enabling favorable rubbing interaction with metal or ceramic seals during engine operation. For optimum performance, the unexposed portion of the particles 18 must be surrounded by matrix metal 16, and the particles 18 must be closely spaced apart from each other. Further, the matrix metal 16 as well as particles 18 must be securely bonded to the blade tip 11. In the invention, at least about 80-90% of the particulate surface area (excluding that surface area

Ex. 1003, Page 189

exposed at the blade tip) is surrounded by matrix metal 16 rather than being in contact with another particle 18. Also, the particles 18 are, in general, evenly and densely spaced on the blade tip 11. Densities of about 35-110 particles per cm^2 of tip surface 11 are obtained with the invention method of application, with about 50 particulates per cm^2 being preferred.

As shown in Fig. 3, the particles 18 have a thickness (length), T, and the matrix thickness W is about 50-90 percent of the particle thickness T. Silicon carbide particles of No. 35-40 U.S. Sieve Series Size (nominally 0.42-0.50 mm) have been found useful in the practice of the invention; up to U.S. No. 20 (0,83 mm) size also appears useful.

Figures 5-8 show how the particulates 18 are laid on the blade tip surface 11 where they will be subsequently permanently adhered. Prior to placing the silicon carbide particulates on the surface 11, they are first coated with about 0.010 mm of vapor deposited alumina according to the aforementioned Johnson et al patent, and then clad with a layer of metal, such as vapor or electrodeposited nickel, to a thickness of about 0.002-0.050 mm. Procedures for applying nickel coatings to ceramic particulates are commercially available and also are revealed in U.S. Patent Nos. 3,920,410, 4,291,089 and 4,374,173. If the ceramic particulate material is inherently resistant to reaction with the matrix material then the alumina coating is not necessary. (Neither the alumina coating nor metal cladding are shown in the Figures).

Just before the particles 18 are laid on the surface of the blade tip 11, a polymer adhesive 48 which is capable of being volatilized at less than about 540° C is applied to the surface 11. The purpose of the adhesive 48 is to hold the abrasive particles 18 in place after they are deposited on the blade tip 11. A 1-20 volume percent polystyrene in toluene solution is preferred.

As noted above, the particles 18 in the abrasive layer 10 should be densely arranged on the blade tip 11 in a uniformly spaced apart pattern. As shown in Figs. 4-8, to deposit the particles 18 in such a manner, a vacuum (suction) is drawn through a transfer tool having a plurality of apertures which are arranged to correspond with the desired, spaced apart pattern of particles on a blade tip. As is shown in Fig. 8, which shows the preferred means for depositing the particles, the apertures are perforations 44 in a rigid plate 42. The size of each perforation 44 or aperture is such that when the plate 42 is brought near a source of the particles 18, the vacuum draws the particles 18 against the plate 42, and only one particle 18 is held over each aperture. While not shown in the Figures, some particles 18 may be attracted to the plate 42 or to other particles 18 by, e.g., electrostatic forces, rather than held thereto by the vacuum. Such excess particles are dislodged from the plate 42 by gently tapping the plate 42, taking care not to dislodge the particles 18 held by the vacuum. The perforations 44 are particularly dimensioned relative to the particle size. The diameter of each perforation 44 must be large enough such that the force of the vacuum is sufficient to draw the particles 18 against the plate 42. At the same time, the peforations 44 must be small enough to prevent the particles 18 from passing therethrough. Preferably, the diameter of each perforation 44 is about 3/4 of the particle size.

As is seen in Fig. 8, the area of the plate 42 covered by the particles 18 is slightly larger than the actual blade tip surface 11. This is accomplished by constructing the container 46, which holds the loosely lying particles, in the shape of an oversized blade tip; once the plate 42 is brought near the container and the vacuum turned on, the particles 18 will be attracted to the plate 42 in the pattern shown in Fig. 8. Preferably, the base 49 of the container 46 is a screen or mesh type material.

Once the particles 18 are securely held by the vacuum to the plate 42 as shown in Figs. 5 and 8, the plate 42 is positioned over the blade tip 11 so that the perforations 44 (and the particles 18 overlying them) are aligned with the desired position of the particles 18 on the blade tip 11, as shown in Figure 8. As noted above, the tip 11 is coated with a layer 48 of adhesive. Once the plate 42 is properly positioned over the blade tip 11, the vacuum level is adjusted so that the particles 18 drop onto the adhesive coated tip 11 (Fig. 7). The distance A between the particles 18 and the tip 11 should be minimized so that when the particles 18 are released from the plate 42, they do not bounce off of the tip 11, or stray significantly from their intended position on the tip 11.

Of course, when the particles 18 are released from the plate 42, it is possible that a few particles 18 will stray from the spaced apart relation they had when held to the plate 42, and as a result, there may be some slight agglomeration of particles 18 on the blade tip 11. Also, when the vacuum is drawn on the plate, it is possible that not every perforation 44 will be overlaid by a particle 18. However, use of the plate 42 as described above allows the deposition of a single layer of generally spaced apart particles 18 on the tip surface 11, in sufficient densities (i.e., particles per cm^2) required for turbine blade tip applications. While interparticle contact on the blade tip surface 11 is undesired, experience has shown that there are typically only a few particles 18 (less than about 15%), which touch each other.

After the particles 18 are deposited on the blade tip surface 11, the blade 14 is heated to volatilize the adhesive 48, which also causes solid state bonding (sintering) of the metal cladding to the tip surface 11. For nickel clad particles, the heat treatment is preferably carried out at about 1080° C for 2 hours, in a vacuum or inert gas atmosphere. Use of such a protective atmosphere precludes oxidation of the cladding and blade tip 11.

Next, the particles 18 are oversprayed with a layer of matrix material 16 deposited by plasma arc spraying to a thickness T′ as shown in Fig. 9. A nickel base superalloy of the type generally described in the aforementioned Johnson et al patent may be used. The preferred matrix composition is, by weight percent, about 25 Cr, 8 W, 4 TA, 6 Al, 1.0 Hf, 0.1 Y, 0.23 C, balance Ni. The blade 14 is positioned with respect to the plasma arc device so that the tip cross section is generally perpendicular to the axis

Ex. 1003, Page 190

along which the molten matrix particles travel. The blade 14 is suitably masked around its periphery that errant spray does not deposit on the sides of the blade 14. The matrix material 16 is applied to a thickness of about 0.6-1.3 mm, preferably 1.1-1.3 mm.

Although the sprayed layer of matrix material 16 will have about 95 percent theoretical density, it will nonetheless be characterized by some porosity or voids. The voids are characteristic of the metal spraying process, and would be produced by any such "line of sight" deposition process. Metal spraying is used because it is one of the few processes capable of applying a superalloy, with all its diverse constituents. Another process that may be used is a physical vapor deposition process, since such process has been shown to be capable of applying MCrAlY coatings and the like. See U.S. Pat. No. 4,153,005 to Norton et al.

Next, the blade is subjected to a hot isostatic pressing procedure. Generally, this comprises deforming the matrix material 16 beyond its yield or creep-limit point at an elevated temperature. Preferably, the part is subjected to argon pressure while at elevated temperature, to close the aforementioned pores and voids, and to enhance the bond between the matrix 16, particles 18, and blade tip 11. For the specific superalloy matrix material described above, a temperature of about 1,100° C and a gas pressure of about 138 MPa applied for two hours is sufficient. Other hot pressing procedures may be used to consolidate the matrix material 16 and achieve the object of densification and bonding.

Next, the rough surface of the abrasive layer 10, as shown in Fig. 9, is machined using a conventional procedure such as grinding to produce a smooth, planar suface, as shown in Fig. 10. After the machining process, the thickness of abrasive particles 18 and matrix material 16 is T. Finally, the surface of the abrasive layer 10 is contacted with an etchant or other substance which will attack and remove some of the matrix material 16, causing the particles 18 to project into space. For example, electrochemical machining can be used, as is described in U.S. Patent No. 4,522,692 to Joslin. This step reduces the matrix thickness to a dimension W, which is about 50-90 percent of the dimension T, and results in the shape schematically shown in Fig. 3.

While Figures 4-8 show the preferred embodiment of the invention, i.e., the use of a perforated plate 42 to deposit the particles 18 on the blade tip 11, the apertures in the transfer tool may be defined by other means. One arrangement within the scope of the invention are hollow cylinders such as tubes or needles, which are spaced apart relative to each other such that the apertures (the cylinder ends) are arranged in the same pattern as the perforations in the plate. When a vacuum is drawn through the cylinders, only one particle will overlie each cylinder end. The particles are dropped from the cylinders in the same manner as they are dropped from the plate, as described above.

It has been found that there is a criticality in the aspect ratio of the particles 18, relevant to obtaining an abrasive layer 10 which contains a high density of uniformly spaced apart particles 18. When the particles are long and thin (i.e., have a high aspect ratio), they will of course tend to lie on their sides either when laid on the blade tip surface 11 initially or in the interval between the volatilization of the adhesive agent and the attainment of a metallic bond. Such laying-at-length disrupts the uniformity of placement and causes undue interparticle contact. Thus, the invention is best practiced when the aspect ratio of the particulates is less than about 1.9 to 1 and preferably is about 1.5 to 1 or less. The aspect ratio is defined herein as the average ratio of the longest particle dimension to the cross sectional dimension, as such is measured on a Quantimet Surface Analyzer (Cambridge Instruments, Cambridge, England).

The present invention is especially useful in providing a more effective abrasive layer (after the matrix is partially chemically milled away) compared to abrasive layers of the prior art. Suppose abrasive layers having the same volume percent of identically sized and shaped particles are made, first using the invention technique and second, using the prior art powder metal technique, as represented in Fig. 2. The area of abrasive particulate exposed at the surface of the invention and prior art layers, after the grinding operation, will be approximately the same. But the particulate deposited according to the invention will be distributed considerably more uniformly. And when the matrix is partially removed from the invention abrasive layer by electrochemical machining, about the same amount of surface area will be exposed on each particle, and the particles will all be about equally well-bonded to the blade tip. In contrast, owing to the non-uniform distribution of particles in prior art structures, when the matrix is electrochemically machined, those particles which were mostly contained in the matrix which is removed will of course be lost. The net result is that there will be less area of abrasive particles remaining at the free surface, and the prior art abrasive layer will be less effective in wearing the ceramic seal it rubs during engine operation.

While in the preferred embodiment of the invention the abrasive particles are placed in a uniform distribution on the blade tip, applications are contemplated in which there need be a greater or lesser concentration of particles at one or another portion of the tip. To achieve such a distribution of particles, the pattern of perforations in the plate is modified accordingly.

Although this invention has been shown and described with respect to a preferred embodiment, it will be understood by those skilled in the art that various changes in form and detail thereof may be made without departing from the spirit and scope of the claimed invention.

Claims

1. A method for depositing a single layer of particles on the surface of an article, wherein the particles are spaced apart from each other

Ex. 1003, Page 191

on said surface in substantially noncontacting relation in a desired pattern, comprising the steps of:

(a) drawing a vacuum through transfer tool means having a plurality of apertures spaced apart from each other in said desired pattern while the tool means is adjacent to a container of the particles, wherein the vacuum holds one particle over each aperture;

(b) positioning the article relative to the apertures such that each particle substantially overlies its desired location on the article surface; and

(c) adjusting the vacuum level such that the particles are released from the tool means and drop onto the article surface to form said desired pattern.

2. The method of claim 1, further comprising the step of applying an organic adhesive to the surface of the article prior to said step of adjusting, such that when the particles drop onto the surface from the plate, they are held on the surface of the article by the adhesive.

3. The method of claim 2, wherein the particles are metal clad ceramic, and further comprising the step of heating the article having the particles thereon to an elevated temperature to remove the adhesive and to bond the particles to the article surface.

4. The method of claim 2, wherein between about 35-110 particles per cm^2 are deposited on the article surface, the particle size being between about Nos. 20 and 40 U.S. Sieve Series.

5. The method of claim 4 wherein the aspect ratio of the particles is less than about 1.9:1.

6. The method of claim 1, wherein the article is a blade for a gas turbine engine.

7. A method for depositing a single layer of particles on the tip surface of a turbine blade, wherein the particles are spaced apart from each other on said tip surface in substantially noncontacting relation in a desired pattern, comprising the steps of:

(a) drawing a vacuum through transfer tool means having a plurality of apertures spaced apart from each other in said desired pattern while said tool means is adjacent to a container of the particles, wherein the vacuum holds one particle over each aperture;

(b) positioning the blade tip relative to the apertures such that each particle substantially overlies its desired location on the blade tip surface;

(c) adjusting the vacuum level through the tool means such that the particles drop onto the blade tip surface to form said desired pattern;

(d) depositing on the blade tip surface article a layer of matrix material to fill in the spaces between the particles; and

(e) removing a portion of the matrix material such that the thickness of the matrix material is less than the thickness of the particles.

Ex. 1003, Page 192

FIG. 1

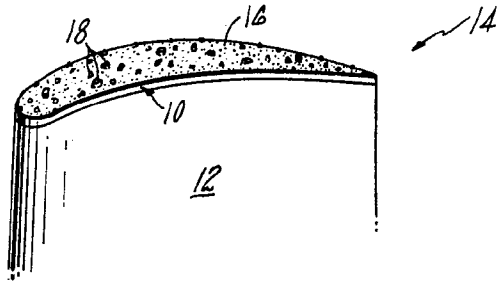
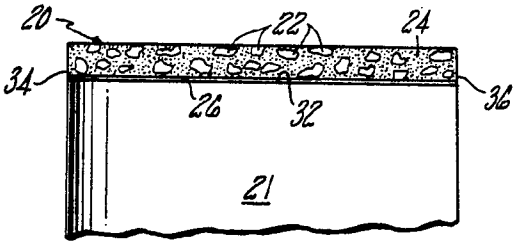


FIG. 2 PRIOR ART



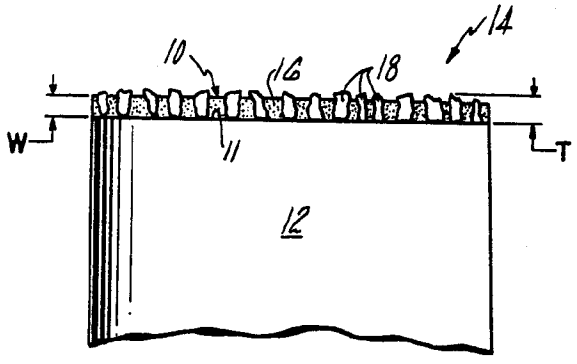


FIG. 3

FIG. 4

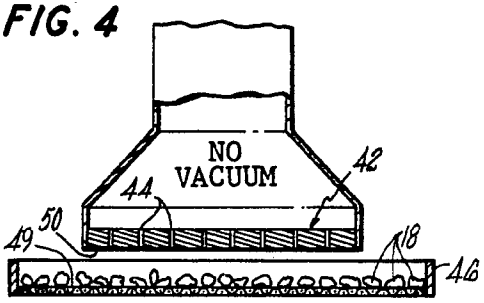


FIG. 5

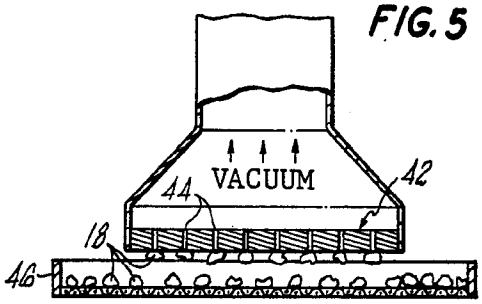


FIG. 6

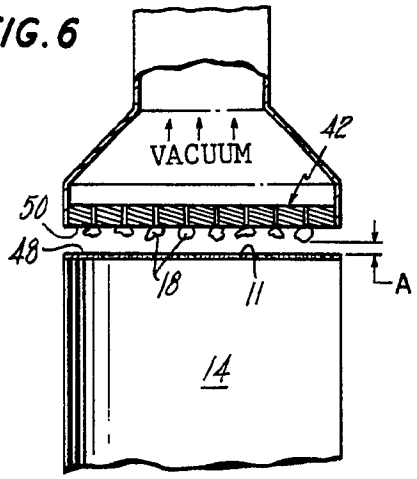
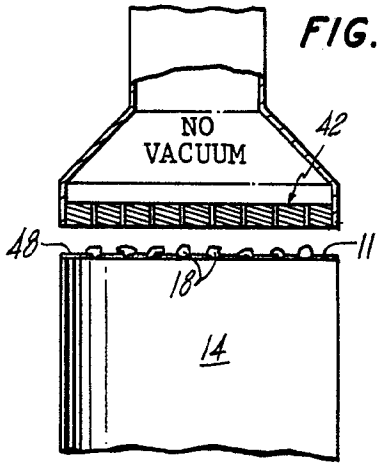


FIG. 7



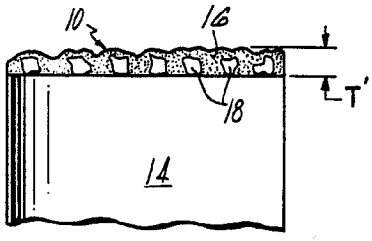


FIG. 9

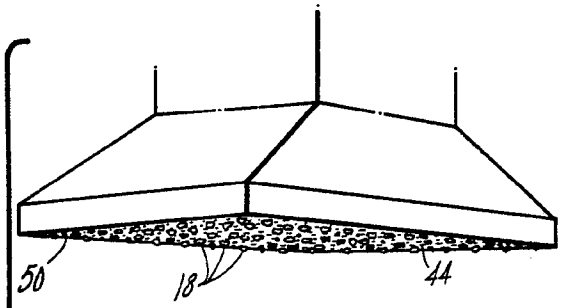
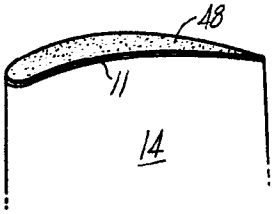


FIG. 8



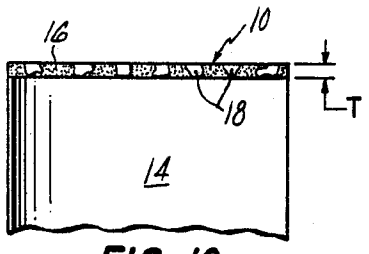


FIG. 10

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(54) Low pressure bonding of PCD bodies and method.

(57) A temperature stable diamond product (30) having a temperature stability up to about 1,200°C comprising a metal matrix carrier, at least one diamond piece (31a-31f) of a predetermined geometrical shape and which is temperature stable up to about 1,200°C having at least one surface (41) contacting at least a portion of said carrier (32), each diamond piece (31a-31f) being coated with a metal which is a carbide former on at least the portion thereof contacting said metal matrix carrier, said carbide forming coating being of a thickness of at least about 1 micron and including a relatively thin portion of a carbide material adjacent to said portion thereof contacting said diamond piece, and said temperature stable diamond product (30) being characterized by a chemical bond between said temperature stable diamond and said coating and said coating and the portion of said matrix carrier (32) which is in contact with said coating.

EP 0 264 674 A2

LOW PRESSURE BONDING OF PCD BODIES AND METHOD

Field of Invention

The present invention relates to an improved synthetic diamond product and method of making the same and more particularly to an improved and novel temperature stable diamond assembly which may be of a variety of sizes, shapes, and thickness, the diamond assembly being formed by an improved and novel bonding method which does not require the comparatively high temperatures and pressures normally used in the synthesis of synthetic diamond products thus to provide a diamond assembly which may be relatively large and in which the diamond, preferably in the form of a temperature stable synthetic polycrystalline diamone, is firmly and chemically bonded on or to a support structure.

Background of the Invention

Various types of synthetic diamond products are known and commercially available. Typically such products are used industrially for material removal purposes, for example drilling products such as core bits, mining bits, drilling bits for oil and gas, saw products, cutting tools, grinding wheels, and products such as wire-drawing and extruding dies, bearing surfaces, dressers, lead bonding tools and abrasives.

These synthetic diamond materials may vary somewhat in their character, one being an unleached polycrystalline product having a temperature stability of up to about 700-750 degrees C., see United States Patents 3,745,623 and 3,608,818. This synthetic diamond product itself is usually formed by diamond-to-diamond bonds and is carried on a substrate which may be cemented tungsten carbide, for example. The substrate is usually preformed and contains cobalt, the diamond product being formed and affixed to the substrate at very high temperatures and pressures, usually referred to as the diamond stable region of temperature and pressure, as set forth in prior patents and other literature, and typically above 50 kbar and at a temperature above 1,200 degrees C.

In this processing the cobalt in the substrate is used as a binder for the tungsten carbide and as a solvent/catalyst for the formation of diamond-to-diamond bonds. The presence of cobalt results in somewhat limited temperature stability of the diamond as noted. It is not uncommon to refer to these products as polycrystalline diamond products (PCD) as contrasted to single crystal diamonds.

PCD elements offer the advantage that upon fracture, only a relatively small fragment of the PCD element is lost as compared to a single crystal diamond in which fracture takes place along a plane with the possibility of losing a relatively large piece of the diamond.

Unleached PCD elements may be formed with the high temperature and high pressure apparatus as disclosed, for example, in United States Patent 2,941,248. PCD products thus far described are available in varoius sizes and shapes, for example circular, half-circles, quarter round, square, rectangular, half-rounds with a pointed section, and the like. The PCD face or disc, joined to a bacing member of tungsten carbide or the like is sometimes mounted, by brazing, on a steel tool body or tungsten carbide slug support. The PCD materials may be as small as several millimeters to as large as 50 millimeters in diameter, see for example United States Patent Application Serial Number 906,169, filed September 11, 1986, and assigned to the same assignee (CHP 6149).

Inclusions such as cobalt, iron or nickel present in unleached PCD elements as residual material initially used as solvents/catalysts in the synthesis of the PCD tend to reduce the temperature stability of the PCD to well below 1,200 degrees C., for example, to about 750 degrees C.

The fabrication of the larger diameter materials requires rather large and expensive processing equipment capable of generating high pressures and temperatures. The temperatures and pressures are those in the diamond forming region of the phase diagram for carbon, see for example United States Patents 4,108,614 and 4,411,672. The length of the substrate or backing is a limitation since the processing equipment must be capable of fabricating a diamond facing on a rather long piece which takes up room in the press. The result is that in at least one instance, the length of the backing, axially to the rear of the PCD face, is rather short and after formation, a longer tungsten carbide member or member of other material is attached to the formed product by any one of several well known procedures, and as described in United States Patent 4,200,159, for example.

In one typical use of these products, for example in oil and gas drilling bits and core bits, the usual practice is to form the bit body and thereafter to mount the PCD cutting element in the bit body. The bit body may be steel and the PCD cutter is a slug type cutter which is press fitted into apertures in the bit body. These procedures are well known. The difficulty with the use of steel bodies is that the body and/or the cutter assembly may be sub-

Ex. 1003, Page 196

ject to erosion by the relatively abrasive flow of the drilling fluid used to clean and cool the bit. Thus, the practice has been to hard face the steel body or to use a carbide slug. In the case of matrix body bits which offer erosion resistance, the practice has been to mount the PCD cutter, normally having an added backing or of a sufficiently long length integrally formed as one piece, by brazing the cutter to the matrix body. One of the problems associated with this procedure, in addition to the increased cost, is the desire to use a relatively high temperature braze material and the need to keep the PCD face cool to prevent thermal damage (above about 750 degrees C.) to the cutter face during the brazing operation. Even so, a large number of bits have been made as described by a variety of bit manufacturers.

Another commercially available form of PCD, sold under the mark GEOSET, is an unbacked leached product, see United States Patents 4,224,380 and 4,288,248, which has a temperature stability of up to 1,200 degrees C., but which may have a porosity of up to about 15% and may not be as impact resistant as desired in a particular application. The temperature stability of these PCD products offers the advantage that they can be processed during the infiltration of the matrix material used to form the bit body and may be exposed to the relatively high tempeatures used during the infiltration procedure without degradation of the PCD, thus eliminating the need for brazing. In general, temperature stability is also an advantage in the cutting action of the PCD during use. For the purposes of this invention, temperature stability means that the PCD element, regardless of type, is stable to about 1,200 degrees C. without significant deterioration in its properties and will be referred to herein as TSPCD. Typical temperature stable leached PCD material is available under the trademark GEOSET, which cobtains porosity.

Temperature stable PCD elements are known which are not porous and are not leached. A typical such material, available under the trademark SYNDAX-3 is a PCD in which silicon is present in the form of silicon carbide.

Significantly, the TSPCD materials are not commercially available as backed products, as contrasted to the previously described low temperature products. The TSPCD materials are available as unbacked materials in a wide variety of shapes and sizes, for example, triangular, square, cylindrical with flat ends or pointed at one end, round discs, rectangular, ovoid, and the like. These products may also be cut to form other shapes by using a laser or other means.

While temperature stability is not required in all uses, there are many applications in which it is required. Further, there are processing steps such as brazing in which the PCD part may be exposed to temperatures above 750 degrees C. In these instances it is necessary to cool the PCD part or to otherwise prevent it from being exposed to the higher temperature. As a result, there are limitations in the processing of such unstable PCD products which tend to limit the use thereof.

TSPCD elements, are available in a limited range of sizes that are generally too small for individual use in many applications. To overcome this problem, TSPCD elements have been formed into a mosaic type of structure in which the individual TSPCD elements are mounted and mechanically affixed in a support structure, e.g., a hot-pressed segment or the like, see United States Patent Applications Serial Number 797,058; 794,569 and 797,569, filed respectively on November 14,1985, November 4, 1985 and November 13,1984, and all assigned to the same assignee as this application.

While these mosaic structures have been operated satisfactorily and provide a large PCD cutter having temperature stability, the mosaic elements lack the advantages of the large one piece PCD cutters (up to 50 mm diameter or more) which are not temperature stable but which have been used in oil and gas drill bits and core bits. Even though the bits using the mosaic cutters avoid the necessity of having to braze the mosaic into the matrix of the bit, the mosaic cutters do not function as a large one piece PCD with the result that the individual mechanically mounted TSPCD elements can be broken free of the supporting structure. A mechanical type of mounting also limits the shape and size and contour of the cutter, even though the assembly is temperature stable.

Another problem with mosaic cutters is that bending stresses tend to cause release of the mechanically mounted TSPCD elements. Moreover, unlike a large integral brazed PCD cutter of the same size in which only small portions of the PCD are fractured off during use, the tendency in the mosaic cutters is to release the TSPCD elements as the supporting structure around the TSPCD is abraded away and support for the TSPCD is reduced, i.e., the individual TSPCD element is lost rather than losing on a small fragment.

In general there is a need for a TSPCD containing product in which one or more TSPCD elements are firmly anchored or locked into or mounted on a suitable support, which optionally may be mounted to another structure. It is desirable to

Ex. 1003, Page 197

mount one or more of such TSPCD elements by more than merely a mechanical mounting, and preferably it is desired that the mounting be by a firm chemical bond.

United States Patent 3,650,714 of March 21, 1972, issued to Farkas, describes a method for coating natural single crystal diamond particles in the range of 200 to 250 mesh with either titanium or zirconium. The coating is applied from dry powder, the resultant material is then heated in a graphite mold, under vacuum conditions for between 10 and 15 minutes at a temperature in the range of 850 to 900 degrees C. The relatively low temperature of heating to form a carbide at the interface indicates that the titanium or zirconium coating is quite thin. The result is said to be a coated diamond particle in which the coating is about 5% by weight of the resultant product and in which a carbide is formed at the interface, with the outer surface of the coating being non-carbided. The calculated thickness of the coating is about 0.445 of a micron for 200 mesh material and 0.344 of a micron for 250 mesh material. Since subsequent processing in an oxidizing atmosphere would result in the formation of an oxide which is not easily wetted by other materials, the coated natural diamond particles are overcoated with either nickel or copper with a film thickness of between 0.002 and 0.005 of an inch (between 50.8 and 127 microns thick). This outer coating is said to serve as a protective coating to prevent oxidation of the titanium or zirconium or their carbides during subsequent processing under oxidizing conditions. The subsequent processing described is hot or cold pressing and subsequent sintering or infiltration with brazing alloys.

An important difficulty with the above procedure and the resulting product is that the carbide containing coating is quite thin, substantially less than one micron, with the result that under certain types of subsequent processing such as with the use of liquid binders in infiltration, as for example in the formation of an infiltrated matrix for earth boring bits, the liquid binder penetrates the protective coating and the relatively thin carbide containing coating. The problem would be particularly acute with PCD elements since their surface is rather "rocky" and irregular and includes a certain amount of surface porosity. Thin coatings such as described in Farkas would contain substantial imperfections and voids that would permit the liquid infiltrating binde to attack the carbide interface between the PCD and the coating and to separate the coating from the PCD In addition, the processing of such coated diamonds, whether PCD elements or natural, in an oxidizing atmosphere is not practical since any imperfection or dissolution of the outer nickel or copper coating, for instance by the action of the liquid binder, would result in the oxidation and destruction of the underlining titanium and titanium carbide layer.

Also known in the prior art are products which might generically referred to as diamond impregnated products. Typically these products are composed of natural or synthetic diamond particles which may be of a variety of mesh sizes. Whereas a large integral PCD element may be between 70% and 97% by volume of diamond material, depending upon the processing, the amount of residual inclusions or porosity, the impregnated products generally have a much lower volume percent of diamond, typically less than 40%. The result is that the impregnated product performs better as an abrasive or cutting element than does the same material without the diamond grit, but the problem is that the small diamond particles are easily lost as the surrounding supporting structure is abraded away. To improve the retention of the diamond grit in the supporting structure it has been proposed to coat the particles of the grit with various metals such as tungsten, tantalum, columbium, niobium or molybdenum, and the like by chemical vapor depostion techniques using a fluidized bed procedure, see for example United States Patents 3,871,840 and 3,841,852. The products there described are about 25% by volume of diamond grit of particle size from 40 to 100 mesh and represent an improved impregnated product in contrast to an improved large diamond product. The coated diamond grit is formed into various shapes by a hot pressing procedure or by infiltration at an elevated temperature.

Coatings have also been used to form diamond products as contrasted to impregnated products. For example, United States Patent 3,879,901 describes the vacuum deposition of a metal such as molybdenum or titanium on diamond materials with subsequent processing at 60 Kbar and between 1,200 and 1,400 degrees C. to form a diamond product in contrast to a diamond impregnated product. In another instance, see United States Patent 4,378,975, an abrasive body with diamond is formed by forming a green body by cold pressing with a nickel based alloy. The green body is then sintered at 950 degrees C. with a diamond volume of between 10% and 40 % by volume.

It is also known in the sintering of synthetic diamond to use a thin coating of between 300 and 6,000 Angstroms of a strong carbide former, with a second coating of copper with subsequent processing at 5 Kbar at a temperature of 600 to 700 degrees C. in an inert atmosphere, see United States Patent 3,356,473. In United States Patent

Ex. 1003, Page 198

3,464,894 it is proposed to form a chemically bonded coating of titanium such that a titanium-carbon bond is formed followed by processing at 62,000 atmospheres of pressure.

In general, the coating procedures of the prior art are related to pretreatment of diamond forming materials which are thereafter processed at relatively high temperatures and pressures to form a diamond element or to form a coating on diamond grit used to form an impregnated product in which the volume of diamond is well below about 50% by volume.

The provision of improved methods for the fabrication of large TSPCD products, which are temperature stable in the range of about 1,200 degrees C. and which may be fabricated at lower pressures and temperatures than have been used in the synthesis of diamond products, would have unique advantages. For example, the cost for fabrication equipment is vastly reduced as well as the cost for each of the products. The ability to fabricate various shapes and sizes and thickness, i.e., to be free of the shape and size limitations of high-temperature and high pressure equipment, would also offer advantages from a processing and application point of view.

One of the significant advantages of the present invention is the ability to fabricate a TSPCD product of a particular configuration and in which the TSPCD starting element or elements are firmly anchored in place. For example, the use of a TSPCD structure in accordance with this invention in various types of cutting tools and lead bond tools as may be used in semiconductor chip processing to form electrical connections to the chip elements through accurate control and positioning of a heated TSPCD faced lead bond tool, or in diamond tracing tools, for example, offers unique advantages as compared to current practices.

Thus, it is an object of this invention to provide a temperature stable diamond product of various sizes and shapes and capable of being used in various products, but which is substantially less expensive than the diamond products currently available and which may be firmly bonded to a supporting structure.

Another object of this invention is to provide an improved and novel process using relatively low temperatures and pressures for the fabrication of a diamond product from smaller temperature stable diamond elements in which the diamond product is temperature stable.

Still another object of this invention is the provision of an improved relatively low temperature and relatively low pressure process for the formation of a temperature stable diamond product.

Yet another object of this invention is the provision of an improved temperature stable diamond cutting element for us in earth boring and core bits which is less expensive than comparable sized elements and which may be fabricated at relatively low temperatures and pressures into shapes and sizes not presently economically feasible.

It is also an object of this invention to provide a mor effective method and means of attaching a temperature stable PCD to a tool or supporting body.

Another object of this invention is to bond TSPCD elements by a chemical bond so that they can be firmly attached to a tool body.

Yet another object of this invention is to bond TSPCD elements to form a specific pattern of cutting elements so that the wear mechanism, such as regenerating sharp cutting edges as the cutter wears, can be controlled.

It is also an object of this invention to form an improved diamond product in which the bonded PCD pieces form an integral part rather than a collection of individual elements.

It is also an object of this invention to provide a strong substrate backing to TSPCD, in which the properties of the backing can be adjusted to suit specific applications.

Brief Description of the Invention

In accordance with the present invention, a temperature stable synthetic polycrystalline diamond (PCD) product is provided which is fabricated from smaller TSPCD elements by improved relatively low pressure and low temperature procedures.

In general the products of the present invention may be formed by either liquid state infiltration or solid state sintering to affix a relatively thin PCD element on a hard ceramic substrate. The method includes the common steps of coating a temperature stable polycrystalline diamond element with a selected carbide former, preferably one which is stable against oxidation, arranging the coated elements in a mold in a predetermined relation with each other. Thereafter, a hard ceramic powder such as tungsten carbide powder containing an organic binder is placed over and around the assembled coated TSPCD elements. The powder is preferably monosized, although bimodial sizes and mixtures may be used.

The resulting assembly is cold pressed to compact the powder to about 50 volume percent, i.e., about 50% of the volume is porosity which will later be filled up by sintering or infiltration. The thus pressed assembly may then optionally be cold

Ex. 1003, Page 199

isostatic pressed to increase the volume percent of the powder to about 60-65%. The cold isostatic pressed part has sufficient integrity to be handled and machined, if that is needed.

Thereafter if liquid state infiltration is used, the formed part is first heated to vaporize off the organic binder and then infiltrated preferably in a vacuum or under inert or reducing atmosphere with a metal binder which melts and fills the voids in the hard ceramic powder and binds it together. The infiltration also causes the metal binder and powder to bond metallurgically to the coating of the TSPCD since the binder wets the outer portion of the coating which partially dissolves in or reacts with the binder for the matrix or matrix carrier powder to form a solid solution of binder and coating metal at the interface. The binder is fluid or near fluid at this point in the processing. It is believed that the bond from the coating is through the binder for the matrix carrier powder.

The metal binder is preferably one which has a melting point within the temperature stable range of the TSPCD and which possesses strength and toughness. In this operation, the coated TSPCD elements are bonded together into a unitary assembly of a TSPCD. The infiltration is carried out at a temperature below the thermal degradation temperature of the TSPCD elements, above the melting point of the binder, and below the melting point of the metal of the coating. In accordance with the invention, a thin carbide layer is formed at the interface between the TSPCD and the carbide former coating. This carbide layer may be formed either before the powder is placed around the elements by heating the elements under carbide forming conditions or simultaneously with the infiltation or sintering. In either case the heating is controlled to promote formation of a relatively thin carbide layer on that portion of the surface of the TSPCD which has been coated by the carbide former. During the controlled heating, under carbide forming conditions, carbon diffuses from the PCD into the coating to form a carbide.

During the heating, it is believed that the temperature used to form the carbide is more important than the rate of heating. The evidence suggests, in the case of a tungsten coating for example, that the coating on the diamond can form a carbide above about 950 degrees C. and preferably at about 1,100 degrees C. no matter how fast the heating is conducted. Thus, heating at 1,100 degrees C. for about 15 minutes is normally sufficient. A longer time of heating at 1,100 degrees C. can increase the thickness of the carbide, but in general longer heating periods at high temperatures may damage the diamond because of too much carbide formation and results in an ineffective chemical bond being formed. If the heating is excessive, the result is that more and more of the carbon from the TSPCD diffuses into the coating with possible damage to the PCD. Thus, while the dimension of the thickness of the coating of the carbide forming metal may vary within wide limits, the dimension of the carbide coating, for a single coating which is stable against oxidation, such as tungsten, should be in the range of at least about 1 micron and up to about 15 to 30 microns, for reasons which will be discussed.

The thickness of the carbide forming metal coating may be as low as one micron, although coating thicknesses above about 3 microns provide stronger bonding strength. Films of above about 30 microns in thickness may be used, but increasing the thickness above about 30 microns does not seem to increase bond strength appreciably over the strengths achieved in the range of 10 to about 30 microns. Film thicknesses of 1 to about 3 microns are acceptable in some applications, but substantially increased bond strength is achieved above about 3 microns fot the binder system tested. Coatings of less tha about 1 micron tend to result in unacceptable separating of the coating from the TSPCD element during infiltration or sintering.

The resulting product of the invention has a chemical bond from the TSPCD to the coating to the matrix carrier. The term "matrix carrier" as used herein describes the powder and binder material which is placed around or in contact with the TSPCD element or elements and which is processed in accordance with this disclosure. The matrix carrier is to be distinguished from the more generic term "matrix" which generally refers to the supporting structure in which the matrix carrier is mounted, for exaple a drill bit body. the term "chemical bond" is used herein to refer to the strong bonds that are formed between the diamond and the metal coating through the medium of the carbide layer at the interface, and between the exterior surface of the metal coating and the matrix carrier. While not fully understood, it is believed that the former is an atom to atom level bond between the carbon of the diamond and the carbide layer. The latter is believed to be a metallurgical bond in which the binder alloy used in the liquid state infiltration or with the sintered material of the matrix carrier if solid state sintering is used. This contrasts sharply with the mechanical bonds that were formed in the prior art systems.

The term "diamond product" used herein and in the claims includes cubic boron nitride.

A solid state sintering operation (also referred to as tungsten powder activated sintering) may also be used to bind the TSPCD element to the matrix carrier. This operation involves vacuum sintering or sintering in a controlled atmosphere or hot pressing

Ex. 1003, Page 200

or hot isostatic pressing of the cold pressed part. If processed by solid state sintering, the materials used for coating and for the backing and the like have to be adjusted to reflect the different but related processing. Here again, if the formation of the carbide layer is simultaneous with the sintering operation, control of the heating and the duration of the heating is such that a relatively thin carbide layer is formed. Since this form of processing does not use a binder for matrix carrier formation, a portion of the coating wets the powder and bonds to the powder during sintering, again forming the chemical bond described. In the case of solid state sintering of tungsten, it is preferred to use a sintering activator such as nickel or nickel-iron. The activator is placed around the tungsten particles and promotes the sintering of the matrix carrier and the bonding to the coating of the TSPCD.

Various shapes and sizes may be formed in accordance with the present invention, and the testing thereof indicates that the bond between adjacent TSPCD elements is stronger than that which may be achieved through merely mechanical mounting of the TSPCD part or parts in a support matrix carrier.

The methods described above permit the formation of a wide variety of shaped cutting elements for use in various cutting and abrading devices. Large and shaped diamond bearings may be fabricated. The larger or shaped TSPCD product of this invention may be processed with a backing or supporting element of a variety of shapes and configurations and compositions. Relative thick and shaped TSPCD products may be fabricated at relatively low pressures and temperatures. In general, however, the principal advantage of this invention is the ability to provide a TSPCD structure in which one or more TSPCD elements are firmly anchored in a support structure by a bond which is significantly stronger than a mere mechanical mounting. The result is that the assembly of several TSPCD pieces in the form of a predetermined pattern will result in action of the part as one large diamond rather than as a plurality of diamonds held together as an aggregrate.

Brief Description of the Drawings

Figure 1 is a flow chart depicting the steps in formation of a TSPCD product in accordance with the present invention;

Figure 2 is a plan view of one form of a TSPCD product in accordance with the present invention;

Figure 3 is a side view as seen along the line 3-3 of Figure 2;

Figure 4 is an enlarged fragmentary view illustrating the bonding path between adjacent TSPCD elements in accordance with this invention;

Figure 5 is a view in perspective of a TSPCD product having a single TSPCD element in accordance with this invention;

Figure 6 is a plan view of one form of bearing in accordance with this invention;

Figure 6a is a sectional view taken along the line 6a-6a of Figure 6;

Figure 7 is a view in perspective of a dressing tool in accordance with the present invention;

Figure 8 is a plan view of a saw blade segment in accordance with this invention;

Figure 8a is a sectional view as seen along the line 8a-8a of Figure 8;

Figure 9 is a sectional view of a lead bonding tool in accordance with this invention;

Figure 10 is a plan view of a TSPCD product in accordance with this invention in which a multiplicity of individual elements is bonded together into a larger TSPCD structure;

Figure 11 is a perspective view of another form of TSPCD product in accordance with this invention;

Figure 11a is a view, partly in section and partly in elevation, illustrating the mounting and use of the product illustrated in Figure 11;

Figures 12a to 12d are diagrammatic views of various structures and the test protocol for three point testing;

Figure 13 is a view in perspective of another form of TSPCD product in accordance with this invention;

Figure 13a is a view similar to that of Figure 13 but illustrating a modified form of product in accordance with this invention;

Figure 14 is a view in section illustrating still another form of product in accordance with this invention;

Figure 15 is a view, in persepctive, of a twist drill in accordance with the present invention;

Figure 16 is a view in perspective of still another product in accordance with this invention;

Figure 17 is a perspective view of one form of earth boring bit in accordance with this invention, and

Figure 18 is an enlarged fragmentary view of the cutters illustrated in Figure 17 and produced in accordance with this invention.

Detailed Description of the Invention

In accordance with this invention the starting diamond material is preferably a temperature stable polycrystalline diamond material composed of aggregate diamond particles having a size in the

Ex. 1003, Page 201

range of submicron to over 100 microns, for example, although the particle sizes need not be uniform; i.e., mixtures of particles of the sizes indicated may be used as is known in the art. The TSPCD should contain at least 60% by volume of diamond aggregates, as described, and the product may be leached or unleached with some diamond to diamond bonding or diamond to metal bonding in the assembly. For products to be used in cutting or abrasive operations where the surface finish of the cut or abraded part is important, it is preferred that the smaller particles of diamond be used, for example, below about 15 microns. By way of exaple, wire drawing dies may require smaller particle sizes in order to provide a certain surface finish on the drawn wire product.

The TSPCD element may be as small as 250 microns, (i.e., 10 mils) on an edge and as large as one carat or more. It is preferred that the edge length of the starting TSPCD be above 1,000 microns (i.e., one millimeter) since pieces of this size are easier to manipulate or handle. Typically, TSPCD elements of a size of three pieces per carat (0.165 of an inch per side triangles) are easily handled.

In some instances the smaller particle sizes of TSPCD material below 250 microns may be used as a grit in the material used to bind the larger TSPCD elements described in order to provide an impregnated support or to increase the amount of diamond material between adjacent larger TSPCD elements.

It is preferred that the TSPCD startng material be of a regular geometrical shape which may be any of the shapes already mentioned. The principal reason for a fixed and known geometrical shape is that it vastly simplifies the arrangement of the TSPCD elements in the desired relationship for processing and thus offers maximum control of placement, spacing and overall configuraton and content of the final part. Since one of the advantages of the present invention is the ability to form large TSPCD sturctures, it is also preferred that the geometrical shape be such that the TSPCD starting elements have side surfaces or faces which can be positioned in a close and controlled spacing to an adjacent TSPCD element, e.g., triangular, square, rectangular and the like, it being understood, as will be discussed in detail, that a TSPCD structure may include TSPCD elements of different geometrical configurations.

In the processing in accordance with this invention, as diagrammatically illustrated in the flow chart of Figure 1, the starting TSPCD material 10 is preferably cleaned by heating in hydrogen at about 800 to 1,000 degrees C. for about 10 to 60 minutes as indicated at 12. Other cleaning procedures may be used provided they are effective to remove oxidants and other surface contaminants. Thereafter the cleaned TSPCD material is surface coated with a metal by any one of several known procedures to form an essentially continuous coating which is essentially free of discontinuities. For reasons which will become apparent, the presence of discontinuities in the coating tends to promote lifting of the carbide containing coating off the surface of the TSPCD or provide a defect through which other materials may migrate to adversely affect the underlying TSPCD.

One such coating procedure is chemical vapor deposition by which a metal compound such as a chloride or a fluoride is heated to decompose the compound to form a mist of the metal which then deposits on the TSPCD material. Another procedure is fused salt bath coating methods.

Tungsten is preferred as the coating metal because it is a strong carbide former, not subject to excessive oxidation damage, has similar thermal expansion characteristics as the TSPCD elements, and is not easily dissoved by the liquid binder metals during later processing. Other metals may be used are any one of the Group IVA, VA, VIA, VIIA metals and alloys and mixtures thereof and silicon, such as tantalum, molybdenum, titanium, niobium, vanadium, zirconium, manganese, molybdenum/manganese, or tungsten/manganese. As a class, these materials have a melting point higher than the temperatures used in subsequent processing and are strong carbide formers and form a strong bond to the diamond. Metals of Group VIIIA, such as cobalt, iron and nickel are less preferable because of the tendency to weaken the TSPCD by back conversion of the diamond or possibly by other means. Zirconium and titanium have been observed to have a volume expansion in response to elevated temperatures which is more than that of diamond but less than that of cobalt and iron, but these materials are subject to oxidation damage in subsequent processing in normal atmospheres unless protected by an overcoating of nickel or copper or some other material. Even so, care must taken to assure that the carbide containing coating is sufficiently thick to prevent penetration by the surrounding metal should the copper or nickel or other coating dissolve into the matrix carrier.

For chemical vapor deposition one may use the hexahalides, tetrahalides, pentahalides, e.g., the hexa-, tetra-and penta-forms of the bromides, chlorides fluorides of the metals mentioned. The thickness of the coating on the TSPCD may vary so long as it is greater than about one micron, but it is preferred that the coating be between about 1

Ex. 1003, Page 202

micron and up to 30 microns. Thicker coatings may be used but do not seem to increase the bond stregth over that acieved in the range of 20 to 30 microns.

The application of the coating does not necessarily result in a chemical bond to the TSPCD at this point in the processing. As near as has been determined, the processing temperature used in chemical vapor deposition is not sufficiently high to form a chemical bond as indicated by the essential absence of any diffused carbon into the metal of the coating and the absence of any metal of the coating being diffused into the TSPCD. While it is believed that the coating is essentially uniform in dimension and essentially covers the entire outer exposed surface of the TSPCD, it is not essential to form an aboslutely continuous coating. In the case of porous leached TSPCD elements, the coating should be thick enough to bridge over the pores in the TSPCD surface so that it is effectively coated with the metal.

Of the carbide forming materials described, tungsten is the most preferred for several reasons. When a tungsten coating, previously converted to a carbide at the interface of the TSPCD as described herein, is furnaced in a graphite mold or oxidative environment, the tungsten is relatively stable against oxidation as compared to titanium, for example, unless the titanium is protected by an overcoating. Another reason for the preferance is that the thermal expansion of tungsten or tungsten carbide is close to the thermal expansion of diamond. Further, tungsten dissolves in the binder at a relatively slow rate. The overcoating requires a second coating operation and thus increases cost. Further, if the overcoating is not continuous, for example if there are defects which expose portions of the underlying oxidation sensitive coating, subsequnet processing in an oxidizing environment may destroy the metal and the carbide interface or a local portion of the underlying diamond material or both. So too, there are some types of matricies, which tend to dissolve the oxidation protective layer deposited over the oxidation sensitive carbide formers with the result that the oxidation protective material dissolves or is otherwise absorbed in the matrix and a gap tends to form between the partially oxidized coating and the surrounding structure, thereby resulting in a marked reduction in the strength of the bond between the coating and the surrounding structure. Ideally, the bond between the outer surface of the coating and the matrix carrier is a metallurgical bond which is not easily formed if the coating is not of compatible material or if it is oxidized.

More particularly, if the matrix or infiltratant includes copper or nickel or alloys thereof, as is normally the case in abraseive resistance matrix carrier, the matrix or infiltrant rapidly dissolves the oxidation protective layer so that the presence of a thin carbide forming layer, even if overcoated with an oxidation resistant material tends not to produce as strong a metallurgical bond as is the case with tungsten.

The use of materials such as zirconium or titanium or chromium may be acceptable provided those coatings are sufficiently thick to essentially prevent adverse reactions which cause damage to the carbide layer or to the underlying diamond since copper and nickel in the binder in contact with the coating will protect the coating from oxidation during infiltration. This is especially true where the matrix composition or the infiltrant is a material as above or hereinafter described. Thus, even if there is penetration of the infiltrant or a component of the matrix through or into the oxidation protective layer, oxidation sensitive material may be used as the coating, provided they are sufficiently thick. Thicknesses of less than one micron tend to be subject to susbsequent processing problems, even if coated with an oxidation protective layer. Thickes layers, especially above about 3 microns tend to provide improved bonding but not as great as that provided by tungsten.

An important advantage of this invention is the ability to form a backing or supporting structure for the TSPCD elements of widely varying configuration and composition. The backing is formed in situ as the TSPCD elements are bonded chemically together andto the support structure.

To assist in handling, it is preferred to compress the powder and the TSPCD elements in an axial compression opertion. Ultimately the backing or support structure will achieve a volume percentage of metal and/or metal carbide and/or abrasive particel (e.g., oxides or non-metal carbides) which is in the range of 40% to 80%. Optionally, the compacted structure may be further compressed by isostatic pressing in which the volume percentage is increased to about 60% to 90%. If the structure is to be sintered, isostatic pressing is preferred.

The hard ceramic powders used to form the matrix carrier may be metal carbide such as tungsten carbide, titanium carbide, tantalum carbide, molybdenum carbide, and cemented tungsten carbide (tungsten carbide cemented with cobalt). Alternatively, it could be made of other hard ceramic powders such as alumina or titanium or silicon nitride. The abrasive particles may be alumina,

Ex. 1003, Page 203

silicon carbide or silicon nitride. For applications such as earth boring bits, where strength and toughness are important, tungsten carbide is the preferred hard ceramic powder.

The particular powder used and the particle size or sizes is related to the desired volume percent of the matrix powder, the final product which in turn is related to the hardness (rigidity) and impact resistance (toughness) of the backing or support structure. Also a consideration is the type of processing, e.g., liquid state infiltration or solid state sintering.

In the case of liquid state infiltration, the hard ceramic powder, for example tungsten carbide, has a particle size which is typically below 325 mesh (Tyler), i.e., below 44 microns (Standard), for example 30 microns. The size distribution and the percentage distribution may vary depending upon the supplier. However, the size may also be varied. For example the same packing efficiency may be obtained by appropriate selection of particle sizes, e.g., 30 microns and 6 microns in a ratio of 70% to 30%.

A preferred infiltrating material is a copper-tin-titanium-manganese alloy. If the alloy does not include manganese or nickel, thinner coatings of about 1 micron may be used, as will be discussed.

In the case of solid state sintering, for example using a tungsten powder, the powder particles are preferably coated with a metal such as nickel, for example 0.35 weight percent. This may be doen by mixing tungsten powder of a particle size of 0.5 microns with a solution of nickel nitrate in alcohol, followed by heating at about 90 degress C. to evaporate the alcohol with the result that the nickel nitrate is deposited on the tungsten powder. the nickel nitrate coated tungsten powder was then heated at 800 degrees C. for 3 hours in a reducing atmosphere (hydrogen) to eliminate the nitrate. The processed tungsten powder was then ball milled for 24 hours using tungsten rods as the milling media and then sieved and screened to 200 mesh (74 microns). The purpose of the nickel, as will be described, is to act as a diffusion activator for the tungsten. Other binder materials which may be used are tantalum, molybdenum, and nickel based alloys, copper-nickel alloys and cobalt based alloys.

In either liquid state infiltration or solid state sintering, the appropriate powder of the appropriate particle size or sizes is mixed with a temporary binder material which is preferably an organic material as indicated at 15. The temporary binder is used to coat the powder material to form a temporary adhesive bond between the powder particles during the initial compression step. Since this temporary binder remains in the processed product through the cold isostatic pressing operation, it should preferably be a material which is present in a relatively low volume percentage. If the volume percentage of the temporary binder needed to achieve a temporary bond is too high, this may affect the volume percentage of the powder in the final product, i.e, too great an amount of the temporary binder may prevent the achievement of higher powder volume percentages. Various organic polymeric or resinous binders which are easily volatilized at elevated tempoerature may be used. A preferred material is paraffin which is easily dissolved in an organic solvent. The amount of paraffin relative to the tungsten carbide is about 2 weight percent. The paraffin is easily and completly removed in the processing. It is also desirable to use a lubricant which may be volatilized completely at elevated temperatures in order to facilitate powder movement during the various pressing operations, especially the optional cold isostatic pressing operation. One such material is mineral oil which may also be dissolved with the temporary binder to coat the powder. A typical useable amount is 20 grams of paraffin per 1,000 grams of cast carbide in 0.2 grams of glycerine per 1,000 grams of cast carbide, by way of example.

The powder is dispersed into the solution of the temporary binder and lubricant in hexane (for the paraffin) and the binder and lubricant coat the powder which is then dried and reground into a powder. Other coating procedures may be used as is known in the art.

The next step in the sequence, as indicated at 20, is to arrange the coated TSPCD elements in the desired relationship as needed to form a product. If the product is to contain multiple TSPCD elements it is preferred to use a template or die configured to position the TSPCD elements with respect to each other. The spacing between adjacent TSPCD elements may vary widely, depending upon the configuration and nature of the final product. Where the part includes multiple TSPCD pieces, the side faces of the TSPCD elements may be spaced from each other to provide a gap of from about less than 1 mil to 500 mils and preferably in the range of 6 to about 30 mils (150 to 750 microns), by way of example. Thereafter, the processed powder is placed in the gaps between the adjacent TSPCD pieces and filled in in contact with the surfaces of the TSPCD elements to be bonded to the matrix carrier.

In some instances it is not necessary to form a gap as described since the TSPCD may be assembled with adjacent surface portions in contact with each other. In this instance where liquid state infiltration is used, the binder penetrates between the TSPCD elements and chemically bonds the adjacen coated TSPCD elements together. Such a butting arrangement o of the TSPCD's may be

Ex. 1003, Page 204

used where erosion is not a potential problem, e.g., in bearings, saw blades and wire dies and the like, for example. Where erosion is likely to be a problem, such as exposure to relatively high velocity flow or the use of abrasive materials, the use of a gap in which powder is present is preferred for erosion resistance.

The thus assembled TSPCD-powder temporary assembly is then assembled into a pressure die of suitable configuration which preferably defines the outside contour of the finished part. One manner of carrying this out is to use an open ended die, invert the die and move the temporary assembly of TSPCD elements up into the bottom of the die, re-invert the die right side up so that the assembly is in the bottom of the die and then add the processed powder material to the required volume. The amount of powder added is related to the thickness of the support or backing and the volume percentage of the metal in the final product.

The assembled material is then axially cold compressed in the die, as indicated at 22, at a pressure of between 10,000 psi and 30,000 psi to increase the tungsten carbide packing density to about 50 to 65 volume percent. As a result of this processing, a compacted intermediate is formed which is fragile but still capable of being handled with care. As another example, if the subsequent processing is solid state sintering, pressures around 10,000 psi may be used and the resultant product has a packing density of about 50 volume percent.

An additional step in the processing is the cold isostatic pressing operation, as shown at 24, in which the compacted intermediate is isostatically cold pressed at a pressure of up to about 30,000 psi for five minutes or longer, for example. The isostatic pressing typically achieves a tungsten carbide packing density of about 62 volume percent. In the case of tungsten powder in solid state sintering, the density is about 65 volume percent. During this operation, the tungsten or the carbide powders are moved and compacted uniformly to a more dense product. The step of cold isostatic pressing is optional although in many cases it is preferred particularly for sintered products.

At this point in the processing, two alternate processing routes are available, as indicated in the dotted lines of Figure 1, liquid state infiltration, as seen at 26 or solid state sintering as seen at 28.

In the case of solid state sintering, the product of the isostatic pressing is sintered in a vacuum furnace at 10 to the minus 5 torr. at 1,200 degrees C. for about 1 hour. The result is a sintered product having a density of about 90 volume percent. The finished product indicates a bond between the tungsten material with the tungsten coating on the TSPCD, i.e., a tungsten-tungsten metal bond. The nickel or other activator is used to promote diffusion and metallurgical bonding. The mechanical properties of this bond are quite strong, and the finished product, although somewhat brittle is quite useable.

There are, however, some limitations of solid state sintering. For example, the limitation of sintering at a temperature below 1,200 degrees C. renders it difficult to find a material with high strength and high modulus. Even if the high modulus materials can be sintered below about 1,200 degrees C., as by activation sintering, the sintered product may contain 5% or more porosity which may act as pre-existing cracks. While this may not be a problem in some cases, it is a consideration where the finished part is in a part which is subject to relatively high impact loads, e.g. a drill bit for example.

In those instances in which the bonded TSPCD product is to be mounted to a high modulus backing, the processing may involve two thermal cycles to bond the TSPCD product to a tungsten carbide back-up element. While effective, the processing steps increase the costs.

Even though these problems exist, the advantage of this form of processing is the ability to provide a high strength, low thermal expansion material to bond the TSPCD together. There are known ductile materials which may be added to the powder or the binder in accordance with this invention to decrease the brittleness of the sintered matrix carrier.

It is preferred to use liquid phase infiltration as the last process step. In this processing, the product from the isostatic pressing operation is heated to cause a metal binder to flow through the compacted powder and bind it together and to the TSPCD elements.

Regardless of the type of processing as herein described, the hard ceramic powder in the matrix carrier can be of relatively high volume percentage, e.g., above 50% and up to 90% by volume. The provision of a relatively high density final product, which is formed in situ at relatively low pressures and temperatures and which is temperature stable to about 1,200 degrees C. offers advantages not heretofore obtained.

The metal binder material should be one which in a liquid state can wet the coating on the TSPCD and the hard ceramic powder particles. It is also preerred that the binder metal have good mechanical properties such as ductility and strength. Typical such binders are copper or nickel based brazing alloys, copper-tin-manganese-nickel alloys, the latter being the preferred binder material.

Ex. 1003, Page 205

The processing above described, as already noted may be used to form a wide variety of products which are characterized as having a strong chemical bond between the coating and the TSPCD and between the coating and the supporting structure. It is this chemical bonding which provides the significant strength over that which can be achieved by merely a mechanical gripping of the TSPCD. The advantage of this invention is that the TSPCD elements or element are effectively locked in or on a support structure to provide a TSPCD cutting element as contrasted to the prior temperature sensitive supported TSPCD elements commercially available.

Referring to the drawings which illustrate preferred forms of the present invention, Figure 2 illustrates a TSPCD product 30 which includes six (6) of initially separate TSPCD elements 31a to 31f of generally triangular shape. The TSPCD elements may be bonded together in or on a support structure 32, see also Figure 3 which may be an infiltrated or sintered powdered material. The support structure may be of various dimensions and of various axial lengths, as indicated by the break line 33.

As seen in Figures 2 and 3, the TSPCD elements include at least one side face 34a which may be positioned adjacent to but spaced a small distance from the side face 34b of the adjacent TSPCD element. The distance between adjacent TSPCD elements, referred to as the gap and illustrted as 35 in Figure 3, may be of the dimensions previously indicated.

Referring to Figure 4, for example, the gap 35 is illustrated with reference to its condition after cold isostatic pressing and before further processing. Each of the TSPCD elements 37 and 38 includes the coating 37a and 38a, respectively, the TSPCD elements being spaced from each other by the gap width. Between the coated surfaces is the powder material 40 such that the gap is TSPCD face-coating-powder-coating-TSPCD face. In subsequent processing, a strong chemical bond is formed from the TSPCD face through the coating, through the powder, through the coating of the adjacent TSPCD face and to the adjacent TSPCD.

Again referring to Figures 2 and 3, in the resulting TSPCD product 30, essentially all surfaces of the TSPCD elements, save one, are in contact with bonded support material, i.e., there is material on the bottom face 41 and peripherally around the outer faces, as indicated at 42 and in the gap 35, while one face 44 is exposed. This form provides the optimum bonding of the TSPCD elements together. This product may be assembled to a bit body during infiltration of the bit to form the matrix bit body. It is also possible to braze the product into the bit body, in which event, higher temperature braze alloys may be used than have heretofor been used.

The form or TSPCD product in accordance with this invention shown diagrammatically in Figure 5 illustrates that not all of the faces need be fully in contact and bonded to the support element. Here a triangular TSPCD element 50 includes side faces 50a, 50b and 50c which are only partially beneath the top surfaces 52 of the support structure 53 shown as triangular for purposes of illustration. Due to the strong chemical bonding between the TSPCD and the support structure, it is possible to expose a substantial portion of the large flat face 50d of the TSPCD while the body of the TSPCD is substantially above the support body. Thus, for example it is possible in accordance with this invention to form large bearings from smaller TSPCD elements.

Referring to Figures 6 and 6a, for example, a TSPCD product 60 is illustrated which may be used as a bearing or the like. This form of product also illustrates that TSPCD elements of different geometrical shapes may be used to form a useful TSPCD products. In this form, TSPCD elements 61 and 63 are half circular disks while TSPCD element 64 is square, although it may also be rectangular. The TSPCD elements are bonded to a support 65 of metal powder processed as already described with gaps 66a and 66b between adjacent elements. As illustrated the gaps, however, are not filled with matrix carrier material thus effectively providing channels between the spaced and facing exposed side faces 61f-64f and 64g-63f, the elements 61, 63 and 64 being positioned such that a substantial portion is exposed as noted with reference to Figure 5. When used as a bearing, the channels may provide for flow of cooling or lubricating fluid, if desired. If channels are not needed, the gaps may be substantially filled as illustrated in Figure 3, i.e., the surface of the support is even with the top surface of the TSPCD elements.

Not all of the side faces need be in contact with and bonded to the support material. Thus, for example as illustrated in Figure 7, a single point dressing tool 70 is shown which includes a TSPCD element 72 mounted on and bonded to a support 74 such that one face 72a is fully contacted by the support material which effectively forms a shoulder 76 to the rear of the element 72. Faces 72b and 72c are fully exposed while the bottom surface is bonded to the support. While element 72 is indicated as triangular, it may be other shapes such as square, rectangular, semi-circular or circular, for example.

Ex. 1003, Page 206

The ability to bond a TSPCD element firmly in a support structure permits the fabrication of unique cutting and abrading products. For example, Figures 8 and 8a illustrate an improved saw blade segment 80 composed of a pluarlity of TSPCD elements 81a through 81k, bonded to a support structure 83. In the form illustrated, the TSPCD elements are circular in shape although other geometrical shapes may be used. Circular shapes, however, have the advantage of providing a relatively large surface area for firmly bonding the elements to the support structure. The diameter of the elements may be in the range of one millimeter or larger depending upon the use of the segment. In prior segments, they were either impregnated with diamond dust or used spherical natural diamonds. In the either case as the supporting matrix carrier was worn due to use, the diamond material was lost due to loss of supporting structure. This problem is largely overcome by the present invention since the TSPCD elements are firmly chemically bonded over a comparatively large surface area.

The TSPCD elements are preferably arranged in a redundant pattern such that elements 81e through 81g are to the rear and in the space between the adjacent lead elements 81a-81d, with elements 81h-81k oriented in the spaces between 81e-81g. Thus, for example if the thickness of the elements is equal to or greater than the gap dimension between adjacent side-by-side elements 81a-81d, the entire face of the cut in the workpiece is traversed by at least one cutter. The redundancy may be two or more, that is, additional elements may be arranged to provide added redundancy as may be needed.

As illustrated in Figure 8a, each element 81a-81k includes an exposed arcuate cutting face, for example 81aa, 81ee and 81hh extending above the surface 84 of the support 83, with a substantial portion of each element firmly and chemically bonded to the support. As the segment wears in use, the elements are retained to a much greater degree than the prior diamond cutting segments. While circular elements have been described, one may use a half-circle or quarter-round TSPCD elements, preferably mounted such that the curved peripheral surface is exposed to form the leading cutting edge. Such materials are commercially available or may be cut off a cylindrical TSPCD part to the desired thickness.

The ability to anchor and firmly bond a TSPCD element to a support structure permits the fabrication of a wide variety of cutting and abrading tools. It also permits the fabricatior of products that are other than abrading or cutting rools, such as bearings, as described and, for example, a lead bonding tool. Heretofore, lead bonding tools have been made of a single crystal diamond cut to a precise shape and dimension with a flat working face. Typically these tools are used to bond lead elements to solid state chips and the like by being brought into contact with the chip and bonding the leads in a precise and exact location to the chip through heat and pressure. Normally the temperature is about 500 degrees C. and natural diamonds are able to withstand this temperature. However, it is difficult to support these natural diamonds in a support structure and if they move even slightly relative to the support structure, problems may be encountered in their use since the bonding operation is a precision operation. In accordance with this invention, an improved lead bonding tool is provided which is firmly anchored in a support structure and which is temperature stable.

Referring to Figure 9 and improved lead bonding tool 90 is illustrated and includes a TSPCD element 92 bonded to a support structure 94, the latter being metallic and including an aperture 95 therein for a heater and including a shank 96 for mounting in the lead bonding machine. The support usually includes a cylindrical section 97 having a frusto-conical end 98 with the element 92 mounted at the tip of the end 98. The element 92 includes a working face 99 which projects beyond the end 98 and which is of a precise flatness and face dimension, i.e., length and width. The face contour may be square or rectangular, or any other shape, as may be desired. Heat may be applied to heat the element in a conventional manner normally used to heat the lead bonding tool. In use the tool is subject to vibration and heat which requires that the element be firmly and accurately mounted and maintained in that accurate mounting during use. By the present invention, the TSPCD element 92 may be chemically bonded to the support structure which may be the support 94 or a separate insert to be received in the support. The advantage of this invention is that a TSPCD element may be used which may be relatively easily affixed to a support structure.

Thus, for example, the element 92 may be shaped to the precise required length and width dimensions and in situ bonded to a support structure. If the support structure is the support 94, the face of the TSPCD may be precision ground to the desired flatness such that the plane of the face 99 is referenced to the support structure, an operation which is difficult to achieve with single crystal diamonds. It is also possible to form a lead bonding tool by formation of a TSPCD support structure which is then assembled to the body 94, rather than forming the entire assembly as herein described.

Ex. 1003, Page 207

One aspect of this invention is the ability to form a TSPCD product of virtually any shape or size from smaller TSPCD elements. The TSPCD product illustrated in Figure 10 is an example. There a large TSPCD product 100 is composed of a multiplicity of triangular TSPCD elements 101 in a TSPCD part which is essentially circular in overall shape, although it may be of any shape. In the form illustrated, there is a surrounding portion 105 of tungsten carbide, for example. The tungsten carbide may be of 70 volume percent tungsten carbide By way of example, the part may be 50 mm in diameter and is useful as a cutter for oil and gas drill and core bits.

Figure 11 illustrates another TSPCD product 110 in accordance with this invention composed of a plurality of TSPCD elements 112 illustrated as being triangular pieces which may be for instance 3 per carat or 1 per carat. The elements 112 are bonded together and to a support 115 such as tungsten carbide by any of the methods already described, so as to form a unitary structure in which the previously separate elements are chemically bonded together and to the tungsten carbide and to the tungsten carbide backing 115. It is understood that the backing may be any of the carbide materials already described.

The form of the invention illustrated in Figure 11 illustrates the variety of TSPCD products that may be fabricated in accordance with this invention. This particular structure provides for a plurality of cutting elements 112a and b, 112c and d, and 112e and f, which form pairs which together provides a series of cutting points 116, 117 and 118. In use in a bit, as shown in Figure 11a, the cutting product 110 may be mounted directly into the body matrix during body matrix infiltration or formation. In this form, there is a back support 121 provided which contacts the backing 115, the cutting direction being indicated by the arrow 123. The tungsten carbide backing 115 provides for increased impact strength.

The cutting product 110 is exposed such that at least one top row of elements 112a-112f are essentially fully exposed. In use as the top row of cutters wear, new cutting surfaces are regenerated from the cutter elements below the top row. This product also illustrates the fact that adjacent elements include at least one side surface which is located adjacent to the adjoining piece, the spacing being as already described.

To improve the cutter performance in terms of regenerating cutter edges and points, the amount of matrix carrier between adjacent TSPCD elements may be reduced in cross-sectional thickness in order to provide controlled weakness planes in the cutter structure thereby providing for fracture of the cutter along predetermined planes of fracture. In this way as the loading on a cutter region increases, the cutter is broken out of the support to provide a new cutter element. Since earth boring, in part, relies upon a kerfing type of cutting, the formation of an uneven overall cutting surface generally tends to improve the cutting action in certain types of formations.

As already noted, the products of the present invention are much stronger than products previously described as mechanical mosaic structures. Referring to Figures 12a to 12d, Figure 12a illustrates diagrammatically a three point bending test in which loads are applied to a part 130 in one direction as indicated by the arrows 131 and 132 and a load is applied in another direction as indicated by the arrow 133. The force necessary to bring about fracture through the joint 135 or through the joined parts 136 is measured and expressed in terms of pounds per square inch to failure.

Figure 12b illustrates a TSPCD part 140 in accordance with this invention made up of triangular TSPCD elements 141 joined together as indicated at the joints 142 by the methods already described. The part 140 was processed by sintering tungsten coated 3 per carat TSPCD elements with tungsten powder which had been compressed to a volume percentage of 90%. In the three point test, the failure mode was in the joint between adjacent elements, as indicated at 145. The load to failure was 21,000 psi.

Figure 12c is similar in structure to that of Figure 12b, but the part 150 was formed from tungsten coated 3 per carat TSPCD elements which were liquid state infiltrated with tungsten carbide using a copper binder, as described, and processed as described. In the three point test, the failure mode was through the TSPCD as indicated at 155 and the load to failure was 26,000 psi.

Figure 12d illustrates a mechanical mosaic structure 160 which is essentially the same as 150, but in which the TSPCD elements were not coated and in which the part was processed using conventional infiltration techniques with the same tungsten carbide and binder used to form the part 150. In the three point test, the failure was through the joint, as indicated at 165, and the load to failure was zero psi.

Other test data indicates that the thickness of the carbide forming coating is significant. These data were derived from the use of a matrix material in which the binder included nickel and manganese. In the case of tungsten coated material, it has been observed that nickel and/or manganese tend to adversely affect the coating if the coating is too thin, for example below about 3 microns. However, if the binder for the matrix is copper or tin or cobminations thereof, thinner coatings of one mi-

Ex. 1003, Page 208

cron may be used. Thus if the binder for the matrix is compatible with the coating, thinner coatings of 1 micron may be used. If the binder for the matrix is from the iron group, e.g., nickel or manganese, somewhat thicker coatings are preferred to prevent the binder from penetrating the coating on the diamonds, for example a tungsten coating. It is thus preferred to use binders which are compatible with tungsten, in the preferred form of this invention, in which instance the coating thickness may be 1 micron in thickness.

For example, in a shear test where coated TSPCD is bonded to a matrix carrier, as described, and subject to a lateral pushing force, the results indicate that in the range of 1 to 5 microns, there is improvement in the bond strength to about 5,000 psi over an uncoated product. The failure mode at this range of coating thickness is by failure at the interface of the coating and the matrix carrier. In the range of about 7 to 10 microns thickness, the fracture is mixed in nature, in that the fracture sometimes is at the stated interface and sometimes through the TSPCD. The stated fractures occur at bond strengths in the range of about 5,000 psi and up to about 22,000 psi. In this range of bond strengths, the TSPCD product in accordance with this invention has a wide variety of uses. Above about 10 microns, the fracture mode is through the TSPCD and not through the bond, i.e., this means that the interfaces between the PCD and the coating, the coating and the bond, the coating itself and the bond itself are all stronger than the PCD. These fractures of the TSPCD, rather than at the interface between the support structure and the TSPCD occur typically at strengths in the range of above 22,000 psi. Above about 15 microns of coating thickness, the bond strengths seem to level off at about 27,000 psi, with fracture through the TSPCD rather than the bond interfaces, even though the coating thickness increases to 20 microns or more. For coating thicknesses above about 30 microns, there does not appear to be any increase in bond strength over that achieved in the range of 15 to 30 microns.

by way of further comparison, a titanium carbide coated TSPCD having a thickness of about 5 to 10 microns, typically at 7 microns, and a total coating thickness (including a nickel oxidation protective coating) of about 30 to 50 microns exhibited mixed failures through the bond and through the TSPCD at about 15,000 psi. These data also tend to establish that a tungsten coating, absent any oxidation protective coating, having the thickness dimensions described, is far superior than products with a titanium carbide coating and a nickel protective coating, wherein the titanium coating was about 7 microns and the nickel coating was about 40 microns, since the failure mode of the latter is a mixed mode, either at the bond rather than only through the TSPCD. The achievement of sufficiently high bond strengths to result in the failure of the TSPCD rather than the bond of the latter to the supporting structure offers considerable advantages, especially in the cases of those products which require a strong bond of a cutter-type product in some form of support structure.

The relatively high strength of the bonded joint between the TSPCD and the supporting structure permits wide variation in the types of products in accordance with this invention. One of the advantages is the ability to provide a TSPCD element which is securely bonded to a support structure. Referring to Figure 13, there is illustrated a product 170 which includes a TSPCD element 172 firmly secured to a tungsten carbide support element 174. While the product 170 superficially resembles the appearance of a STRATAPAX brand cutter, it is vastly different.

More specifically, the TSPCD element may be a product available commercially as SYNDAX, TSPCD material in the form of a thin one-piece disc. The dimensions of the disc 172 may vary widely from a diameter of 2 to 3 mm to as much as 50mm or more. The disc 172 is firmly chemically bonded to the support element 174 through the chemical bond formed through the coating applied to the disc 172 which in turn is bonded to the infiltrated or sintered material 175.

The support 174 may be separately formed and the disc 172 and the material 175 may be assembled and processed as described. The result is the chemical bonding of a TSPCD to a preformed support. The advantage is that the length, diameter and shape of the support is not limited by the size of the high pressure high temperature presses normally used to form STRATAPAX types of products. The cost for making a product as shown and described in Figure 13 is less than that for the temperature sensitive product, but the more important advantage is the ability to form the support of essentially any length, as indicated by the break line 176, without requiring larger presses. This form of product has the advantage of being temperature stable up to about 1,200 degrees C. and may thus be infiltrated simultaneously with the formation of a supporting matrix, for example a matrix body for a drill bit. Alternatively, the product may be brazed into the bit body by the use of higher temperature brazing materials than have been heretofore used. It is also understood that the coated diamond may be brazed to a suitable support such as cemented tungsten carbide, and the like, since the braze joint is between the coating on the diamond and the support element. Since the

Ex. 1003, Page 209

diamond is temperature stable, higher melting point brazing alloys may be used as compared to those alloys which are used with diamond product which are not temperature stable, as herein defined.

As illustrated in Figure 13, the side surface 178 of the disc is free or sintered or infiltrated material. In the form illustrated in Figure 13a, the sintered or infiltrated material may cover the sides of the disc 172a, as indicated at 179 for added strength.

Figure 14 illustrates a structure in which one or more TSPCD elements 180 are affixed to a supporting body such that the back side 183 of the elements are in contact with the support body while the side faces are adjacent to each other or include a fully exposed side face, as indicated at 187. In this particular case, the support body may be a saw blade segment or a preformed and preshaped cutter for an earth boring bit. While the configuration of the product illustrated in this figure may appear similar to some of the illustrations of United States Patent 3,938,599 issued to Horn, there are significant differences. Unlike the Horn structure, that of the present invention is chemically bonded to the supporting matrix carrier and is far stronger than a mere mechanical bond as described in the referenced patent, see also the data previously described. This strong bonding allows for the provision of a variety of products.

Thus, for example, Figure 15 illustrates a twist drill 190 having segments 192 and 194 mounted thereon. Segments 192 and 194 may be a TSPCD product in accordance with this invention containding one or more TSPCD elements in a support structure. The drill bit may be a 1/4 inch or larger size.

Figure 16 illustrates a form of the invention similiar to that of Figure 13 wherein the TSPCD elements 200 are square in shape, although rectangular shapes and mixtures of geometrical shapes may be used. The TSPCD elements are bonded to each other through the material 201 and to the base 202, as described. The base may be of a shape other than square and may be of any length as indicated by the break line 205.

Figures 17 and 18 illustrate a drill bit 220 in accordance with the present invention. The bit includes a body 222, preferably of matrix material such as carbide, having a plurality of waterways 225 associated with a plurality of blades 230. It is to be understood that in referring to the bit body as a matrix body, the matrix material of the bit body may be relatively thin coating on a suitable support structure. The bit also includes nozzles 235 for flow of fluid form the interior of the body to the waterways to cool and clean the bit.

As shown in Figure 18, each blade includes a plurality of cutters 240 having a configuration as illustrated in Figure 2, for example. The cutters 240 are mounted in the matrix body during infiltation of the body thus eliminating the need to braze the individual cutters into the bit body. As mentioned. each of the cutters, by virtue of the production thereof is temperature stable to 1,200 degrees C. It is understood that other cutter configuration in accordance with this invention may be used, for example that illustrated in Figure 10 or in Figure 13. for example. It is also possible to provide cutters of selected geometry, e.g., quarter circles, half circles and the like. It is also possible to have temperature stable cutters which are larger in diameter than about 14mm, the usual maximum size for the STRATAPAX brand products.

Drill bit or core bit bodes may be of any of a number of types, as is known in the art. Thus, for example, stepped bit designs may be used with a significant cutter exposure, to mention only one variant. The ability to fabricate cutter to different desired geometries as may be needed for effective cutting action in various formations is now possible in accordance with this invention.

One of the features of the present invention is the fact that a wide variety of shapes and sizes is possible in the provision of a temperature stable PCD assembly through the use of processing steps that do not involve the high temperature and high pressure regime used in the synthesis of PCD materials.

Typical processing of TSPCD elements in accordance with this invention is set forth in the following specific example, by way of illustration.

Triangular temperature stable TSPCD elements of a size of 3 per carat were coated with tungsten metal to a thickness of about 6 microns. The powder used to form the matrix carrier was tungsten carbide powder of 3 to 5 micron particle size. About 1,000 grams of this powder were completely and thoroughly mixed with a solution of 200 ml. of hexane in which 20 grams of paraffin and mineral oil had been dissolved. The resultant mixture was heated to between 80 to 100 degrees C. for 45 minutes to evaporate the hexane completely. The processed powder was then ball milled, as described, for thirty minutes to break up the flocculated powder and then screened through a 200 mesh screen.

The tungsten coated TSPCD elements were arranged in a suitable hexagonal die and 4.5 grams of the processed tungsten carbide powder was placed in the mold to fill the space between the TSPCD elements and to cover them. The resulting assembly was then cold pressed at 20,000 psi to form a hexagonal preform.

Ex. 1003, Page 210

The hexagonal preform was then loaded into a rubber mold which was then evacuated and sealed and then isostatically pressed at 29,000 psi. The resulting product was then ground to specific size.

The isostatically processed hexagonal product was the placed on a crucible with 2.6 grams of a copper based alloy (Cu-Sn-Mn-Ni) on the powder side. The preform was then heated in a vacuum furnace (10 to the minus 5 torr) at the rate of 5 degrees C. per minute up to 400 degrees C. for one hour and then at 10 degrees C. per minute to a temperature of 1160 degrees C. for one hour for infiltration, later followed by cooling.

Heating at a temperature over 1100 degrees C. for one hour is important because it is at this stage that tungsten carbide formation takes place. Typically the tungsten carbide layer thus formed is about 3 microns thick.

If the preform is heated quickly to 1,000 degrees C. and held for 1.5 hours, the formed tungsten carbide layer is about 0.3 microns thick. Heating to the carbide forming temperature at the rate of 10 degree C. per minute may also be used.

After the thermal cycle, the finished product is surface ground to a thickness of about 0.315 of an inch and may be used.

As a variant, cemented tungsten carbide grit (94% by weight of tungsten carbide bonded with 6% by weight of cobalt, a commercially available material) of 400 mesh size was mixed with a solution of 2% by weight paraffin in hexane.

It is within the scope of this invention to form the isostatically pressed part and then process that part so that it is simulataneously bonded to a supportng structure such as a matrix bit body or other support structure while effecting conversion to the carbide layer described. For example, carburized tungsten powder in the nominal size range of 3 to 5 microns was ball milled with 1% by weight of nickel powder (-325 mesh) for one hour. The milled powder mixture was then mixed with a solution of 2% by weight of paraffin wax in hexane.

The mixture was dried by heating and then the processed powders were used with triangular TSPCD elements, coated as already described, to form a much larger assembly, as described. The larger assembly was cold axially pressed and then isostatically pressed as described. The resulting product was a "green" hexagonal cutter as shown in Figure 2. The green assembly was then heated to burn off the organic material and held at a temperature of 1,000 degrees C. for one hour.

Thereafter, the processed green assembly, sufficiently strong to be handled was assembled to a matrix powder material in a mold, e.g. the matrix bit forming operation, and the matrix infiltrated at 1,200 degrees C. During the matrix infiltration, the green assembly was bonded to the matrix and the coating was formed into the carbide layer. In this processing there was a strong bond between the metal binder of the matrix and that in the green assembly and between the coating, powder and matrix supporting structure. Essentially the same processing may be used to form other products already described in or on a matrix carrier support element.

Another form of one step infiltration involves the use of powders processed as follows: As received carburized tungsten carbide powder in a particle size range of from 3 to 5 microns was screened through a mesh screen to break up the agglomerates which tend to form in the fine powder. The screened powders were mixed with 7 weight percent of nickel powder having a particle size in the range of 3 to 7 microns and mixed in a suitable mixer for one hour. A second tungsten carbide powder of a particle size in the range of 3 to 5 microns was screened through a 200 mesh screen to break of the larger agglomerates. The screened material was then ball milled with 3 weight percent of nickel powder of a particle size range of from 3 to 7 microns with 2 weight percent of paraffin in a hexane solution for about eight hours. The ball milled powders were then dried and coated with paraffin.

Six pieces of tungsten coated triangular TSPCD elements, coated as already described, of a size of 3 per carat were arranged in a hexagonal punch and about 0.8 grams of the second powder was placed between the TSPCD elements, spaced as already described. About 4 grams of the first powder was then placed in the die and a hexagon product was formed under pressure of 15, 000 psig. The cold pressed hexagaonal product was then isostatically pressed at 29,000 psig. After isostatic pressing the hexagonal product was infiltrated directly into a matrix, such as a drill bit matrix using a copper-manganese-tin-nickel alloy.

In another example, tungsten carbide powder in the size range of 20 to 30 microns and in the range of 60 to 70 microns was ball milled with 3 weight percent of nickel powder, of the type and size described, with 2 weight percent of paraffin in a hexane solution. The powders were dried, screend through a 200 mesh screen, assembled to TSPCD elements as already described and cold pressed in the fixture.

The cold pressed material with the TSPCD elements was then transferred to a high vacuum furnace, optionally a hydrogen furnace, to burn off the organic components by heating relatively slowly. This was accomplished by heating at a rate of about 5 degrees C. per minute to a temperature of

Ex. 1003, Page 211

800 degres C. The burned off hexagonal shapes were then assembled in a mold, and infiltrated into a matrix to provide a finished part, in this case a drill bit.

It is to be understood that the one-step infiltration process described may be used to form any of a variety of products in accordance with this invention.

In those cases where it is contemplated that the product of the present invention is to be assembled to a matrix material supporting structure, such as a matrix body drill or core bit, it is desirable to coat the product of this invention with a carbide forming material, as already described. Such processing has the advantage of of providing a coating, of the thickness described, which may in turn form a carbide layer for effective bonding to the matrix carrier in which the part is to be mounted. The carbide layer may be formed by a separate preheating operation or by controlled heating during body matrix formation.

The abrasive character of the products in accordance with the present invention may be enhanced somewhat by adding to the powder materials relatively small particles of diamond or TSPCD material in the form of particles having the approximate size of the powders used. The volume percent of such diamond powders may be as high as 40% and the resulting material is processed as already described with the bonding as already described. The starting diamond material is preferably synthetic polycrystalline diamond which is temperature stable through natrual diamond material may also be used in some instances.

It will be apparent to those skilled in the art that there may be variations and alterations, based on the above detailed disclosures, of the various procedures and product here described. It is to be considered that the same may come within the scope of the present invention as set forth in the appended claims.

Claims

Claim 1. A temperature stable diamond product having a temperature stability up to about 1,200 degrees C. comprising:
a metal matrix carrier;
at least one diamond piece of a predetermined geometrical shape and which is temperature stable up to about 1,200 degrees C. having at least one surface contacting at least a portion of said carrier,
each diamond piece being coated with a metal which is a carbide former on at least the portion thereof contacting said metal matrix carrier,
said carbide forming coating being of a thickness of at least about 1 micron and including a relatively thin portion of a carbide material adjacent to said portion thereof contacting said diamond piece, and
said temperature stable diamond product being characterized by a chemical bond between said temperature stable diamond and said coating and said coating and the portion of said matrix carrier which is in contact with said coating.

Claim 2. A temperature stable diamond product as set forth in Claim 1 wherein said diamond piece is a synthetic diamond piece which includes a face, at least a portion of said face being in contact with said matrix carrier, and
said metal matrix carrier being tungsten carbide and extending to the rear of the face a predetermined distance and forming a backing integral with said diamond piece.

Claim 3. A temperature stable diamond product as set forth in Claim 1 wherein said temperature stable diamond includes at least one side face, and said side face being coated with said metal which is a carbide former and being at least partially in contact with said matrix carrier.

Claim 4. A temperature stable diamond product as set forth in claim 1 wherein said product includes a plurality of initially separate temperature stable diamond pieces arranged in a predetermined pattern and including opposed surface portions forming a small gap therebetween, and
matrix carrier being located in said gap and contacting at least a portion of the surface portions of said temperature stable diamond elements.

Claim 5. A temperature stable diamond product as set forth in claim 4 wherein at least some of said temperature stable diamond elements are generally triangular in shape.

Claim 6. A temperature stable diamond product as set forth in claim 1 wherein said product is generally circular in shape and said temperature stable diamond is generally circular in shape.

Claim 7. A temperature stable diamond product as set forth in claim 1 wherein said product is a cutter for an earth boring bit.

Claim 8. A temperature stable diamond product as set forth in claim 7 in which said product includes more than one initially separate temperature stable diamond elements.

Claim 9. A temperature stable diamond product as set forth in claim 4 in which said gap is between 6 and 30 mils.

Claim 10. A temperature stable diamond product as set forth in claim 1 in which said coating has a thickness of up to about 30 microns.

Claim 11. A temperature stable diamond product as set forth in claim 1 in which said matrix carrier includes a carbide and a binder.

Claim 12. A temperature stable diamond product as set forth in claim 1 in which said matrix carrier is a sintered metal.

Ex. 1003, Page 212

Claim 13. A temperature stable diamond product as set forth in claim 1 in which said coating includes at least one metal selected from the metals of Group IVA, VA, VIA, VIIA and alloys and mixtures thereof.

Claim 14. A temperature stable diamond product as set forth in claim 11 in which said carbide is selected from the group consisting of the carbides of tungsten, chromium, niobium, titanium, tantalum, zirconium and molybdenum.

Claim 15. A temperature stable diamond product as set forth in claim 12 in which said sintered metal is selected from the group consisting of tungsten, nickel, tantalum, molybdenum, cobalt and sintered tungsten body infiltrated by a copper based alloy.

Claim 16. A temperature stable diamond product as set forth in claim 1 wherein said diamond piece is composed of at least 60% by volume of diamond aggregates.

Claim 17. A temperature stable diamond product having a temperature stability up to about 1,200 degrees C. comprising:
a metal matrix carrier selected from the group consisting of tungsten metal powder and the carbides of tungsten, titanium, tantalum and molybdenum,
at least one diamond piece of a predetermined geometrical shape and which is temperature stable up to about 1,200 degrees C. at least some of which have at least one surface contacting at least a portion of said carrier,
said diamond being a polycrystalline diamond composed of at least 60% by volume of diamond aggregates,
each diamond piece being coated with a metal which is a carbide former on at least the portion thereof contacting said metal matrix carrier,
said coating on said temperature stable diamond having a thickness of at least about one micron and including a carbide layer which is of a thickness of less than about 20 microns, said carbide former being a metal selected from the group consisting of Groups IVA, VA, VIA and VIIA metals and alloys and mixtures thereof, and
said temperature stable diamond product being characterized by a chemical bond between said temperature stable diamond and said coating and said coating and the portion of said matrix carrier which is in contact with said coating.

Claim 18. A temperature stable diamond product as set forth in claim 17 which includes a plurality of initially separate temperature stable diamond elements arranged in a predetermined relationship and including opposed surface portions forming a gap therebetween,
said gap being in the range of between less than 1 mil and 500 mils, and
said matrix carrier being located in said gap and contacting at least a portion of the surface portions of said temperature stable diamond elements.

Claim 19. A temperature stable diamond product as set forth in claim 17 in which said product is a saw blade segment.

Claim 20. A temperature stable diamond product as set forth in claim 17 in which said product is a bearing element.

Claim 21. A temperature stable diamond product as set forth in claim 17 in which said product is a dressing tool.

Claim 22. A temperature stable diamond product as set forth in claim 17 in which said product is a cutter adapted to be mounted on an earth boring bit.

Claim 23. A temperature stable diamond product as set forth in claim 17 in which said product is a component of a twist drill.

Claim 24. A temperature stable diamond product as set forth in claim 17 in which said product is a lead bonding tool.

Claim 25. A temperature stable diamond product as set forth in claim 17 in which said product is a single point dressing tool.

Claim 26. In an earth boring bit including a body, the improvement comprising:
at least one cutter element supported by said body,
said cutter element including at least one polycrystalline diamond element which is temperature stable to at least about 1,200 degrees C. supported by a carrier matrix, said temperature stable diamond element having a coating thereon at least on that surface thereof which is in contact with said carrier matrix,
said coating including being of a thickness of at least about 1 micron and including a relatively thin carbide layer adjacent to said surface, and
said temperature stable diamond element being bonded to the contacting portion of said matrix carrier by a chemical bond between said temperature stable diamond element and said coating and said coating and the portion of said matrix carrier which is in contact with said coating.

Claim 27. An earth boring bit as set forth in claim 26 wherein said cuter element includes a plurality of polycrystalline diamond elements.

Claim 28. An earth boring bit as set forth in claim 26 wherein said body includes a surface of matrix carrier material.

Claim 29. An earth boring bit as set forth in claim 27 wherein at least some of said polycrystalline diamond elements are triangular in shape.

Claim 30. An earth boring bit as set forth in claim 26 wherein said polycrystalline diamond element is in the form of a facing on said carrier matrix and chemically bonded thereto.

Ex. 1003, Page 213

Claim 31. An earth boring bit as set forth in claim 26 wherein said cutter element is of a diameter greater than 14mm.

Claim 32. An earth boring bit as set forth in claim 26 in which said bit includes a plurality of said cutter elements.

Claim 33. A temperature stable diamond product having a temperature stability up to about 1,200 degrees C. comprising:
a metal matrix carrier including a powdered material and a binder therefor,
a plurality of diamond pieces of a predetermined geometrical shape each of which is temperature stable up to about 1,200 degrees C. and at least some of which have at least one surface contacting at least a portion of said carrier,
at least some of said diamond pieces being in butting relation with an adjacent diamond piece,
each diamond piece being coated with a metal which is a carbide former on at least the portion thereof contacting said metal matrix carrier and on the surface portion which is adjacent to another diamond piece,
the coating on said diamond piece being at least about 3 microns in thickness, and
said temperature stable diamond product being characterized by a chemical bond between said temperature stable diamond and said coating and said coating and the binder for said matrix carrier which is in contact with said coating.

Claim 34. A method of forming a temperature stable diamond product having a temperature stability of up to about 1,200 degrees C. from at least one initially separate temperature stable diamond element of a predetermined geometric shape, comprising the steps of:
coating at least a portion of the surface of said temperature stable diamond element with a metallic carbide former,
said coating being of a thckness of at least about 3 microns,
assembling with said coating temperature stable diamond element a matrix carrier powder such that the powder contacts at least the coated surface of said temperature stable diamond,
cold pressing said assembled owder and said temperature stable diamond to compact said powder to a volume density of more than 40% to form a cold pressed intermediate,
cold isostatic pressing said cold pressed intermediate to compact said powder to a volume density of greater than 50% to provide an isostatic pressed intermediate, and
heating said isostatic pressed intermediate at an elevated temperature and in a controlled atmosphere for a period of time sufficient form of a chemical bond between the coating on the surface of said temperature stable diamond element and said diamond element and between said coating and the matrix carrier material in contact with said coating.

Claim 35. A method as set forth in Claim 34 wherein heating of said isostatic pressed intermediate includes heating at a rate not greater than about 10 degrees C. per minute in the range of between about 900 degrees C. and 1,100 degrees C. to effect conversion of a portion of said carbide forming coating to a carbide.

Claim 36. A method as set forth in Claim 34 wherein said matrix carrier powder is a mixture of a carbide and a binder therefor.

Claim 37. A method as set forth in Claim 34 wherein said assembly step includes the assembly of a plurality of coated temperature stable diamond elements in a predetermined configuration.

Claim 38. A method as set forth in Claim 37 wherein said elements are assembled such that there is a gap between adjacent elements.

Claim 39. A method as set forth in Claim 37 wherein said elements are assembled such that at least some of them are in butting relation.

Claim 40. A method as set forth in Claim 34 wherein said metallic carbide former is a metal selected from the group consisting of the metals of Group IVA, VA, VIA,VII and alloys and mixtures thereof.

Claim 41. A method as set forth in Claim 34 further including the step of assembling said isostatic pressed intermediate to a powdered support material, and
said heating operation including heating the isostatic pressed intermediate and said powdered material to effect the chemical bonding and to effect formation of said powdered material into a support for said temperature stable synthetic diamond element.

Ex. 1003, Page 214

Fig. 1

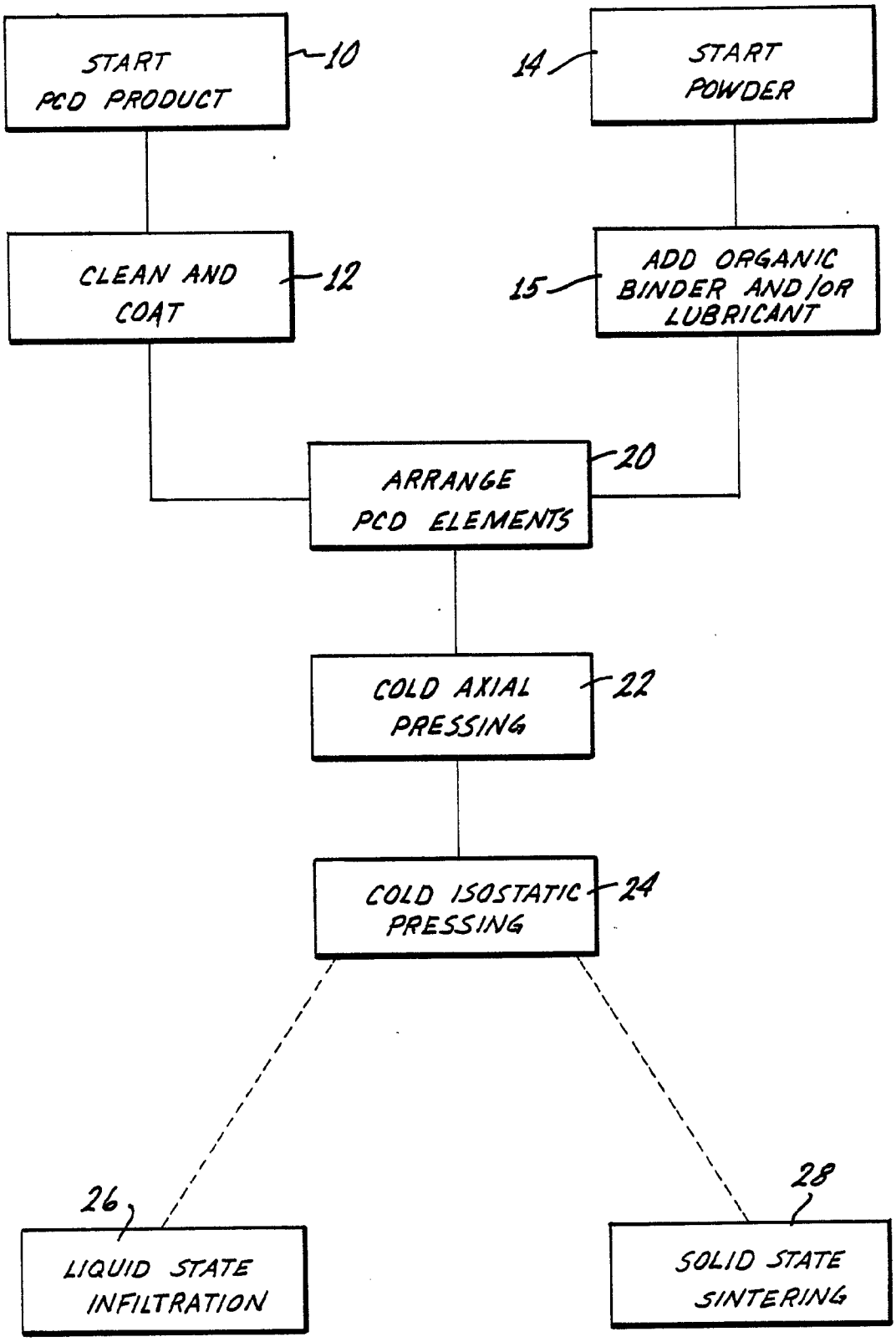

START PCD PRODUCT
10
14
START POWDER
CLEAN AND COAT
12
15
ADD ORGANIC BINDER AND/OR LUBRICANT
ARRANGE PCD ELEMENTS
20
COLD AXIAL PRESSING
22
COLD ISOSTATIC PRESSING
24
26
LIQUID STATE INFILTRATION
28
SOLID STATE SINTERING

Fig. 2

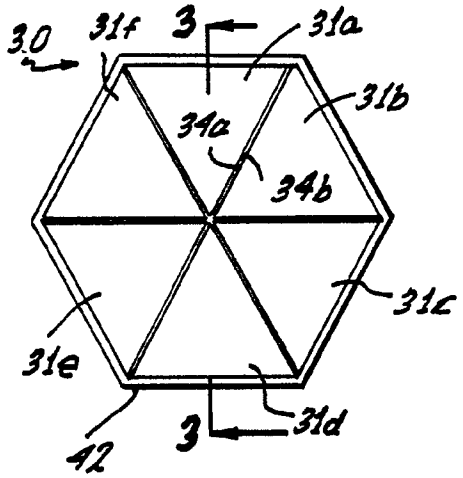
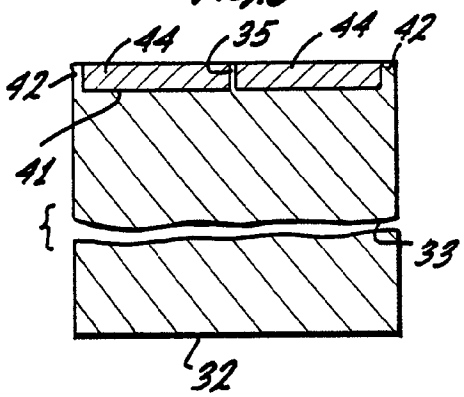


Fig. 3



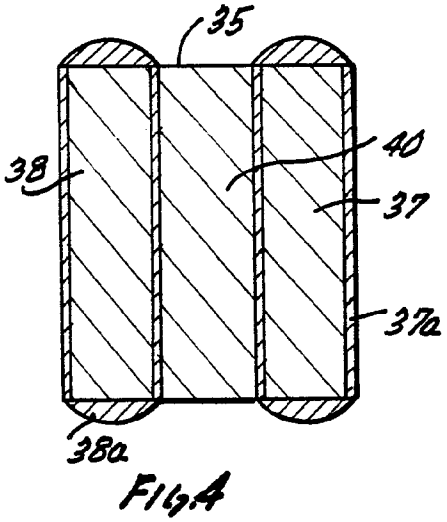


Fig. 4

Fig. 5

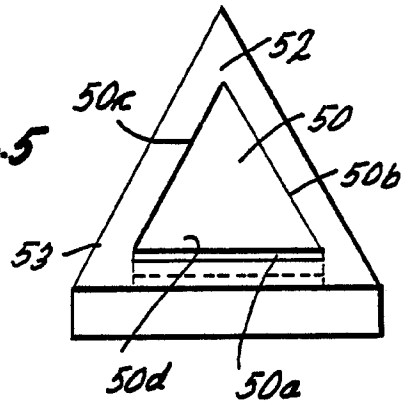
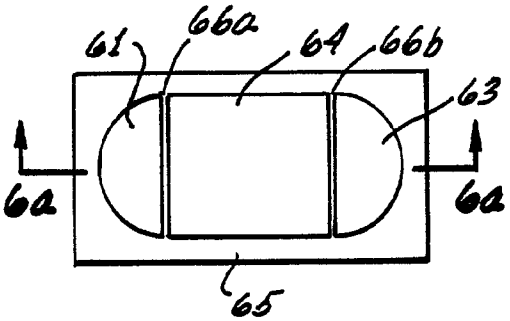


Fig. 6



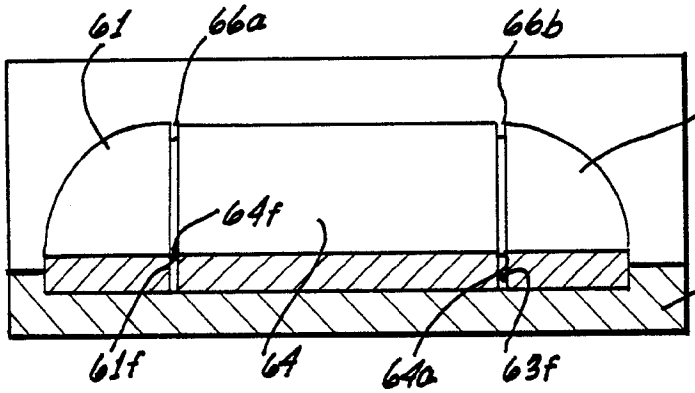
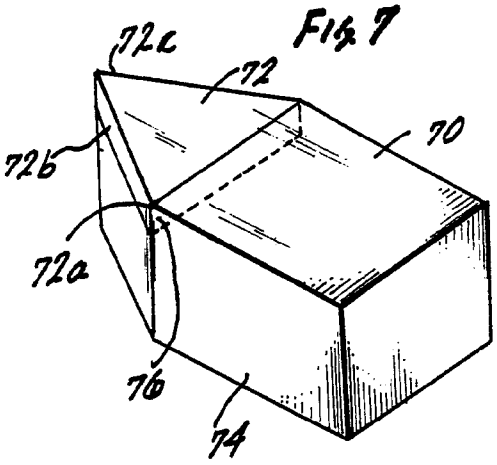


Fig. 6a

BAD ORIGINAL

Fig. 7



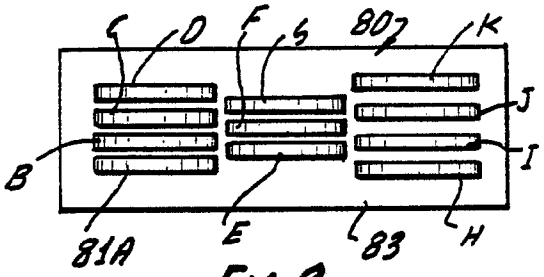


Fig. 8

Fig. 8a

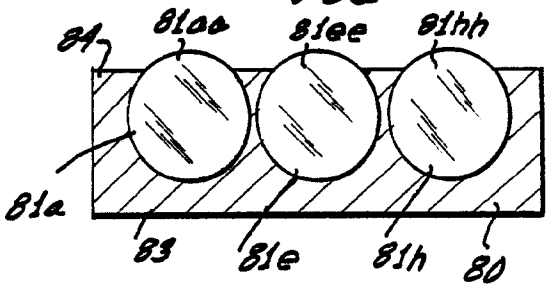


Fig. 9

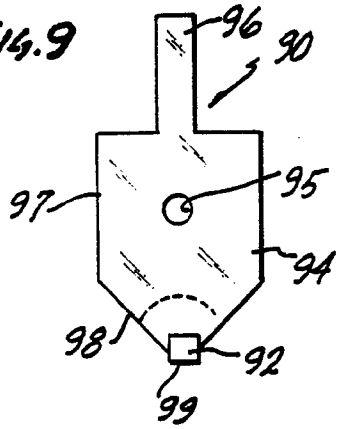
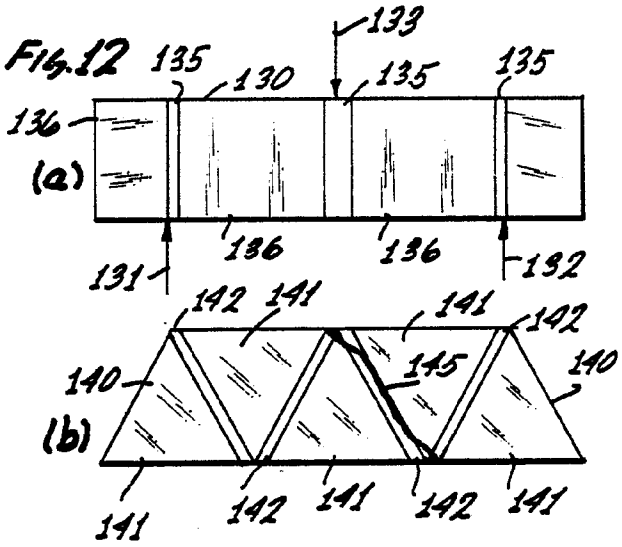


Fig. 12



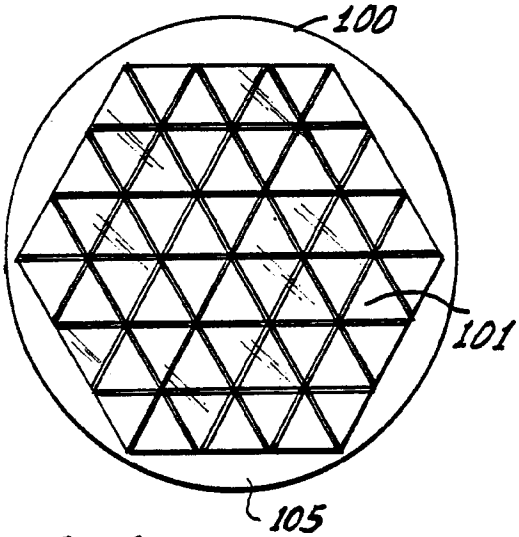
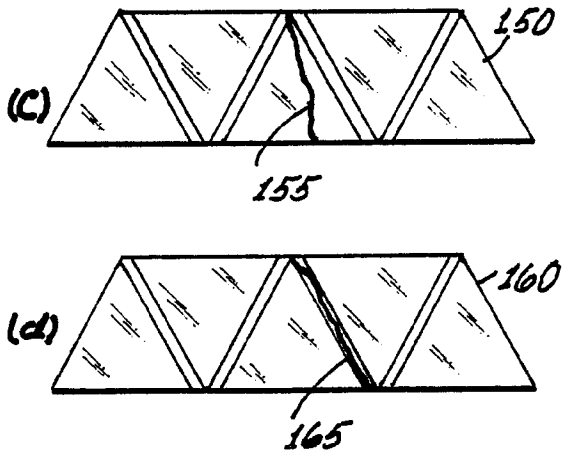


Fig. 10



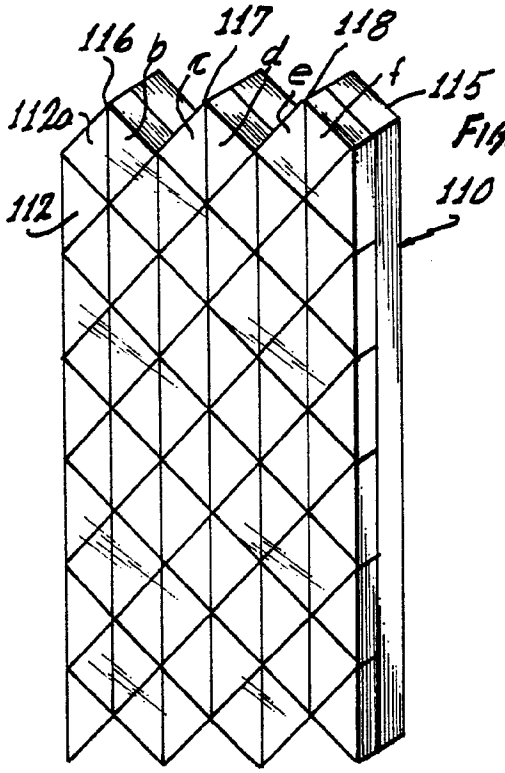


Fig. 11

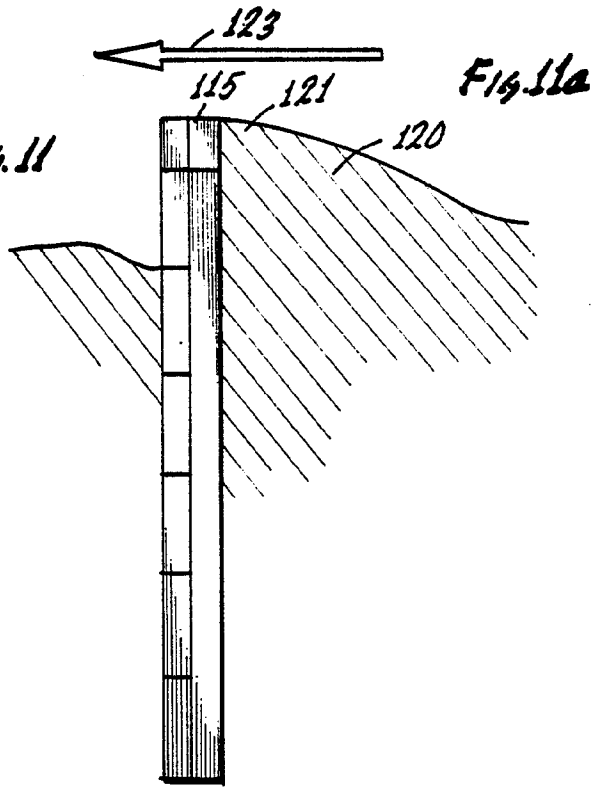


Fig. 11a

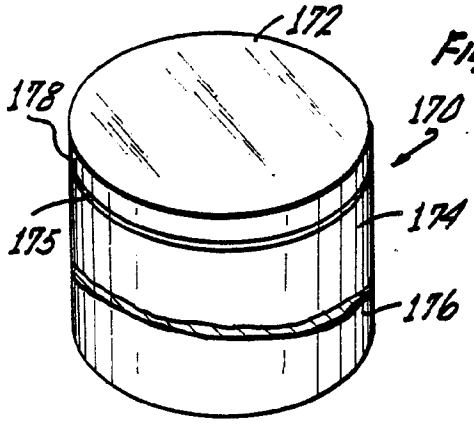


Fig. 13

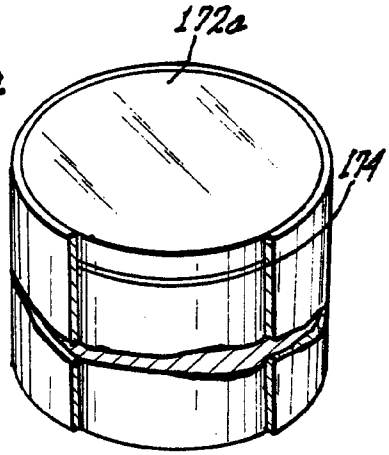


Fig. 13a

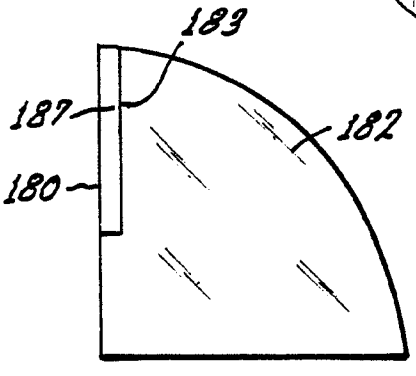


Fig. 14

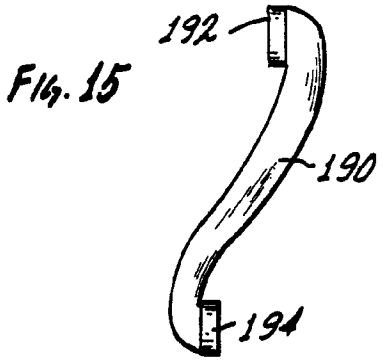


Fig. 15

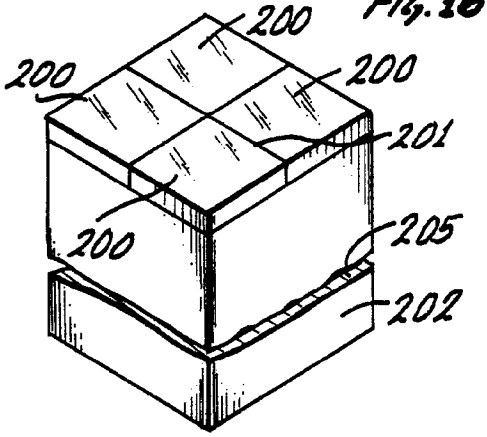


Fig. 16

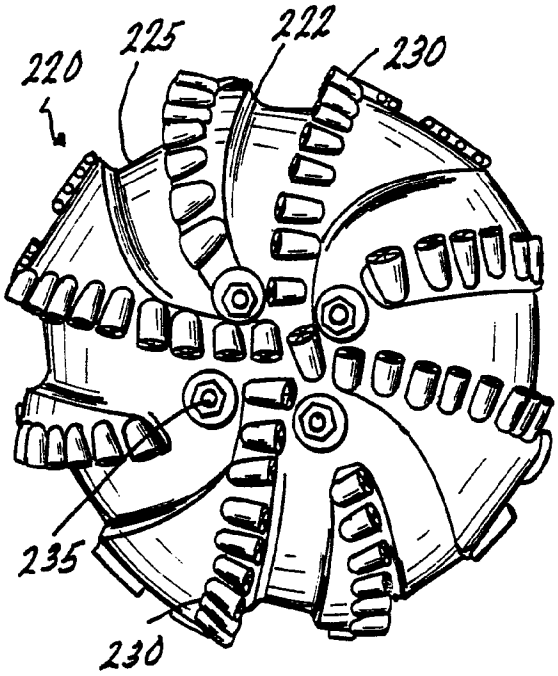


Fig. 17

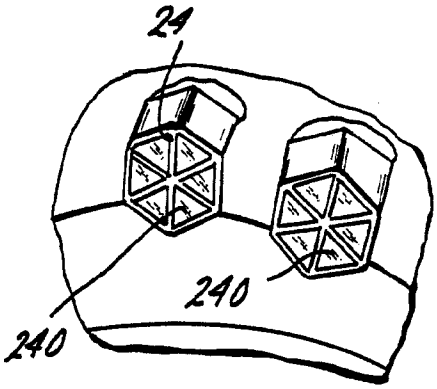


Fig. 18

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(54) **Flexible abrasives.**

(57) A method of forming an abrasive member comprises fixedly attaching a metal film to one surface of a flexible sheet, applying a mask of plating resistant material to the exposed surface of the metal film, said plating resistant material having a multitude of discrete openings therein, and electrodepositing metal through said discrete openings onto said metal film in the presence of particulate abrasive material so that the material adheres directly to said metal film and the abrasive becomes embedded in the metal deposits.

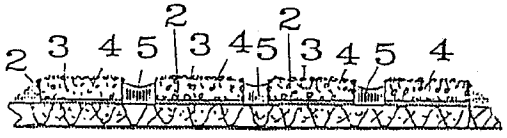


Fig. 1

EP 0 280 657 A2

Description

Flexible abrasives.

This invention relates to a flexible abrasive member particularly suitable for abrading, grinding, smoothing, and finishing operations on stone, glass and other materials in heavy-duty applications.

United States patent no.4,256,467, issued August 17, 1981, to Ian Gorsuch discloses a flexible abrasive member comprising a flexible non-conductive mesh carrying a multitude of nickel deposits in which abrasive material, such as diamond grit, is embedded.

According to the Gorsuch patent, the flexible abrasive member is manufactured by first laying a sheet of flexible non-conductive mesh material onto a smooth electrically conductive surface, suitably masked to expose only those surface portions where electrodeposition is desired, so that the mesh material is in immovable relationship with the conductive surface. Nickel is then electrodeposited onto the exposed portions of the smooth surface through the mesh material in the presence of abrasive material so that the abrasive material becomes embedded in the metal layer and the mesh becomes embedded in the nickel deposits. Finally. the mesh is stripped from the electrically conductive surface and cut into the desired shape.

There are, however, a number of disadvantages associated with the process. The preparation of the cylinder prior to each deposition is expensive and complex. The process is slow and can only operate on a batch basis because a sheet of flexible mesh material of specific size must be attached to the cylinder, applied under tension, and maintained in immovable relationship therewith.

More importantly, the product produced by the Gorsuch process is structurally weak and only suitable for light-duty operations, such as lens grinding. If the product is used in heavier duty applications, such as abrading belts, the mesh has to be bonded to a suitable substrate. The heat generated during the abrading operation makes it difficult to provide a satisfactory bond, and difficulties have been experienced due to the belts breaking, the nickel deposits chipping off the intrinsically weak mesh, and delamination of the belts.

Our co-pending Canadian application no. 518,201, filed on September 15. 1986, describes a method which overcomes the problems relating to the preparation of the conductive cylinder and permits continuous operation of the process. In this method the mask is applied directly to the mesh, which is rendered conductive, instead of to the conductive surface. When a mesh is employed, however, the abrasive member must still be bonded to a strong substrate for heavy-duty applications.

An object of the invention is to provide a process for producing a flexible abrasive member, which is faster and more economical to operate than the one set forth in co-pending Canadian application No. 518. 201, and which lends itself readily to automation. Furthermore, the invention can provide, by such a process, abrasive sheets which can be made into as pads, discs, or belts, capable of operating at higher abrading speeds and presenting a clean surface with clearly defined spaces between the metal deposits. This gives a more efficient abrading action and requires less metal or abrasive material, making the product more economical to manufacture. More significantly, the process can produce directly an abrasive sheet for use in heavy-duty applications without the need for subsequent lamination to a backing material. The abrading member produced by the inventive process dissipates heat efficiently and thus has a longer life.

According to the present invention there is provided a method of making an abrasive member, comprising fixedly attaching a metal film to one surface of a flexible sheet, applying a mask of plating resistant material to the exposed surface of the metal film, said plating resistant material having a multitude of discrete openings therein, and depositing metal directly through said discrete openings onto said metal film in the presence of a particulate abrasive material so that the metal adheres directly to said metal film and the abrasive material becomes embedded in the metal deposits.

The deposition is preferably carried out by electrodeposition although electroless deposition can be employed. The preferred metal for the film is copper and for the metal deposits nickel, although other combinations can be employed.

The abrasive member produced by this process is useful per se. However, in order to reduce the heat buildup in the member during use and thus increase its efficiency and life expectancy, in a preferred embodiment of the present invention , the mask is stripped from the sheet after electrodeposition of the metal to expose the metal film, and the metal film between the discrete metal electrodeposits is etched away to expose the sheet.

The mask can be applied to the metal film by coating with a layer of a photopolymer and exposing the photopolymer to ultra violet light through a screen defining the openings to decompose the polymer. The coating is then developed, preferably by treatment with an alkali, such as sodium hydroxide. The photopolymer can be a dry film photopolymer, such as a dry film photopolymer supplied under the name Riston by Dupont, a laminar dry film resist supplied by Dynachem, or dry film resist supplied by Herculestic, or a liquid film resist supplied by Kodak, GAF, Dynachem, Dupont, or Fuji film. The photopolymer is desirably exposed to ultra-violet light. However, any other type of radiation which degrades the polymer such that it can be developed is suitable.

In a further aspect of the invention there is provided a method of making a flexible abrasive member, comprising applying to an electrically conductive metal surface of a flexible substrate a coating of a photopolymer, exposing the photopolymer to light through a screen having discrete openings to decompose said polymer, developing the coating to provide a mask having a multitude of discrete openings therein, and

Ex. 1003, Page 221

electrodepositing metal directly through said discrete openings onto said metal surface in the presence of a particulate abrasive material so that the metal adheres directly to said metal surface and the abrasive becomes embedded in the metal deposits. As before, in this method it is desirable after electrodeposition of the metal to strip the mask from the substrate sheet to expose the metal surface and etch the metal surface between the deposits to expose the substrate.

Alternatively, the mask may be applied by silk screening, in which case the mask may be made of ultra-violet light curable or thermally curable inks such as infra-red heat curable inks. Such curable plating resists and etching resists may be supplied by McDermid Inc., Dynachem and M&T Chemicals.

The flexible substrate is preferably in the form of a woven fabric, but it may be fibre glass epoxy laminate of the type used for printed circuit board applications, supplied by Westinghouse and GE, when it is desired to make abrasive pads and disks. The sheet may also be formed of a phenolic resin, such as a phenol formaldehyde resin or it may be a polyester fibre glass laminate also supplied for printed circuit board applications. Such sheets suitably have an overall thickness of about 8 to 12 mils.

For forming a flexible abrasive member suitable for use as an abrasive belt, a copper clad, fibre free resin system such as that supplied under the trademark Kapton (by Dupont), which is used for flexible printed circuits may be used. However, in a particularly desirable embodiment of the present invention, the sheet is formed of a strong woven fabric on which the metal film is deposited. A particularly suitable fabric is made of polyaramid yarn, such as p-poly (phenylene) terephthalamide yarn, which is supplied under the trademark Kevlar.

The metal film is fixedly attached to the surface of the sheet and is laminated as a film or deposited by electroless plating, vapour deposition, sputtering, or electrochemical deposition, such as electroplating. The metal may be any electrically conductive metal such as copper, aluminum, nickel, steel, rhodium or gold, but is preferably copper. Suitably the metal film has a thickness from 3/20 to 14 thousanths of an inch preferably 7/10 to 2.8 thousanths of an inch.

The abrasive material is a conventional particulate abrasive such as diamond grit or cubic boron nitride, and preferably industrial diamond. The metal can be any metal which can be deposited from a suitable bath by electrodeposition or electroless deposition and is preferably nickel or copper, more preferably nickel.

In a preferred embodiment, the sheet with the metal film attached thereto is continuously passed through an electrolytic bath to form a cathode, the anodes of which are formed by the metal, whereby the metal is continuously deposited in the discrete openings and during said electrodeposition the particulate abrasive is released into the bath. In order to ensure that the sheet is present in the bath as a cathode, it is connected to a source of negative potential. The sheet is preferably in contact with a smooth non-conductive surface such as a plastic surface, in the bath, which is suitably a nickel sulphamate bath. The mask, which is in the form of a very thin sheet a few thousanths, e.g. 3 to 4 thousanths of an inch thick, defines a lattice with a large number of openings, for example 1/16 of an inch in diameter.

After removal from the bath, the sheet is stripped and etched with alkaline solution. A further very significant feature of the invention is that the Kevlar® sheet bearing the diamond-embedded nickel deposits is coated with a resin, such as a two-part polyurethane resin sold under the trade designation UR 2139X-1 and UR 2139X-1A by Elecbro Inc. After stripping and etching, the Kevlar sheet consists of a multitude of nickel nodules carried by copper segments bonded to the Kevlar fabric. The nodules hold quite well onto the fabric during use, but their tendency to chip off can be dramatically reduced by coating with the polyurethane resin. This fills the interstices between the nodules, thereby reducing the shearing forces as the fabric is moved over the working surface. It has been further found that the use of a filled resin, i.e. a resin filled with a solid particulate material, particularly silicon carbide powder further inhibits the lateral movement of the nodules reducing even further their tendencies to chip off.

In a still further feature of the invention, the nickel nodules are given predetermined characteristic shapes. In one embodiment, the nodules have a crescent-moon shape. This has the effect of minimizing the use of diamond without impairing the abrasive properties. The removal of abraded material can also be assisted by careful design of the shapes of the nodules. The photographic and silk-screen processes described above lend themselves particularly well to the fabrication of shaped nodules.

The invention will now be described in more detail, by way of example only, with reference to the accompanying drawings, in which:

Figure 1 shows in cross-section a short length of Kevlar fabric carrying diamond-bearing nickel deposits;

Figure 2 shows a laminated substrate bearing a surface mask defining a regular pattern of crescent-shaped holes;

Figure 3a shows a detail of one of the shaped holes; and

Figure 3b shows a detail of a group of holes.

Example 1

A copper clad, fibre glass epoxy laminate, sold for printed circuit board applications by Westinghouse or GE, having a thickness of 8 mils to 12 mils had its copper surface mechanically cleaned with a scrubber. A dry film photopolymer supplied by Dynachem was laminated to the copper surface at about 220°F and then exposed to ultra violet light through an apertured screen defining the holes with a Scannex exposure unit. The protective Mylar sheet, which comes with the dry film, was removed and the exposed film developed with

Ex. 1003, Page 222

potassium hydroxide solution.

The product bearing the photographically formed mask was then treated in a commercial electrolytic nickel sulphamate bath, supplied under the trademark SNR 24 by Hansen, operating at 170 amps and 9 volts DC at a temperature of 140-C.

The flexible abrasive member leaving the bath, though suitable for cutting and use without further treatment, was treated with a Chemalex stripper to strip off the dry photofilm and then etched with alkaline-based copper etching solution supplied by Hunt Chemicals, by spray etching.

The abrasive member had a clear translucent aesthetically pleasing appearance with well defined protuberances containing the diamond abrasive and substantially no intermediate diamond-containing metal between the protuberances. This is in contrast to the product obtained according to the process described in our copending Canadian application no. 518.210, which displayed a more untidy appearance and tended to have metal and diamond particles present between the protuberances. The clean appearance of the abrasive member has consumer appeal, particularly in the do-it-yourself market, but it also provides a more efficient abrading member. In addition it makes the product cheaper to manufacture as there is less waste of metal and abrasive material.

The presence of the copper layer has a number of advantages: It provides a smooth surface on which deposition can take place, which is important to prevent break-through of the mask and to permit even distribution of diamond grit. When the mask and copper bridging regions between the nodules are removed, the remaining copper segments under the nodules, by which the nodules are attached to the substrate, form part of the protruberances. To achieve a protuberance of given height, the electrodeposition time can be shortened due to the presence of the underlying metal segments. The metal deposits should stand proud of the substrate by an amount sufficient to permit adequate removal of abraded material and avoid undue wear.

Example 2

A 10 ounce Kevlar fabric 24 × 24 inches in size was subjected to electroless copper plating by passing through the standard electroless copper plating process known under the trademark Ethone System CU 701. Such a process is conventionally used for producing printed circuit boards with a copper coating of a thickness of 80 to 120 microns deposited on both sides.

The copper coated fabric was then subjected to masking and nickel and diamond deposition by the method described in example 1. The copper clad sheets can be treated in a manner similar to the fibre glass epoxy laminate.

Upon removal from the electrolytic bath, and after stripping and etching, the Kevlar sheets were coated with polyurethane resin to fill the interstices between the nickel nodules. The sheets were then cut and formed into belts after the reverse surface was covered with a rubberized epoxy resin system to prevent fraying and cutting of the belt.

Example 3

A Sheet of Barrday F-2160/175 Kevlar 29-1500 denier scoured fabric was impregnated with BO800 LOMOD™ copolyester elastomeric resin. The resin was in liquid form and applied with rollers. A layer of 10oz. copper foil was then applied to the impregnated sheet and the assembly maintained in a press under 250 psi pressure for approximately one hour at room temperature.

Upon removal from the press, the exposed surface of the foil was mechanically scuffed to improve adhesion. A plating-resistant mask with a multitude of openings was then applied to the copper foil in the manner described above, and the laminate placed in an electrolytic deposition bath. Nickel was deposited onto the copper foil through the openings in the mask with diamond particles sprinkled into the tank during the electrodeposition.

The mask was stripped from the foil and the intervening copper etched away to leave upstanding diamond-bearing nickel deposits lying on small copper discs. The interstices between the nickel deposits were then filled with a flexible polyurethane resin, such as Elecbro UR 2139X-1 and UR 2139X-1A, so that the abrasive product presented a continuous surface on the abrasive side. As discussed above, the use of a resin coating has the important advantage that during use the tendency of the deposits to be chipped off the backing fabric is minimized. Other flexible resins can be employed.

The LOMOD™ resin substantially enhances the properties of the fabric. It prevents degradation of the fabric due to fraying and scuffing during heavy industrial use without impairing the flexibility of the belt. It has good physical, mechanical, thermal, electrical and flame-resistant properties.

Of equal significance is the fact that the LOMOD™ has sufficient strength to permit lamination of the copper foil to the fabric and good retention of the residual copper segments after stripping and etching during use.

The advantage of this technique is that unlike the copper spray, the laminated foil has a smooth surface. The uniformity of the abrasive can be accurately controlled and the tendency of the electrolytic deposits to break through the masked portions minimized.

The physical data for these LOMOD™ resins are as follows:

Ex. 1003, Page 223

Property	LOMOD BO800	LOMOD BO852
Specific Gravity	1.23	1.30
Flexural Modulus,psi	85,000	95,000
Tensile Strength,psi	3,350	3,475
Tensile Elongation,% @ Break	250	125
Dielectric Strength	415	405

Belts, discs and other types of abrasive product made with LOMOD℠ impregnated sheets in the manner described have exceptional strength and abrasive properties.

EXAMPLE 4

A sheet of 10 ounce KevlarTM [a trade mark of DuPont for p-poly-(phenylene)terephthalamide yarn] 24 by 24 fabric was bonded under heat and pressure with LomodTM (available from General Electric) resin to a copper sheet having a surface density of one ounce per square foot. The surface of the copper sheet was cleaned and scrubbed with an abrasive brush in a scrubbing machine.

The cleaned laminate was passed through a dry film laminator made by Thiokol/Dynachem Company (Model 30) to apply a Riston (a trade mark of DuPont) photo-resist film (an alternative is Dynachem film).

Laminate with the applied photo-resist film was placed in a Scannex II exposure unit with a screen defining the desired pattern of crescent-shaped holes. The screen can be produced photographically.

After exposure to ultra violet light, the image was developed and the protective Mylar film, applied by the laminator, removed.

The electrodeposition took place in the presence of diamond grit in an electrolytic bath in a similar manner to that described above to form crescent-shaped diamond-embedded nickel pellets. Other abrasive particulate material, such as cubic boron nitride, can be employed,

After electrodeposition, the mask and exposed copper were removed with an alkaline stripping and etching solution.

The product was then roller coated with polyurethane protective resin, having the trade designation UR 2139X-1 and UR 2139X-1A by Elecbro Inc, to fill the interstices between the nickel deposits.

The sheet was then cut into strips and the strips formed into belts ready for use as an abrasive.

Instead of using photo-resist materials to form the mask, the mask can be applied by a silk screening process. In this case, the mask is made of enplate UR 2311B silk screening material which is ultra-violet cured after application in the silk-screening process.

Referring to Figure 1, a length of Kevlar fabric 1 is impregnated with Lomod℠ and has bonded thereto copper discs 2. These discs were applied as a copper foil in the manner described above but are all that remain of the original foil after the stripping and etching operation described above.

The nickel nodules 3 are electrolytically deposited on the copper discs 1 and have diamond particles 4 embedded therein.

The voids between the nodules 3 are filled with polyurethane resin 5 in the manner described above. The resin 5 reduces lateral movement of the nodules 4 and has a profound effect on their tendency to chip off during the abrasion process. The resin has a greater effect than would result merely from its adhesive action due to the way in which it stabilizes the nodules in operation. One of the factors inhibiting widespread use of this type of abrasive product in the past has been the difficulty of retaining the nodules on the substrate in the hostile environment of an industrial abrading machine.

The sheets are cut into strips and formed into belts by making a butt joint and applying a tape on the rear side with Bostik 7070℠ adhesive. To minimize wear, the rear side should be slightly scuffed in the region where the tape is to be located so as to avoid a noticeable bump when the tape is in place. The edges should desirably be cut in a wavy line to reduce lateral movement.

The laminate 11, shown in figure 2, comprises a Kevlar℠ fabric resin bonded to a copper sheet 12 covered

with a surface mask 13 of photo-resist material defining crescent-shaped holes 14 through which electrodeposition occurs. The laminate shown in Figure 2 is subsequently placed in an electrolytic tank to permit deposition of nickel in the presence of diamond grit through the shaped holes 14. This process produces crescent-shaped pellets at the locations of the holes with diamond grit embedded in the nickel.

After removal from the tank, the mask and exposed copper are striped from the Kevlar™ to leave a sheet consisting of a regular pattern of crescent-shaped pellets firmly attached to the Kevlar™ backing. Each pellet consists of an electrodeposit of nickel bearing the diamond grit carried on a crescent-shaped segment of copper bonded to the underlying fabric.

Figure 3a shows in detail the shape of the holes. The crescent-shapes are defined by overlapping circles of slightly different radii. Figure 3b shows how the holes are arranged in a symmetrical arrangement.

The manufactured sheet is subsequently cut into strips, which in turn are formed into belts. The crescent-shaped modules make the belts unidirectional, in that the convex edge has to face the direction of movement a of the belt. This is generally a significant disadvantage.

The use of crescent-shapes permits significant savings in diamond grit, since the surface area of the pellets is less than for circular pellets, without deterioration in the abrasive properties, and furthermore the removal of abraded matter is improved.

The holes can have other shapes. For example, honeycomb shapes provide the belt with greater rigidity.

The spacing and size of the pellets can be varied to fine tune the properties of the abrasive product according to the intended application. A much greater degree of control can be exercised over the abrasive properties than was previously possible. For rough grinding purposes, the pellets are spaced further apart and larger diamonds employed. For smooth grinding applications, the pellets are brought closer together and smaller diamonds used.

Kevlar™ is a particularly useful material for making abrasive belts. For disks on the other hand, the copper foil can be bonded onto fiberglass or other semi-rigid material and the fiberglass then laminated onto a firm backing, for example a polyester backing.

Claims

1. A method of forming an abrasive member, characterized in that a metal film is fixedly attached to one surface of a flexible sheet, a mask of plating resistant material is applied to the exposed surface of the metal film, said plating resistant material having a multitude of discrete openings therein, and metal is electrodeposited through said discrete openings onto said metal film in the presence of particulate abrasive material so that the material adheres directly to said metal film and the abrasive becomes embedded in the metal deposits.

2. A method according to claim 1 characterized in that after electrodeposition of metal, the mask is stripped from the sheet to exposed the metal film and the metal film between the discrete metal electrodeposits is etched away to expose the sheet.

3. A method according to claim 1 or 2 characterized in that the voids between the metal deposits are at least partially filled with resin in particular polyurethane resin, to reduce lateral movement of the deposits.

4. A method according to any of claims 1 to 3 characterized in that the mask is applied to the metal film by coating the film with a layer of photopolymer and the photopolymer is exposed to light through a screen having discrete openings therein to decompose said polymer, said coating then being developed to remove the decomposed polymer and expose the underlying metal film.

5. A method according to claim 1 characterized in that the mask is applied by silk-screening through a mesh.

6. A method according to claim 5 characterized in that the mask is made of an ultra-violet light curable or thermally curable ink.

7. A method according to any of claims 1 to 6 characterized in that the metal film is laminated onto the sheet or is deposited by electroless plating, vapour deposition, sputtering, or electrochemical deposition onto the sheet.

8. A method according to any of claims 1 to 7 characterized in that the sheet is a flexible woven fabric, in particular polyaramid yarn or p-poly (phenylene) terephthalamide yarn.

9. A method according to any of claims 1 to 8 characterized in that the mask defines a multitudinous pattern of holes having a predetermined shape, in particular a crescent shape, whereby said metal deposits form shaped metal pellets.

10. A method according to any of claims 1 to 9 characterized in that said metal film is copper foil, said substrate is a polyaramid fabric resin bonded to sail copper foil, said electrodeposited metal is nickel, and said abrasive particulate material is diamond grit.

11. A method according to any of claims 1 to 10 characterized in that the flexible sheet is coated with a copolyester elastomeric resin.

12. A method according to claim 11 characterized in that the metal film is bonded to the fabric with said copolyester resin under pressure.

13. A method according to claim 3 characterized in that the resin is filled with a particulate solid filler

Ex. 1003, Page 225

material.

14. A method according to claim 3 characterized in that the resin is filled with silicon carbide powder.

15. An abrasive member characterized in that it comprises a flexible sheet having a multitude of discrete metal protuberances fixedly attached to one surface thereof, each of the protuberances comprising a lower thin layer of a first metal fixedly attached to the sheet and an upper electrodeposited layer of a second metal having a particulate abrasive material embedded therein.

16. An abrasive member according to claim 15 characterized in that the first metal is copper, the second metal is nickel, and the particulate abrasive material is diamond grit.

17. A flexible abrasive product characterized in that it comprises a flexible substrate, in particular a polyaramid fabric, having a multitude of metal pellets bearing particulate abrasive material attached thereto, said pellets being arranged in a pattern and having a predetermined shape, said pellets comprising electrodeposited metal on copper segments resin-bonded to said substrate.

18. A flexible abrasive product according to claim 17 characterized in that said segments are bonded to said fabric with copolyester resin.

19. An abrasive product characterized in that it com prises a multitude of discrete metal pellets bearing particulate abrasive material, said pellets being fixedly attached to a flexible substrate and the voids between the pellets being at least partially filled with flexible filler material to reduce the shearing forces on the pellets during use.

20. An abrasive product according to claim 19 characterized in that the filler material is polyurethane resin.

21. An abrasive product according to claim 20 characterized in that the resin is filled with a particulate solid filler material

22. An abrasive product according to claim 20 characterized in that the resin is filled with silicon carbide powder.

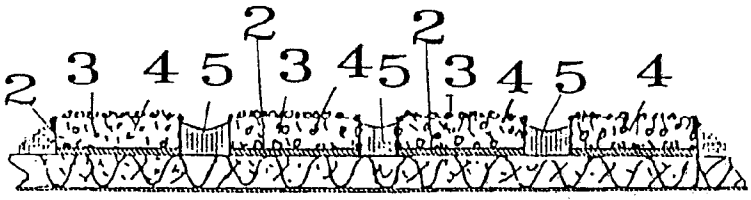


Fig. 1

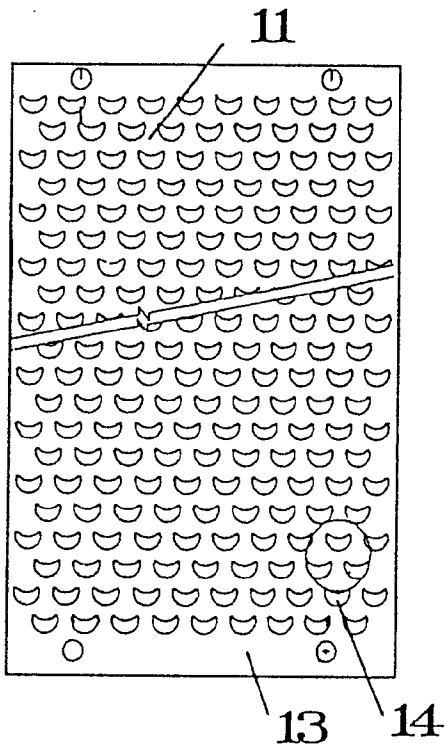


Fig. 2

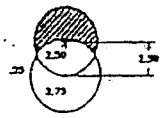


Fig. 3a

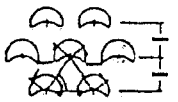


Fig. 3b

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(54) Abrasive sheeting having individually positioned abrasive granules.

(57) Abrasive sheeting can produce fine finishes at surprisingly high cutting rates when its abrasive granules are individually positioned in a predetermined pattern, with an uncoated portion of virtually every granule protruding from the surface of the binder layer. Each of the abrasive granules preferably is a spherical composite of a large number of abrasive grains in a binder. For example, abrasive grains having a mean dimension of about 4 µm can be bonded together to form spherical abrasive granules of virtually identical diameters, preferably within a range of from 25 to 100 µm.

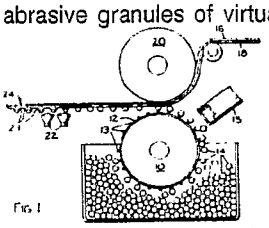


Fig. 1

EP 0 331 344 A2

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ABRASIVE SHEETING HAVING INDIVIDUALLY POSITIONED ABRASIVE GRANULES

Background of the Invention

Field of the Invention

The invention concerns abrasive sheeting or coated abrasives of the type having a backing which usually is flexible and carries abrasive grains or granules embedded in a binder layer and usually is flexible.

Description of the Related Art

At least as early as 1905, as taught in U.S. Patent No.794,495 (Gorton), it was discovered that unbroken "abrading surfaces soon become clogged and gummed by the fine grit and minute particles worn from the abracing surface and from the metal-work (or other work) being held to the abrading-surface" and that these materials can be carried away more effectively by applying the abrasive grit in a dot pattern. In U.S. Patent No. 1,657,784 (Bergstrom), a dot pattern is obtained by applying a binder to a backing, applying abrasive grains or granules to the binder, and then scraping off portions of the binder and adhered abrasive granules. More commonly, an adhesive is applied in a dot pattern so that the abrasive granules adhere only in that pattern. In U.S. Patent No. 4,317,660 (Kramis et al.), the adhesive dot pattern is obtained by applying the adhesive through stencil holes. After attracting a large number of abrasive granules to each dot, the abrasive granules are covered with a size coat.

In abrasive sheeting that has appeared on the market with such a dot pattern, there is a heap or a pile of abrasive granules at each dot, and that heap of granules is covered with a size coat. Because the height of each heap is more or less random, the individual heaps and the individual granules of each heap are loaded differently and hence produce uneven cutting. The size coat interferes with the cutting action of the abrasive granules and also results in uneven cutting due to variations in the extent to which the size coat covers the abrasive granules.

Uneven cutting of prior abrasive sheetings also emanates from irregular sizes and shapes of their abrasive granules. This effect has been minimized by using spherical granules of equal size. Another type of spherical abrasive granule consists of a large number of abrasive grains in either an organic or inorganic matrix. Because of their uniformity, such composite granules renew themselves as they wear away, thus maintaining relatively uniform abrasive cutting action for longer periods of time than has been possible when using abrasive sheeting coated with irregular abrasive granules. Such composite abrasive granules are disclosed in U.S. Pats. No. 3,916,584 (Howard et al.); No. 4,112,631 (Howard); and No. 4,541,842 (Rostoker). U.S. Pat. No. Re. 29,808 (Wagner) shows hollow balls consisting of abrasive grains bonded onto the outer surface of a friable matrix. Even though Fig. 1 of the Wagner patent shows those spheres uniformly positioned in a binder layer, the patent says that the hollow balls "are mixed with a bonding material and brought into the shape of the grinding body, after which the bonding material is allowed to harden out, and during the production of an abrasive belt the hollow bodies are bonded in the usual manner to a base material" (col. 6, lines 43-48). In Example 1, ready-prepared abrasive grain balls are uniformly strewn onto a layer of resin on a cotton twill fabric.

Even when prior abrasive sheetings employ composite spherical granules of equal size, sheetings made at one time tend to have different cutting rates than do those made at other times due to variables in their manufacture. Accordingly, when prior abrasive sheetings have been used under numerical or robotic control, it has been necessary to test the cutting rate of each jumbo roll before putting it to use.

Abrasive sheeting ordinarily is manufactured in great lengths that are wound into rolls for storage and shipment. Eventually, the sheeting is die-cut into desired sizes and shapes. For example, it may be cut to form daisy pads that are used to polish lenses. In doing so, the die contacts the abrasive particles which cause its cutting edge to become dull and to require resharpening within a short period of time.

Other Prior Art

Ex. 1003, Page 229

Abrasive tools are often made by handsetting abrasive granules such as diamonds, but such granules are quite large. It is believed that handsetting has never been employed in abrasive sheeting that has appeared in the market.

U.S. Pat No. 4,536,195 (Ishikawa) concerns a method of making grinding stones, the abrasive grains of which are distributed in a controlled manner so that the load working on each grain is even, making the stone more efficient and of longer life. In a first variation of the Ishikawa method, an electrically conductive pattern is formed on a resinous binder sheet which is then immersed into an electroplating path containing metallic ions mixed with abrasive grains that are attracted to the pattern. The electrically conductive pattern may be formed by photoetching or printing techniques. A number of the abrasive-bearing sheets are placed in layers and molded into a grinding stone by warm or hot pressure molding. Another variation is the same as the first except that the sheet is metallic and a surface is masked so that the abrasive grains are attracted only to the unmasked areas. An example of distribution of abrasive grains on the surface of a grinding stone is shown in Fig. 15 wherein the grains are located in rows and uniformly spaced from adjacent grains.

Summary of the Invention

The invention provides abrasive sheeting or coated abrasive that can produce finer finishes at faster cutting rates than could be attained in the prior art. Furthermore, the novel abrasive sheeting is believed to provide a more predictable cutting rate, thus minimizing the need to test its cutting rate before using it in robotic or numerically controlled machines.

Briefly, the abrasive sheeting of the invention has a backing carrying a binder in which abrasive granules are strongly bonded and lie substantially in a plane and at a predetermined lateral spacing. The granules should be of substantially equal size, i.e., the mean dimension of 90% of the granules should differ by less than 2:1. Each of the abrasive granules should pass a screen with 300 μm openings, because substantially larger granules would not provide the fine finishes desired in uses for which the novel abrasive sheeting is intended.

The granules preferably are in a predetermined pattern (or patterns when using granules of differing sizes or types) that provides spaces between the granules of sufficient width to carry off detritus. In a preferred pattern, single granules are uniformly spaced and aligned in rows extending both longitudinally and transversely (i.e., in the X and Y directions). In another preferred pattern, abrasive sheeting having circular rows of uniformly spaced granules can be cut into discs, the centers of which are concentric with the circular rows.

Preferably the abrasive granules are equiax, and the diameter of substantially every granule is within 10% of the mean diameter so that the granules protrude from the surface of the binder layer to substantially the same extent and so can be loaded equally upon contacting a workpiece, thus providing an extraordinarily uniform finish. By "equiax" is meant that each granule has approximately the same thickness in every direction. An equiax granule can be considered to have a diameter, whether or not it is spherical.

The abrasive granules can have various populations at various areas of the novel abrasive sheeting in order to remove material at differing rates from selected faces of a workpiece. In one technique for determining the populations required to accomplish this, the wear of abrasive sheets of uniform density is studied, and the novel abrasive sheeting is made to have increased granule population at areas showing the most wear. The predetermined granule pattern also can be selected to leave the novel abrasive sheeting free from abrasive granules in areas to be die-cut, thus allowing the die to remain sharp much longer than has heretofore been possible. This also minimizes waste.

The invention also concerns a novel method of making abrasive sheeting by the sequential steps of

1) attracting small abrasive granules only to dots in a predetermined pattern on a carrier,

2) while advancing a backing that carries a tacky binder layer in synchronism with said carrier, transferring the attracted granules into the binder layer in said pattern, and

3) rendering the binder nontacky.

When the carrier is at the surface of a rotating cylinder, this 3-step method can produce abrasive sheeting of almost unlimited length which can be wound up in roll form for convenient storage and shipment. In such a roll, the predetermined pattern of abrasive granules repeats many times. Such a roll can be converted into a variety of articles such as discs, daisies, sheets, and belts.

When the abrasive granules have irregular shapes, the 3-step method causes the major axis of each

granule to lie substantially in a plane parallel to the backing. Hence, when irregular granules are of substantially the same size, they tend to protrude from the binder to substantially the same extent and to abrade uniformly to afford uniform finishes.

Preferably the binder is selected so that subsequent to step 2) is an additional step of softening the binder, usually by being heated, to form a meniscus at each granule, thus enhancing the bonding of each granule to the backing. Doing so should make it unnecessary to overcoat the granules, thus leaving the cutting surface of each granule free from material that could otherwise interfere with its abrasive function. When heat is used to form menisci, the extent to which the abrasive granules protrude from the binder can be controlled by adjusting the time and temperature at which the menisci are formed. When a meniscus is to be formed at each abrasive granule, the pressure applied in step 2) can be very light, just enough to tack the granules to the binder layer.

Instead of, or in addition to, forming a meniscus at each granule, a size coat can be applied over the novel abrasive sheeting to enhance the bonding of the abrasive granules. Usually this is unnecessary, unless the abrasive granules are rather large, e.g., are retained by a screen with 100 μm openings. When the abrasive granules are expensive, e.g., diamonds, a size coat may be desirable to ensure that they are not dislodged and lost.

Although the individual abrasive granules that lie in a predetermined pattern should be of substantially equal size, selected areas of the novel abrasive sheeting can have abrasive granules of one size in one predetermined pattern while other areas have granules of a different size in another predetermined pattern, each to provide a desired rate of cutting and degree of finish at a particular area of a workpiece. For the same reason, the novel abrasive sheeting may employ abrasive granules of two or more different types, each type being individually positioned in a predetermined pattern. In order to make abrasive sheeting having two sizes or two types of abrasive granules, steps 1) and 2) are repeated with the second size or type of granule prior to step 3).

Detailed Description

Each of the abrasive granules of the novel sheeting preferably is an equiax composite of a large number of abrasive grains in an inorganic or organic binder matrix. For example, abrasive grains having a mean dimension of about 4 μm can be bonded together to form spheres of virtually identical diameters, preferably within a range of from 25 to 100 μm. Because of their uniform diameter, each equiax granule can be positioned to protrude to the same extent from the binder layer. By individually positioning the equiax granules to be spaced equally from adjacent granules, the granules each bear the same load and hence wear at substantially identical rates and tend to continue to be equally effective as long as uncoated portions protrude from the binder layer. Consequently, workpieces continue to be polished uniformly, in contrast to the tendency of prior abrasive sheeting to provide uneven polishing upon becoming worn.

Preferably the thickness of the binder layer of the novel abrasive sheeting is from 25 to 150 μm. Thicknesses above that range may be uneconomical, while thicknesses below that range may not bind the abrasive granules as strongly as desired. When the binder layer is coated from solution or emulsion, it may be difficult to obtain uniform layers much greater than 50 μm.

A carrier that can be used in the above-outlined 3-step process is a printing plate marketed by Toray Industries as "Toray Waterless Plate." It has a flexible sheet of aluminum bearing a layer of photosensitive material covered with a layer of silicone rubber. Upon exposure to light through a half-tone screen, the silicone rubber of a positive-acting plate causes the photosensitive material to bind itself firmly to the silicone rubber in areas where the light strikes, after which the silicone rubber in unexposed areas can be brushed off, leaving silicone rubber dots in the predetermined pattern provided by the light exposure. The printing plate is then wrapped onto a cylinder, and the cylinder is rotated through a fluidized bed of abrasive granules. The granules are attracted to the printing plate only where the silicone rubber remains and not to the ink-receptive areas. Upon moving a binder-carrying backing in synchronism with the rotating printing plate, the granules are picked up by and become embedded into the binder layer in the pattern of the printing plate. That pattern repeats many times when the backing is long.

When a "Toray Waterless Plate" is used in the above-outlined 3-step process and the tacky binder layer would stick to the silicone rubber of the printing plate, a transfer roll can be positioned between the binder layer and the printing plate. The surface of the transfer roll should be selected to cause the abrasive granules to transfer from the silicone rubber of the printing plate, while acting as a release surface in relation to the tacky binder.

For most applications of the novel sheet, the breadth of the dots formed in step 1) of the above 3-step

Ex. 1003, Page 231

process should be small enough that only one abrasive granule is attracted to each position, but when each dot is large enough to make it fairly certain that there will be a granule at every dot, it can be expected two or possibly three granules will be deposited side by side at a few positions. When only one abrasive granule per dot is desired, each dot preferably is roughly circular and has a diameter within the range of 30 to 90% of the mean dimension of the abrasive granules. When using spherical abrasive granules 80 μm in diameter, good results have been attained with dots 62 μm in diameter. When the diameter of each dot substantially exceeds the diameter of an abrasive granule, a monolayer of several granules may be attracted to each dot.

The use of a printing plate mounted on a cylinder in the above-outlined 3-step process may result in a seam that may produce discontinuities in the pattern of abrasive granules. Abrasive sheeting of the invention, that has no seam in its pattern, can be made by sequentially coating onto a cylinder formulations that provide a cylindrical printing plate, preferably including a silicone rubber layer. Preferred sequential coating formulations are those of U.S. Pat. No. 3,511,178 (Curtin).

If a seam in the pattern is not objectionable, step 1) of the above-outlined 3-step process for making abrasive sheeting of the invention can use a carrier prepared by the steps of

1) coating one face of a sheet of metal foil with rubber and the other face with a photoresist,
2) exposing the photoresist to light in a predetermined pattern,
3) removing areas of the photoresist corresponding to said pattern, and
4) etching away the metal foil in said areas to expose the rubber in said predetermined pattern.

It is also preferred, particularly if the abrasive granules would be attracted to the remaining photoresist, to include after the etching step the step

5) removing the remaining photoresist.

Among classes of binders that can be used in the novel abrasive sheeting are thermoplastic resins such as ethylene/acrylic acid copolymer, polyethylene, and poly(ethylmethylacrylic) acid, which is available from E.I. duPont Company under the trade designation "Surlyn". Another useful class of binders is acrylic pressure-sensitive adhesives which cure to a nontacky state. Also useful are thermosetting binders which have a tacky state such as epoxy resins, phenolics, and polyurethanes.

The backing of the novel abrasive sheeting can be fabric (e.g., woven or non-woven fabric such as paper) which may be saturated with a filled binder material, a polymer film such as that formed of oriented heat-set polypropylene or poly(ethylene terephthalate) which may be first primed, if needed, with a priming material, or any other conventional backing material.

In the novel abrasive sheeting, the addition of a grinding aid over the surface of the abrasive granules may provide improved grinding performance. Grinding aids may also be added to the size coat or as particulate material. The preferred grinding aid is KBF_4, although other grinding aids are also believed to be useful. Other useful grinding aides include NaCl, sulfur, K_2TiF_6, polyvinyl chloride, polyvinylidene chloride, cryolite and combinations and mixtures thereof. The preferred amount of grinding aid is on the order of 50 to 300 g, preferably 80 to 160 g, per square meter of coated abrasive product.

The Drawing

The invention may be more understandable by reference to the drawing wherein:

Fig. 1 schematically shows a method of printing abrasive granules onto a binder layer carried by a flexible backing, thus providing preferred abrasive sheeting of the invention;

Fig. 2 is an electronmicrograph showing abrasive granules having been attracted to a tackified binder layer using apparatus illustrated in Fig. 1;

Fig. 3 is an electronmicrograph showing a fragment of abrasive sheeting of the invention made by heating the intermediate product of Fig. 2 to form a binder meniscus at each granule; and

Fig. 4 schematically shows a fragment of an abrasive sheeting of the invention after it has been cut to form daisy pads.

In Fig. 1, attached to a rotatable, heated metal cylinder 10 is a printing plate 12, the outer surface of which has been developed to leave roughly circular rubber dots 13. The cylinder is rotated through a fluidized bed of spherical abrasive granules 14, each of uniform diameter somewhat larger than the diameter of the rubber dots. After removing excess granules by suction at 15, substantially one abrasive granule 14 adheres to each of the rubber dots 13. Moving at the same speed as the surface of the cylinder

Ex. 1003, Page 232

10 is a flexible backing 16 carrying a heat-activatable binder layer 18 which is pressed against the printing plate 12 by a heated nip roll 20. Heat from the cylinder 10 and rubber-covered nip roll 20 tackify the binder layer 18 to permit the attracted granules 14 to be adhered superficially to the binder layer 18 in spaced rows extending in the X and Y directions as seen in Fig. 2. In the upper left corner of Fig. 2, a few abrasive granules have fallen out, leaving small craters in the binder layer. The abrasive granules have been deposited in the lower part of Fig. 2 at twice the density of the upper part. At most positions, only one abrasive granule has been deposited, but at a few dots, there are two granules side by side.

Referring again to Fig. 1, the granule-bearing backing 16 is passed across a bank of infrared lamps 22 by which the binder is heated to wet the surfaces of the abrasive granules, thus causing the binder layer to flow and form a meniscus 23 around the base of each granule as shown in the electronmicrograph of Fig. 3. This causes the abrasive granule to become strongly bonded to the flexible backing 16.

The resulting abrasive sheeting 24 of the invention contains abrasive granules 14 individually positioned to permit the sheeting to be die-cut into daisy pads 26 as shown in Fig. 4. The abrasive granules have been positioned in concentric rows such that their density in areas 28 adjacent the outer edges of each petal is twice the density at the central areas 30 of the daisy pad. The abrasive sheeting from which the daisy pads 26 were cut was left free from abrasive granules adjacent the phantom line 32 at the peripheries of the petals along which the sheeting 24 is to be die-cut so that the die does not contact any abrasive granules. By leaving the area 34 between the daisy pads free from abrasive granules, no granules have been wasted.

In the following examples, all parts are given by weight.

Example 1

Used to make abrasive sheeting of this example were:

1) a negative-acting "Toray Waterless Plate" which had been exposed to a half-tone screen to produce roughly circular dots, each about 63 µm in diameter and arranged in rows extending parallel to both the lengthwise and widthwise edges of the plate, 20 rows per inch (7.9 per cm) in both directions;

2) spherical abrasive granules, each 80 µm in diameter and made of 77 parts Al_2O_3 grains having a mean particle size of 4 µm in 23 parts of a matrix based on phenol-formaldehyde resin;

3) a long roll of biaxially oriented poly(ethyleneterephthalate) polyester film 0.05 mm in thickness bearing a 0.05 mm binder layer of ethylene/acrylic acid copolymer.

The dot-containing plate was mounted on the metal cylinder 10 of the apparatus shown in Fig. 1, and spherical abrasive granules 14 were fluidized by a mechanical vibrator and became attached to the silicone dots 13. Excess granules were removed by suction at 15. The binder layer 18 was heated by the rubber-covered nip roll 20 to about 70 °C to become tacky so that the abrasive granules transferred to it, with a force of 79 N applied to the nip roll per cm of width. Additional heating by infrared lamps 22 caused the binder to form a meniscus around the base of each of the granules.

The exposed face of the polyester film backing of the resulting abrasive sheeting was laminated with a double-coated pressure-sensitive adhesive tape and this composite was die-cut into daisy pads 7.6 cm in diameter and similar in shape to the daisy pads 26 of Fig. 4 except having six petals. The pads were used as a second fining pad in the polishing of lenses formed of polycarbonate of the type commercially available from PPG under the trade designation CR39. A "Coburn" #506 cylinder machine was used at a load of 20 pounds (89 N) with a water flood on the high speed spindle setting. The test was conducted using two types of lapping tools, a 6.25/8.25 dioptral and a 2.12 dioptral. In both cases the amount of lens removed after two minutes was measured. The results are in Table I.

Examples 2-6

Abrasive sheetings of Examples 2-6 were made as in Example 1 except having different spacings of their rows in both directions as indicated in Table I. Also in Table I are results from using a "Control" daisy pad made with the same spherical abrasive granules coated from slurry in a manner used for current commercial production and having about 870 granules/cm^2. This granule density had been selected based upon extensive experimentation for general purpose use and was intermediate the granule densities of the abrasive sheetings of Examples 3 and 4.

Ex. 1003, Page 233

TABLE I

Ex.	Rows per inch	Rows per cm	mm of lens removed in 2 mins	
			2.12 d. lens	6.25/8.25 d. lens
1	20	7.9	0.001	0.000
2	40	15.7	0.091	0.058
3	65	25.6	0.025	0.078
4	85	33.5	0.026	0.124
5	150	59.0	0.012	0.032
6	200	78.7	0.009	0.025
Control			0.026	0.056

Example 7

A negative-acting "Toray Waterless Plate" was exposed to produce 63 µm silicone rubber dots in rows extending in the X and Y directions at a density of 65 rows per inch (25.6 rows/cm). The plate was covered with the spherical abrasive granules of Example 1, and excess granules were then removed by turning the plate over and tapping it. Examination of the plate showed that there was at least one granule at each of the silicone dots. Against those granules was laid the binder layer of the polyester film backing of Example 1, and the composite was put through the nip of a heated two-roll laminator at about 73° C while a force of 79 N was applied to the nip roll per cm of width. The composite was then placed in an oven at 112° C for 10 minutes to cause the binder to form a meniscus around the base of each granule, resulting in abrasive sheeting of the invention.

Examples 8-11

A round-dot litho contact screen was exposed to form a pattern of spots, each 61 µm in diameter and equally spaced in rows extending in the X and Y directions. The number of rows of spots per unit distance within an inner circle 6.0 cm in diameter differed from the number of rows between that circle and an outer circle 15.24 cm in diameter. In other words, the spot population changed at the junction of the two circles as it does in Fig. 2. Several different combinations of populations were prepared in this manner as shown in Table II.

A negative-acting "Toray Waterless Plate" was exposed using each contact screen and developed in the usual manner. The developed plate was mounted on a metal cylinder and employed in the same way as in Example 1 to transfer spherical abrasive granules 83 µm in diameter from a fluidized bed to create 6-petal daisy pads, the centers of which coincided with the centers of the circles. The daisy pads were tested as in Example 1 with results in Table II in comparison to the same "Control".

Ex. 1003, Page 234

TABLE II

Ex.	Inner Circle Rows/cm	Outer Circle Rows/cm	mm of lens removed	
			212 lens	625/825 lens
8	19.7	33.5	0.034	0.040
9	25.6	19.7	0.108	0.086
10	25.6	33.5	0.058	0.102
11	25.6	39.4	0.070	0.103
Control			0.025	0.056

Example 12

A round-dot litho contact screen was exposed to form an opaque spiral pattern using the standard formula $X = k\phi$ and $X = k\phi + 0.317$ mm, starting from a circle 2.1 cm in diameter and extending to a circle 15.2 cm in diameter. This produced opaque and transparent spiral areas of equal size between the two circles. Then using the developed contact screen as a mask, a laser was used to expose a negative-acting "Toray Waterless Plate" through the transparent areas of the mask to produce a pattern of dots 63 μm in diameter equally spaced in rows extending in the X and Y directions. There were 100 rows of silicone dots per inch (39.4 dots/cm) within a spiral pattern on the developed plate.

The developed plate was used as in Example 7 (except using abrasive granules 83 μm in diameter) to provide abrasive sheeting of the invention which was cut into a disc on which was centered the spiral pattern of abrasive granules in spaced rows.

Example 13

Abrasive sheeting was prepared as in Example 7 except as follows:

a) the abrasive granules were Al_2O_3 grains of irregular shape which had been screened to pass 100 mesh (150 μm openings) and retained on 120 mesh (125 μm openings);

b) there were 65 rows/inch (25.6 rows/cm) of silicone dots;

c) the abrasive sheeting, after being removed from the oven, was overcoated with a size coat of phenolformaldehyde resin which after curing in an oven had a dry weight of 80 g/m^2.

The backing of this abrasive sheeting was laminated with a double-coated pressure-sensitive adhesive tape and then die-cut to 3-inch (7.5 cm) discs. Each disc was adhered by its pressure-sensitive adhesive to a "Coburn" No. 507 cylinder machine using the following settings: spindle stroke set at 7, spindle speed 100%, cross stroke 0, and a load of 30 pounds (133 N). The workpiece was a 1018 mild steel ring 4.45 cm I.D. and 5.4 cm O.D. The ring was abraded in an operation normally called flat lapping after being mounted on a bracket that fixed its axis perpendicular to the abrasive surface as the machine oscillated the abrasive surface in a circular motion. The test was run in one minute cycles at a rate endpoint of 0.3 g/min. The total cut and surface finish at endpoint are given in Table III in comparison to a "Control" made in the same way except that the abrasive granules were coated from slurry to provide a binder layer containing approximately the same mass of granules (i.e., about 0.40g).

Ex. 1003, Page 235

TABLE III

Example	Total Cut	R_a (μm)	R_t (μm)
13	2.94 g	0.30	2.87
Control	1.44 g	0.45	3.45

R_a = average surface roughness
R_t = maximum peak-to-valley height

Claims

1. Abrasive sheeting comprising a backing carrying a binder layer of substantially uniform thickness in which abrasive granules are bonded substantially in a plane and characterized by the abrasive granules lying at a predetermined lateral spacing and being of substantially equal size and passing a screen with 300 μm openings, and a portion of virtually every granule protruding from the surface of the binder layer.

2. Abrasive sheeting as defined in claim 1 further characterized by substantially every granule being equiax and of substantially the same diameter, and each granule protruding from the binder to substantially the same extent.

3. Abrasive sheeting as defined in claim 1 further characterized by single granules being aligned in uniformly spaced rows.

4. Abrasive sheeting as defined in claim 3, further characterized by a great length of which being wound upon itself in roll form, said granules being in a predetermined pattern that repeats a large number of times over the length of the sheet.

5. Abrasive sheeting as defined in claim 4 further characterized by said predetermined pattern leaving areas of the binder layer free from abrasive granules in positions to be contacted by a tool for cutting the sheeting into articles of desired shapes, thus protecting the cutting tool from contacting abrasive granules.

6. Abrasive sheeting as defined in claim 1 further characterized by additional abrasive granules of substantially equal size different from the size of the first-mentioned abrasive granules being individually positioned in a second predetermined pattern and an uncoated portion of virtually every of said additional granules protruding from the surface of the binder layer.

7. Method of making abrasive sheeting of claim 1 by the sequential steps of

1) attracting small abrasive granules only to dots laterally spaced in a predetermined pattern on a carrier,

2) while advancing a backing that carries a tacky binder layer in synchronism with said carrier, transferring the attracted granules into the binder layer in said pattern, and

3) rendering the binder nontacky.

8. Method as defined in claim 7, further characterized in step 2 of which the granules being pressed into the binder layer.

9. Method as defined in claim 7 further characterized by said carrier covering the cylindrical exterior of a rotating drum, and in step 1) the drum being rotated through a fluidized bed of said granules.

Ex. 1003, Page 236

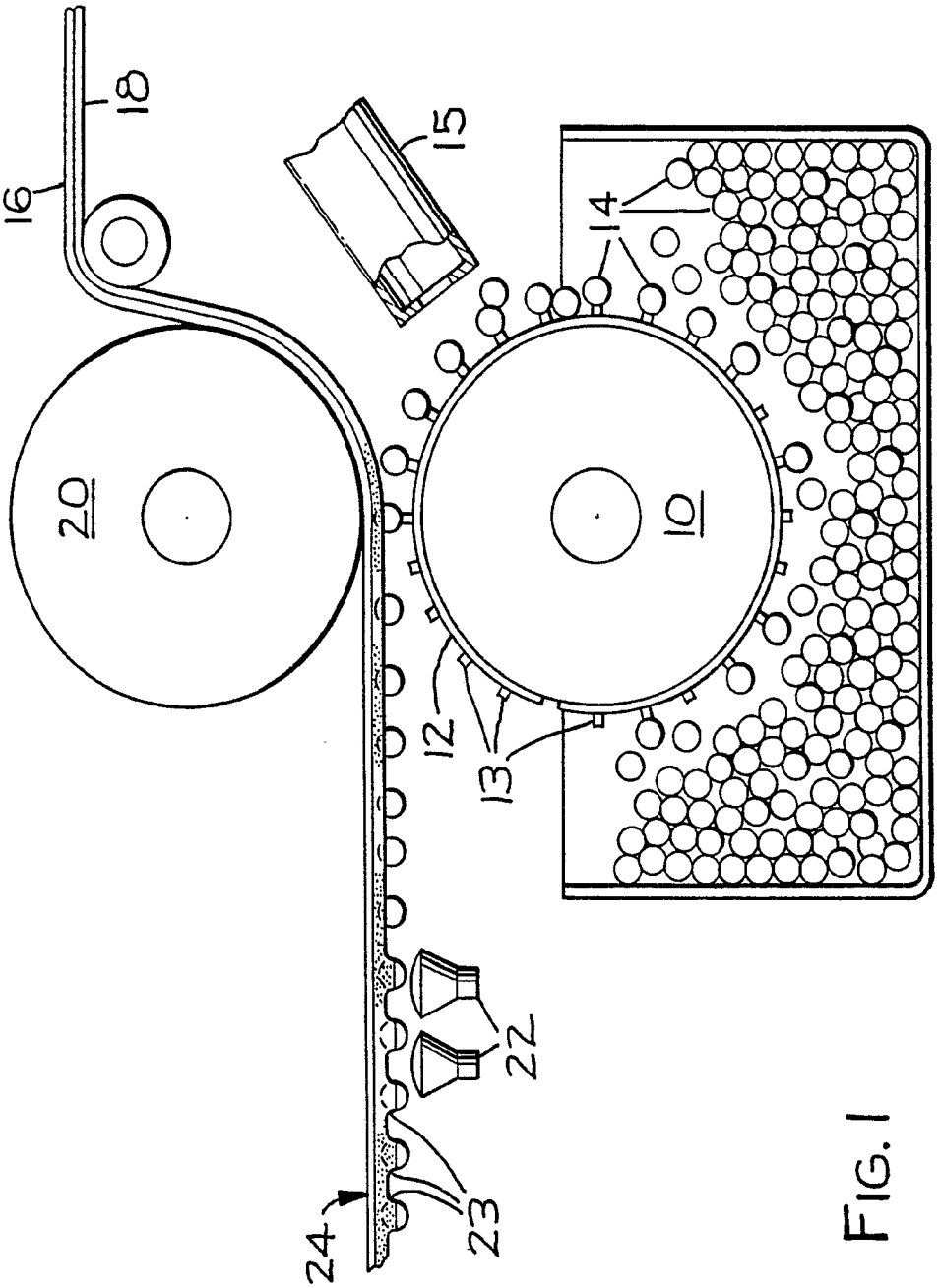


FIG. 1

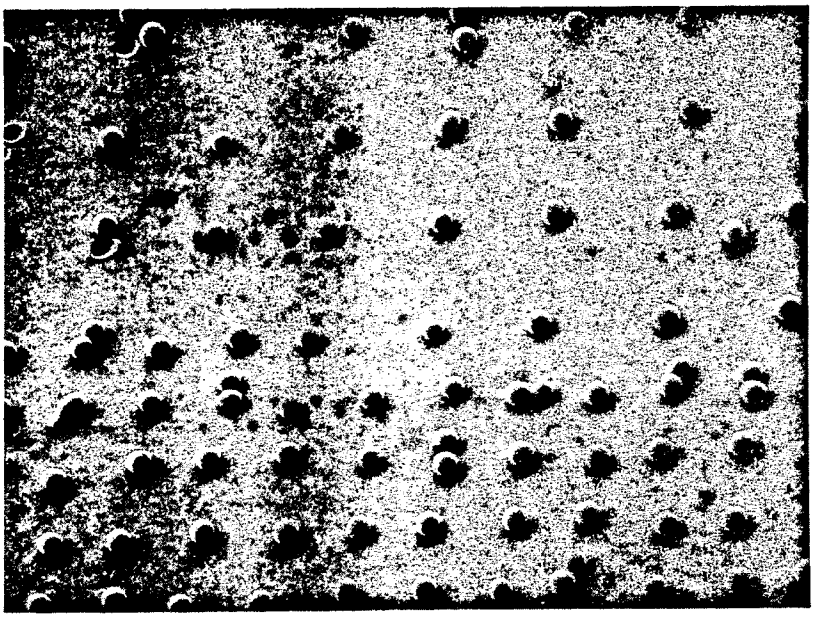


FIG. 2

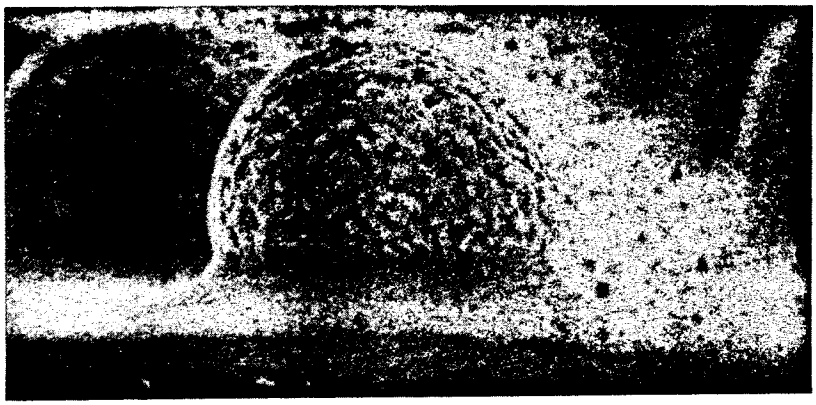


FIG. 3

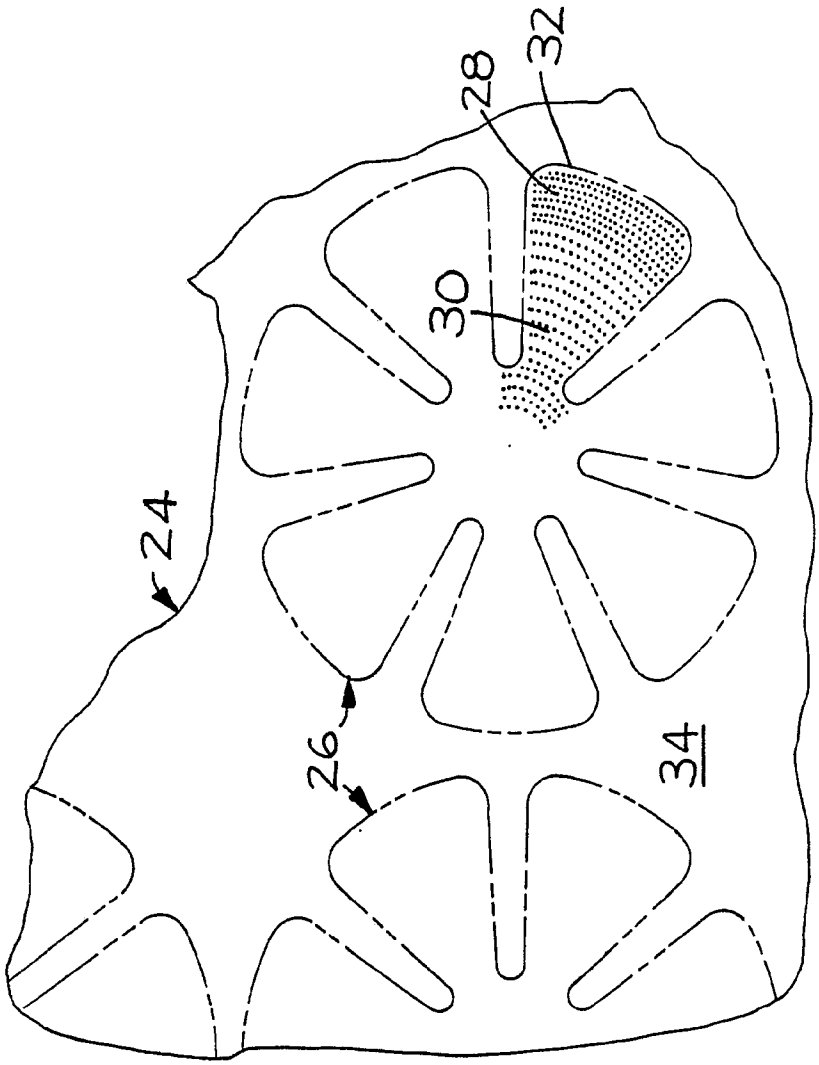


FIG. 4

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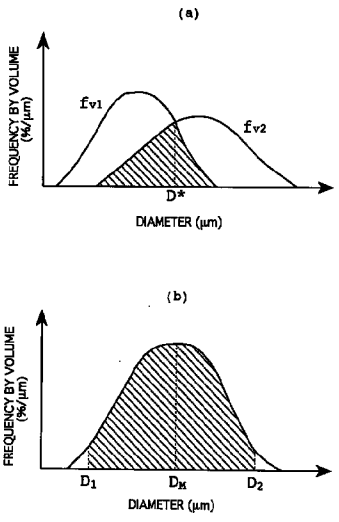
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(54) Diamond sinter, high-pressure phase boron nitride sinter, and processes for producing those sinters

(57)A powder of diamond or high-pressure phase boron nitride core particles is charged into a coating space as it is dispersed to a dispersity that is preset in accordance with the average diameter of those core particles and a precursor of a coat forming substance that is either generated via the vapor phase or in a vapor-phase state is allowed to contact and/or impinge against the particles in said powder of core particles so that their surfaces are covered with the coat forming substance, thereby preparing coated diamond or high-pressure phase boron nitride particles, and these particles or mixtures containing them or mixtures of these particles with bonding materials are sintered under appropriate sintering conditions at a predetermined high temperature and pressure. The thusly produced diamond or high-pressure phase boron nitride sinter is composed of coated core particles of high performance that are superhard, uniform, dense and sintered firmly and which have a controlled microstructure. If sintering is done at pressures less than 2000 MPa, the invention offers the advantage of eliminating the constraint of the need to use equipment capable of creating ultrahigh pressures of at least 2000 MPa. The use of ultrahigh pressure HIP apparatus or other types of HIP apparatus offers the added advantage of enabling the production of shaped sinters, thereby presenting significant benefits to manufacture on an industrial scale.

FIG.1



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Description

BACKGROUND OF THE INVENTION

This invention relates to a diamond sinter produced by sintering a mixture containing at least coated diamond particles having a coat forming substance formed on the surfaces of diamond particles and a high-pressure phase boron nitride sinter produced by sintering a mixture containing at least coated high-pressure phase boron nitride particles having a coat forming substance formed on the surfaces of high-pressure phase boron nitride particles. The invention also relates to processes for producing those sinters.

Diamond, with strong covalent bonding, has many excellent properties. On the other hand, because of this covalent bonding, diamond has such a small self-diffusion coefficient that it is very difficult to sinter. Another problem with diamond is that it is thermodynamically stable only under ultrahigh pressures so that it will experience a phase transition to graphite if the pressure is insufficient under elevated temperatures. Therefore, without addition of bonding materials or sintering aids, strong sinters cannot be produced unless a ultrahigh pressure of about 8500 MPa is applied simultaneously with exposure to a very high temperature of about 2440 K.

High-pressure boron nitrides consist of cubic boron nitride and/or wurtzite boron nitride. Cubic boron nitride (c-BN) has similar characteristics to diamond; on account of strong covalent bonding, it has many excellent properties but, on the other hand, because of this covalent bonding, c-BN has such a small self-diffusion coefficient that it is very difficult to sinter. Further, c-BN is thermodynamically stable only under ultrahigh pressures and it will experience a phase transition to hexagonal boron nitride (h-BN) of a graphitic phase (if the pressure is insufficient under elevated temperatures.) Wurtzite boron nitride (w-BN) has generally the same properties as the cubic boron nitride; it is a difficult-to-sinter substance and it is thermodynamically stable only at ultrahigh pressures and will experience a phase transition to hexagonal boron nitride (h-BN) of a graphitic phase if the pressure is insufficient under elevated temperatures. It should be noted that if the wurtzite boron nitride is sintered under ultrahigh pressures and at elevated temperatures where the cubic boron nitride is thermodynamically stable, a phase transition to the cubic boron nitride occurs depending on the conditions, yielding in this case a sinter containing the cubic boron nitride or a sinter that is solely comprised of the cubic boron nitride. In either case, without addition of bonding materials or sintering aids, strong sinters cannot be produced unless a ultrahigh pressure of about 7000 MPa is applied simultaneously with exposure to a very high temperature of about 2000 K.

However, irrespective of whether diamond or high-pressure phase boron nitride is to be sintered, the sintering conditions set forth in the preceding paragraphs for the case where neither bonding materials nor sintering aids are added are extremely hostile and are not suitable for the purpose of producing diamond or high-pressure phase boron nitride sinters of high density on an industrial scale.

Therefore, in order to produce these high-density sinters, sintering must be done under industrially applicable conditions and to this end the addition of bonding materials and/or sintering aids is essential.

The first thing to be stated in this connection is that in recent years, a particular need exists for enhancing the performance of diamond or high-pressure phase boron nitride sinters of high density and to this end, it is required that diamond or high-pressure phase boron nitride particles such as those not larger than 10 μm be used as the starting material and sintered to produce a diamond or high-pressure phase boron nitride sinter of high density.

Conventionally, diamond or high-pressure phase boron nitride sinters have been produced mainly by a method in which bonding materials and/or sintering aids are added in a powder form. In this case, even if the bonding materials and/or sintering aids are of a fine particulate form, it is extremely difficult to insure ideal uniform addition or uniform dispersion in such a way that they cover all of the individual diamond or high-pressure phase boron nitride particles present. Even if such uniform dispersion is realized, the extent of "uniformity" that can be attained is limited since the bonding materials and/or sintering aids are added on a particle basis. It should particularly be noted that if the bonding materials or sintering aids are added in small quantities, the resulting sinter will inevitably contain those areas where the bonding materials or sintering aids are absent.

In many actual cases, those powder particles or the particles of the bonding material and/or sintering aid agglomerate to either form lumps in the sinter or occur unevenly in the sinter. Hence, particularly in those areas where diamond or high-pressure phase boron nitride particles agglomerate, the structure is essentially the same as what is produced without addition of bonding materials or sintering aids and unsintered portions will locally occur. In those areas where the bonding materials and/or sintering aids agglomerate, the microscopic structure contains portions where diamond or high-pressure phase boron nitride particles are absent from the sinter. Either phenomenon produces imperfections that cause marked deterioration in the performance of the produced diamond or high-pressure phase boron nitride sinter.

Therefore, in order to produce diamond or high-pressure phase boron nitride sinters of high performance that are free from the above-mentioned imperfections, it is necessary that the bonding material and/or sintering aid be positively distributed on individual diamond or high-pressure phase boron nitride particles and, to this end, it is strongly desired to produce diamond or high-pressure phase boron nitride sinters of high performance using coated diamond or high-pressure phase boron nitride particles which are prepared by coating uniformly a bonding material forming substance

Ex. 1003, Page 241

and/or a sintering aid forming substance on the individual particles of diamond or high-pressure phase boron nitride, respectively.

Secondly, as already mentioned, both diamond and high-pressure phase boron nitride are substances that are extremely difficult to sinter and which are stable only under ultrahigh pressures and, hence, in order to prevent the diamond or high-pressure phase boron nitride from experiencing a phase transition to the lower-pressure phase (graphite or h-BN) and to produce a dense sinter of the diamond or high-pressure phase boron nitride, manufacturers have adopted the practice of using bonding materials and/or sintering aids under ultrahigh pressures in excess of 2000 MPa and at elevated temperatures. According to the report of T. Noma et al. (J. Master. Scie., 19 (1984) 2319-2322), diamond is sintered at 6000 MPa (60 kb) and at 1300°C or above. Cubic boron nitride may be sintered at 4000 MPa (40 kb) and at 1200°C or above, as taught in Unexamined Published Japanese Patent Application (kokai) Sho 63-35456. These sintering conditions refer to extreme states and they are both rigorous indeed in that they cannot be created without using a ultrahigh pressure apparatus such as of the girdle or belt type.

This constraint of using a ultrahigh pressure apparatus such as of the girdle or belt type for creating ultrahigh pressures in excess of 2000 MPa has made it difficult to achieve mass production of diamond or high-pressure phase boron nitride sinters and not only has the production cost been high but it has also been impossible to manufacture large shapes.

However, speaking of diamonds, Hall conducted an experiment under ultrahigh pressures and reported that when a diamond was thermodynamically metastable if not stable, it took such a long time for a phase transition to occur that it remained stable for practical purposes; he then drew line ② as shown in Fig. 33a to indicate the upper limit beyond which the diamond would no longer exist in a practically stable manner (H. T. Hall, Science, 169 (1970) 868-869). Hence, according to his report, even under low-pressure and high-temperature conditions that are not stable with respect to thermodynamic equilibrium line ① in Fig. 33a, a diamond will exist practically stable if it is at temperatures that do not exceed line ② in Fig. 33a, say, up to about 1400 K (ca. 1100°C), given a pressure of up to about 35 kb.

Speaking of high-pressure phase boron nitrides, Wakatsuki et al. conducted an experiment on a cubic boron nitride under ultrahigh pressures and reported that when the cubic boron nitride was thermodynamically metastable if not stable, it took such a long time for a phase transition to occur that it remained stable for practical purposes; they then drew line ② as shown in Fig. 33b to indicate the upper limit beyond which the cubic boron nitride would no longer exist in a practically stable manner (Wakatsuki, Ichise, Aoki and Maeda, Abstracts of the Lectures Read at the 14th Symposium on High-Pressure Technology (1972) p. 78). Hence, according to their report, even under low-pressure and high-temperature conditions that are not stable with respect to thermodynamic equilibrium line ① in Fig. 33b, a cubic boron nitride will exist practically stable if it is at temperatures that do not exceed line ② in Fig. 33b, say, not higher than 1200°C.

Based on these facts, one may conclude that if diamond sinters containing diamonds or high-pressure phase boron nitride sinters containing high-pressure phase boron nitrides at least one of which is a cubic boron nitride can be produced under the stable conditions mentioned above, he can effectively use a ultrahigh pressure apparatus such as a piston-cylinder (PC) type ultrahigh pressure apparatus that is capable of creating comparatively moderate ultrahigh pressures less than 2000 MPa, or a ultrahigh pressure HIP apparatus as a known technique capable of pressurization up to 1000 MPa, or hot isostatic press (HI) apparatus excluding the ultrahigh pressure HIP apparatus, or a hot press (HP) apparatus.

Thusly, unlike in the case of ultrahigh pressure apparatus of such types as the aforementioned girdle and belt types that create ultrahigh pressures in excess of 2000 MPa, not only does it become easy to achieve mass production but it also becomes possible to produce sinters of large shapes.

In recent years, it is strongly desired that diamond or high-pressure phase boron nitride sinters of high density and performance that contain comparatively large amounts of diamond or high-pressure phase boron nitride particles such as those not larger than 10 μm should be manufactured in high volumes or in large shapes using a ultrahigh pressure apparatus such as the aforementioned piston-cylinder (PC) type ultrahigh pressure apparatus which is capable of creating comparatively moderate ultrahigh pressures less than 2000 MPa, or the ultrahigh pressure HIP apparatus capable of pressurization up to 1000 MPa, the hot isostatic press (HIP) apparatus excluding the ultrahigh pressure HIP apparatus, or the hot press (HP) apparatus.

In fact, however, it is not easy to assure that diamond or high-pressure phase boron nitride sinters containing fairly large amounts of diamond or high-pressure phase boron nitride, respectively, are effectively densified under those limited pressures.

Stated more specifically, this can be realized only when the bonding material which is added in the form of a feed powder for that material deforms or migrate during sintering to fill the gaps between diamond or high-pressure phase boron nitride particles so that the applied pressure will work effectively for the displacement and loss of pores or the voids which are surrounded by the bonding material and the diamond or high-pressure phase boron nitride particles. To this end, the bonding material and/or the feed powder for that material which reacts with diamond or high-pressure phase boron nitride to generate the bonding material must be present in such a way that they contact and surround the diamond or high-pressure phase boron nitride particles.

It should particularly be noted that under the sintering pressure and temperature conditions where the pressure is less than 2000 MPa and where diamond or high-pressure phase boron nitride is thermodynamically metastable if not stable, there is little chance for the particles of diamond or high-pressure phase boron nitride to bind directly and, hence,

Ex. 1003, Page 242

those portions of the diamond or high-pressure phase boron nitride particles in the resulting sinter where the feed powder for the bonding material is absent will remain unsintered. As a result, the performance of the sinters produced will deteriorate considerably. If the bonding material is added only in the form of a feed powder for that material, it is extremely difficult to insure ideal uniform addition or uniform dispersion in such a way that the particles of the feed powder cover all of the individual diamond or high-pressure phase boron nitride particles present even if the particles of the feed powder are fine. Even if such uniform dispersion is realized, the extent of "uniformity" that can be attained is limited since the feed powder for the bonding material is added on a particle basis. Thus, a serious problem is presented in the manufacture of diamond or high-pressure phase boron nitride sinters that contain large amounts of diamond or high-pressure phase boron nitride, respectively. Therefore, even in the case of mixing diamond or high-pressure phase boron nitride particles with the feed powder for the bonding material, one may compensate for the insufficiency by using coated diamond or high-pressure phase boron nitride particles which are prepared by coating the surfaces of bare diamond or high-pressure phase boron nitride particles with a bonding material forming substance that is of the same and/or different type from the feed powder for the bonding material; as a consequence, the distribution of the bonding material can be made sufficiently uniform. Hence, the coating method for preparing such coated diamond or high-pressure phase boron nitride particles bears technologically great importance.

Stated more specifically, irrespective of whether the bonding material is added in the form of a feed powder for bonding material forming substance or not, the bonding material and/or a substance that reacts with diamond or high-pressure phase boron nitride to form the bonding material is allowed to be present covering the surfaces of diamond or high-pressure phase boron nitride particles. Only by following this approach, one can assure that diamond or high-pressure phase boron nitride sinters of high density and performance that contain comparatively large amounts of diamond or high-pressure phase boron nitride particles are manufactured in high volumes or large shapes using a ultrahigh pressure apparatus such as the piston-cylinder (PC) type ultrahigh pressure apparatus which is capable of creating comparatively moderate ultrahigh pressures less than 2000 MPa, or the ultrahigh pressure HIP apparatus which is capable of pressurization up to 1000 MPa, or the hot isostatic press (HIP) apparatus excluding the ultrahigh pressure HIP apparatus, or the hot press (HP) apparatus.

While the foregoing description is directed to diamond or high-pressure phase boron nitride sinters that use the respective particles with a particle diameter not greater than 10 μm, it should be noted that if the sinters are to be used in definite applications, it is very effective to intentionally use diamond or high-pressure phase boron nitride particles of comparatively large particle diameters, say, in excess of 10 μm.

Consider, for example, a wear-resistant sinter that takes advantage of the ultrahigh hardness of diamond or high-pressure phase boron nitride. This sinter can be produced by sintering a dispersion of a comparatively large quantity of diamond or high-pressure phase boron nitride particles in excess of 10 μm in diameter and, in this case, using diamond or high-pressure phase boron nitride particles larger than 10 μm in diameter as a starting material is extremely important.

The diamond or high-pressure phase boron nitride sinter which has dispersed therein diamond or high-pressure phase boron nitride particles exceeding 10 μm in diameter is characterized in that the effect of dispersion of the dispersed particles is marked because those dispersed particles are sintered as they are closely bound to the surrounding microstructure without imperfections or pores. However, as already mentioned, both diamond and high-pressure phase boron nitride are extremely difficult to sinter and, therefore, it is essential to use a sintering aid or a bonding material that accelerate the sintering of the diamond or high-pressure phase boron nitride particles larger than 10 μm in diameter together with the surrounding microstructure.

Conventionally, such sintering aids and bonding materials have been added solely by the powder mixing method. However, the incorporation of impurities during mixing is unavoidable in this method; what is more, there is theoretically a limit on the extent to which a uniform structure can be realized. Even if the particles of a sintering aid or a bonding material are extremely fine, it is very difficult to insure ideal uniform mixing or uniform dispersion in such a way that the particles of a sintering aid or bonding material powder cover all of the diamond or high-pressure phase boron nitride particles present. Even if such uniform dispersion is realized, the extent of "uniformity" that can be attained is limited since the particles of the sintering aid or bonding material powder are mixed on a particle basis. Particularly in the case where those particles are used in relatively small amounts, an uneven distribution will occur inevitably.

In many actual cases, the dispersed diamond or high-pressure phase boron nitride particles lump together or the particles of the sintering aid or bonding material powder agglomerate, as a result, the diamond or high-pressure phase boron nitride particles will form lumps in the sinter or they occur unevenly in the diamond or high-pressure phase boron nitride sinter, eventually leading to marked deterioration in the performance of the sinters.

Therefore, it is necessary that the sintering aid or the bonding material be distributed positively to all individual diamond or high-pressure phase boron nitride particles exceeding 10 μm in diameter. Further, in order to assure that the diamond or high-pressure phase boron nitride particles are sintered as they are closely bound to the surrounding microstructure, it is required to provide a highly controlled uniform coating on the surfaces of the diamond or high-pressure phase boron nitride particles; the coating is uniform in that it leaves no surfaces of the individual diamond or high-pressure phase boron nitride particles left uncovered and it is highly controlled in that this uniform coating is applied to all individual diamond or high-pressure phase boron nitride particles. What is more, the greater the size of these

Ex. 1003, Page 243

particles, the more uniform this highly controlled coating should be in order to further reduce the uncovered portions of the particles.

Thus, the production of coated diamond or high-pressure phase boron nitride particles of desired diameters in which diamond or high-pressure phase boron nitride particles are covered with uniform coatings that are highly controlled in accordance with the particle diameter, as well as the manufacture of high-performance sinters using those coated particles are strongly desired.

While coat forming substances could be applied by various techniques such as vapor-phase processes and wet plating methods, the vapor-phase approach in which inorganic materials, metallic materials and other coat forming substances are applied to provide coatings as films and various other forms theoretically have major features that are unattainable by other coating techniques, such as: (1) easy control of the atmosphere; (2) the selection of coat forming substances is basically unlimited and various kinds of substances including elemental metallic substances (e.g. active metals), alloys, nitrides, carbides, borides and oxides can be applied; (3) the desired substance can be applied in high purity; and (4) the coating weight of the coat forming substance can be controlled freely.

However, for the reasons to be set forth below, none of the various coating apparatus and methods heretofore proposed as known techniques have been capable of forming the above-described highly controlled uniform coatings.

Particularly in the case of diamond or high-pressure phase boron nitride particles not larger than 10 μm, the diamond or high-pressure phase boron nitride particles to be coated are so fine that they are cohesive enough to have a great tendency to agglomerate together, whereby almost all single particles form agglomerates. Since these agglomerates cannot be disintegrated unless they are subjected to a special action greater than their cohesive force, they cannot be simply coated as such to insure that the surfaces of the individual particles are covered with the coating of a bonding material and/or sintering aid, eventually yielding coated agglomerates in which the surfaces of the agglomerates are covered with the coating of the bonding material and/or sintering aid. This has caused a problem with the individual diamond or high-pressure phase boron nitride particles forming the agglomerates in that the surfaces of the particles located on the surfaces of the agglomerates have large coating weights but suffer from uneven coating whereas the particles located within the agglomerates are not covered at all.

With a view to solving these problems, attempts have already been made to coat the particles in a dispersed state in order to assure the coating of the individual particles in the powder of the core particles to be coated. However, for the reasons stated below, none of the various coating apparatus and methods heretofore proposed as known techniques have been capable of forming the intended uniform coating.

For instance, Unexamined Published Japanese Patent Application (kokai) Sho 58-31076 teaches an apparatus and method, according to which a vessel placed in PVD equipment is charged with the particles in a powder of core particles and vibrated by an electromagnetic means so that the core particles in the vessel are rolled as they are coated by a PVD process. Unexamined Published Japanese Patent Application (kokai) Sho 61-30663 teaches an apparatus, according to which a vessel placed in PVD equipment is charged with the particles in a powder of core particles and vibrated by a mechanical means so that the core particles in the vessel are rolled as they are coated by a PVD process. However, in the actual practice with those apparatus or methods in which the vessel is vibrated so that the particles in the powder of core particles are rolled as they are provided with coatings, the necessary action for disintegrating the agglomerates of diamond or high-pressure phase boron nitride particles with an average diameter not greater than 10 μm by applying a force exceeding their cohesive force cannot be produced and, hence, the agglomerates cannot be disintegrated; to the contrary, a granulating action develops to form agglomerates that are greater in number or size than before the powder of core particles is supplied into the vessel. On the other hand, diamond or high-pressure phase boron nitride particles having an average diameter in excess of 10 μm are simply subjected to a sliding action as they form many layers in superposition and it has been impossible to achieve the desired coating of single separate particles.

Unexamined Published Japanese Patent Application (kokai) Hei 3-153864 teaches an apparatus and method, according to which a rotating vessel having barriers and/or ridges and grooves in the inner surface is charged with core particles and rotated as the surfaces of the particles are coated by an evaporation method. The problem with this apparatus and method is that the necessary action for disintegrating the agglomerates of diamond or high-pressure phase boron nitride particles with an average diameter not greater than 10 μm by applying a force exceeding their cohesive force cannot be produced and, hence, the agglomerates cannot be disintegrated and, what is more, an increased number or size of agglomerates will simply form. On the other hand, diamond or high-pressure phase boron nitride particles having an average diameter in excess of 10 μm are simply subjected to a gentle stirring action as many of them contact one another forming many layers in superposition and it has been impossible to achieve the desired coating of single separate particles.

Unexamined Published Japanese Patent Application (kokai) Sho 58-141375 teaches an apparatus in which the particles of a powder in a reactive gas atmosphere are suspended by the flow of the reactive gas under gravity and in which the surfaces of the particles are coated with the precipitating substance that forms by the chemical reaction involving the reactive gas. Unexamined Published Japanese Patent Application (kokai) Hei 2-43377 teaches a method in which particles placed under vacuum are fluidized as they are subjected to coating by a thermochemical reaction treatment. Unexamined Published Japanese Patent Application (kokai) Sho 64-80437 teaches a method in which the agglom-

Ex. 1003, Page 244

erates of core particles in powder are disintegrated by a sound wave that is a composite of low and high frequency waves, so that the agglomerates are fluidized to improve the coating efficiency. However, these techniques which utilize the fluidized bed of the particles in a powder of core particles which is formed by a gas flow or vibrations have had the problem that with diamond or high-pressure phase boron nitride particles having an average diameter of no more than 10 μm, it is practically impossible to fluidize the separate individual particles, thus failing to disintegrate the agglomerates of these particles. On the other hand, with diamond or high-pressure phase boron nitride particles exceeding 10 μm in average diameter, it is practically impossible to insure that all of these particles are similarly and independently fluidized and suspended as single separate entities and one has been incapable of eliminating uneven coating of the particles which is due to the hiding of one particle by another.

Unexamined Published Japanese Patent Application (kokai) Sho 54-153789 teaches an apparatus in which a powder material is dropped within a vacuum vessel, where the metal vapor is generated to form a metal coating on the particles. Unexamined Published Japanese Patent Application (kokai) Sho 60-47004 teaches a method in which a monomer gas and the particles of a powder are introduced into a high-frequency plasma region in a vacuum vessel, where a coating film of an organic substance is formed by plasma-assisted polymerization. If diamond or high-pressure phase boron nitride particles with an average diameter of no more than 10 μm are simply introduced as in the techniques described above, agglomerates cannot be disintegrated. On the other hand, diamond or high-pressure phase boron nitride particles exceeding 10 μm in average diameter will simply drop while forming agglomerates which are not single separate particles and various problems occur, such as uneven coating due to the hiding of one particle by another, the total failure of the particles within an agglomerate to be coated, and differences in the coating weights of individual particles.

Unexamined Published Japanese Patent Application (kokai) Sho 64-80437 teaches a method in which the agglomerates of core particles in powder are disintegrated by a sound wave consisting of low and high frequency waves so that they are fluidized to improve the coating efficiency. However, this method which imparts vibrations to the fluidized bed has had the problem that with diamond or high-pressure phase boron nitride particles having an average diameter of no more than 10 μm, it is practically impossible to fluidize the separate individual particles, thus failing to disintegrate the agglomerates of these particles. On the other hand, with diamond or high-pressure phase boron nitride particles exceeding 10 μm in average diameter, it is practically impossible to insure that all of these particles are similarly and independently fluidized and suspended as single separate entities and one has been incapable of eliminating uneven coating of the particles which is due to the hiding of one particle by another.

Unexamined Published Japanese Patent Application (kokai) Sho 62-250172 teaches an apparatus and method, according to which a powder that has been preliminarily treated by jet milling is allowed to stay within a chamber for heat treatment under vacuum, where it is subjected to a heat treatment and thence dropped under gravity through a powder feeder into a cylinderal sputtering chamber equipped with a vertical target, whereby the particles in powder are provided with a coating. Unexamined Published Japanese Patent Application (kokai) Hei 2-153068 teaches an apparatus and method, according to which a powder that has been preliminarily treated by jet milling is allowed to stay within a chamber for heat treatment under vacuum, where it is subjected to a heat treatment and thence introduced through a powder feeder into a rotary vessel accommodating a sputter source within a sputtering chamber in the form of a powder (not as single particles), with sputtering being effected as the vessel is rotated. These techniques involve a heating step which is performed before coating so that the jet-milled powder of core particles is allowed to stay for heat treatment and because of this staying of the powder in the heating step, the diamond or high-pressure phase boron nitride particles of any diameter will form agglomerates again which are not single particles and, eventually, such agglomerates will not revert to single particles in the coating step.

Thus, none of the so far proposed techniques have successfully solved the problems associated with the apparatus or method for providing coatings on the core particles in powder which are diamond or high-pressure phase boron nitride particles. In actual cases, diamond or high-pressure phase boron nitride particles having an average diameter of not more than 10 μm form agglomerates which cannot be disintegrated and, hence, no methods or apparatus have been available for producing coated diamond or high-pressure phase boron nitride particles in which said diamond or high-pressure phase boron nitride particles, being dispersed as single particles, are covered on their surfaces with coat forming substances including a bonding material forming substance and/or a sintering aid forming substance.

Speaking of diamond or high-pressure phase boron nitride particles exceeding 10 μm in average diameter, these particles are in actual cases subjected to a coating treatment in the form of agglomerates in which they remain in mutual contact and, hence, those portions which are blocked by other particles remain uncoated. As already mentioned, highly controlled uniform coatings are needed and although even the small cohesive force discussed above has been so much influential as to cause a very serious problem, no methods have been available for producing the satisfactory coated diamond or high-pressure phase boron nitride particles, nor have been apparatus for implementing such methods.

In short, irrespective of the diameter of starting diamond or high-pressure phase boron nitride particles, it has been impossible to prepare coated diamond or high-pressure phase boron nitride particles by providing each of those starting particles with a controlled uniform coat by vapor-phase coating techniques using coat forming substances including bonding material forming substances and/or sintering aid forming substances.

Ex. 1003, Page 245

Hence, it has been impossible to manufacture diamond or high-pressure phase boron nitride sinters of high performance that are uniform, dense and firmly sintered and which have a highly controlled microstructure using small (e.g., ≦ 10 μm) coated diamond or high-pressure phase boron nitride particles. In addition, diamond or high-pressure phase boron nitride sinters of high density and performance that contain comparatively large amounts of diamond or high-pressure phase boron nitride based on those coated particles could not be manufactured in high volumes or large shapes. Further, diamond or high-pressure phase boron nitride sinters of high density and performance that contain comparatively large amounts of diamond or high-pressure phase boron nitride as derived from a mixture of those coated particles and a separately added bonding material could not be manufactured in high volumes or large shapes.

It has also been impossible to manufacture wear-resistant sinters that have dispersed therein comparatively large amounts of coated diamond or high-pressure phase boron nitride particles exceeding, for example, 10 μm in diameter.

BRIEF SUMMARY OF THE INVENTION

Under the circumstances described on the foregoing pages, it is strongly desired to develop and commercialize the following sinters and processes for producing them: a diamond or a high-pressure phase boron nitride sinter of high performance derived from coated diamond or high-pressure phase boron nitride particles in which the particles in a powder of core particles that are diamond or high-pressure phase boron nitride particles to be coated in practice having an average diameter of no more than 10 μm are coated on a single particle basis with a coat forming substance selected from the group consisting of a bonding material forming substance and a sintering aid forming substance, as well as processes for producing such diamond and high-pressure phase boron nitride sinters; a diamond or a high-pressure phase boron nitride sinter of high performance that contains a comparatively large amount of diamond or high-pressure phase boron nitride particles as produced by sintering the coated diamond or high-pressure phase boron nitride particles under pressure and temperature conditions where diamond or high-pressure phase boron nitride is thermodynamically metastable if not stable, as well as processes for producing such diamond and high-pressure phase boron nitride sinters; a diamond or a high-pressure phase boron nitride sinter of high performance that is produced by sintering a mixture of the coated diamond or high-pressure phase boron nitride particles and a separately added bonding material under pressure and temperature conditions where diamond or high-pressure phase boron nitride is thermodynamically metastable if not stable, as well as processes for producing such diamond and high-pressure phase boron nitride sinters; the production of coated diamond or high-pressure phase boron nitride particles in which bare diamond or high-pressure phase boron nitride particles in a powder of core particles exceeding 10 μm in average diameter are provided with a highly controlled uniform coating, which is uniform in that it leaves no surfaces of the individual diamond or high-pressure phase boron nitride particles left uncovered and which is highly controlled in that this uniform coating is applied to all individual diamond or high-pressure phase boron nitride particles, said highly controlled uniform coating being further characterized in that the greater the size of those particles, the more uniform it should be in order to further reduce the uncovered portions of the particles, as well as a diamond or a high-pressure phase boron nitride sinter that uses the thus coated diamond or high-pressure phase boron nitride particles, and processes for producing such diamond and high-pressure phase boron nitride sinters.

Thus, an object of the present invention is to provide a diamond or a high-pressure phase boron nitride sinter of high performance that are uniform, dense and firmly sintered and which have a highly controlled microstructure using coated diamond or high-pressure phase boron nitride particles in which diamond or high-pressure phase boron nitride particles that are either the particles in a powder of fine core particles or the particles in a powder of core particles the greater part of which is comprised of fine particles are coated on a single particle basis with a bonding material forming substance and/or a sintering aid forming substance.

Another object of the invention is to provide processes for producing said diameter or high-pressure phase boron nitride sinters of high performance.

A further object of the invention is to provide a diamond or a high-pressure phase boron nitride sinter that contain comparatively a large amount of diamond or high-pressure phase boron nitride particles comprised of high-performance fine particles, which are uniform, dense and firmly sintered and which have a highly controlled microstructure, which sinters are produced by sintering at pressures less than 2000 MPa and at temperatures not exceeding 1850°C those coated diamond or high-pressure phase boron nitride particles in which diamond or high-pressure phase boron nitride particles that are either the particles in a powder of fine core particles or the particles in a powder of core particles the greater part of which is comprised of fine particles are coated on a single particle basis with a coat forming substance selected from between a bonding material forming substance and a sintering aid forming substance, or a mixture containing said coated diamond or high-pressure phase boron nitride particles.

A still further object of the invention is to provide processes for producing said diamond or high-pressure phase boron nitride sinters.

Yet another object of the invention is to provide a diamond or a high-pressure phase boron nitride sinter that contains comparatively a large amount of diamond or high-pressure phase boron nitride particles comprised of high-performance fine particles, which are uniform, dense and firmly sintered and which have a highly controlled microstructure, which

Ex. 1003, Page 246

sinters are produced by sintering, at pressures less than 2000 MPa and at temperatures not exceeding 1850°C under such pressure and temperature conditions that diamond or high-pressure phase boron nitride is thermodynamically metastable, a mixture of a bonding material and those coated diamond or high-pressure phase boron nitride particles in which diamond or high-pressure phase boron nitride particles that are either the particles in a powder of fine core particles or the particles in a powder of core particles the greater part of which is comprised of fine particles are coated on a single particle basis with coat forming substances comprising not only a bonding material forming substance and/or a sintering aid forming substance but also a surface modifier forming substance capable of controlling grain boundaries.

Still another object of the invention is to provide processes for producing such diamond and high-pressure phase boron nitride sinters.

A further object of the invention is to provide coated quasi-fine diamond or high-pressure phase boron nitride particles in which the particles in a powder of quasi-fine core particles that are quasi-fine diamond or high-pressure phase boron nitride particles or the particles in a powder of core particles the greater part of which is comprised of such quasi-fine particles are provided with a highly controlled uniform coating of a coat forming substance selected from between a bonding material forming substance and a sintering aid forming substance, which coating is uniform in that it leaves no surfaces of the individual diamond or high-pressure phase boron nitride particles left uncovered and which is highly controlled in that this uniform coating is applied to all individual diamond or high-pressure phase boron nitride particles, said highly controlled uniform coating being further characterized in that the greater the size of those particles, the more uniform it should be in order to further reduce the uncovered portions of the particles.

A still further object of the invention is to provide a diamond or a high-pressure phase boron nitride sinter using said coated quasi-fine diamond or high-pressure phase boron nitride particles.

A yet further object of the invention is to provide processes for producing said diamond and high-pressure phase boron nitride sinters.

In order to attain these objects, the present inventors conducted intensive studies and found the following: in order to insure that the particles in a powder of core particles which comprise diamond or high-pressure phase boron nitride particles are coated, on a single particle basis, with coat forming substances including a bonding material and/or a sintering aid, a mixture of a gas with a powder of highly dispersed core particles in which the particles in said powder of core particles are present in a gaseous atmosphere mainly in a single-particle state must be introduced into the coating start region of a coating space while the particles are in a highly dispersed state such that the dispersity β is at least 70%, 80%, 90%, 95%, 97% or 99% depending upon the diameter of the introduced particles and then a coating operation must be started on those particles.

Stated more specifically, the present invention has been accomplished on the basis of the finding of the following two facts: (I) In a mixture of a gas with a powder of highly dispersed core particles in which the particles in said powder of core particles are present in a gaseous atmosphere mainly in a single-particle state, those dispersed particles have a tendency to reagglomerate with the passage of time, chiefly due to turbulent agglomeration, even if they are not allowed to stay within a certain area and once they have formed reagglomerates, it is difficult to disintegrate the latter to a highly dispersed state, namely, to redisperse the agglomerated particles to separate single particles unless they are dispersed by a dispersing means having especially high dispersing performance (the situation is the same as with the agglomerates that formed before the treatment conducted to prepare the highly dispersed core particles) and, to this end, those particles have to be introduced into the coating start region of a coating space while the particles are in a highly dispersed state such that the dispersity β is at least 70%, 80%, 90%, 95%, 97% or 99% depending on the diameter of the introduced particles; and to this end, (II) it is necessary to employ one or more dispersing means that have especially high dispersing performance such that the agglomerates composed of the powder of those core particles can be disintegrated and dispersed in a gaseous atmosphere to provide a very high dispersity in accordance with the specific diameter of the particles under treatment.

The present inventors also found that the coated diamond or high-pressure phase boron nitride particles described above behaved during sintering as if they were the particles of the aforementioned coat forming substances. Consequently, they found that such coated diamond or high-pressure phase boron nitride particles could be sintered in the presence of an added fibrous substance comprising at least one metal or compound that was capable of reinforcing diamond or high-pressure phase boron nitride sinters, which had a width of no more than 500 μm and which was of such a shape that the length to width ratio was at least two. The present invention has been accomplished on the basis of these findings.

According to the present invention, there is provided a process for producing a diamond or high-pressure phase boron nitride sinter comprising the step of preparing coated diamond or high-pressure phase boron nitride particles and sintering said coated diamond or high-pressure phase boron nitride particles or mixtures containing such particles or mixtures of coated diamond or high-pressure phase boron nitride particles with bonding material,

sintering said coated diamond or high-pressure phase boron nitride particles or said mixtures containing such particles at pressures no less than 2000 MPa and at elevated temperatures; or

sintering said coated diamond or high-pressure phase boron nitride particles or said mixtures containing such particles at pressures of less than 2000 MPa and at temperatures not exceeding 1850°C under such sintering pressure

and temperature conditions that diamond or high-pressure phase boron nitride is thermodynamically metastable if not stable; or

sintering said mixtures of coated diamond or high-pressure phase boron nitride particles with a bonding material in a volume ratio of (1 - 90):(99 - 10), said bonding material being sintrable to a density of at least 85% at pressures less than 2000 MPa and at temperatures not exceeding 1850°C under such conditions that diamond and/or high-pressure phase boron nitride particles are thermodynamically metastable, at pressures less than 2000 MPa and at temperatures not exceeding 1850°C under such sintering pressure and temperature conditions that diamond and/or high-pressure phase boron nitride is thermodynamically metastable if not stable.

wherein said coated diamond or high-pressure phase boron nitride particles are prepared by a method comprising the steps of charging a powder of diamond or high-pressure phase boron nitride core particles into a coating space and then permitting a precursor of a coat forming substance which is generated via the vapor phase and/or a precursor of a coat forming substance in a vapor-phase state to contact and/or impinge against the particles in said powder of core particles so that their surfaces are coated with the coat forming substance and

said coated diamond or high-pressure phase boron nitride particles are prepared by coating means comprising:

(A) a dispersing step in which the particles in the powder of core particles are dispersed by a group of means for high dispersion treatment of particles in a gaseous atmosphere to form a mixture of a gas and the particles in a powder of highly dispersed particles, the final treating means in said group of means for high dispersion treatment of particles being selected from between:

(a) dispersing means for dispersing the particles in the powder of core particles in a gaseous atmosphere; and
(b) means for selecting a mixture of a gas and the particles in a powder of highly dispersed particles which comprises:

(b-1) means for selecting a mixture of a gas and the particles in a powder of highly dispersed particles that separates a portion of the powder that is composed of less highly dispersed particles from the mixture of a gas and the particles in the powder of core particles in which the particles in the powder of core particles are dispersed in a gaseous atmosphere, thereby selecting a mixture of a gas and the particles in a powder of highly dispersed core particles in which the particles in the powder of core particles are present in the gaseous atmosphere mainly in a single-particle state; and
(b-2) feedback means by which the portion of the powder that is composed of less highly dispersed particles and which has been separated by said means for selecting a mixture of a gas and the particles in a powder of highly dispersed particles is transported to the final dispersing means among the dispersing means in the group of means for high dispersion treatment of particles and/or treating means upstream of the final dispersing means; and

(B) a coating step in which the particles in the powder of core particles that have been dispersed in the dispersing step (A) start to be coated by being allowed to contact and/or impinge against the precursor of the coat forming substance in the coating start region of the coating space as they are dispersed to satisfy the following conditions:

the dispersity β should be at least 70% if those particles have an average diameter of no more than 10 μm in the frequency distribution by volume;

the dispersity β should be at least 80% if those particles have an average diameter in excess of 10 μm but no more than 20 μm in the frequency distribution by volume;

the dispersity β should be at least 90% if those particles have an average diameter in excess of 20 μm but no more than 50 μm in the frequency distribution by volume;

the dispersity β should be at least 95% if those particles have an average diameter in excess of 50 μm but no more than 300 μm in the frequency distribution by volume;

the dispersity β should be at least 97% if those particles have an average diameter in excess of 300 μm but no more than 800 μm in the frequency distribution by volume; and

the dispersity β should be at least 99% if those particles have an average diameter in excess of 800 μm in the frequency distribution by volume, and wherein said sintering step is performed by

sintering said coated diamond or high-pressure phase boron nitride particles or said mixtures containing such particles at pressures no less than 2000 MPa and at elevated temperatures; or

sintering said coated diamond or high-pressure phase boron nitride particles or said mixtures containing such particles at pressures of less than 2000 MPa and at temperatures not exceeding 1850°C under such sintering pressure and temperature conditions that diamond or high-pressure phase boron nitride is thermodynamically metastable if not stable; or

sintering said mixtures of coated diamond or high-pressure phase boron nitride particles with a bonding material in a volume ratio of (1 - 90):(99 - 10), said bonding material being sintrable to a density of at least 85% at

pressures less than 2000 MPa and at temperatures not exceeding 1850°C under such conditions that diamond and/or high-pressure phase boron nitride particles are thermodynamically metastable, at pressures less than 2000 MPa and at temperatures not exceeding 1850°C under such sintering pressure and temperature conditions that diamond and/or high-pressure phase boron nitride is thermodynamically metastable if not stable.

Preferably, said coated diamond or high-pressure phase boron nitride particles are prepared through the following additional step(s):

a step of disagglomeration or otherwise size reduction in which lumps of the coated diamond and/or high-pressure phase boron nitride particles that form as a result of association of several particles that contact one another via the coat forming substance on the coated particles are disagglomerated and/or otherwise reduced in size; and/or

a step of selective separation in which the associating lumps of the coated diamond or high-pressure phase boron nitride particles are selectively separated from the coated diamond or high-pressure phase boron nitride particles on a primary particle basis.

Preferably, the particles in the powder of diamond and/or high-pressure phase boron nitride core particles to be coated with the coat forming substance are coated by a dip method using a molten salt bath with one or more layers of the coat forming substance that derives from the dip method.

Preferably, the dispersing step is performed by:

a group of means for high dispersion treatment of particles that has such dispersing performance that a powder of core particles having an average diameter of no more than 10 μm in the frequency distribution by volume is dispersed in a gaseous atmosphere by the final treatment by said group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and that the dispersity β of the particles in said powder of core particles is adjusted to at least 70%;

a group of means for high dispersion treatment of particles that has such dispersing performance that a powder of core particles having an average diameter in excess of 10 μm but no more than 20 μm in the frequency distribution by volume is dispersed in a gaseous atmosphere by the final treatment by said group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and that the dispersity β of the particles in said powder of core particles is adjusted to at least 80%;

a group of means for high dispersion treatment of particles that has such dispersing performance that a powder of core particles having an average diameter in excess of 20 μm but no more than 50 μm in the frequency distribution by volume is dispersed in a gaseous atmosphere by the final treatment by the group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and that the dispersity β of the particles in said powder of core particles is adjusted to at least 90%;

a group of means for high dispersion treatment of particles that has such dispersing performance that a powder of core particles having an average diameter in excess of 50 μm but no more than 300 μm in the frequency distribution by volume is dispersed in a gaseous atmosphere by the final treatment by the group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and that the dispersity β of the particles in said powder of core particles is adjusted to at least 95%;

a group of means for high dispersion treatment of particles that has such dispersing performance that a powder of core particles having an average diameter in excess of 300 μm but no more than 800 μm in the frequency distribution by volume is dispersed in a gaseous atmosphere by the final treatment by the group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and that the dispersity β of the particles in said powder of core particles is adjusted to at least 97%; and

a group of means for high dispersion treatment of particles that has such dispersing performance that a powder of core particles having an average diameter in excess of 800 μm in the frequency distribution by volume is dispersed in a gaseous atmosphere by the final treatment by the group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and that the dispersity β of the particles in said powder of core particles is adjusted to at least 99%; and

said coated diamond or high-pressure phase boron nitride particles being prepared by:

a method in which the mixture of a gas and the particles in the powder of highly dispersed core particles as formed by one of the means in said group of means for high dispersion treatment of particles is either discharged directly to the coating step or transported thereto via at least one member indispensable to transport that is selected from the group consisting of a hollow member, an intermediate member composed of a member that forms a hollow portion, and a pipe and which is provided between the dispersing and coating steps, as the mixture emerges from a discharge section that discharges said mixture of a gas and the particles; and/or

a method in which the mixture of a gas and the particles in the powder of highly dispersed core particles as formed by one of the means in said group of means for high dispersion treatment of particles is transported to the coating step via at least one means selected from the group consisting of means for maintaining the dispersion in a gaseous atmosphere of the highly dispersed core particles in the mixture of a gas with the core particles which have been dispersed in the gaseous atmosphere to attain one of the dispersities set forth above, means for enhancing the dispersion in a

Ex. 1003, Page 249

gaseous atmosphere of the highly dispersed core particles in the mixture of a gas with the core particles which have been dispersed in the gaseous atmosphere to attain one of the dispersities set forth above, and means for separating a mixture of a gas and the particles in a powder of less highly dispersed core particles from said gas-particle mixture so as to select a mixture of a gas and the particles in a powder of highly dispersed core particles in which the core particles are present in the gaseous atmosphere mainly in a single-particle state.

Preferably, the dispersing step is performed by:

a group of means for high dispersion treatment of particles that has such dispersing performance that a powder of core particles having an average diameter of no more than 10 μm in the frequency distribution by volume is dispersed in a gaseous atmosphere by the final treatment by said group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and that the dispersity β of the particles in said powder of core particles is adjusted to at least 70%;

a group of means for high dispersion treatment of particles that has such dispersing performance that a powder of core particles having an average diameter in excess of 10 μm but no more than 20 μm in the frequency distribution by volume is dispersed in a gaseous atmosphere by the final treatment by said group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and that the dispersity β of the particles in said powder of core particles is adjusted to at least 80%;

a group of means for high dispersion treatment of particles that has such dispersing performance that a powder of core particles having an average diameter in excess of 20 μm but no more than 50 μm in the frequency distribution by volume is dispersed in a gaseous atmosphere by the final treatment by the group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and that the dispersity β of the particles in said powder of core particles is adjusted to at least 90%;

a group of means for high dispersion treatment of particles that has such dispersing performance that a powder of core particles having an average diameter in excess of 50 μm but no more than 300 μm in the frequency distribution by volume is dispersed in a gaseous atmosphere by the final treatment by the group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and that the dispersity β of the particles in said powder of core particles is adjusted to at least 95%;

a group of means for high dispersion treatment of particles that has such dispersing performance that a powder of core particles having an average diameter in excess of 300 μm but no more than 800 μm in the frequency distribution by volume is dispersed in a gaseous atmosphere by the final treatment by the group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and that the dispersity β of the particles in said powder of core particles is adjusted to at least 97%; and

a group of means for high dispersion treatment of particles that has such dispersing performance that a powder of core particles having an average diameter in excess of 800 μm in the frequency distribution by volume is dispersed in a gaseous atmosphere by the final treatment by the group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and that the dispersity β of the particles in said powder of core particles is adjusted to at least 99%; and

said coated diamond or high-pressure phase boron nitride particles being prepared by performing at least one part of the dispersing step in such a way that it shares one or more parts of the space with at least one part of the coating step.

Preferably, said coated diamond or high-pressure phase boron nitride particles are prepared with the coating start region of the coating space being located in either one of the following space regions that includes planes through which all particles in the powder of highly dispersed core particles as mixed with a gas will pass:

a space region in which a powder of core particles that have an average diameter of no more than 10 μm in the frequency distribution by volume is dispersed in a gaseous atmosphere by the final treatment by said group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and that the dispersity β of the particles in said powder of core particles is adjusted to at least 70%;

a space region in which a powder of core particles that have an average diameter in excess of 10 μm but no more than 20 μm in the frequency distribution by volume is dispersed in a gaseous atmosphere by the final treatment by said group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and that the dispersity β of the particles in said powder of core particles is adjusted to at least 80%;

a space region in which a powder of core particles that have an average diameter in excess of 20 μm but no more than 50 μm in the frequency distribution by volume is dispersed in a gaseous atmosphere by the final treatment by the group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and that the dispersity β of the particles in said powder of core particles is adjusted to at least 90%;

a space region in which a powder of core particles that have an average diameter in excess of 50 μm but no more than 300 μm in the frequency distribution by volume is dispersed in a gaseous atmosphere by the final treatment by the

Ex. 1003, Page 250

group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and that the dispersity β of the particles in said powder of core particles is adjusted to at least 95%;

a space region in which a powder of core particles that have an average diameter in excess of 300 μm but no more than 800 μm in the frequency distribution by volume is dispersed in a gaseous atmosphere by the final treatment by the group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and that the dispersity β of the particles in said powder of core particles is adjusted to at least 97%; and

a space region in which a powder of core particles that have an average diameter in excess of 800 μm in the frequency distribution by volume is dispersed in a gaseous atmosphere by the final treatment by the group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and that the dispersity β of the particles in said powder of core particles is adjusted to at least 99%, or wherein

said coated diamond or high-pressure phase boron nitride particles are prepared with the coating start region of the coating space being located in either one of the following space regions that includes planes through which all particles to be recovered into the recovery section of the recovery means will pass:

a space region in which a powder of core particles that have an average diameter of no more than 10 μm in the frequency distribution by volume is dispersed in a gaseous atmosphere by the final treatment by said group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and that the dispersity β of the particles in said powder of core particles is adjusted to at least 70%;

a space region in which a powder of core particles that have an average diameter in excess of 10 μm but no more than 20 μm in the frequency distribution by volume is dispersed in a gaseous atmosphere by the final treatment by said group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and that the dispersity β of the particles in said powder of core particles is adjusted to at least 80%;

a space region in which a powder of core particles that have an average diameter in excess of 20 μm but no more than 50 μm in the frequency distribution by volume is dispersed in a gaseous atmosphere by the final treatment by the group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and that the dispersity β of the particles in said powder of core particles is adjusted to at least 90%;

a space region in which a powder of core particles that have an average diameter in excess of 50 μm but no more than 300 μm in the frequency distribution by volume is dispersed in a gaseous atmosphere by the final treatment by the group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and that the dispersity β of the particles in said powder of core particles is adjusted to at least 95%;

a space region in which a powder of core particles that have an average diameter in excess of 300 μm but no more than 800 μm in the frequency distribution by volume is dispersed in a gaseous atmosphere by the final treatment by the group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and that the dispersity β of the particles in said powder of core particles is adjusted to at least 97%; and

a space region in which a powder of core particles that have an average diameter in excess of 800 μm in the frequency distribution by volume is dispersed in a gaseous atmosphere by the final treatment by the group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and that the dispersity β of the particles in said powder of core particles is adjusted to at least 99%.

Preferably, the particles in the powder of core particles to be used have a size distribution of ($[D_M/5, 5D_M]$, ≧ 90%) in terms of frequency distribution by volume, where D_M represents the average particle diameter.

According to another aspect of the present invention, there is provided a diamond or high-pressure phase boron nitride sinter that are produced by the processes for producing a diamond or high-pressure phase boron nitride sinter as recited above.

Thus, in accordance with the present invention, diamond or high-pressure phase boron nitride sinters are produced by one of the following methods: the particles in a powder of core particles that are diamond or high-pressure phase boron nitride particles and the surfaces of which are coated with a coat forming substance or mixtures containing such particles are sintered at a pressure of 2000 MPa or higher and at an elevated temperature; or those particles or mixtures containing them are sintered at a pressure less than 2000 MPa and at a temperature not exceeding 1850°C under such sintering pressure and temperature conditions that diamond or high-pressure phase boron nitride is thermodynamically metastable if not stable; or a mixture of the coated diamond or high-pressure phase boron nitride particles and a bonding material in a volume ratio of (1 - 90):(99 - 10), which bonding material is sinterable to a density of at least 85% at a

Ex. 1003, Page 251

pressure of less than 2000 MPa and at a temperature not exceeding 1850°C under such conditions that diamond or high-pressure phase boron nitride is thermodynamically metastable, is sintered at a pressure of less than 2000 MPa and at a temperature not exceeding 1850°C under such sintering pressure and temperature conditions that diamond or high-pressure phase boron nitride is thermodynamically metastable if not stable. In the practice of this invention, the diamond or high-pressure phase boron nitride particles the surfaces of which are coated with the coat forming substance are prepared by the following method: a precursor for the coat forming substance that has just formed via the vapor phase by a vapor-phase process and/or a precursor for the coat forming substance in a vapor-phase state and a mixture of a gas with a powder of highly dispersed core particles that have been dispersed in a gaseous atmosphere by the final treating means in a group of means for high dispersion treatment of particles are allowed to combine with each other in the coating start region of a coating space while the particles in the mixture of a gas and the powder of highly dispersed core particles are in a dispersed state with their dispersity being adjusted to one of the values set forth in preceding paragraphs depending upon their diameter, whereupon the precursor and the mixture are allowed to contact and/or impinge against each other so that the surfaces of the diamond or high-pressure phase boron nitride particles are coated with the coat forming substance. By so doing, one can produce a heretofore unattainable diamond or high-pressure phase boron nitride sinter of high performance that has no surfaces of diamond or high-pressure phase boron nitride particles left unsintered, that is uniform, dense and firmly sintered and which has a highly controlled microstructure. The precursor for the coat forming substance which is to be used in preparing the coated core particles is in a vapor-phase state as comprised of atoms, molecules, ions, clusters, atomic clusters, molecular clusters, cluster ions, etc. or it has just formed via the vapor phase; having started to contact and/or impinge against the diamond or high-pressure phase boron nitride particles in the already-discussed highly dispersed state, the coat forming substance binds firmly to the surfaces of the individual core particles in the state of primary particles, thereby yielding coated diamond or high-pressure phase boron nitride particles in which those core particles in powder form are covered with the coat forming substance on a single particle basis.

As will be understood from the above description, when the coated diamond or high-pressure phase boron nitride particles are sintered at a pressure of 2000 MPa or above and at an elevated temperature under such sintering conditions that diamond or high-pressure phase boron nitride is thermodynamically stable, the coats on the diamond or high-pressure phase boron nitride particles are bursted by the pushing force from inside and any two contiguous diamond or high-pressure phase boron nitride particles will bind directly to each other, thereby yielding an extremely uniform and dense sinter.

The invention also contemplates the case where the coated diamond or high-pressure phase boron nitride particles, either taken alone or in combination with a bonding material, are sintered at a pressure less than 2000 MPa and at a temperature not exceeding 1850°C under such sintering pressure and temperature conditions that diamond or high-pressure phase boron nitride is thermodynamically metastable if not stable. In this case, the diamond or high-pressure phase boron nitride particles may contact each other but will not bind directly to each other; nevertheless, except in areas where the coated diamond or high-pressure phase boron nitride particles are in direct contact with each other, the coating substance which is to form a bonding material or a sintering aid is sure to surround those diamond or high-pressure phase boron nitride particles, and the resulting sinter has no part left unsintered, is uniform, dense and firmly sintered, and has an extremely highly controlled microstructure.

BRIEF DESCRIPTION OF THE DRAWINGS

Fig. 1a is a diagram showing the dispersity β of particles in a powder;

Fig. 1b is a diagram showing the frequency by volume vs diameter relationship of a powder 90% of the volume of which is occupied by particles with sizes in the range from D_1 - D_2;

Figs. 2a, 2b and 2c are block diagrams showing three basic arrangements of means for high dispersion treatment of particles;

Figs. 3a - 3g are block diagrams showing in greater detail the arrangements of means for high dispersion treatment of particles;

Figs. 4a - 4e are diagrams showing how the coating operation is started on core particles in powder;

Figs. 5a - 5g are block diagrams showing the compositions of various apparatus for producing coated diamond or high-pressure phase boron nitride particles;

Fig. 6 is a diagram showing the apparatus used in Example 1;

Fig. 7 is a partial enlarged view of the apparatus used in Example 1;

Fig. 8 is a scanning electron micrograph showing one of the coated diamond particles produced in Example 1;

Fig. 9 is an electron micrograph showing a polished surface of the sinter produced in Example 1;

Fig. 10 is an electron micrograph showing a polished surface of a commercial sintered diamond tool;

Fig. 11 is a diagram showing the apparatus used in Example 2;

Fig. 12 is a partial enlarged view of the apparatus used in Example 2;

Fig. 13 is a diagram showing the apparatus used in Example 3;

Ex. 1003, Page 252

Fig. 14 is a partial enlarged view of the apparatus used in Example 3;

Fig. 15 is a scanning electron micrograph showing one of the coated cubic boron nitride particles produced in Example 5-1;

Fig. 16 is a graph showing Vickers microhardness of the cubic boron nitride sinters;

Fig. 17 is an electron micrograph showing a polished surface of the sinter produced in Example 5-1;

Fig. 18 is an electron micrograph showing a polished surface of a commercial sintered cubic boron nitride tool;

Fig. 19 is a scanning electron micrograph showing one of the coated diamond particles produced in Example 6-1;

Fig. 20 is an electron micrograph showing a polished surface of the sinter produced in Example 6-1;

Fig. 21 is an electron micrograph showing a polished surface of a sinter comprising uncoated diamond particles;

Fig. 22 is a scanning electron micrograph showing one of the coated cubic boron nitride particles produced in Example 7-1;

Fig. 23 is an electron micrograph showing a polished surface of the sinter produced in Example 7-1;

Fig. 24 is an electron micrograph showing a polished surface of a sinter comprising uncoated cubic boron nitride particles;

Fig. 25 is a scanning electron micrograph showing one of the coated diamond particles produced in Example 8-1;

Fig. 26 is an electron micrograph showing a polished surface of the sinter produced in Example 8-1;

Fig. 27 is a scanning electron micrograph showing one of the coated cubic boron nitride particles produced in Example 9-1;

Fig. 28 is an electron micrograph showing a polished surface of the sinter produced in Example 9-1;

Fig. 29 is a scanning electron micrograph showing one of the coated diamond particles produced in Example 10-1;

Fig. 30 is an electron micrograph showing a polished surface of the sinter produced in Example 10-1;

Fig. 31 is a scanning electron micrograph showing one of the coated cubic boron nitride particles produced in Example 11-1;

Fig. 32 is an electron micrograph showing a polished surface of the sinter produced in Example 11-1;

Fig. 33a is a pressure-temperature diagram showing the region where diamond is stable; and

Fig. 33b is a pressure-temperature diagram showing the region where high-pressure phase boron nitride is stable.

DETAILED DESCRIPTION OF THE INVENTION

The present invention is described below in detail. Before going into details of the invention, let us define the various terms, phrases and expressions that are used herein, and describe, if necessary, the specific implications of those terms, phrases and expressions, and then proceed to the description of the technical means for preparing diamond or high-pressure phase boron nitride particles coated with coat forming substances.

Coated Diamond or High-Pressure Boron Nitride Particles

The term "coated diamond particles" refers to the diamond particles to be described below which are provided with coats. The term "coated high-pressure phase boron nitride particles" refers to the high-pressure phase boron nitride particles to be described below which are provided with coats. To give one specific example, the terms refer to those coated particles in which diamond or high-pressure phase boron nitride core particles are provided with the coats of coat forming substances in the form of at least one type selected from among ultrafine particles, islands, continuous phase, uniform membranes, projections, etc.

Feed Powder Particles for Diamond or High-Pressure Boron Nitride Sinters

Natural and/or synthetic diamond powder particles are used as feed powder particles for diamond sinters in the present invention. On the other hand, cubic and/or wurtzite boron nitride particles are used as feed powder particles for high-pressure phase boron nitride sinters in the present invention.

To produce diamond or high-pressure phase boron nitride sinters of high performance which suit the purposes of the present invention, diamond or high-pressure phase boron nitride particles with diameters of no more than 10 μm are advantageously used as feed powders. However, in the case where wear resistance is of particular importance, particles exceeding 10 μm in average diameter are advantageous. Stated specifically, the particle diameter can be expressed in terms of frequency distribution by volume such that the average particle diameter D_M satisfies the relation ($[D_M/5, 5D_M]$, ≧ 90%). However, depending on use, diamond or high-pressure phase boron nitride particles of a comparatively narrow distribution which, when expressed in terms of frequency distribution by volume, satisfies the relation ($(D_M/3, 3D_M]$, ≧ 90%) can preferably be selected. More advantageously, one may select diamond or high-pressure phase boron nitride particles that have been controlled, by sieve classification or other techniques, in the diameter of diamond or high-pressure phase boron nitride particles to satisfy the relation ($[D_M/2, 3D_M/2]$, ≧ 90%) in terms of frequency distribution by volume.

Ex. 1003, Page 253

If diamond or high-pressure phase boron nitride particles having such distributions are used as feed powder particles and, by sintering those diamond or high-pressure phase boron nitride particles or mixtures containing those coated diamond or high-pressure phase boron nitride particles, one can manufacture diamond or high-pressure phase boron nitride sinters of high performance that are uniform, dense and firmly sintered and which have an extremely highly controlled microstructure, those sinters being characterized in that the diamond or high-pressure phase boron nitride particles are sure to be surrounded by a bonding material and/or a sintering aid and/or a surface modifier except in areas where the coat forming substance is bursted by the pushing force from inside and any two contiguous diamond or high-pressure phase boron nitride particles bind directly to each other and also characterized in that no part of the surfaces of the diamond or high-pressure phase boron nitride particles in the coated diamond or high-pressure phase boron nitride sinters is left unsintered.

It should be particularly noted when sintering is done under sintering pressure and temperature conditions where diamond or high-pressure phase boron nitride is thermodynamically metastable if not stable, the diamond or high-pressure phase boron nitride is advantageously of as high purity as to inhibit a phase transition to the low-pressure phase (graphite or hBN). As for diamond, grades of ultrahigh purity can be selected from natural products; however, high-pressure phase boron nitride does not occur naturally. Hence, with synthetic products of diamond or high-pressure phase boron nitride, any substance that has a catalytic action and which is used during synthesis is advantageously removed thereafter as much as possible. Most advantageously, one can select, for example, a diamond or cubic boron nitride of ultrahigh purity that has been synthesized via the vapor phase by a suitable technique such as a physical vapor deposition (PVD) process or a chemical vapor deposition (CVD) process. When the synthesized product is in the form of a thin film, it is used after being reduced in size with care being taken to avoid contamination with impurities. If the synthesized product is in a particulate or powder form, it may be used as such. Another example of high-purity product that can be selected is a high-purity single-crystal one which has been grown over time. Alternatively, diamond or cubic boron nitride particles that have been positively freed of impurities may be selected.

Vapor-Phase Coating Method

The term "vapor-phase coating method" means a method in which coating is applied with the feed of a coat forming substance being passed at least once through a vapor-phase state that consists of at least one member selected from among a molecular stream, an ionic stream, a plasma, a gas, a vapor and an aerosol; alternatively, the term refers to a method in which coating is applied from the feed of a coat forming substance in either one of the vapor-phase states mentioned above.

Core Particles

The term "core particles" refers to those particles which are to be provided with coatings. Such particles may sometimes be referred to as "base particles", "seed particles" or "particles to be coated".

The core particles are diamond or high-pressure phase boron nitride particles and the high-pressure phase boron nitride particles consist specifically of cubic boron nitride particles and/or wurtzite boron nitride particles.

Powder of Core Particles

The term "powder of core particles" means a powder consisting of core particles. The phrase "the particles in a powder of core particles" refers to those particles which compose the powder of core particles. The particles in the powder of core particles to be coated which are used in the present invention range from fine particles the average diameter of which is not more than 10 μm in terms of frequency distribution by volume to those particles the average diameter of which exceeds 800 μm in terms of frequency distribution by volume.

Preferred particles are those which have such a particle size distribution that the average diameter D_M satisfies the relation ($[D_M/5, 5D_M]$, $\geqq$ 90%) in terms of frequency distribution by volume.

With powders having such comparatively narrow distributions, their dispersion or cohesion characteristics are characterized by average particle diameter and the powders can be dispersed by operating a group of means for high dispersion treatment of particles under conditions that suit the specific value of D_M.

With a powder of core particles the particle size distribution of which is either broad or has a plurality of separate spaced peaks, a suitable selective separation treatment, say, classification may advantageously be performed and the individual classified powder portions are subjected to the coating treatment according to the invention. By so doing, coating can be started on the individual classified powder portions under the above-stated conditions in the coating start region of the coating space with the dispersity β adjusted to at least 70%, 80%, 90%, 95%, 97% or 99% depending on the average particle diameter, whereupon the individual particles in the powder of core particles can be provided with coatings.

Ex. 1003, Page 254

Coat Forming Substance

The term "coat forming substance" means a substance that forms a coat on the particles to be coated. This may specifically be exemplified by those substances which provide the particles in the powder of core particles with coatings in the form of at least one kind selected from among ultrafine particles, islands, continuous phase, uniform membranes, projections, etc.

Particularly in the case where the coat forming substance assumes the form of ultrafine particles, their diameter may range from 0.005 μm to 0.5 μm.

The coat forming substance may form a coating by itself or, alternatively, it reacts with the diamond or high-pressure phase boron nitride in the core particles and/or dissolves in the diamond or high-pressure phase boron nitride particles to form a solid solution and/or two or more coat forming substances may be reacted and/or alloyed with each other and/or dissolved in each other to form coatings. To this end, the coat forming substance is selected from among elemental substances and/or compounds that generate one or more of the desired inorganic compounds, alloys, intermetallic compounds, etc. to form such coatings, thereby serving as sintering aids and/or bonding materials which promote the sintering of the coated diamond or high-pressure phase boron nitride particles and/or as surface modifiers for the diamond or high-pressure phase boron nitride particles.

When sintering is done under sintering pressure and temperature conditions where diamond or high-pressure phase boron nitride is thermodynamically metastable if not stable, the diamond or high-pressure phase boron nitride particles are to be coated with a coat forming substance that is selected from among those which will not promote the phase transition of diamond or high-pressure phase boron nitride to the lower-pressure phase (graphite or hBN). The coat forming substance is also selectable as a surface modifier which controls the grain boundaries of the diamond or high-pressure phase boron nitride particles. Various modifications may be performed as required and they include, for example: enhancing the chemical bonding between the diamond or high-pressure phase boron nitride particles and said sintering aid and/or bonding material; isolating the individual diamond or, high-pressure phase boron nitride particles from a certain substance, thereby inhibiting the phase transition of diamond or high-pressure phase boron nitride to the lower-pressure phase (graphite or hBN); or inhibiting the reaction between diamond or high-pressure phase boron nitride with a certain substance. These methods contribute to an outstanding increase in the latitude in the selection of the coat forming substance as a sintering aid and/or bonding material, which certainly is a definite advantage for the purposes of the invention.

These coat forming substances can be those which contain at least one member selected from among metals, semiconductors, metalloids, rare earth metals and nonmetals of groups 1a, 2a, 3a, 4a, 5a, 6a, 7a, 1b, 2b, 3b, 4b, 5b, 6b, 7b and 8 of the periodic table, as well as oxides, nitrides, carbides, oxynitrides, oxycarbides, carbonitrides, oxycarbonitrides, borides and silicides thereof, as exemplified by the following: Al, B, Si, Fe, Ni, Co, Ti, Nb, V, Zr, Hf, Ta, W, Re, Cr, Cu, Mo, Y, La, TiAl, Ti_3Al, $TiAl_3$, TiNi, NiAl, Ni_3Al, SiC, TiC, ZrC, B_4C, WC, W_2C, HfC, VC, TaC, Ta_2C, NbC, Mo_2C, Cr_3C_2, Si_3N_4, TiN, ZrN, Si_2N_2O, AlN, HfN, VxN (x = 1 ~ 3), NbN, TaN, Ta_2N, TiB, TiB_2, ZrB_2, VB, V_3B_2, VB_2, NbB, NbB_2, TaB, TaB_2, MoB, MoB_2, MoB_4, Mo_2B, WB, W_2B, W_2B_5, LaB_6, $B_{13}P_2$, $MoSi_2$, BP, Al_2O_3, ZrO_2, $MgAl_2O_4$ (spinel) and Al_2SiO_5 (mullite).

The amounts in which the coat forming substances are added to form the coats on the surfaces of the coated diamond or high-pressure phase boron nitride particles are not limited in any particular way and suitable values can be selected that suit specific uses; advantageously, any desired values are selected as long as they are capable of densifying the diamond or high-pressure phase boron nitride sinters.

Uniform Coating

In the case where the coat forming substance takes the form of a uniform membrane, the term "uniform coating" means that the coating film is uniform in every portion of a single particle. In the case where the coat forming substance takes the form of an ultrafine particle, an island or a projection, the term "uniform coating" means that the coat forming substance in the form of an ultrafine particle, an island or a projection provides a coating of uniform distribution. It should be understood that any unevenness that is unavoidable in the process of generation of the coat forming substance will be embraced within the category of "uniformity".

Definition of "Charging into the Coating Space"

The phrase "charging into the coating space" means introducing the powder of core particles into the coating space by a certain method of falling such as free fall. In the case of charging by means of a carrier gas, the phrase means introducing the powder of core particles as it is carried in the direction of a mixture of a gas and the particles in the powder of core particles, or carried by a gas in the direction of its flow, or carried by a gas to change the direction of flow. Alternatively, the phrase means introduction under the action of a carrier gas, for example, through the wave motion, specifically, a nonlinear wave motion, of the carrier gas. Alternatively, the phrase means introduction into the coating

Ex. 1003, Page 255

space by sound waves, ultrasonic waves, magnetic fields, electron beams, etc. in the gas. The phrase also means introduction in an applied field such as an electric field, a magnetic field or electron beams. Specifically, the powder particles may be charged or magnetized in an electric field, a magnetic field, by electron beams, etc. and introduced into the coating space by attractive or repulsive forces. Also contemplated by the phrase is introduction by aspiration under the back pressure of the gas or in vacuo.

Coating Space

The term "coating space" refers to a space in which the precursor of the coat forming substance which is generated from the feed of the coat forming substance via the vapor phase and/or the precursor of the coat forming substance in a vapor-phase state contacts and/or impinges against the particles in the powder of core particles. Alternatively, the term refers to a space region in which the surfaces of the particles in the powder of core particles are coated with the coat forming substance.

Coating Chamber

The term "coating chamber" means a compartment having the coating space in at least one part. More specifically, the coating chamber is a partitioned or generally partitioned (generally closed or semi-closed) compartment including the coating space and it is a compartment including the coating space in at least one part.

Within Gaseous Atmosphere

The expression "within a gaseous atmosphere" means within a space under vacuum or in a vapor-phase state. The term "vapor-phase state" as used herein means various states such as a molecular stream, an ionic stream, a plasma, a gas and a vapor. Technically speaking, the term "vacuum" refers to a state under reduced pressure. Strictly speaking, gases, molecules, atoms, ions, etc. are contained at any values of reduced pressure.

Precursor of the Coat Forming Substance

The term "precursor of the coat forming substance" means a precursor for the coat forming substance. More specifically, it refers to the feed per se of the coat forming substance in a vapor-phase state or, alternatively, it means a substance that is formed and/or synthesized from the feed of the coat forming substance via the vapor phase and which exists until just before it forms a coating on the core particles to be coated. The precursor of the coat forming substance is not limited to any state as long as it is formed and/or synthesized from the feed of the coat forming substance via the vapor phase. If the feed of the coat forming substance is in the vapor phase, the feed itself can be used as the precursor of the coat forming substance. The precursor of the coat forming substance may itself be in the vapor phase. If the precursor of the coat forming substance is a reactive substance, it may be prior to, or during or after the reaction. Specific examples of the precursor of the coat forming substance include ions, atoms, molecules, clusters, atomic clusters, molecular clusters, cluster ions, superfine particles, gases, vapors, aerosols, etc.

Feed of the Coat Forming Substance

The term "feed of the coat forming substance" refers to a feed material that passes through the vapor phase to become a coat forming substance. Specific examples of the state in which the feed of the coat forming substance may exist include solid lumps, powder particles, gases, liquids, etc.

Dispersity β

Dispersity β was proposed by Masuda, Gotoh et al. as an index for evaluating the dispersing performance of powder dispersing equipment [see KAGAKU KOGAKU, Summaries of Speeches and Lectures Delivered at the 22th Autumn Conference, p. 349 (1989)] and is defined as the proportion of the weight of particles in the state of apparent primary particles relative to the total weight of the particles present. The term "particles in the state of apparent primary particles" as used herein refers to the proportion of the overlap between the frequency distribution by mass of powder particles in a given state of dispersion f_{m2} and the frequency distribution by mass of completely dispersed powder particles f_{m1} and is expressed by β according to the following equation:

Ex. 1003, Page 256

$$\beta = \int_0^{D_m^*} f_{m2}(D)dD + \int_{D_m^*}^{\infty} f_{m1}(D)dD$$

where

D: particle diameter (μm);
D_m^*: the particle diameter (μm) for the point of crossing between f_{m1} and f_{m2};
f_{m1}: the frequency distribution by mass (%/μm) representing the particle size distribution for complete dispersion;
f_{m2}: the frequency distribution by mass (%/μm) representing the particle size distribution of particles in a given state of dispersion; and
β: dispersity (%).

In the equation set forth above, the unit of measurement (μm) of the particle diameter is not limited to any particular value.

This equation evaluates dispersity on the basis of the particle size distribution as expressed by mass; however, dispersity should in principle be evaluated on the basis of the particle size distribution expressed by volume. But given the same density of powder particles, the particle size distribution as expressed by mass is equal to the particle size distribution expressed by volume. Therefore, in practice, the particle size distribution by mass which is easy to measure is determined and used as an equivalent of the particle size distribution by volume. Therefore, the dispersity β in the true sense of the term is expressed by the following equation and the area of the hatched portion shown in Fig. 1a:

$$\beta = \int_0^{D_v^*} f_{v2}(D)dD + \int_{D_v^*}^{\infty} f_{v1}(D)dD$$

where

D: particle diameter (μm);
D_v^*: the particle diameter (μm) for the point of crossing between f_{v1} and f_{v2};
f_{v1}: the frequency distribution by volume (%/μm) representing the particle size distribution for complete dispersion;
f_{v2}: the frequency distribution by volume (%/μm) representing the particle size distribution of particles in a given state of dispersion; and
β: dispersity (%).

In the equation set forth above, the unit of measurement (μm) of the particle diameter is not limited to any particular value.

In the following description, the distribution of the particles in the powder of core particles and their average diameter should, in principle, be based on volume unless otherwise noted.

Frequency Distribution by Volume

The frequency distribution by volume represents the distribution of particle diameter in terms of the relative volume of particles in a certain range of diameters.

Definition of ([D1,D2], ≧ 90%)

A ([D1,D2], ≧ 90%) distribution represents one in which particles from D1 to D2 (D1 and D2 each represent the particle diameter, provided that D1 < D2) account for at least 90% of the total volume of the particles present; a powder having this distribution is shown graphically in Fig. 1b, in which the hatched portion accounts for at least 90% of the area under the curve.

Ex. 1003, Page 257

Definition of Frequency Distribution by Volume ($[D_M/5, 5D_M]$, ≧ 90%)

A particle size distribution that is expressed by ($[D_M/5, 5D_M]$, ≧ 90%) in terms of frequency distribution by volume represents one in which particles ranging in diameter from one fifth of D_M to five times D_M (D_M is the average particle diameter by volume) account for at least 90% of the total volume of the particles present. Consider, for example, particles having an average diameter D_M of 10 μm on a volume basis; a particle size distribution that is expressed by ($[D_M/5, 5D_M]$, ≧ 90%) in terms of frequency distribution by volume represents one in which particles ranging in diameter from 2 μm to 50 μm account for at least 90% of the total volume of the particles present. The average particle diameter D_M on a volume basis is expressed by:

$$D_M = \frac{\int_0^\infty Df(D)dD}{\int_0^\infty f(D)dD}$$

where f(D) is the frequency distribution by volume. Or D_M is technically expressed by:

$$DM = \Sigma(V_i D_i)/\Sigma V_i$$

where Vi is the volume of a group of particles within a given interval of diameters $D_i \pm \Delta D_i/2$ (ΔD_i is the width of interval).

Coating Start Region

The region where coating is first started after the final treatment by the group of means for high dispersion treatment of particles is called the "coating start region". Therefore, prior to the final treatment by the group of means for high dispersion treatment of particles, no region is called the "coating start region" according to this definition even if coating is first started there.

Dispersity β in the Coating Start Region

In the present invention, the coating chamber is provided in such a way that the coating start region of the coating space is located in a region that satisfies one of the following conditions:

(1) a powder of core particles that have an average diameter of no more than 10 μm in the frequency distribution by volume should be dispersed in a gaseous atmosphere by the final treatment by the group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and the dispersity β of the particles in said powder of core particles should be adjusted to at least 70%;
(2) a powder of core particles that have an average diameter in excess of 10 μm but no more than 20 μm in the frequency distribution by volume should be dispersed in a gaseous atmosphere by the final treatment by the group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and the dispersity β of the particles in said powder of core particles should be adjusted to at least 80%;
(3) a powder of core particles that have an average diameter in excess of 20 μm but no more than 50 μm in the frequency distribution by volume should be dispersed in a gaseous atmosphere by the final treatment by the group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and the dispersity β of the particles in said powder of core particles should be adjusted to at least 90%;
(4) a powder of core particles that have an average diameter in excess of 50 μm but no more than 300 μm in the frequency distribution by volume should be dispersed in a gaseous atmosphere by the final treatment by the group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and the dispersity β of the particles in said powder of core particles should be adjusted to at least 95%;
(5) a powder of core particles that have an average diameter in excess of 300 μm but no more than 800 μm in the frequency distribution by volume should be dispersed in a gaseous atmosphere by the final treatment by the group

Ex. 1003, Page 258

of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and the dispersity β of the particles in said powder of core particles should be adjusted to at least 97%; and

(6) a powder of core particles that have an average diameter in excess of 800 μm in the frequency distribution by volume should be dispersed in a gaseous atmosphere by the final treatment by the group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and the dispersity β of the particles in said powder of core particles should be adjusted to at least 99%.

Given one of those dispersities in the coating start region of the coating space, one can disperse the particles in the powder of core particles to such a state that they are substantially single particles in a gaseous atmosphere, so that the precursor of the coat forming substance will uniformly contact and/or impinge against the surfaces of all particles in the powder of core particles that pass through the coating start region of the coating space, whereby the coat forming substance can be deposited on single particles in equal amounts.

In the present invention, dispersity β varies continuously with the average diameter of the particles in the powder of core particles but because of the difficulty involved in expressing it, the convenient stepwise expression is adopted.

Advantageously, the coating chamber is provided in such a way that the coating start region of the coating space is located in a space region that satisfies one of the following conditions:

(1) a powder of core particles that have an average diameter of no more than 10 μm in the frequency distribution by volume should be dispersed in a gaseous atmosphere by the final treatment by the group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and the dispersity β of the particles in said powder of core particles should be adjusted to at least 80%;

(2) a powder of core particles that have an average diameter in excess of 10 μm but no more than 20 μm in the frequency distribution by volume should be dispersed in a gaseous atmosphere by the final treatment by the group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and the dispersity β of the particles in said powder of core particles should be adjusted to at least 90%;

(3) a powder of core particles that have an average diameter in excess of 20 μm but no more than 50 μm in the frequency distribution by volume should be dispersed in a gaseous atmosphere by the final treatment by the group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and the dispersity of the particles in said powder of core particles should be adjusted to 95%;

(4) a powder of core particles that have an average diameter in excess of 50 μm but no more than 300 μm in the frequency distribution by volume should be dispersed in a gaseous atmosphere by the final treatment by the group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and the dispersity β of the particles in said powder of core particles should be adjusted to 97%; and

(5) a powder of core particles that have an average diameter in excess of 300 μm in the frequency distribution by volume should be dispersed in a gaseous atmosphere by the final treatment by the group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and the dispersity β of the particles in said powder of core particles should be adjusted to at least 99%. Given the coating chamber in which the coating start region of the coating space is located in such a way as to satisfy those conditions, the particles in the powder of core particles are at least free from areas that are substantially closed by two adjacent core particles and it is possible to assure that the precursor of the coat forming substance is allowed to contact and/or impinge against the surfaces of individual particles from every direction, whereby coating with the coat forming substance can be accomplished more uniformly on a single particle basis and, accordingly, coating can be done to provide more uniform coating weights for the individual core particles.

The particles in a powder of core particles that have an average diameter exceeding 10 μm in the frequency distribution by volume are subjected to a cohesive action in a gaseous atmosphere, although not as intense as in the case of the particles in a powder of core particles that have an average diameter no more than 10 μm, and they contact and/or impinge against one another, causing unevenness in the distribution of the particles in the powder of highly dispersed core particles which form a mixture with a gas. However, if the coating of the particles in the powder of core particles is started as they are dispersed to provide one of the above-specified dispersities in accordance with their diameter, the particles in the powder of core particles or the particles in a powder that consists chiefly of the core particles can be coated with the coat forming substance more uniformly on a single particle basis and, accordingly, the individual particles can be coated with the coat forming substance in more uniform weights.

More advantageously, in the coating start region of the coating space,

Ex. 1003, Page 259

(1) a powder of core particles that have an average diameter of no more than 10 μm in the frequency distribution by volume should be dispersed in a gaseous atmosphere by the final dispersing treatment by the group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and the dispersity β of the particles in said powder of core particles should be adjusted to at least 90%, or

(2) a powder of core particles that have an average diameter in excess of 10 μm but no more than 20 μm in the frequency distribution by volume should be dispersed in a gaseous atmosphere by the final dispersing treatment by the group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and the dispersity β of the particles in said powder of core particles should be adjusted to at least 95%, or

(3) a powder of core particles that have an average diameter in excess of 20 μm but no more than 50 μm in the frequency distribution by volume should be dispersed in a gaseous atmosphere by the final dispersing treatment by the group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and the dispersity β of the particles in said powder of core particles should be adjusted to at least 97%, or

(4) a powder of core particles that have an average diameter in excess of 50 μm in the frequency distribution by volume should be dispersed in a gaseous atmosphere by the final dispersing treatment by the group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and the dispersity β of the particles in said powder of core particles should be adjusted to at least 99%.

If one of these dispersities is attained in the coating start region of the coating space, the core particles are practically free from agglomeration and practically uniform coating can be performed on all surfaces of the individual particles.

If, in a particular case, high-quality coating is desired at the sacrifice of the efficiency of treatment, the dispersity is preferably at least 95%. In this case, the throughput of the treatment of the particles in the powder of core particles may be reduced to a very small level, namely, the concentration by number of the particles in the powder of core particles in the gaseous atmosphere may be reduced so that the particles in the powder of core particles are dispersed completely, whereby uniform coating can thoroughly be accomplished on the entire surfaces of the individual particles.

Group of Means for High Dispersion Treatment of Particles

The group of means for high dispersion treatment of particles which is to be used in the present invention is such that: (A) it has at least one dispersing means; and (B) it has, as the final treating means,

(a) dispersing means for dispersing the particles in a powder of core particles in a gaseous atmosphere or

(b) means for selecting a mixture of a gas and the particles in a powder of highly dispersed particles which comprises

(b-1) means for selecting a mixture of a gas and the particles in a powder of highly dispersed particles that separates a portion of the powder that is composed of less highly dispersed particles from the mixture of a gas and the particles in the powder of core particles in which the particles in the powder of core particles are dispersed in a gaseous atmosphere, thereby selecting a mixture of a gas and the particles in a powder of highly dispersed core particles in which the particles in the powder of core particles are present in the gaseous atmosphere mainly in a single-particle state and

(b-2) feedback means by which the portion of the powder that is composed of less highly dispersed particles and which has been separated by the means for selecting a mixture of a gas and the particles in a powder of highly dispersed particles is transported to the final dispersing means among the dispersing means in the group of means for high dispersion treatment of particles and/or treating means upstream of the final dispersing means.

Advantageously, the group of means for high dispersion treatment of particles has such dispersing performance that one of the following conditions is satisfied:

(1) a powder of core particles that have an average diameter of no more than 10 μm in the frequency distribution by volume should be dispersed in a gaseous atmosphere by the final treatment by said group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and the dispersity β of the particles in said powder of core particles should be adjusted to at least 70%;

(2) a powder of core particles that have an average diameter in excess of 10 μm but no more than 20 μm in the frequency distribution by volume should be dispersed in a gaseous atmosphere by the final treatment by said group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder

Ex. 1003, Page 260

of highly dispersed core particles and the dispersity β of the particles in said powder of core particles should be adjusted to at least 80%;

(3) a powder of core particles that have an average diameter in excess of 20 μm but no more than 50 μm in the frequency distribution by volume should be dispersed in a gaseous atmosphere by the final treatment by the group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and the dispersity β of the particles in said powder of core particles should be adjusted to at least 90%;

(4) a powder of core particles that have an average diameter in excess of 50 μm but no more than 300 μm in the frequency distribution by volume should be dispersed in a gaseous atmosphere by the final treatment by the group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and the dispersity β of the particles in said powder of core particles should be adjusted to at least 95%;

(5) a powder of core particles that have an average diameter in excess of 300 μm but no more than 800 μm in the frequency distribution by volume should be dispersed in a gaseous atmosphere by the final treatment by the group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and the dispersity β of the particles in said powder of core particles should be adjusted to at least 97%; and

(6) a powder of core particles that have an average diameter in excess of 800 μm in the frequency distribution by volume should be dispersed in a gaseous atmosphere by the final treatment by the group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and the dispersity β of the particles in said powder of core particles should be adjusted to at least 99%.

By insuring that the group of means for high dispersion treatment of particles have dispersing capabilities comparable to or better than the various dispersities to be attained in the above-described coating start region and by providing them in correspondence to those dispersities, high-quality coating can be applied in the coating start region to comply with the respective dispersities.

Final Treating Means

If the final treating means in the group of means for high dispersion treatment of particles is a dispersing means, said dispersing means is referred to as the final treatment means in the group of means for high dispersion treatment of particles. If the final treating means in the group of means for high dispersion treatment of particles is a means for selecting a mixture of a gas and the particles in a powder of highly dispersed particles which is equipped with a feedback means by which the portion that has been separated selectively on account of less highly dispersed state during the step of treatment for selecting the mixture of a gas and the particles in the powder of highly dispersed core particles is to be transported to the final dispersing means at the last stage of the means for high dispersion treatment of particles, or if said final treating means is a means for selecting a mixture of a gas and the particles in a powder of highly dispersed particles which is equipped with a feedback means by which the portion that has been separated selectively on account of less highly dispersed state during the step of treatment for selecting the mixture of a gas and the particles in the powder of highly dispersed core particles is to be transported to a treating means upstream of the final dispersing means, said means for selecting a mixture of a gas and the particles in a powder of highly dispersed core particles is referred to as the final treating means in the group of means for high dispersion treatment of particles.

It should be noted here that any means for selecting a mixture of a gas and the particles in a powder of highly dispersed particles that is provided upstream of the means for selecting a mixture of a gas and the particles in a powder of highly dispersed particles that is equipped with a feedback means and which is the final treating means in the group of means for high dispersion treatment of particles ("upstream" may typically mean between the means for selecting a mixture of a gas and the particles in a powder of highly dispersed particles that is equipped with a feedback means and the final dispersing means, or upstream of the final dispersing means) is a constituent element of the group of means for high dispersion treatment of particles and this is true irrespective of whether the feedback means is used or not.

Dispersing Means

The means used to disperse particles is named a dispersing means. Any mechanism that has even a small or slight dispersing effect is usable as a dispersing means and it is used as a dispersing means in the invention. Consider, for example, a rotary feeder and an injection feeder for pneumatic conveying which are commonly used as supply means ["Funtai Kogaku Binran (A Handbook of Particle Technology)", Ed. by Funtai Kogakukai (Society of Particle Technology), published by Nikkan Kogyo Shinbunsha in 1986, pp. 568 and 571]. These apparatus also have a dispersing effect and, hence, they are dispersing means if used as means to achieve dispersion. The dispersion maintaining and promoting means which are to be discussed later are also dispersing means if they are used to achieve dispersion (for increasing

Ex. 1003, Page 261

β). Such dispersing means may be a single apparatus or machine or it may be a complex of apparatus and machines; to cover all of these cases, the dispersing means is collectively referred to as the "group of means for high dispersion treatment of particles".

The group of means for high dispersion treatment of particles is such that it operates on at least one selected mechanism of dispersion, such as dispersing the particles in a powder of core particles by a gas stream which accelerates and/or places them in a velocity gradient, dispersing the particles in a powder of core particles by impingement against a static obstacle and/or an obstacle composed of a rotating body, and dispersing the particles in a powder of core particles by mechanical disagglomeration which consists of a fluidized bed and/or pulsation and/or a rotary drum and/or vibrations and/or scraping.

Specifically, the group of means for high dispersion treatment of particles is equipped with at least one selected dispersing means, such as an ejector-type disperser, a Venturi-type disperser, a disperser using a capillary, an agitator, and an obstacle in a gas stream, a disperser using a jet blow, a disperser using a spiral tube and rotating blades, a disperser using rotating pins (Kady mill), a fluidized bed-type disperser, a disperser using pulsation, a disperser using a rotary drum, a disperser using vibrations, a disperser using a vibrating screen and the scraping action of a scraper, an SAEI Gonnel-type disperser, a Chujo-type disperser, a Roller-type disperser, an office-type disperser, a B.M. type disperser, a Timbrell-type disperser, and a Wright-type disperser ["Funtai Kogaku Binran (A Handbook of Particle Technology)", Ed. by Funtai Kogakukai (Society of Particle Technology), published by Nikkan Kogyo Shinbunsha in 1986, p. 430].

Also usable are the dispersers described in official gazettes and exemplified blow: the disperser using agitating blades which is described in Unexamined Published Japanese Patent Application (kokai) Sho 56-1336; the disperser using a high-speed gas stream and dispersing nozzles which is described in Unexamined Published Japanese Patent Application (kokai) Sho 58-163454; the disperser using the dispersing action of rotating blades and the dispersing action of plasma ions which is described in Unexamined Published Japanese Patent Application (kokai) Sho 59-199027; the disperser using the dispersing action of plasma ions which is described in Unexamined Published Japanese Patent Application (kokai) Sho 59-207319; the disperser using an ejector and the dispersing action of plasma ions which is described in Unexamined Published Japanese Patent Application (kokai) Sho 59-216616; the disperser using an ejector and the dispersing action of ion streams which is described in Unexamined Published Japanese Patent Application (kokai) Sho 59-225728; the disperser using the dispersing action of plasma ions which is described in Unexamined Published Japanese Patent Application (kokai) Sho 59-183845; the disperser using the dispersing action of dispersing blades and a pressure gas which is described in Unexamined Published Japanese Patent Application (kokai) Sho 63-166421; the disperser using lineal or annular slit jet ports which is described in Unexamined Published Japanese Patent Application (kokai) Sho 62-176527; the disperser using mesh blades which is described in Unexamined Published Japanese Patent Application (kokai) Sho 63-221829; the disperser using the dispersing action of high-speed gas streams issuing from jet nozzles which is described in Unexamined Published Japanese Patent Application (kokai) Sho 63-1629; the disperser using many small holes which is described in Unexamined Published Utility Model Application (kokai) Sho 63-9218; the ejector-type disperser which is described in Unexamined Published Japanese Utility Model Application (kokai) Sho 62-156854; and the disperser using small holes and orifices which is described in Unexamined Published Japanese Utility Model Application (kokai) Sho 63-6034.

Dispersing means advantageous for use in the group of means for high dispersion treatment of particles include the apparatus that are described in Japanese Patent Application Sho 63-311358, Japanese Patent Application Hei 1-71071, Japanese Patent Application Hei 2-218537, etc.

Means for Selecting a Mixture of a Gas and the Particles in a Powder of Highly Dispersed Core Particles

The means for selecting a mixture of a gas and the particles in a powder of highly dispersed core particles is a means that separates a mixture of a gas and the particles in a powder of less highly dispersed core particles from the mixture of a gas and the particles in the powder of core particles so as to select a mixture of a gas and the particles in a powder of highly dispersed core particles that is chiefly composed of those particles which are in a single-particle state. Agglomerating particles which are association of primary particles have a greater apparent diameter than the primary particles and, hence, they can be separated by a suitable means such as dry classifying means. An example of the means for selecting a mixture of a gas and the particles in a powder of highly dispersed core particles is at least one dry classifying means selected from among the following: classifying means using gravity, classifying means using inertia force, classifying means using centrifugal force, classifying means using static electricity, classifying means using a fluidized bed, etc.

Specific examples of the means for selecting the mixture of a gas and the particles in a powder of highly dispersed core particles include: a gravity classifier, an inertia classifier, a centrifugal classifier, a cyclone, an air separator, a micron separator, a microplex, a multiplex, a zigzag classifier, an acu-cut, a conical separator, a turbo classifier, a super separator, a dispersion separator, an elbow jet, a fluidized bed classifier, a virtual impactor, an O-Sepa, a sieve, a vibrating

Ex. 1003, Page 262

screen, a sifter ["Funtai Kogaku Binran (A Handbook of Particle Technology)", Ed. by Funtai Kogakukai (Society of Particle Technology), published by Nikkan Kogyo Shinbunsha in 1986, p. 514].

Mixture of a Gas and the Particles in a Powder of Core Particles

The mixture of a gas and the particles in a powder of core particles refers to (a) a homogeneous flow characterized in that the particles in a powder of core particles are suspended uniformly in a gaseous atmosphere (which may be called a "uniform suspension flow"), (b) a heterogeneous flow characterized in that the particles in a powder of core particles show a nonuniform distribution in certain regions of a gaseous atmosphere (which may be called an "inhomogeneous suspension flow"), (c) a flow involving a sliding layer of the particles in a powder of core particles (which may be called a "sliding flow"), or (d) a flow involving a stationary layer of the particles in a powder of core particles.

Mixture of a Gas and the Particles in a Powder of Less Highly Dispersed Core Particles

The mixture of a gas and the particles in a powder of less highly dispersed core particles refers to that portion of a mixture of a gas and the particles in a powder of core particles in which the powder particles are present in a gaseous atmosphere mainly in states other than the single-particle state.

Mixture of a Gas and the Particles in a Powder of Highly Dispersed Core Particles

The mixture of a gas and the particles in a powder of highly dispersed particles refers to a mixture of a gas and the particles in a powder of core particles in which the powder particles are present in a gaseous atmosphere mainly in a single-particle state. No matter how much high the degree of dispersion is, the mixture of a gas and the particles in a powder of highly dispersed core particles contains agglomerating particles in practice. The mixture of a gas and the particles in a powder of less highly dispersed particles contains non-agglomerating single particles in practice and can be selectively separated into a mixture of a gas and the particles in a powder of less highly dispersed particles and a mixture of a gas and the particles in a powder of highly dispersed particles. The mixture of a gas and the particles in a powder of less highly dispersed particles is converted to a mixture of a gas and the particles in a powder of highly dispersed particles by selective separation and/or redispersion of the agglomerating particles.

Recovery Means

The recovery means is a means for picking up the coated particles that have been prepared in the coating space. The part of the recovery means in which the recovery treatment is performed is called the "recovery section". The coated particles the coating on which has been provided by passage through the coating start region of the coating space are recovered by direct pickup from the gaseous atmosphere or after temporary storage following pickup from the gaseous atmosphere or together with a gas.

Usable as the recovery section of the recovery means is one of the following members: the recovery section of the recovery means that uses barriers (obstacles), the recovery section of the recovery means that uses gravity, the recovery section of the recovery means that uses inertia forces, the recovery section of the recovery means that uses centrifugal forces, the recovery section of the recovery means that uses coulombic attractive forces; the recovery section of the recovery means that uses thermophoretic forces, the recovery section of the recovery means that uses Brownian diffusion, the recovery section of the recovery means that uses the pulling force of the back pressure, evacuation, etc.

Advantageous examples of the recovery section of the recovery means include a gravity dust collector, an inertia dust collector, a centrifugal dust collector, a filtration dust collector, an electrostatic precipitator, a scrubbing dust collector, a particle packed layer, a cyclone, a bag filter, a ceramic filter, a scrubber, etc.

Bonding Material

The bonding material to be used to manufacture diamond or high-pressure phase boron nitride sinters in accordance with the invention is selected from among those bonding materials which can be sintered densely to a density of at least 85% by sintering at pressures less than 2000 MPa and at temperatures not exceeding 1850°C. Advantageously, those bonding materials are selected which will not promote the phase transition of diamond or high-pressure phase boron nitride to the lower-pressure phase (graphite or hBN). Alternatively, those bonding materials are selected which, when sintered at pressures less than 2000 MPa and at temperatures not exceeding 1850°C, will react with diamond or boron nitride to yield dense reaction products that have densities of at least 85%. More preferably, those substances are selected which, when sintered at pressures less than 2000 MPa and at temperatures not exceeding 1850°C, will produce bonding materials that are dense (density ≧ 90%) and/or of high hardness (Vickers hardness ≧ 600). Feed powders which, when sintered at pressures less than 2000 MPa and at temperatures not exceeding 1850°C, will yield dense

Ex. 1003, Page 263

bonding materials having densities of at least 85% are selected from powders or particles that contain at least one of the metals, semiconductors, metalloids and rare earth metals of groups 1a, 2a, 3a, 4a, 5a, 6a, 7a, 1b, 2b, 3b, 4b, 5b, 6b, 7b and 8 of the periodic table and/or at least one compound containing at least one of those metals, semiconductors, metalloids and rare earth metals. More specifically, such powders or particles can be those of at least one member which is selected from among B, Ti, Zr, Hf, Ta, Nb, V, SiC, TiC, ZrC, B_4C, WC, HfC, TaC, NbC, Si_3N_4, TiN, ZrN, AlN, HfN, TaN, TiB, TiB_2, ZrB_2, HfB, HfB_2, LaB_6 $MoSi_2$, BP, Al_2O_3, Al_2SiO_5 (mullite), ZrO_2 (PSZ: partially stabilized zirconia with Y_2O_8, MgO or CaO, or TZP: tetragonal zirconia polycrystalline compound), and $MgAl_2O_4$ (spinel). To take alumina as an example, a suitable example is a high-purity, easily sintrable fine feed, such as the alumina described in Unexamined Published Japanese Patent Application (kokai) Sho 63-151616 which is produced by pyrolysis of an ammonium aluminum carbonate and this is preferred since it can be densified at a temperature of about 1400°C even by normal sintering at atmospheric pressure. Another suitable example is a fine, high-purity alumina powder that contains up to 10% by volume of magnesia (MgO) and/or titania (TiO_x, x = 1 - 2) which are effective in promoting the sintering of alumina and if this condition is met, high-purity aluminas other than the alumina described in Unexamined Published Japanese Patent Application (kokai) Sho 63-151616, supra may be used, as exemplified by high-purity (≧ 99%) and easily sintrable aluminas that are composed of fine (≦ 1 μm) particles and which are typically produced by the Bayer process, hydrolysis of organic aluminum, pyrolysis of aluminum alum, the ethylene chlorohydrin process, submerged arc discharge, etc.

Besides the aluminas mentioned above, zirconium oxides may also be selected and advantageous examples include the easily sintrable yttria partially stabilized zirconia (2-4 mol% Y_2O_3-ZrO_2) powder which is produced by coprecipitation, and an alumina-zirconia base powder [FC Report, 1(5) (1983) 13-17]. Also selectable is a titanium oxide or titania powder [TiO_2; Summaries of Lectures Read at the 15th Symposium on High-Pressure Technology (1973) p. 174].

Another example that can be selected is titanium nitride [TiN; Yamada et al., Yogyo Kyokaishi (Journal of The Ceramic Society of Japan, 89, (1981) 621-625].

In the next place, the group of means for high dispersion treatment of particles which is adopted to prepare the coated diamond or high-pressure phase boron nitride particles is described below with reference to accompanying drawings.

Description of the Drawings of the Group of Means for High Dispersion Treatment of Particles

Fig. 2a is a block diagram showing an example of the basic composition of the group of means for high dispersion treatment of particles as it is used to prepare coated diamond or high-pressure phase boron nitride particles. The group is composed of a final dispersing means A for dispersing the particles in a powder of core particles and a constituent element d of the group of means for dispersion treatment which is upstream of the final dispersing means. Shown by ε is a mixture of a gas and the particles in a powder of highly dispersed core particles which are part of the particles in the powder of core particles and which are present in a gaseous atmosphere mainly in a single-particle state. Constituent element d may be any treating means such as dispersing means, supply means, and means for selecting a mixture of a gas and the particles in a powder of highly dispersed core particles, which may be used either individually or in combination. Constituent element d is optional and may be omitted. The group of means for high dispersion treatment of particles is advantageously of such a design that after treatment by the final treating means A, one of the following values of dispersity β can be realized: (1) at least 70% for a powder of core particles that have an average diameter of no more than 10 μm in the frequency distribution by volume; (2) at least 80% for a powder of core particles that have an average diameter in excess of 10 μm but no more than 20 μm in the frequency distribution by volume; (3) at least 90% for a powder of core particles that have an average diameter in excess of 20 μm but no more than 50 μm in the frequency distribution by volume; (4) at least 95% for a powder of core particles that have an average diameter in excess of 50 μm but no more than 300 μm in the frequency distribution by volume; (5) at least 97% for a powder of core particles that have an average diameter in excess of 300 μm but no more than 800 μm in the frequency distribution by volume; and (6) at least 99% for a powder of core particles that have an average diameter in excess of 800 μm in the frequency distribution by volume.

Fig. 2b is a block diagram showing a second example of the basic composition of the group of means for high dispersion treatment of particles as it is used to prepare coated diamond or high-pressure phase boron nitride particles. The group is composed of a final dispersing means A for dispersing the particles in a powder of core particles, final means B for selecting a mixture of a gas and the particles in a powder of highly dispersed core particles at the last stage which is equipped with feedback means C by which a mixture of a gas and the particles in a powder of less highly dispersed core particles η as separated from a mixture of a gas and the particles in a powder of highly dispersed core particles in which the powder particles are present in a gaseous atmosphere mainly in a single-particle state is fed back to the final dispersing means A, a constituent element d of the group of means for dispersion treatment which is upstream of the final dispersing means, and a constituent element e of the group of means for high dispersion treatment of particles which is provided between the final dispersing means and the final selecting means. As for the mixture ε and the constituent element d, the explanation made in connection with Fig. 2a will apply and, hence, no detailed description is made. Constituent element e may be any treating means other than the dispersing means, such as supply means and

Ex. 1003, Page 264

selecting means, which may be used either individually or in combination. Constituent elements d and e are optional and may be omitted. The group of means for high dispersion treatment of particles is advantageously of such a design that after treatment by selecting means B which is the final treating means, the above-mentioned values of dispersity can be realized for powders of core particles having the above-mentioned average diameters.

Fig. 2c is a block diagram showing a third example of the basic composition of the group for high dispersion treatment of particles as it is used to prepare coated diamond or high-pressure phase boron nitride particles. The group is composed of a final dispersing means A for dispersing the particles in a powder of core particles, final means B for selecting a mixture of a gas and the particles in a powder of highly dispersed core particles at the last stage which is equipped with feedback means C by which a mixture of a gas and the particles in a powder of less highly dispersed core particles η as separated from a mixture of a gas and the particles in a powder of highly dispersed core particles in which the powder particles are present in a gaseous atmosphere mainly in a single-particle state is fed back to a treating means upstream of the final dispersing means A, a constituent element d of the group of means for high dispersion treatment of particles which is upstream of the final dispersing means, and a constituent element e of the group of means for high dispersion treatment of particles which is provided between the final dispersing means and the final selecting means. As for the mixture ε, constituent elements d and e, and the group of means for high dispersion treatment of particles, the foregoing explanation will apply and no detailed description is made.

Having the construction described above, the group of means for high dispersion treatment of particles under discussion may include powder supply sources such as a supply vessel and core particle generating means. To take the case of Fig. 2c as an example, the group of means for high dispersion treatment of particles may of course adopt such a composition that the mixture η is fed back to a supply vessel by the feedback means C. It also goes without saying that the step of dispersion by the group of means for high dispersion treatment of particles may be preceded by a disintegrating step in which the particles in the powder of core particles are disintegrated and/or otherwise reduced in size.

Typical specific examples of these basic compositions of the group of means for high dispersion treatment of particles are described below in greater detail with reference to more detailed block diagrams.

Design 1

Fig. 3a is a block diagram illustrating a first specific design of the group of means for high dispersion treatment of particles as it is used to prepare coated diamond or high-pressure phase boron nitride particles and the drawing corresponds to Fig. 2a. The group shown in Fig. 3a is composed of a supply vessel 100 for supplying the powder of core particles to be coated, and the final dispersing means A for dispersing the powder of core particles to be coated.

Design 2

Fig. 3b is a block diagram illustrating a second specific design of the group of means for high dispersion treatment of particles as it is used to prepare coated diamond or high-pressure phase boron nitride particles and the drawing corresponds to Fig. 2a. The group shown in Fig. 3b is composed of a supply vessel 100 for supplying the powder of core particles to be coated, dispersing means a for dispersing the powder of core particles to be coated, and the final dispersing means A for dispersing the powder of core particles to be coated.

Design 3

Fig. 3c is a block diagram illustrating a third specific design of the group of means for high dispersion treatment of particles as it is used to prepare coated diamond or high-pressure phase boron nitride particles and the drawing corresponds to Fig. 2a. The group shown in Fig. 3c is composed of a supply vessel 100 for supplying the powder of core particles to be coated, dispersing means a for dispersing the powder of core particles to be coated, feedback means C by which a mixture of a gas and the particles in a powder of less highly dispersed core particles η as separated from a mixture of a gas and the particles in a powder of highly dispersed core particles in which the powder particles are present in a gaseous atmosphere mainly in a single-particle state is fed back from the mixture of a gas and the particles in the powder of core particles (which have been dispersed by the dispersing means a) to the dispersing means a, means b for selecting the mixture of a gas and the particles in a powder of highly dispersed particles, by which the mixture of a gas and the particles in a powder chiefly composed of highly dispersed core particles is introduced into the final dispersing means A, and the final dispersing means A for dispersing the powder of core particles to be coated.

Design 4

Fig. 3d is a block diagram illustrating a fourth specific design of the group of means for high dispersion treatment of particles as it is used to prepare coated diamond or high-pressure phase boron nitride particles and the drawing in Fig. 3d corresponds to Fig. 2b. The group shown in Fig. 3d is composed of a supply vessel 100 for supplying the powder

Ex. 1003, Page 265

of core particles to be coated, a final dispersing means A for dispersing the powder of core particles to be coated, feedback means C by which a mixture of a gas and the particles in a powder of less highly dispersed core particles η as separated from a mixture of a gas and the particles in a powder of highly dispersed particles in which the powder particles are present in a gaseous atmosphere mainly in a single-particle state is fed back from the mixture of a gas and the particles in the powder of core particles (which have been dispersed by the final dispersing means A) to the dispersing means A, and final means B for selecting the mixture of a gas and the particles in the powder of highly dispersed core particles at the final stage and which discharges the mixture of a gas and the particles in the powder of highly dispersed core particles.

Design 5

Fig. 3e is a block diagram illustrating a fifth specific design of the group of means for high dispersion treatment of particles as it is used to prepare coated diamond or high-pressure phase boron nitride particles and the drawing corresponds to Fig. 2b. The group shown in Fig. 3e is composed of a supply vessel 100 for supplying the powder of core particles to be coated, dispersing means a for dispersing the powder of core particles to be coated, a final dispersing means A for dispersing the powder of core particles to be coated, feedback means C by which a mixture of a gas and the particles in a powder of less highly dispersed core particles η as separated from a mixture of a gas and the particles in a powder of highly dispersed particles in which the powder particles are present in a gaseous atmosphere mainly in a single-particle state is fed back from the mixture of a gas and the particles in the powder of core particles (which have been dispersed by the final dispersing means A) to the dispersing means A, and final means B for selecting the mixture of a gas and the particles in the powder of highly dispersed core particles at the final stage and which discharges the mixture of a gas and the particles in the powder of highly dispersed core particles.

Design 6

Fig. 3f is a block diagram illustrating a sixth specific design of the group of means for high dispersion treatment of particles as it is used to prepare coated diamond or high-pressure phase boron nitride particles and the drawing corresponds to Fig. 2b. The group shown in Fig. 3f is composed of a supply vessel 100 for supplying the powder of core particles to be coated, means b for selecting a mixture of a gas and the particles in a powder of highly dispersed core particles, which rejects a mixture of a gas and the particles in a powder chiefly composed of less highly dispersed core particles from the mixture of a gas and the particles in the powder of core particles and which introduces into dispersing means A the mixture of a gas and the particles in a powder chiefly composed of highly dispersed core particles, final dispersing means A for dispersing the particles in the selectively separated powder of core particles, feedback means C by which a mixture of a gas and the particles in a powder of less highly dispersed core particles as separated from a mixture of a gas and the particles in a powder of highly dispersed particles in which the powder particles are present in a gaseous atmosphere mainly in a single-particle state is fed back from the mixture of a gas and the particles in the powder of core particles (which have been dispersed by the final dispersing means A) to the dispersing means A, and final means B for selecting the mixture of a gas and the particles in the powder of highly dispersed core particles at the final stage and which discharges the mixture of a gas and the particles in the powder of highly dispersed core particles.

Design 7

Fig. 3g is a block diagram illustrating a seventh specific design of the group of means for high dispersion treatment of particles as it is used to prepare coated diamond or high-pressure phase boron nitride particles and the drawing corresponds to Fig. 2c. The group shown in Fig. 3g is composed of a supply vessel 100 for supplying the powder of core particles to be coated, dispersing means a for dispersing the powder of core particles to be coated, a final dispersing means A for dispersing the powder of core particles to be coated, feedback means C by which a mixture of a gas and the particles in a powder of less highly dispersed core particles as separated from a mixture of a gas and the particles in a powder of highly dispersed particles in which the powder particles are present in a gaseous atmosphere mainly in a single-particle state is fed back from the mixture of a gas and the particles in the powder of core particles (which have been dispersed by the final dispersing means A) to the dispersing means A, and final means B for selecting the mixture of a gas and the particles in the powder of highly dispersed core particles at the final stage and which discharges the mixture of a gas and the particles in the powder of highly dispersed core particles.

To maintain the thus achieved high dispersive state of the particles, means for maintaining dispersion in a gaseous atmosphere may be added to the group of means for high dispersion treatment of particles. The term "means for maintaining dispersion in a gaseous atmosphere" as used herein refers to means that maintains a desired value of dispersity β by preventing reagglomeration of the particles in the powder of core particles that have been carried dispersively in a gaseous atmosphere. To promote the thus achieved high dispersive state of the core particles, means for promoting dispersion in a gaseous atmosphere may be added between the group of means for high dispersion treatment of particles

Ex. 1003, Page 266

and the coating chamber. The term "means for promoting dispersion in a gaseous atmosphere" as used herein refers to means which is primarily intended to promote the redispersion of that part of the particles in the powder of core particles carried dispersively in a gaseous atmosphere which have agglomerated again, thereby retarding the drop in the dispersive state of the powder; the term also refers to means that promotes redispersion in such a way that the state of dispersion that has dropped once can be restored to the initial high dispersive state.

Advantageous examples of said means for maintaining dispersion in a gaseous atmosphere or promoting dispersion in a gaseous atmosphere include a pipe vibrator, a pipe heater, a plasma generator, an electric charger, etc.

The pipe vibrator is means in which a pipe equipped with an oscillator is vibrated to provide the particles dispersed in a gaseous atmosphere with vibrations uncomparable to those imparted by dispersers, whereby reagglomeration is suppressed to maintain the high dispersive state or the dispersion of reagglomerating particles is promoted.

The pipe heater is means in which a heated pipe imparts heat to a carrier gas from outside, thereby expanding the carrier gas so that its flow velocity is accelerated to a level that is too fast to be comparable to that achieved by dispersers, whereby reagglomeration of the particles is suppressed while promoting the dispersion of reagglomerating particles.

The plasma generator is means in which a plasma is generated in the gaseous atmosphere which dispersively caries the powder of core particles, with the resulting plasma ions being allowed to impinge against the core particles, whereby reagglomeration is suppressed to maintain the high dispersive state or the dispersion of reagglomerating particles is promoted.

The electric charger is means in which corona discharge, electron beams, radiations or some other method is used to generate monopolar ions in the gaseous atmosphere which dispersively carries the powder of core particles, which are subsequently passed through the monopolar ion atmosphere to be charged to a single polarity, whereupon electrostatic repulsive forces either suppress reagglomeration to maintain the high dispersive state or promote the dispersion of reagglomerating particles.

The powder of core particles which have been rendered to assume a highly dispersive state in the manner described on the foregoing pages is sent to the coating chamber so that the surfaces of the particles are coated with the coat forming substance. The coating compartment is provided with the coating space including the coating start region.

The group of means for high dispersion treatment of particles is desirably coupled directly to the coating chamber. If necessary, they may be connected together by means of a hollow member and/or a pipe that are indispensable to transport. In this case, too, it is essential that the dispersity β in the coating start region be adjusted to one of the values within the ranges set forth hereinabove.

If the group of means for high dispersion treatment of particles is coupled to the coating chamber located separately, the powder of core particles suffices to be introduced into the coating chamber as it maintains the state of dispersion that has been achieved by said group of means for high dispersion treatment of particles. To this end, means for maintaining dispersion in a gaseous atmosphere (which is a device for maintaining the state of dispersion of the powder of core particles) and/or means for promoting dispersion in a gaseous atmosphere (which is a device for enhancing the state of dispersion) and/or means for selecting a mixture of a gas and the particles in a powder of highly dispersed particles (which separates the powder portion of less highly dispersed core particles from the mixture of a gas and the particles in the powder of core particles, thereby selecting a mixture of a gas and the particles in a powder of highly dispersed core particles, the major part of which comprises particles in a single-particle state) may be provided between the group of means for high dispersion treatment of particles and the coating chamber.

The invention also contemplates that when used to prepare coated diamond or high-pressure phase boron nitride particles, the group of means for high dispersion treatment of particles may share one or more portions of the space with (1) the coating chamber, (2) the coating space or (3) the coating start region.

For example, the group of means for high dispersion treatment of particles may share its dispersing space with the coating chamber, or with the coating space having the coating start region, or with the coating start region.

The term "coating start region" as used herein refers to the region where the precursor of the coat forming substance that has been generated via the vapor phase and/or the precursor of the coat forming substance in a vapor-phase state contacts and/or impinges against the powder of highly dispersed core particles which has been transported in the state of dispersion that has one of the following values of dispersity β, whereupon coating of the individual particles is started: (1) at least 70% for a powder of core particles that have an average diameter of no more than 10 μm in the frequency distribution by volume; (2) at least 80% for a powder of core particles that have an average diameter in excess of 10 μm but no more than 20 μm in the frequency distribution by volume; (3) at least 90% for a powder of core particles that have an average diameter in excess of 20 μm but no more than 50 μm in the frequency distribution by volume; (4) at least 95% for a powder of core particles that have an average diameter in excess of 50 μm but no more than 300 μm in the frequency distribution by volume; (5) at least 97% for a powder of core particles that have an average diameter in excess of 300 μm but no more than 800 μm in the frequency distribution by volume; and (6) at least 99% for a powder of core particles that have an average diameter in excess of 800 μm in the frequency distribution by volume. Five typical embodiments of the coating start region are shown schematically in Figs. 4a - 4e.

In Figs. 4a - 4e, the coating start region is indicated by numeral 2.

Ex. 1003, Page 267

In the case shown in Fig. 4a, the coating start region 2 of the coating space in which the coating of core particles is started as they are dispersed to one of the above-listed values of dispersity β in accordance with the average diameter of those core particles is provided in such a way that it surrounds the group of means for high dispersion treatment of particles or its discharge section 1.

In the case shown in Fig. 4b, the coating start region 2 of said coating space is provided in such a way that it is passed by all the particles 4 in the powder of core particles as they are discharged from the group of means for high dispersion treatment or its discharge section 1.

By adopting these arrangements, the particles in all powders of core particles can start to be coated as they are dispersed to the above-listed values of dispersity β.

In the case shown in Fig. 4c, the coating start region 2 of said coating space is provided in such a way that it is passed in all cases by those particles 4 in the powder of core particles which are discharged from the group of means for high dispersion treatment of particles or its discharge section 1 to enter the recovery section 5.

In the case shown in Fig. 4d, the coating start region 2 of said coating space is provided in such a way that it surrounds the recovery section 5.

In the case shown in Fig. 4e, the recovery section 5 is provided in a position that can be reached solely by the particles in the mixture of a gas and the particles in the powder of highly dispersed core particles. Hence, the region indicated by 6 in Fig. 4e is selection means using gravity. The coating start region 2 of said coating space is provided in the hatched area of Fig. 4e so that it is passed in all cases by those particles in the mixture of a gas and the particles in the powder of highly dispersed core particles which enter the recovery section. This arrangement insures that only the core particles that started to be coated as they were dispersed to the above-listed values of dispersity β can be recovered and there is no chance that the core particles that did not pass through the coating start region will intermingle with the coated particles which emerge from the coating start region.

As one can understand from the foregoing description, the apparatus for implementing the present invention are composed of the group of means for high dispersion treatment of particles and the coating chamber or, alternatively, the group of means for high dispersion treatment of particles, the coating chamber and the recovery means. The constituent elements of the apparatus can be combined in various manners and several designs of these apparatus are described below with reference to drawings.

Apparatus Design 1

Fig. 5a is a block diagram showing the construction of a first apparatus for producing coated diamond or high-pressure phase boron nitride particles. As shown, the apparatus is composed of the main body of the production equipment 2-A (coating apparatus), coating chamber 2-B1, coating space 2-B2, coating start region 2-B3, the group of means for high dispersion treatment of particles 2-C1, and recovery means 2-D. The group of means for high dispersion treatment of particles 2-C1 is coupled directly to the coating chamber 2-B1.

Apparatus Design 2

Fig. 5b is a block diagram showing the construction of a second apparatus for producing coated diamond or high-pressure phase boron nitride particles. As shown, the apparatus is composed of the main body of the production equipment 2-A (coating apparatus), coating chamber 2-B1, coating space 2-B2, coating start region 2-B3, the group of means for high dispersion treatment of particles 2-C1, indispensable hollow member 2-C2, and recovery means 2-D. The group of means for high dispersion treatment of particles 2-C1 is connected to the coating chamber 2-B1 via the indispensable hollow member 2-C2.

Apparatus Design 3

Fig. 5c is a block diagram showing the construction of a third apparatus for producing coated diamond or high-pressure phase boron nitride particles. As shown, the apparatus is composed of the main body of the production equipment 2-A (coating apparatus), coating chamber 2-B1, coating space 2-B2, coating start region 2-B3, the group of means for high dispersion treatment of particles 2-C1, means for maintaining dispersion in gaseous atmosphere 2-C3, and recovery means 2-D. The group of means for high dispersion treatment of particles 2-C1 is connected to the coating chamber 2-B1 via the means for maintaining dispersion in gaseous atmosphere 2-C3.

Apparatus Design 4

Fig. 5d is a block diagram showing the construction of a fourth apparatus for producing coated diamond or high-pressure phase boron nitride particles. As shown, the apparatus is composed of the main body of the production equipment 2-A (coating apparatus), coating chamber 2-B1, coating space 2-B2, coating start region 2-B3, the group of means

Ex. 1003, Page 268

for high dispersion treatment of particles 2-C1, and recovery means 2-D. The group of means for high dispersion treatment of particles 2-C1 shares space with the coating chamber 2-B1.

Apparatus Design 5

Fig. 5e is a block diagram showing the construction of a fifth apparatus for producing coated diamond or high-pressure phase boron nitride particles. As shown, the apparatus is composed of the main body of the production equipment 2-A (coating apparatus), coating chamber 2-B1, coating space 2-B2, coating start region 2-B3, the group of means for high dispersion treatment of particles 2-C1, and recovery means 2-D. The group of means for high dispersion treatment of particles 2-C1 is provided within the coating chamber 2-B1.

Apparatus Design 6

Fig. 5f is a block diagram showing the construction of a sixth apparatus for producing coated diamond or high-pressure phase boron nitride particles. As shown, the apparatus is composed of the main body of the production equipment 2-A (coating apparatus), coating chamber 2-B1, coating space 2-B2, coating start region 2-B3, the group of means for high dispersion treatment of particles 2-C1, and recovery means 2-D. The coating chamber 2-B1 is provided within the dispersing space of the group of means for high dispersion treatment of particles 2-C1.

Apparatus Design 7

Fig. 5g is a block diagram showing the construction of a seventh apparatus for producing coated diamond or high-pressure phase boron nitride particles. As shown, the apparatus is composed of the main body of the production equipment 2-A (coating apparatus), coating chamber 2-B1, coating space 2-B2, coating start region 2-B3, the group of means for high dispersion treatment of particles 2-C1, recovery means 2-D, and recoating supply means 2-E. The coated particles issuing from the recovery means 2-D can be transported by the recoating supply means 2-E to the group of means for high dispersion treatment of particles 2-C1 for performing repeated coating treatments.

Either one of the apparatuses of these designs can produce coated diamond or high-pressure phase boron nitride particles.

The coated particles in which the powder of core particles (diamond or high-pressure phase boron nitride particles) has its surface coated with the coat forming substance may be coated again with the coat forming substance or subjected to repeated recoating operations. In these cases, the coated particles are sent to the recoating supply means. The term "recoating supply means" as used herein refers to means for transporting the coated particles to the group of means for high dispersion treatment of particles to perform recoating. Specifically, the recoating supply means is furnished with (a) means for recovering the coated particles and (b) means for transporting said coated particles from the recovery means (a) to the group of means for high dispersion treatment of particles. Alternatively, the recoating supply means is furnished with (a) means for recovering the coated particles, (b) means for transporting said coated particles from the recovery means (a) to the group of means for high dispersion treatment of particles, and (c) means for classifying the coated particles. Given large coating weights, the particle size distribution of the coated particles will vary from that of the particles in the powder of uncoated core particles. Under these conditions, it is effective to perform the recoating operation after the particle size distribution of the coated particles is adjusted by the classifying means (c).

The recoating operation may be repeated depending on the need and the coating weight of the coat forming substance can be set to a desired level. If necessary, the coating operation may be repeated with the type of the coat forming substance varied and, thus, a substance of more than one component can be coated in multiple layers as the coat forming substance.

The equipment for producing coated particles that is to be used in the present invention is in no way limited as long as it is capable of applying the coat forming substance over the surfaces of the particles in the powder of core particles by vapor-phase processes involving transition through the vapor phase. For example, chemical vapor deposition (CVD) equipment can be used, as exemplified by a thermal CVD apparatus, a plasma-assisted CVD apparatus, apparatus for CVD utilizing electromagnetic waves (i.e., visible light CVD, laser CVD, ultraviolet CVD, infrared CVD and far-infrared CVD) and a MOCVD apparatus. Alternatively, physical vapor deposition (PVD) equipment can be used, as exemplified by a vacuum evaporation apparatus, an ion sputtering apparatus and an ion plating apparatus. Stated more specifically, a suitable apparatus for producing coated particles is described in the official gazette of Unexamined Published Japanese Patent Application (kokai) Hei 3-75302, entitled "Particles Coated with Superfine Grains on the Surfaces and A Process for Producing Such Coated Particles".

As described on the foregoing pages, the process of the present invention for producing coated diamond or high-pressure phase boron nitride comprises the steps of charging into the coating space the particles in the powder of core particles which are diamond or high-pressure phase boron nitride particles and then permitting the precursor of the coat forming substance which is generated via the vapor phase and/or the precursor of the coat forming substance in a vapor-

Ex. 1003, Page 269

phase state to contact and/or impinge against the particles in said powder of core particles so that their surfaces are coated with the coat forming substance. Five basic processes for coating the core particles with the coat forming substance are summarized below.

I. A coating method comprising:

(A) a dispersing step in which the particles in the powder of core particles are dispersed in a gaseous atmosphere by the group of means for high dispersion treatment of particles so as to make a mixture of a gas and the particles in a powder of highly dispersed core particles; and
(B) a coating step in which the core particles in said gas-particle mixture, as they have been dispersed with the dispersity β adjusted to the values within the ranges set forth above, are allowed to contact and/or impinge against the coat forming substance in the coating start region of the coating space, thereby starting the coating operation.

II. A coating method comprising:

(A) a dispersing step in which the particles in the powder of core particles are dispersed in a gaseous atmosphere by the group of means for high dispersion treatment of particles which realize the adjustment of dispersity β to the values within the ranges set forth above, thereby making a mixture of a gas and the particles in a powder of highly dispersed core particles; and
(B) a coating step in which the core particles in said gas-particle mixture, as they have been dispersed with the dispersity β adjusted to the values within the ranges set forth above, are allowed to contact and/or impinge against the coat forming substance in the coating start region of the coating space, thereby starting the coating operation.

III. A coating method comprising:

(A) a dispersing step in which the particles in the powder of core particles are dispersed in a gaseous atmosphere by the group of means for high dispersion treatment of particles which realize the adjustment of dispersity β to the values within the ranges set forth above, thereby making a mixture of a gas and the particles in a powder of highly dispersed core particles;
(B) a transport step in which the highly dispersed core particles in said gas-particle mixture are transported directly to a subsequent coating step; and
(C) the coating step in which the transported core particles in said gas-particle mixture, as they have been dispersed with the dispersity β adjusted to the values within the ranges set forth above, are allowed to contact and/or impinge against the coat forming substance in the coating start region of the coating space, thereby starting the coating operation.

IV. A coating method comprising:

(A) a dispersing step in which the particles in the powder of core particles are dispersed in a gaseous atmosphere by the group of means for high dispersion treatment of particles which realize the adjustment of dispersity β to the values within the ranges set forth above, thereby making a mixture of a gas and the particles in a powder of highly dispersed core particles;
(B) a transport step in which the highly dispersed core particles in said gas-particle mixture are transported via at least one member indispensable to transport that is selected from the group consisting of a hollow member, an intermediate member composed of a member that forms a hollow portion, and a pipe; and
(C) a coating step in which the transported core particles in said gas-particle mixture, as they have been dispersed with the dispersity β adjusted to the values within the ranges set forth above, are allowed to contact and/or impinge against the coat forming substance in the coating start region of the coating space, thereby starting the coating operation.

V. A coating method comprising:

(A) a dispersing step in which the particles in the powder of core particles are dispersed in a gaseous atmosphere by the group of means for high dispersion treatment of particles which realize the adjustment of dispersity β to the values within the ranges set forth above, thereby making a mixture of a gas and the particles in a powder of highly dispersed core particles;
(B) a transport step in which the highly dispersed core particles in said gas-particle mixture are transported to a subsequent coating step via at least one means selected from the group consisting of means for maintaining the dispersion in a gaseous atmosphere of the highly dispersed core particles in said gas-particle mixture, means for

Ex. 1003, Page 270

enhancing the dispersion in a gaseous atmosphere of the highly dispersed core particles in said gas-particle mixture, and means for separating a mixture of a gas and the particles in a powder of less highly dispersed core particles from said gas-particle mixture so as to select a mixture of a gas and the particles in a powder of highly dispersed core particles in which the core particles are present in the gaseous atmosphere mainly in a single-particle state; and

(C) the coating step in which the transported particles in said mixture of a gas and the powder of highly dispersed core particles, as they have been dispersed with the dispersity β adjusted to the values within the ranges set forth above, are allowed to contact and/or impinge against the coat forming substance in the coating start region of the coating space, thereby starting the coating operation.

In each of the coating methods I - V, it is preferred that the coating start region of the coating space is located in either one of the following portions of the space region in which the dispersity β of the particles in the powder of highly dispersed core particles as mixed with a gas and which have been dispersed by the group of means for high dispersion treatment of particles is adjusted to the values within the ranges set forth above:

the space region that includes planes through which all particles in the powder of highly dispersed core particles as mixed with a gas will pass; or

the space region that includes planes through which all particles to be recovered into the recovery section of the recovery means will pass.

Alternatively, in each of the coating methods I and II, it is preferred that at least one part of the dispersing step, in which the particles in the powder of core particles are dispersed in a gaseous atmosphere by the group of means for high dispersion treatment of particles which realize the adjustment of dispersity β to the values within the ranges set forth above, thereby making a mixture of a gas and the particles in a powder of highly dispersed core particles, is performed sharing one or more parts of the space with at least one part of the coating step.

It should be noted here that the coated diamond or high-pressure phase boron nitride particles occasionally form lumps as a result of association of several particles that contact one another via the coat forming substance on the coated particles. Thus, the powder of the coated diamond or high-pressure phase boron nitride particles consists of coated particles in a single-particle state, lumps formed by the association of from several to several tens of such discrete coated particles as a result of contacting one another, and larger lumps formed by the association of more of such discrete coated particles as a result of contacting one another, and the shapes and sizes of the particles and lumps are nonuniform and irregular. The lumps which are the associations of the discrete coated particles are preferably disagglomerated and/or otherwise reduced in size before they are subjected to the shaping (molding) or sintering process. To disagglomerate and/or otherwise reduce in size the associating lumps of the coated diamond or high-pressure phase boron nitride particles, various disagglomerating means may be utilized, as exemplified by ball mills, vibrating ball mills, mortars and jet mills, etc. Alternatively, the discrete coated particles may be selectively separated from the associating lumps of such discrete coated particles, so that only the discrete coated particles can be subjected to the shaping or sintering process.

In accordance with the present invention, the thus coated diamond or high-pressure phase boron nitride particles or mixtures containing such particles are then sintered at pressures no less than 2000 MPa and at elevated temperatures, or at pressures of less than 2000 MPa and at temperatures not exceeding 1850°C under such sintering pressure and temperature conditions that diamond or high-pressure phase boron nitride is thermodynamically metastable if not stable.

Alternatively, the coated diamond or high-pressure phase boron nitride particles are mixed with a bonding material in volume ratios of (1 - 90):(99 - 10) that is sintrable to a density of at least 85% at pressures less than 2000 MPa and at temperatures not exceeding 1850°C under such pressure and temperature conditions that diamond or high-pressure phase boron nitride is thermodynamically metastable, and the mixture is then sintered at pressures less than 2000 MPa and at temperatures not exceeding 1850°C under such sintering pressure and temperature conditions that diamond or high-pressure phase boron nitride is thermodynamically metastable if not stable.

If an auxiliary agent, or a substance that develops another function, is to be added, the substance may be in a powder, plate or particulate form. More specifically, the substance may be selected from among powders and/or particles, etc. that are composed of at least one member selected from the group consisting of metals, semiconductors, metalloids, and rare earth metals of groups 1a, 2a, 3a, 4a, 5a, 6a, 7a, 1b, 2b, 3b, 4b, 5b, 6b, 7b and 8 of the periodic table, as well as oxides, nitrides, carbides, oxynitrides, oxycarbides, carbonitrides, oxycarbonitrides, borides and silicides of these elements, as exemplified below:

Al, B, Si, Fe, Ni, Co, Ti, Nb, V, Zr, Hf, Ta, W, Re, Cr, Cu, Mo, TiAl, Ti_3Al, $TiAl_3$, TiNi, NiAl, Ni_3Al, SiC, B_4C, Cr_3C_2, TiC, ZrC, WC, W_2C, HfC, TaC, Ta_2C, NbC, VC, Mo_2C, Si_3N_4, TiN, ZrN, Si_2N_2O, AlN, HfN, VxN (x = 1 ~ 3), NbN, TaN, Ta_2N, TiB, TiB_2, ZrB_2, VB, V_3B_2, VB_2, NbB, NbB_2, TaB, TaB_2, MoB, MoB_2, MoB_4, Mo_2B, WB, W_2B, W_2B5, LaB_6, BP, $B_{13}P_2$, $MoSi_2$, Al_2O_3, ZrO_2 (PSZ: partially stabilized zirconia with the addition of Y_2O_3, MgO or CaO; or TZP: tetragonal zirconia polycrystalline compound), $MgAl_2O_4$ (spinel) and Al_2SiO_5 (mullite).

The auxiliary agent may be a fibrous substance. The fibrous substance to be mixed with the coated diamond or high-pressure phase boron nitride particles is composed of at least one of elemental metals and compounds and has such a morphology that the width is no more than 500 μm, with the length to width ratio being at least 2. The fibrous

Ex. 1003, Page 271

substance comprises bar- or rod-like substances having widths of no more than 500 μm, with the length to width ratio being at least 2, and/or filaments which are continuous fibers formed by melt spinning and/or short fibers produced by crystals themselves to give shape and/or whiskers produced by unidirectional crystal growth into a fibrous form. The whiskers comprise intrinsic whiskers which, according to the definition, are formed without experiencing any phase transition or any phenomenon such as chemical reactions that affect the overall volume and/or whiskers in the broad sense of the term which refer to single crystals that are formed as long needles by the growth of only one crystal face of the crystal that is created by a phase transition or a chemical change that affects the overall volume and/or other whiskers that are single crystals having cross-sectional areas of no more than 8×10^{-5} in^2, with the length being at least 10 times the average diameter.

The fibrous substance to be used may be those which contain at least one compound containing at least one member of the group consisting of metals, semiconductors, metalloids, rare earth metals and nonmetals of groups 1a, 2a, 3a, 4a, 5a, 6a, 7a, 1b, 2b, 3b, 4b, 5b, 6b, 7b and 8 of the periodic table and which have widths of no more than 500 μm, with the length to width ratio being at least 2. More specifically, the fibrous substances to be used are those which are composed of at least one member selected from the group consisting of metals, semiconductors, metalloids, and rare earth metals of groups 1a, 2a, 3a, 4a, 5a, 6a, 7a, 1b, 2b, 3b, 4b and 8 of the periodic table, as well as oxides, nitrides, carbides, oxynitrides, oxycarbides, carbonitrides, oxycarbonitrides, borides and silicides and which have widths of no more than 500 μm, with the length to width ratio being at least 2. Preferably, the following fibrous substances may be selected: those which are composed of at least member one selected from the group consisting of Al, B, Si, Fe, Ni, Co, Ti, Nb, V, Zr, Hf, Ta, W, Re, Cr, Cu, Mo, TiAl, Ti_3Al, $TiAl_3$, TiNi, NiAl, Ni_3Al, SiC, B_4C, Cr_3C_2, TiC, ZrC, WC, W_2C, HfC, TaC, Ta_2C, NbC, VC, Mo_2C, Si_3N_4, TiN, ZrN, Si_2N_2O, AlN, HfN, VxN ($x = 1 \sim 3$), NbN, TaN, Ta_2N, TiB, TiB_2, ZrB_2, VB, V_3B_2, VB_2, NbB, NbB_2, TaB, TaB_2, MoB, MoB_2, MoB_4, Mo_2B, WB, W_2B, W_2B5, LaB_6, BP, $B_{13}P_2$, $MoSi_2$, Al_2O_3, ZrO_2 (PSZ: partially stabilized zirconia with the addition of Y_2O_3, MgO or CaO; or TZP: tetragonal zirconia polycrystalline compound), $MgAl_2O_4$ (spinel), Al_2SiO_5 (mullite), etc., and which have widths of no more than 500 μm, with the length to width ratio being at least 2.

As described above, the coated diamond or high-pressure phase boron nitride particles to be used in the present invention have their surfaces coated with the coat forming substance by vapor-phase processes and, hence, the coat forming substance that can be used is basically unlimited. While materials design of diamond or high-pressure phase boron nitride sinters can be made in any suitable manners depending on their use, the coating of diamond or high-pressure phase boron nitride particles with the coat forming substance may be preceded by preliminary coating of the surfaces of those surfaces with a similar and/or dissimilar coat forming substance by a similar and/or dissimilar coating technique.

Take, for example, the case where the surfaces of diamond or high-pressure phase boron nitride particles are to be eventually provided with coats formed of a desired metal carbide; in this case, those particles may be preliminarily coated with carbon. While the preliminary coating technique is not limited in any particular way, suitable examples include not only the molten salt dip method which is described in Unexamined Published Japanese Patent Application (kokai) Hei 2-252660 and the molten salt disproportionation method which is described in Unexamined Published Japanese Patent Application (kokai) Hei 1-207380 but also electroplating, electroless plating, cladding, physical vapor deposition processes (e.g., sputtering and ion plating) and chemical vapor deposition processes. The metals in the metal components to be eventually coated are not limited to any particular types as long as they are within the class of substances that can be applied as bonding materials and/or sintering aids for the purpose of the invention.

The coating films formed by the molten salt dip method are composed of dense substances having high hardness and melting point and, having typically the capability of isolating diamond or high-pressure phase boron nitride particles from other substances, these films offer the advantage of markedly increasing the latitude in the choice of binders. The diamond or high-pressure phase boron nitride particles provided with coating films by the molten salt dip method are difficult to sinter densely by the conventional process for producing diamond or high-pressure phase boron nitride sinters at pressures less than 2000 MPa under such sintering pressure and temperature conditions that diamond or high-pressure phase boron nitride is thermodynamically metastable. However, if diamond or high-pressure phase boron nitride particles that are coated with the films of high-hardness and high-melting substances which have been formed by the molten salt dip method are coated by the vapor-phase coating techniques of the invention with one or more layers in suitable amounts of substances that can be applied as bonding materials that are sintrable to assure satisfactory density and high hardness, favorable results are attained that are advantageous for the purpose of promoting sintering.

In accordance with the present invention, coated diamond or high-pressure phase boron nitride particles which have the coat forming substance applied to the surfaces of bare diamond or high-pressure phase boron nitride particles by vapor-phase processes may be mixed with a bonding material or, alternatively, the mixture of the coated diamond or high-pressure phase boron nitride particles and the bonding material may be mixed with the aforementioned auxiliary substance in a powder, plate or particulate form and/or the fibrous substance also mentioned above which has a width of no more than 500 μm, with the length to width ratio being at least 2, and either mixture may be sintered in a powder form or after being shaped into a compact.

Ex. 1003, Page 272

Diamond or high-pressure phase boron nitride is thermodynamically stable only at ultrahigh pressures and if the pressure is inadequate under elevated temperatures, they will experience a phase transition to the lower-pressure phase (graphite or hexagonal boron nitride (hBN)).

Therefore, in order to prevent the phase transition of diamond or high-pressure phase boron nitride to the lower-pressure phase (graphite or hBN) and to insure sintering to high density, the sinters containing diamond or high-pressure phase boron nitride are produced at ultrahigh pressures exceeding 2000 MPa and at elevated temperatures under such conditions that diamond or high-pressure phase boron nitride is thermodynamically stable.

Therefore, if one selects those sintering pressure and temperature conditions where the pressure is at least 2000 MPa and where diamond or high-pressure phase boron nitride is thermodynamically stable, the temperature is limited by pressure in accordance with the thermodynamic equilibrium line in the phase diagram of diamond or high-pressure phase boron nitride. However, if one selects those sintering pressure and temperature conditions where the pressure is at least 2000 MPa and where diamond or high-pressure phase boron nitride is thermodynamically metastable if not stable, temperatures will suffice if they deviate slightly from the region that is limited by pressure in accordance with the thermodynamic equilibrium line in the phase diagram of diamond or high-pressure phase boron nitride.

On the other hand, if one selects those sintering pressure and temperature conditions where the pressure is less than 2000 MPa and where diamond or high-pressure phase boron nitride is thermodynamically metastable if not stable, one should note that there is a pressure-dependent temperature in the region where diamond or high-pressure phase boron nitride is thermodynamically metastable.

Stated more specifically, in the case of selecting those sintering pressure and temperature conditions where diamond or high-pressure phase boron nitride is thermodynamically metastable if not stable, the upper limit of the sintering temperature will vary with the quality of the diamond or high-pressure phase boron nitride particles to be sintered.

Take, for example, the case of diamonds; Hall conducted an experiment under ultrahigh pressures and reported that when a diamond was thermodynamically metastable if not stable, it took such a long time for a phase transition to occur that it remained stable for practical purposes; he then indicated the temperature 1100°C as the upper limit beyond which the diamond would no longer exist in a practically stable manner (see H.T. Hall, Science, 169 (1970) 868-869, as well as Fig. 15a). Speaking of high-pressure phase boron nitrides, Wakatsuki et al. conducted an experiment on a cubic boron nitride under ultrahigh pressures and reported that when the cubic boron nitride was thermodynamically metastable if not stable, it took such a long time for a phase transition to occur that it remained stable for practical purposes; they then indicated the temperature 1200°C as the upper limit beyond which the cubic boron nitride would no longer exist in a practically stable manner (see Wakatsuki, Ichise, Aoki and Maeda, Abstracts of the Lectures Read at the 14th Symposium on High-Pressure Technology (1972) p. 78, as well as Fig. 33b).

Therefore, if high-purity diamonds (e.g. natural products of high quality synthetic diamonds produced via the vapor phase by means of PVD or CVD processes, or diamonds synthesized by application of ultrahigh pressures over a long time) are especially selected and if they are sintered either by HIP, optionally under ultrahigh pressures, as they are confined within a degassed, pressure-transmitting capsule or by PC or HP under vacuum or in an inert gas, diamonds will actually exist up to 1850°C which is much higher than 1100°C, or the temperature reported by Hall, even if they are not in a thermodynamically stable state. However, if the temperature exceeds 1850°C, the diamonds will turn to the graphite phase in a short time. Generally speaking, synthesis catalysts or the like will sometimes remain as impurities in synthetic diamonds. However, if synthetic diamonds are such that impurities have been removed positively to achieve high purity, the diamond will exist up to 1700°C by application of similar sintering techniques and this is the same as in the case of natural diamonds. Alternatively, with known diamonds that do not contain fine grains smaller than 1 μm, preferably those which are at least 3 μm and whose size is within the class contemplated by the present invention, the diamond will exist up to 1600°C irrespective of whether they are natural or synthetic products. It should also be noted that in the case of known diamonds of comparatively high purity, both natural and synthetic products will exist up to 1500°C. However, with commonly known types of synthetic diamonds, the suitable temperature is up to 1400°C. Therefore, the upper limit of sintering temperatures is 1850°C.

Speaking of high-pressure phase boron nitrides, if those of high purity (e.g. cubic boron nitrides of ultrahigh purity that are produced via the vapor phase by means of PVD or CVD processes, or cubic boron nitrides of ultrahigh purity that are synthesized by application of ultrahigh pressures over a long time) are especially selected and if they are sintered either by HIP (hot isostatic pressing), optionally under ultrahigh pressures, as they are confined within a degassed, pressure-transmitting capsule or by PC (piston-cylinder) or HP (hot pressing) under vacuum or in an inert gas, cubic boron nitrides will actually exist up to 1850°C which is much higher than 1200°C, or the temperature reported by Wakatsuki et al., even if they are not in a thermodynamically stable state. However, if the temperature exceeds 1850°C, the cubic boron nitrides will turn to a graphitic phase in a short time. If conventionally known cubic boron nitrides are used after impurities have been removed positively to achieve high purity, the cubic boron nitride will exist up to 1700°C by application of similar sintering techniques. Alternatively, with known cubic boron nitrides that do not contain fine grains, preferably those which are at least 3 μm in size, the cubic boron nitride will exist up to 1600°C by application of similar sintering techniques. It should also be noted that in the case of known cubic boron nitrides of comparatively high purity, the cubic boron nitride will exist up to 1500°C by application of similar sintering techniques. However, in the case where

Ex. 1003, Page 273

commonly known types of cubic boron nitrides or wurtzitic boron nitrides are contained, the suitable temperature is up to 1400°C.

Consequently, if one selects those sintering pressure and temperature conditions where the pressure is less than 2000 MPa and where diamond or high-pressure phase boron nitride is thermodynamically metastable if not stable, the upper limit of sintering temperatures is 1850°C. It should be remembered that the temperature at which the diamond or high-pressure phase boron nitride is not in a thermodynamically stable state but exists stably for practical purposes varies with the quality of the diamond or high-pressure phase boron nitride to be used and that, therefore, the sintering temperature must be set in accordance with the quality of the feed of diamond or high-pressure phase boron nitride. If it is necessary that the sintering temperature be set to a value that is close to the right temperature which matches the quality of the feed of diamond or high-pressure phase boron nitride, said sintering temperature must be controlled with close tolerance.

The diamond or high-pressure phase boron nitride sinters of the present invention are produced by sintering processes that adopt two kinds of conditions with different pressures. To produce diamond or high-pressure phase boron nitride sinters by performing sintering for a suitable period of time at ultrahigh pressures of 2000 MPa or above and at elevated temperatures, any conventional equipment that produces ultrahigh pressures can be applied without particular limitations, as exemplified by the cubic, tetragonal, girdle, and belt type ultrahigh pressure apparatus.

Known apparatus and methods may be applied without any particular limitations to produce dynamic ultrahigh pressures (≧ 2000 MPa) and elevated temperatures and they include: an explosive charge lens (planar explosion generator)_, a cylindrical explosion generator, a method involving collision with a flying object, a magnetic implosion method, a Mach explosion method, an excessive explosion method, a conical implosion method, a single-action shock gun, a double-action light gas gun, etc. According to these apparatus and methods which generate dynamic ultrahigh pressures and elevated temperatures, the pressure that can be produced is at least 10 GPa (10,000 MPa), preferably at least 40 GPa and, therefore, they can be selected as preferred examples in cases where particularly high pressures need be exerted for short periods of time.

Depending on the object, for example, in the case where samples need be pressurized while giving results with good reproducibility, the above-mentioned cubic apparatus and various other types of equipment that are capable of generating ultrahigh temperatures in a static manner may be used to produce pressures of 2000 MPa and above. The sintering temperature condition may deviate slightly from the region where diamond or high-pressure phase boron nitride is thermodynamically stable.

But, more preferably, one may select those sintering pressure and temperature conditions where the pressure is at least 2000 MPa and where diamond or high-pressure phase boron nitride is thermodynamically stable.

To produce diamond or high-pressure phase boron nitride sinters by performing sintering for a suitable period of time at pressures less than 2000 MPa and at temperatures not exceeding 1850°C under such sintering pressure and temperature conditions that diamond or high-pressure phase boron nitride is thermodynamically metastable if not stable, any conventional equipment can be applied without particular limitations, as exemplified by PC (piston-cylinder) type ultrahigh pressure apparatus, ultrahigh pressure HIP (hot isostatic pressing) apparatus, HIP apparatus and HP (hot pressing) apparatus.

If the PC type ultrahigh pressure apparatus is to be used, pressures less than 2000 MPa may be applied but, considering the durability of this apparatus, the pressure does not preferably exceed 1500 MPa. Conventionally known technologies concerning the production of pressures include ultrahigh pressure HIP apparatus which is capable of operating at HIP pressures up to 1000 MPa, as well as HIP apparatus (excluding the ultrahigh pressure HIP apparatus) and HP apparatus which are each capable of operating at pressures up to 200 MPa.

The diamond or high-pressure phase boron nitride sinters produced by the methods described above have high performance with highly controlled microstructures. While their most common use is as machine parts, it is possible to manufacture diamond or high-pressure phase boron nitride sinters that are superhard, with the Vickers hardness (Hv) being preferably at least 600, and which are dense, with the density being at least 85%. Preferably, it is possible to manufacture diamond or high-pressure phase boron nitride sinters that are superhard, with the Vickers hardness (Hv) being preferably at least 800, and/or which are dense, with the density being at least 90%. More preferably, considering the application to highly wear-resistant machine parts, superhard (Hv ≧ 1000) diamond or high-pressure phase boron nitride sinters can be manufactured by both increasing the relative content of diamond or high-pressure phase boron nitride and sintering to high density. For use in tools and other applications that require even higher levels of wear resistance, diamond or high-pressure phase boron nitride sinters having Vickers hardness values of at least 2000 can be manufactured by further increasing the content of diamond or high-pressure phase boron nitride while sintering to high density.

In a typical case, a diamond sinter may be produced from a diamond powder feed in the following manner using a cubic apparatus to produce ultrahigh pressures. First, the particles in the diamond powder are coated with a suitable coat forming substance on the surfaces. The coated diamond particles are then shaped into a pellet by stamping. The pellet is surrounded by a shaped hexagonal boron nitride (h-BN) part which, in turn, is enclosed with a graphite tubular heater. Provided around the heater as a solid pressure transmitting medium is pyrophyllite from which the water of

Ex. 1003, Page 274

crystallization has been removed by heat treatment at 700°C. The sample thus prepared is placed in the cubic apparatus; the pressure is increased to a predetermined level and, then, the temperature is also elevated to a predetermined level and sintering is effected for a suitable time. Thereafter, the temperature is lowered and the pressure is also reduced. The pressure-transmitting medium is taken out of the cubic ultrahigh pressure apparatus and the sample is then recovered from the medium. In this way, one can produce a diamond sinter of high performance that is composed of diamond and which is characteristic in that it has a controlled distribution of the bonding material and/or sintering aid and/or surface modifier, that is uniform and dense, and that it has a highly controlled microstructure.

As described in detail on the foregoing pages, a powder of fine diamond or high-pressure phase boron nitride core particles having an average diameter of no more than 10 μm in terms of frequency distribution by volume was dispersed in a gaseous atmosphere and the core particles thus dispersed to a dispersity β of at least 70% were allowed to contact or impinge against a precursor of a coat forming substance, whereby the core particles were covered on their surfaces with the coat forming substance in a single-particle state. In accordance with the invent ion, the thus coated core particles or mixtures containing them were sintered at ultrahigh atmospheres of at least 2000 MPa and at elevated temperatures, whereby coated diamond or high-pressure phase boron nitride sinters of high performance were produced that had uniform, dense, firmly sintered and highly controlled microstructures.

According to another aspect of the invention, said core particles or mixtures containing them or mixtures of said core particles with bonding materials are sintered at pressures less than 2000 MPa and at temperatures not exceeding 1850°C under such sintering pressure and temperature conditions that diamond or high-pressure phase boron nitride is thermodynamically metastable if not stable, whereby one can produce coated diamond or high-pressure phase boron nitride sinters of high performance that have uniform, dense, firmly sintered and highly controlled microstructures. This offers the advantage of eliminating the constraint of the need to use equipment capable of creating ultrahigh pressures of at least 2000 MPa. It should be particularly noted that the use of ultrahigh pressure HIP apparatus or other types of HIP apparatus offers the added advantage of enabling the production of complexly shaped sinters, thereby presenting significant benefits to manufacture on an industrial scale.

According to yet another aspect of the invention, even pseudo-fine diamond or high-pressure phase boron nitride particles having an average diameter greater than 10 μm in terms of frequency distribution by volume may be covered with coat forming substances after being dispersed to a dispersity as determined by their average diameter. By sintering the thus coated pseudo-fine particles, one can produce sinters that are substantially free from graphite or a graphitic phase and which have the same features as fine-particulate sinters having an average particle diameter of no more than 10 μm.

EXAMPLES

The diamond or high-pressure phase boron nitride sinters of the present invention, as well as the processes for their production are described below with reference to examples.

Example 1

Diamond particles having an average diameter (D_M) of 1 μm and a frequency distribution by volume of ([D_M/2, 3D_M/2], ≧ 90%) were coated with elemental silicon.

The construction of the apparatus used is shown in Fig. 6 and Fig. 7 which is a partial enlarged view of Fig. 6; the apparatus is a specific example of the design shown in Fig. 5a.

As shown, the apparatus comprises: a plasma torch 3-A including a plasma chamber 3-a, a vessel 3-B for cooling a coat forming substance precursor generating chamber 3-b, a coating chamber cooling vessel 3-C in the narrow sense of the term, a coating chamber 3-c in the narrow sense of the term, a vessel 3-D for cooling a coated particle cooling chamber 3-d, a supply unit 3-E1 on the side where the feed of the coat forming substance is supplied, an agitating disperser 3-F1 and an ejector-type disperser 3-H1 which are on the side where a powder of core particles is supplied, a capillary disperser 107, and a coated particle recovery section 3-G. The supply unit 3-E1 is coupled to a feeder 112 equipped with a vessel for supplying the feed powder of the coat forming substance, and the agitating disperser 3-F1 is coupled to a feeder 111 equipped with a vessel for supplying the powder of core particles. According to the definition, the coating chamber used in Example 1 is composed of the plasma chamber 3-a, the coat forming substance precursor generating chamber 3-b, the coating chamber 3-c in the narrow sense of the term, and the coated particle cooling chamber 3-d. These make up the coating chamber in the broad sense of the term and the chamber 3-c where most part of the coating operation is performed is referred to as the "coating chamber in the narrow sense of the term".

The group of means for high dispersion treatment of particles α used in Example 1 is composed of the feeder 111 equipped with a supply vessel, agitating disperser 3-F1, ejector-type disperser 3-H1, and the capillary disperser 107 which is made of a stainless steel pipe having an inside diameter of 4 mm. The basic concept of the group is shown in Fig. 2a and it is a specific example of the group of means for high dispersion treatment of particles that is of the design shown in Fig. 3b. The group is designed in such a way that it can realize an output value β of at least 70% (β ≧ 70%)

Ex. 1003, Page 275

given the particles in a powder of diamond core particles that are characterized by ($[D_M/5, 5D_M]$, ≧ 90%) where D_M = 1 μm. The capillary disperser 107 which is the final means of treatment in the group of means for high dispersion treatment of particles is coupled directly to the coating chamber 3-c and designed in such a way as to realize β ≧ 70% in the coating start region 3-L1 of the coating space 3-L2.

A gas jet port 101 is provided on top of the plasma torch 3-A and an argon gas is supplied from a supply source 102 through this port at a rate of 20 L/min. The supplied argon gas is turned into a plasma by applied high-frequency waves, thereby forming a plasma flame in the plasma chamber 3-a within the plasma torch 3-A.

A silicon powder comprising particles with an average diameter of 10 μm serves as the feed of the coat forming substance and is supplied from the feeder 112 equipped with a vessel for supplying the feed powder of the coat forming substance. The supplied silicon powder is carried by a carrier gas 103 flowing at a rate of 5 L/min so that it is introduced into the plasma flame at a rate of 0.2 g/min via an inlet 104 provided at the bottom of the plasma torch 3-A. Being evaporated by the heat of the plasma flame, the silicon powder goes through the vapor phase to become the precursor of the coat forming substance in the precursor generating chamber 3-b.

The diamond core particles with an average diameter of 1 μm are supplied at a rate of 1.2 g/min from the feeder 111 equipped with a supply vessel of the core particles and they are dispersed with the agitating disperser 3-F1 while being carried by a carrier gas 105 that is supplied at a rate of 5 L/min. The particles are then dispersed to a dispersity (β) of 82% by means of the ejector-type disperser 3-H1 and the capillary disperser 107 with the aid of a dispersing gas 106 that is supplied at a flow rate of 10 L/min. The thusly dispersed core particles are introduced into the coating chamber.

The highly dispersed diamond particles entering the coating start region 3-L1 of the coating space 3-L2 start to contact and/or impinge against the precursor of the coat forming substance as they maintain the dispersity β at 82%.

Thus, the diamond particles are given the coat of the coat forming substance on their surfaces and they descend down the coated particle cooling chamber 3-d together with the gas, eventually reaching the recovery section 3-G. The product comprising the coated particles is separated from the gas 109 by means of a filter 110 and collected for recovery.

The recovered fine diamond particles having silicon coats on the surfaces were examined with a scanning electron microscope. As Fig. 8 shows, all of these particles were uniformly coated with superfine silicon particles of about 0.005 μm in size. The silicon coating weight was 20% by volume.

The resulting powder of diamond particles coated with 20 vol% silicon was stamped into a pellet having an outside diameter of 6 mm and a height of 2 mm. The pellet was buried in a pressure-transmitting medium surrounded by a shaped hexagonal boron nitride (h-BN) and it was vacuum-dried overnight at 200°C and at 10^{-3} torr to remove low-boiling impurities. The pellet was then set in a cubic ultrahigh pressure apparatus: the pressure was raised to 5.5 GPa at room temperature, then the temperature was elevated to 1450°C, which was held for 30 min; thereafter, the temperature was lowered and the pressure reduced.

The resulting sinter was examined by X-ray diffractiometry; the elemental silicon reacted with the diamond to change to both diamond and SiC while the elemental silicon disappeared. Quantification by X-ray analysis showed that the volume proportions of the diamond sinter and SiC were about 78% and 22%, respectively.

The surface of the sinter was polished with a diamond paste and its Vickers microhardness (Hv) was measured. The diamond sinter synthesized in Example 1 was superhard with the Hv (0.5/10) being about 12,000 and this value was comparable to or higher than that of a commercial cutting tool made of sintered diamond. Furthermore, the hardness of the sinter of Example 1 was uniform over the entire surface subjected to the hardness measurement and it was substantially free from variations.

The polished surface of the sinter resulting of the example 1 was coated with a gold film by a customary evaporation technique and examined with an electron microscope. The result is shown in the Fig. 9 electron micrograph (X5000), from which one can see that the sinter had a relative density of at least 99%. In addition, the sinter had no portion left unsintered. In areas where diamond particles contacted each other via a thin layer of the coat forming substance, the latter was bursted by the contiguous diamond particles, which hence were coupled directly as a result of sintering. In all other areas, SiC was distributed to surround the diamond particles, rendering the sinter to be characteristic in that it was dense, uniform and had the bonding material distributed provide a highly controlled microstructure. It should also be noted that the diamond particles in the sinter did not grow at all compared to the particles in the feed powder and that the coating on the diamond was formed by chemical reaction between the diamond and the elemental silicon which served as the coat forming substance; these facts combined together to offer the characteristic feature that the diamond particles in the sinter were finer than those in the feed although they were coated with silicon. This is very favorable and, hence, ideal for the mechanical characteristics of the sinter. The differences can be clearly seen by comparing Fig. 9 with Fig. 10 which is an electron micrograph (X5000) of a polished surface of the above-mentioned commercial cutting tool that was made of sintered diamond and which was coated with a gold film by a customary evaporation technique for examination. Obviously, the bonding material and the sintering aid were distributed irregularly in the diamond sinter of the commercial cutting tool to form lumps in certain areas. On the other hand, the number of areas that were devoid of the necessary sintering aid and bonding material was by no means small and unsintered portions were found there. Another major difference from the sinter of Example 1 was that every diamond particle in the cutting tool was coarse.

Ex. 1003, Page 276

Diamond is the most difficult to sinter of all substances that exist on the earth. In spite of this fact, the coated particles in a diamond powder that were synthesized in Example 1 in accordance with the invention behaved as if they were comparatively easy-to-sinter particles under industrially feasible ultrahigh pressures and elevated temperatures, thereby forming a dense, rigid and superhard structure.

Example 2

Cubic boron nitride particles having an average diameter (D_M) of 1 μm and a frequency distribution by volume of ($[D_M/2, 3D_M/2]$, ≧ 90%) were coated with alumina.

The construction of the apparatus used is shown in Fig. 11 and Fig. 12 which is a partial enlarged view of Fig. 11; the apparatus is a specific example of the design shown in Fig. 5d.

As shown, the apparatus comprises: a plasma torch 5-A including a plasma chamber 5-a, a vessel 5-B for cooling a coat forming substance precursor generating chamber 5-b, a coating chamber cooling vessel 5-C in the narrow sense of the term, a coating chamber 5-c in the narrow sense of the term, a vessel 5-D for cooling a coated particle cooling chamber 5-d, a supply unit 5-E1 on the side where the feed of the coat forming substance is supplied, an agitating disperser 5-F1 which is on the side where a powder of core particles is supplied, a capillary disperser 211 and a disperser 5-H2 that utilizes an impingement plate, and a coated particle recovery section 5-G. That part of this apparatus which was to generate the precursor of the coat forming substance was of the same design as in Example 1. The group of means for high dispersion treatment of particles α was composed of a feeder 214 equipped with a supply vessel, the agitating disperser 5-F1, the capillary disperser 211, and the disperser 5-H2 that utilizes an impingement plate. The basic concept of the group α is shown in Fig. 2a and it is a specific example of the group of means for high dispersion treatment of particles that is of the design shown in Fig. 3b. The capillary disperser 211 is made of a stainless steel pipe having an inside diameter of 4 mm. The disperser 5-H2 is the final means of treatment in the group of means for high dispersion treatment of particles α and it consists of a SiC impingement plate 213 supported by a stainless steel holder 212. The disperser 5-H2 is contained in the coating chamber 5-c in the narrow sense of the term, which shares the space with the group of means for high dispersion treatment of particles α. A coating space 5-L1 and the coating start region 5-L2 of the coating space are also contained in the coating chamber 5-c in the narrow sense of the term. The group of means α in the apparatus of Example 2 has such performance that the particles in a powder of core particles having an average diameter (D_M) of 1 μm and a frequency distribution by volume of ($[D_M/5, 5D_M]$, ≧ 90%) can be dispersed to a dispersity (β) of at least 70% (β ≧ 70%) right after impingement against the plate 213 in the disperser 5-H2 at the last stage of dispersion treatment. Therefore, coating of the core particles starts with the dispersity β held at 70% and above.

A gas jet port 201 is provided on top of the plasma torch 5-A and an argon gas is supplied from a supply source 202 through this port at a rate of 20 L/min. The supplied argon gas is turned into a plasma by applied high-frequency waves, thereby forming a plasma flame in a plasma chamber 5-a within the plasma torch 5-A.

An alumina powder comprising particles with an average diameter of 2 μm serves as the feed of the coat forming substance and is supplied at a rate of 1.0 g/min from a feeder 215 equipped with a vessel for supplying the feed powder of the coat forming substance. The supplied alumina powder is carried by a carrier gas 203 flowing at a rate of 5 L/min so that it is introduced into the plasma flame via an inlet 204 of the feed of the coat forming substance provided at the bottom of the plasma torch 5-A. Being evaporated by the heat of the plasma flame, the alumina powder goes through the vapor phase to become the precursor of the coat forming substance in a coat forming substance precursor generating chamber 5-b.

The core particles of cubic boron nitride are supplied at a rate of 0.7 g/min from the feeder 214 equipped with a vessel for supplying the powder of core particles and they are dispersed with the agitating disperser 5-F1 while being carried by a carrier gas 205 that is supplied at a rate of 20 L/min. The particles then pass through the capillary disperser 211 to enter the disperser 5-H2 with the impingement plate in the coating chamber, in which they are dispersed in a gaseous atmosphere to a dispersity (β) of 82%.

The highly dispersed core particles of cubic boron nitride, entering the coating start region 5-L1 of the coating space 5-L2 start to contact and/or impinge against the precursor of the coat forming substance as they maintain the dispersity β of 82%.

Thus, the cubic boron nitride particles are given the coat of the coat forming substance on their surfaces and they descend down a coated particle cooling chamber 5-d together with the gas, eventually reaching a coated particle recovery section 5-G. The product comprising the coated cubic boron nitride particles is separated from the gas 209 by means of a filter 210 and collected for recovery. The recovered cubic boron nitride particles had alumina coats in an amount of 50% by volume.

The recovered fine cubic boron nitride particles having alumina coats on their surfaces were examined with a scanning electron microscope. All of those particles were uniformly coated with superfine alumina particles of about 0.005 μm in size.

Ex. 1003, Page 277

The resulting cubic boron nitride particles with 50 vol% alumina were stamped into a pellet having a diameter of 8 mm and a thickness of 5 mm. The pellet was put into a Pyrex glass capsule of generally the same design as in Example 1 and sealed after degassing by heating.

Thereafter, the encapsulated pellet was set in a ultrahigh pressure HIP apparatus and sintered by holding at a temperature of 1200°C and at a pressure of 1000 MPa for 3 h.

The sinter of Example 2 was checked for its crystalline phase by powder X-ray diffraction and no diffraction peaks were observed except for the cubic boron nitride and α-form alumina.

AS Table 4 shows, the sinter was considerably dense, with a density of 95.8%, and at the same time it was superhard, with the Vickers microhardness being Hv (0.5/10) = 3750.

It should also be mentioned that the cubic boron nitride particles in the sinter were surrounded by alumina to provide a uniform distribution and a highly controlled microstructure.

Example 3

Quasi-fine diamond particles having an average diameter (DM) of 17 μm and a frequency distribution by volume of ([DM/2, 3DM/2], ≧ 90%) were coated with metallic zirconium.

The construction of the apparatus used is shown in Fig. 13 and Fig. 14 which is a partial enlarged view of Fig. 13; the apparatus is a specific example of the design shown in Fig. 5b.

As shown, the apparatus comprises: a plasma torch 6-A including a plasma chamber 6-a, a vessel 6-B for cooling a coat forming substance precursor generating chamber 6-b, a coating chamber cooling vessel 6-C in the narrow sense of the term, a coating chamber 6-c in the narrow sense of the term, a vessel 6-D for cooling a coated particle cooling chamber 6-d, a supply unit 6-E1 on the side where the feed of the coat forming substance is supplied, an agitating disperser 6-F1, a cyclone 6-I, a hopper 6-J and a rotary value 6-K which are on the side where a powder of core particles is supplied, a pipe 307 inevitable to transport, and a coated particle recovery section 6-G. That part of this apparatus which was to generate the precursor of the coat forming substance was of the same design as in Example 1. The group of means for high dispersion treatment of particles α was composed of a feeder 313 equipped with a supply vessel, the agitating disperse 6-F1 which is dispersing means, and the cyclone 6-I which is means for selecting a mixture of a gas and the particles in a powder of highly dispersed core particles. The basic concept of the group α is shown in the Fig. 2b block diagram. The section of cyclone 6-I discharging the mixture of a gas and the particles in a powder of highly dispersed core particles is connected to the coating chamber 6-c in the narrow sense of the term by means of the pipe 307 inevitable to transport, whereas the section of cyclone 6-I discharging the powder portion consisting of less highly dispersed core particles is connected to the agitating disperser 6-F1 by means of a transport pipe 310 via the hopper 6-J and the rotary valve 6-K. Using the group of means α in the apparatus of Example 3, the particles in a powder of core particles having an average (DM) of 17 μm and a frequency distribution by volume of ([DM/5, 5DM], ≧ 90%) can be dispersed to a dispersity (β) of at least 85% in the section of cyclone 6-I (i.e., the means of final treatment) discharging the powder stream of highly dispersed particles. As shown, the coating space 6-L2 and the coating start region 6-L1 of this coating space are contained in the coating chamber 6-c in the narrow sense of the term. Because of the constraint by the flange portion coupling a coating chamber cooling vessel 6-C in the narrow sense of the term with the vessel 6-D for cooling the coated particle cooling chamber 6-d, the pipe 307 was indispensable to transport, but the resulting drop in dispersity β could be held at a reduced level. Hence, coating of the core particles starts with the dispersity β held at 80% and above in the coating start region.

A gas jet port 301 is provided on top of the plasma torch 6-A and an argon gas is supplied from a supply vessel 302 through this port at a rate of 20 L/min. The supplied argon gas is turned into a plasma by applied high-frequency waves, thereby forming a plasma flame in the plasma chamber 6-a within the plasma torch 6-A.

A zirconium powder serving as the feed of the coat forming substance is supplied at a rate of 0.5 g/min from a feeder 314 equipped with a vessel for supplying the feed powder of the coat forming substance. The supplied zirconia powder is carried by a carrier gas 303 flowing at a rate of 5 L/min so that it is introduced into the plasma flame via an inlet 304 the feed of the coat forming substance provided at the bottom of the plasma torch 6-A. Being evaporated by the heat of the plasma flame, the zirconia powder goes through the vapor phase to become the precursor of the coat forming substance in the coat forming substance precursor generating chamber 6-b.

Diamond core particles are supplied at a rate of 2.0 g/min from the feeder 313 equipped with a vessel for supplying the powder of core particles and they are dispersed with the agitating disperser 6-F1 while being carried by a carrier gas 305 that is supplied at a rate of 15 L/min. The particles then pass through a pipe 306 and thence enter the cyclone 6-I. The cyclone 6-I is adjusted in such a way that the fines produced will have a maximum particle diameter of 20 μm; a mixture of the gas and the particles in a powder of highly dispersed (β = 92%) core particles which are mostly single particles passes through the pipe 307 which is indispensable to transport, so that it is discharged into the coating chamber 6-c in the narrow sense of the term via a discharge port 308. On the other hand, the powder portion consisting of less highly dispersed core particles which have been selectively separated by cyclone 6-I passes through hopper 6-J and

Ex. 1003, Page 278

rotary valve 6-K to be transported through a pipe 310 by a carrier gas 309 flowing at a rate of 10 L/min so that it is fed back to the agitating disperser 6-F1.

The highly dispersed diamond particles entering the coating start region 6-L1 of the coating space 6-L2 start to contact and/or impinge against the precursor of the coat forming substance as they maintain the dispersity (β) of 89%. Thus, the diamond particles are given the coat of the coat forming substance on their surfaces and they descend down a coated particle cooling chamber 6-d together with the gas, eventually reaching a coated particle recovery section 6-G. The product comprising the coated diamond particles is separated from the gas 311 by means of a filter 312 and collected for recovery.

The recovered diamond particles having zirconium coats on the surfaces were examined with a scanning electron microscope. All of these particles were uniformly coated with superfine zirconium particles of about 0.005 μm in size. The zirconium coating weight was 10% by volume.

To provide a bonding material for binding the resulting zirconium-coated diamond particles, another lot of diamond particles were coated with titanium carbide under generally the same conditions as those used to produce the zirconium-coated diamond particles, thereby producing a coated diamond powder in which fine diamond particles of diameters ranging from 0.5 to 2 μm (average diameter = 1 μm) were coated with 15 vol% of titanium carbide. Thirty percent by volume of the previously prepared zirconium-coated diamond particles were wet mixed in acetone with seventy percent by volume of the thus prepared titanium carbide coated diamond particles. The mixture was stamped to a pellet having an outside diameter of 6 mm and a height of 2 mm. The pellet was buried in a pressure-transmitting medium surrounded by a shaped hexagonal boron nitride (h-BN) part and it was vacuum-dried overnight at 200°C and at 10^{-3} torr to remove low-boiling impurities. The pellet was then set in a cubic ultrahigh pressure apparatus; the pressure was raised to 5.5 GPa at room temperature, then the temperature was elevated to 1450°C, which was held for 30 min; thereafter, the temperature was lowered and the pressure reduced.

The resulting sinter was examined by X-ray diffractiometry and only diamond, zirconium carbide and titanium carbide were found in it. The sinter was dense with a relative density of at least 99%. Quantification by X-ray analysis showed that the volume proportions of diamond, zirconium carbide and titanium carbide were about 86%, 3% and 11%, respectively.

The sinter was dense and rigid, forming a very highly controlled microstructure.

Example 4

Coated diamond particles were prepared by coating the bare diamond particles of Example 1 with various coat forming substances using apparatus of the same types as used in Examples 1 - 3. The names of the coat forming substances used to prepare the coated diamond particles and their coating weights (relative proportions by vol%), as well as the specific types of the apparatus used and the conditions for their use (coating operation) are shown in Table 1 together with the data for Example 1.

The coated diamond particles thus obtained were examined with a scanning electron microscope. All of the particles prepared in Runs 4-1 and 4-2 were uniformly covered with superfine particles of coat forming substances in sizes of about 0.005 μm.

The apparatus used in Run 4-1 was of the same type as used in Example 2; a metallic zirconium powder having an average diameter of 12 μm was supplied as a feed of coat forming substance from the feeder 215 at a rate of 0.6 g/min, and the core diamond particles were supplied from the feeder 214 at a rate of 1.2 g/min. The other conditions in Run 4-1 were the same as in Example 2.

The apparatus used in Run 4-2 was of the same type as used in Example 3; the group of means for high dispersion treatment of particles in this apparatus was to treat core diamond particles having an average diameter D_M of 1 μm and a distribution by volume of ($[D_M/5, 5D_M]$, ≧ 90%), so the particles in the powder of core particles could be dispersed to a dispersity of 75% or more (β ≧ 75%) at the discharge section of cyclone 6-I, making it possible to start the coating operation at a dispersity of 70% or more (β ≧ 70%) in the coating start region. In Run 4-2, a metallic titanium powder was supplied as a feed of coating forming substance from the feeder 314 at a rate of 0.6 g/min and the core diamond particles were supplied from the feeder 313 at a rate of 1.2 g/min; the cyclone 6-I was adjusted in such a way that the fines produced would have a maximum particle diameter of 1.5 μm and the dispersity that could be attained in the discharge section was 85% (β = 85%) whereas the dispersity attainable in the coating start region was 82% (β = 82%); the other conditions in Run 4-2 were the same as in Example 3.

The apparatus used in Runs 4-7 and 4-8 were both of the same type as used in Example 1 and operated under generally the same conditions as in Example 1 except that aluminum and metallic nickel were deposited in that order to give a total coating weight of 15% in Run 4-7 as calculated for NiAl (Al to Ni molar ratio being 1:1) and 20% in Run 4-8 for Ni_3Al (Al to Ni molar ratio being 1:3).

The thus prepared powders of coated diamond particles or mixtures thereof with silicon carbide whiskers (addition aids) were shaped as in Example 1 and set in a cubic ultrahigh pressure apparatus; the pressure was raised to 5.5 GPa

at room temperature, then the temperature was elevated to 1450 - 1460°C, which was held for 30 min; thereafter, the temperature was lowered and the pressure reduced.

In Runs 4-9 and 4-10, 30% by volume of the titanium carbide and silicon carbide coated diamond particles prepared in Runs 4-4 and 4-6, respectively, were wet mixed in acetone with 10% by volume of silicon carbide whiskers as a fibrous substance (SiC; width = 0.5 μm; average length = 30 μm) and subsequently vacuum dried to produce dry blends.

Each of the sinters thus produced was entirely free from pores, could be densified to a relative density of 100% and had no portion left unsintered. As in Example 1, the surfaces of the sinters were polished with a diamond paste and their Vickers microhardness Hv (0.5/10) values were measured. The results are shown in Table 1 together with the sintering conditions including the data for Example 1.

The crystalline phases of the sinters were examined by powder X-ray diffraction. In Run 4-1, only diamond and ZrC were observed and quantification by X-ray analysis showed that the volume proportions of diamond and ZrC were about 78% and 22%, respectively. In Run 4-2, only diamond and TiC were observed and quantification by X-ray analysis showed that the volume proportions of diamond and TiC were about 76% and 24%. In Runs 4-3 to 4-8, only diamond and the coat forming substances used (TiC, TiC, SiC, SiC, NiAl and Ni_3Al, respectively) were observed; in Run 4-9, only diamond and TiC were observed in addition to SiC; in Run 4-10, only diamond and SiC were observed.

All of the sinters thus produced were superhard, dense, uniform and had a uniform distribution of bonding materials to establish highly controlled microstructures. In particular, the composite sinters produced in Runs 4-9 and 4-10 had highly controlled microstructures in which the TiC bonding material (Run 4-9) or SiC bonding material (Run 4-10) and the SiC whiskers were dispersed uniformly between coated diamond particles.

Ex. 1003, Page 280

Table 1

Run No.	Relative Proportions (vol %)				Coating Apparatus Ex. No.	Coating Conditions (Approximate) Ex.or Run. No.	Sintering Conditions			Sinter		
	Core Particle	Coat Forming Substance		Whisker			Pressure	Temperature	Time	Sinter apparatus	Density	Hardness
	Diamond	Name	Amount	SiC			GPa	°C	min		%	Hv
1	80	Si	20	-	1	1	5.5	1450	30	CB	≥99	12000
4-1	80	Zr	20	-	2	4-1	5.5	1450	30	CB	100	11000
4-2	80	Ti	20	-	3	4-2	5.5	1450	30	CB	100	11500
4-3	85	TiC	15	-	1	1	5.5	1460	30	CB	100	12000
4-4	80	TiC	20	-	1	4-3	5.5	1460	30	CB	100	11000
4-5	80	SiC	20	-	1	4-4	5.5	1450	30	CB	100	12000
4-6	70	SiC	30	-	2	4-5	5.5	1450	30	CB	100	11000
4-7	85	NiAl	15	-	1	1	5.5	1450	30	CB	100	8900
4-8	80	Ni_3Al	20	-	1	4-7	5.5	1450	30	CB	100	6700
4-9	72	TiC	18	10	1	4-4	5.5	1460	30	CB	100	9800
4-10	72	SiC	18	10	1	4-6	5.5	1450	30	CB	100	9400

CB: Cubic ultrahigh pressure apparatus

Example 5

Using apparatus of the same types as used in Examples 1 - 3, high-pressure phase boron nitride powder particles, or cubic boron nitride (cBN) particles of the same type as used in Example 2 that had an average diameter (D_M) of 1 μm and a frequency distribution by volume of ($[D_M/2, 3D_M/2]$, ≧ 90%) were covered with various coat forming substances. The names of the coat forming substances used to prepare the coated cubic boron nitride particles and their coating weights (relative proportions by vol%), as well as the specific types of the coating apparatus used and the conditions for their use (generally the same) are shown in Table 2.

The apparatus used in Run 5-1 was of the same type as used in Example 1; a metallic zirconium powder having an average diameter of 12 μm was supplied as a feed of coat forming substance from the feeder 112 at a rate of 0.6 g/min, and the cubic boron nitride core particles were supplied from the feeder 111 at a rate of 1.2 g/min. The other conditions in Run 5-1 were the same as in Example 1. The apparatus used in Run 5-2 was of the same type as used in Example 2; a metallic titanium powder having an average diameter of 25 μm was supplied as a feed of coat forming substance at a rate of 0.4 g/min whereas the core particles were supplied at a rate of 1.2 g/min; the other conditions in Run 5-2 were the same as in Example 2.

The apparatus used in Run 5-3 was of the same type as used in Example 3. Notwithstanding the difference in the core particles used (diamond or cubic boron nitride), the values of dispersity (β) attained in the discharge section of cyclone 6-I and in the coating start region were the same as in Run 4-2 and titanium nitride as a feed of coat forming substance was supplied at a rate of 0.6 g/min whereas the core particles were supplied at a rate of 2.0 g/min. The other conditions in Run 5-3 were the same as in Example 3.

The coated cubic boron nitride particles thus prepared were examined with a scanning electron microscope. All of the particles prepared in Runs 5-1 to 5-3 were uniformly covered with superfine particles (ca. 0.005 μm) of coat forming substances as Fig. 15 shows (particularly for the case of Run 5-1).

The coating with NiAl in Run 5-14 and with Ni_3Al in Run 5-13, as well as the production of mixtures with SiC whiskers in Runs 5-15 and 5-16 were performed as in Runs 4-7 to 4-10, respectively.

The thus prepared powders of coated cubic boron nitride particles or mixtures thereof with SiC whiskers were shaped as in Example 1 and set in a cubic ultrahigh pressure apparatus; the pressure was raised to 5.1 - 5.6 GPa at room temperature, then the temperature was elevated to 1450 - 1500°C, which was held for 30 min; thereafter the temperature was lowered and the pressure reduced.

Each of the thus produced sinters was entirely free from pores, could be densified to a relative density of 100% and had no portion left unsintered. As in Example 1, the surfaces of the sinters were polished with a diamond paste and their Vickers microhardness Hv (0.5/10) values were measured. The results are shown in Table 2 together with the sintering conditions.

The crystalline phases of the sinters were examined by powder X-ray diffraction. In Run 5-1, Zr reacted with the cubic boron nitride, whereupon metallic Zr disappeared, changing to cubic boron nitride, ZrN and ZrB_2 which were found to be present in respective volume proportions of about 77%, 14% and 9% according to quantification by X-ray analysis. In the sinter of Run 5-2, only cubic boron nitride, TiN and TiB_2 were observed and their respective volume proportions were about 74%, 15% and 11%. In Runs 5-3 to 5-14, only the cubic boron nitride and the coating forming substances used (TiN, TiN, AlN, AlN, α- and β-Si_3N_4, α- and β-Si_3N_4, α-Al_2O_3, α-Al_2O_3, Co, Co, Ni_3Al and NiAl, respectively) were observed; in Runs 5-15 and 5-16, only TiN and α- and β-Si_3N_4, respectively, were observed in addition to the aid SiC and the cubic boron nitride core particles. It should be noted that the sinter produced in Run 5-11 and 5-12 were entirely free from the pool of metallic Co.

Fig. 16 is a graph showing the Hv (0.5/10) value of the cubic boron nitride sinter of Run 5-1 in comparison with the hardness data of four typical commercial cutting tools made from cubic boron nitride sinters using ceramic bonding materials or sintering aids. The sinter produced in Run 5-1 was superhard with the Hv (0.5/10) being about 5500 and this value was comparable to or higher than those of the commercial cutting tools made of sintered cubic boron nitride. Furthermore, the hardness of the sinter of Run 5-1 was uniform over the entire surface subjected to the hardness measurement and it was substantially free from variations.

The polished surface of the sinter of Run 5-1 was coated with a gold film by a customary evaporation technique and examined with an electron microscope. The result is shown in the Fig. 17 electron micrograph (X5000), in which the dark area represents the cubic boron nitride and the light area the ZrN-ZrB_2 base substance. As one can see from Fig. 17, the sinter was entirely free from pores and could be sintered to a relative density of 100%. In addition, it had no portion left unsintered. In areas where cubic boron nitride particles contacted each other via a thin layer of the coat forming substance, the latter was bursted by the contiguous cubic boron nitride particles, which hence were coupled directly as a result of sintering. In all other areas, the ZrN-ZrB_2 base material was distributed to surround the cubic boron nitride particles, rendering the sinter to be characteristic in that it was dense, uniform and had the bonding material distributed to provide a highly controlled microstructure. It should also be noted that the cubic boron nitride particles in the sinter did not grow at all compared to the particles in the feed powder and that the coating on the cubic boron nitride was formed by chemical reaction between the cubic boron nitride and the metallic zirconium which served as the coat

Ex. 1003, Page 282

forming substance; these factors combined together to offer the characteristic feature that the cubic boron nitride particles in the sinter were finer than those in the feed although they were coated zirconium. This is very favorable and, hence, ideal for the mechanical characteristics of the sinter. The differences can be clearly seen by comparing Fig. 17 with Fig. 18 which is an electron micrograph (X5000) of a polished surface of one of the above-mentioned commercial cutting tools that was made of sintered cubic boron nitride and which was coated with a gold film by a customary evaporation technique for examination. Obviously, the bonding material and the sintering aid were distributed irregularly in the cubic boron nitride sinter of the commercial cutting tool to form lumps in certain areas. On the other hand, the number of areas that were devoid of the necessary sintering aid and bonding material was by no means small and unsintered portions were found there. Another major difference from the sinter of Run 5-1 was that every cubic boron nitride particle in the cutting tool was coarse.

The cubic boron nitride is inherently very difficult to sinter. In spite of this fact, the coated particles in a cubic boron nitride powder that were produced in Example 5 in accordance with the invention behaved as if they were comparatively easy-to-sinter particles under industrially feasible ultrahigh pressures and elevated temperatures, thereby forming dense, rigid and superhard structures.

The sinters thus produced were similar to those produced in Examples 1 and 4 in that they were sinters or composite sinters that were superhard and which had dense, uniform and highly controlled microstructures with substantially uniform distributions of the bonding materials.

Ex. 1003, Page 283

Table 2

Run No.	Relative proportions (vol %)				Coating	Coating Conditions (Approxi-mate)	Sintering Conditions			Sinter		
	Core Particle	Coat Forming Substance		Whisker	Appara-tus		Pres-sure	Temper-ature	Time	Sinter appara-tus	Density	Hard-ness
	cBN	Name	Amount	SiC	Ex. No.	Ex.or Run. No.	GPa	°C	min		%	Hv
5-1	80	Zr	20	-	1	5-1	5.5	1450	30	CB	100	5500
5-2	80	Ti	20	-	2	5-2	5.3	1480	30	CB	100	5600
5-3	85	TiN	15	-	3	5-3	5.3	1480	30	CB	100	6400
5-4	80	TiN	20	-	3	5-3	5.3	1480	30	CB	100	5900
5-5	80	AlN	20	-	1	5-1	5.4	1450	30	CB	100	5800
5-6	70	AlN	30	-	1	5-5	5.4	1450	30	CB	100	5200
5-7	80	Si_3N_4	20	-	1	5-1	5.3	1470	30	CB	100	5900
5-8	70	Si_3N_4	30	-	1	5-7	5.3	1470	30	CB	100	5100
5-9	85	Al_2O_3	15	-	2	5-2	5.5	1450	30	CB	100	6100
5-10	80	Al_2O_3	20	-	2	5-9	5.6	1450	30	CB	100	5900
5-11	90	Co	10	-	1	5-1	5.5	1450	30	CB	100	6000
5-12	70	Co	30	-	1	5-1	5.5	1450	30	CB	100	2800
5-13	85	Ni_3Al	15	-	1	5-1	5.1	1450	30	CB	100	4300
5-14	80	NiAl	20	-	1	5-13	5.1	1450	30	CB	100	3300
5-15	72	TiN	18	10	3		5.5	1500	30	CB	100	5000
5-16	72	Si_3N_4	18	10	1		5.3	1470	30	CB	100	5100

CB: Cubic ultrahigh pressure apparatus

Example 6

Coated diamond particles were prepared by coating the bare diamond particles of Example 1 with various coat forming substances using apparatus of the same types as used in Examples 1 - 3. The names of the coat forming substances used to prepare the coated diamond particles and their coating weights (relative proportions by vol%) are shown in Table 3.

The apparatus used in Run 6-1 was of the same type as used in Example 1; an alumina powder having an average diameter of 2 μm was supplied as a feed of coat forming substance from the feeder 112 at a rate of 1.5 g/min and the core diamond particles were supplied from the feeder 111 at a rate of 0.3 g/min. The other conditions in Run 6-1 were the same as in Example 1. The apparatus used in Run 6-2 was of the same type as used in Example 2; an alumina powder having an average diameter of 2 μm was supplied as a feed of coat forming substance at a rate of 1.0 g/min and the diamond particles were supplied at a rate of 0.7 g/min. The other conditions in Run 6-2 were the same as in Example 2. The apparatus used in Run 6-3 was of the same type as in Example 3; the values of dispersity (β) attained in the discharge section of cyclone 6-I and in the coating start region were the same as in Run 4-2 and an alumina powder was supplied at a rate of 0.6 g/min whereas the diamond particles were supplied at a rate of 1.5 g/min. The other conditions in Run 6-3 were the same as in Example 3.

The apparatus used in Run 6-4 was of the same type as used in Example 1 and operated to perform the coating operation under generally the same conditions as in Run 6-1. In Runs 6-5 to 6-9, inorganic salts consisting of KCl, $BaCl_2$ and NaF were mixed with TiO_2 and ferroalloy (Fe-Ti) to prepare an immersing bath. In this bath, diamond powder particles (up to 2 μm in diameter) were dipped for 1 h by the molten salt disproportionation method with the bath being melted at 900°C. As a result, the surfaces of the diamond powder particles were coated with TiC in such a way that the amount of internal addition would be 5% by volume. The surfaces of diamond particles were further coated with the apparatus of Example 1 under generally the same conditions as in Run 6-1.

The coated diamond particles thus prepared were examined with a scanning electron microscope. All of the particles prepared in Runs 6-1 to 6-3 were uniformly covered with superfine particles (ca. 0.005 μm) of coat forming substances as Fig. 19 shows (particularly for the case of Run 6-1).

In Runs 6-10 and 6-11, the powders of alumina-coated diamond particles prepared in Runs 6-1 and 6-2, respectively, were wet mixed with SiC whiskers at a volume ratio of 90:10 in acetone for 2 h in an alumina ball mill. In Run 6-12, a powder of diamond particles was coated with 53% by volume of alumina using the apparatus of Example 1 under generally the same conditions as in Run 6-1 and then wet mixed with SiC whiskers at a volume ratio of 85:15 in acetone for 2 h in an alumina ball mill. The SiC whiskers had their surfaces coated with the target TiC by sputtering in a coating weight of 33% by volume in terms of internal addition. The mixtures was vacuum dried at 10^{-6} torr at 200°C to make dry blends.

The coated diamond particles thus prepared or mixtures thereof with SiC whiskers were stamped into disks having a diameter of 16 mm and a thickness of 5 mm in Runs 6-1 and 6-10 and into disks having a diameter of 8 mm and a thickness of 5 mm in Runs 6-2 and 6-11. The disks were placed into Pyrex glass capsules packed with a h-BN powder, degassed to 10^{-6} torr at 400°C over 12 h and then sealed. The sealed capsules were set in a HIP or a ultrahigh pressure HIP apparatus using an argon gas as a pressure-transmitting medium and sintering was performed under the conditions shown in Table 3. In Runs 6-9 to 6-12, the coated diamond particles or mixtures thereof with whiskers were stamped into pellets having a diameter of 10 mm and a thickness of 8 mm and the pellets were buried in a pyrophyllite pressure-transmitting medium surrounded by a h-BN shaped part; the assemblies were set in a piston-cylinder (PC) ultrahigh pressure apparatus and sintering was performed under the conditions shown in Table 3. Following furnace cooling, the pressure was opened to atmosphere and the sinters were recovered.

The recovered sinters were measured for their density and Vickers microhardness Hv (0.5/10) as in Example 1 and the results are shown in Table 3.

The sinters of Runs 6-1 to 6-4 were checked for their crystalline phases by powder X-ray diffraction and no diffraction peaks were observed except for the diamond and α-form alumina. Similar results were obtained in the other runs: only diffraction peaks for diamond, titanium carbide and α-form alumina were observed in Runs 6-5 to 6-7; only diffraction peaks for diamond, titanium carbide and the monoclinic and tetragonal zirconium oxide each composing the PSZ were observed in Runs 6-8 and 6-9; only diffraction peaks for diamond, α-form alumina and silicon carbide were observed in Runs 6-10 and 6-11; and only diffraction peaks for diamond, α-form alumina, silicon carbide and titanium carbide were observed in Run 6-12. No other diffraction peaks were observed.

The surface of the sinter produced in Run 6-1 was polished with a diamond paste; the polished surface was coated with a gold film by a customary evaporation technique and examined with an electron microscope. The result is shown in the Fig. 20 micrograph (X5000), in which the dark area represents the diamond and the light area the alumina bonding material.

For comparison, bare diamond particles of the same type as used in Example 6 were mixed with an alumina powder at a ratio of 70:30 in volume percent and the mixture was sintered under the same conditions as in Example 6. The resulting sinter had a density of 92% and a Vickers microhardness of 2200. The difference is clearly visible by comparing

Ex. 1003, Page 285

Fig. 20 with Fig. 21 which is an electron micrograph (X5000) of a polished surface of the comparative sinter that was coated with a gold film by a customary evaporation technique for examination. Obviously, the bonding material and the sintering aid were distributed irregularly in the comparative sinter to form lumps in certain areas. On the other hand, the number of areas that were devoid of the necessary sintering aid and bonding material was by no means small and unsintered portions were found there. Thus, the sinter produced in Run 6-1 in accordance with the invention was significantly superior to the comparative sinter in terms of density, hardness and microstructure.

Each of the sinters produced in Runs 6-5 to 6-7 presented considerable difficulty in forming a polished surface for the hardness measurement and this fact shows that those sinters had high wear resistance, which was probably due to the formation of the titanium carbide coating film by dip coating.

The foregoing data, when considered with particular reference to Figs. 20 and 21, demonstrate that the sinters produced in Example 6 were entirely free from unsintered portions, pores, defects, etc., highly dense, superhard and comprised of highly controlled microstructures in which the diamond particles were surrounded by uniform distributions of the bonding material(s), i.e., α-form alumina, the combination of titanium carbide and α-form alumina, or the combination of titanium carbide and PSZ, or in which these coated diamond particles were further surrounded with a uniform dispersion of SiC.

Ex. 1003, Page 286

Table 3

Run No.	Relative Proportions (vol%)						Sintering Conditions				Sinter	
	Coated Diamond Particles				Fibrous Substance							
	Diamond	Coat Forming Substance			Whisker	Coating Substance	Pressure	Temperature	Time	Apparatus	Density	Hardness
		TiC	Al_2O_3	PSZ	SiC	TiC	MPa	°C	hr.		%	Hv
6-1	30	-	70	-	-	-	150	1200	3	HIP1	96.0	2740
6-2	50	-	50	-	-	-	1000	1200	3	HIP2	95.0	3350
6-3	50	-	50	-	-	-	1500	1200	3	PC	95.3	3380
6-4	50	-	50	-	-	-	1500	1300	3	PC	95.5	3400
6-5	55	5	40	-	-	-	1500	1200	6	PC	94.8	3670
6-6	50	5	45	-	-	-	1500	1300	3	PC	95.0	3700
6-7	60	5	35	-	-	-	1500	1300	6	PC	94.6	3730
6-8	55	5	-	40	-	-	1500	1300	6	PC	99.3	2360
6-9	50	5	-	45	-	-	1500	1300	3	PC	99.6	2120
6-10	27	-	63	-	10	-	150	1200	3	HIP1	94.8	2660
6-11	45	-	45	-	10	-	1000	1200	3	HIP2	94.0	3220
6-12	40	-	45	-	10	5	1500	1300	3	PC	95.1	3170

HIP1: HIP apparatus incapable of producing ultrahigh pressures

HIP2: Superhigh pressure HIP apparatus

PC: Piston-cylinder (PC) ultrahigh pressure apparatus

Ex. 1003, Page 287

Example 7

Cubic boron nitride (cBN) particles of the same type as used in Example 2 were coated with various coat forming substances using apparatus of the same types as used in Examples 1 - 3. The names of the coat forming substances used to prepare the coated cubic boron nitride particles and their coating weights (relative proportions by vol%) are shown in Table 4.

The apparatus used in Runs 7-1 and 7-2 were of the same types as used in Examples 1 and 3, respectively; an alumina powder having an average diameter of 2 μm was supplied as a feed of coat forming substance at respective rates of 1.5 and 2.1 g/min and the cubic boron nitride core particles were supplied at respective rates of 0.3 and 1.5 g/min. The other conditions in Runs 7-1 and 7-2 were the same as in Example 1 and Run 4-2, respectively. The apparatus used in Runs 7-3 and 7-4 was of the same type as used in Example 1 and the other conditions were the same as in Run 7-1. In Runs 7-5, 7-6 and 7-8, the procedures of Runs 6-6, 6-7 and 6-9 were repeated, except that the surfaces of cubic boron nitride particles were dip coated with the combination of titanium nitride (TiN) and titanium diboride (TiB_2) in place of titanium carbide and, further, alumina or PSZ coating was performed using the same apparatus as in Example 1 under generally the same conditions as in Run 7-1. In Run 7-7, the apparatus of Example 1 was used and coating was performed under generally the same conditions as in Run 7-1, except that alumina was replaced by PSZ. In Runs 7-9 and 7-10, the alumina coated cubic boron nitride particles as prepared in Run 7-1 and Example 2, respectively, were each mixed with SiC whiskers as in Runs 6-10 and 6-11. In Run 7-11, cubic boron nitride particles were coated with 53% alumina using the apparatus of Example 1 under generally the same conditions as in Run 7-1 and then mixed with TiC coated SiC whiskers that were prepared as in Run 6-12.

The coated cubic boron nitride particles thus prepared were examined with a scanning electron microscope. All of the particles prepared in Runs 7-1 to 7-3 were uniformly covered with superfine particles (ca. 0.005 μm) of coat forming substances as Fig. 22 shows (particularly for the case of Run 7-1).

The powders of coated cubic boron nitride particles thus prepared in Runs 7-1 to 7-8 or the mixtures thereof with SiC whiskers which were prepared in Runs 7-9 to 7-11 were shaped by stamping as in Runs 6-1, 6-3 to 6-12, respectively, and sintered with the sinter apparatus shown in Table 4 under the sintering conditions shown in Table 4.

The sinters thus produced were measured for their density and Vickers microhardness Hv (0.5/10) as in Example 1 and the results are shown in Table 4.

The crystalline phases of the sinters were examined by powder X-ray diffraction; as in Runs 6-1 to 6-12, no diffraction peaks were observed except for the cubic boron nitride (core particles), the bonding materials or coat forming substances (α-alumina, titanium nitride, titanium diboride, as well as the PSZ composing monoclinic and tetragonal zirconium oxide), and whiskers (silicon carbide) and the titanium carbide coating thereon.

The polished surface of the sinter of Run 7-1 was coated with a gold film by a customary evaporation technique and examined with an electron microscope. The result is shown in the Fig. 23 electron micrograph (X5000), in which the dark area represents the cubic boron nitride and the light area the bonding material alumina. As one can see from Fig. 23, the sinter was entirely free from unsintered portions, pores, defects, etc. and it was comprised of a highly controlled microstructure in which the bonding material alumina was distributed uniformly around the cubic boron nitride particles.

For comparison, bare cubic boron nitride particles of the same type as used in Run 7-1 were mixed with an alumina powder at a ratio of 70:30 in volume percent and the mixture was sintered under the same conditions as in Run 7-1. The resulting sinter had a density of 94% and a Vickers microhardness of 2300. From an electron micrograph (X5000) of a polished surface of the comparative sinter that was coated with a gold film by a customary evaporation technique for examination. Obviously, the bonding material and the sintering aid were distributed irregularly in the comparative sinter to form lumps in certain areas. On the other hand, in the comparative sinter, the number of areas that were devoid of the necessary sintering aid and bonding material was by no means small and unsintered portions were found there.

In Runs 7-5, 7-6 and 7-9, the TiN-TiB_2 coating film formed by dip coating were found to be as effective as the TiC coating film formed by dip coating in Runs 6-5 to 6-7.

Thus, the sinters produced in Example 7 were entirely free from unsintered portions, pores, defects, etc., highly dense, superhard and comprised of highly controlled microstructures in which the cubic boron nitride particles were surrounded by uniform distributions of the bonding material(s), i.e., α-alumina, PSZ, the combination of TiN-TiB_2 and α-alumina, or the combination of TiN-TiB_2 and PSZ, or in which these coated cubic boron nitride particles were further surrounded with a uniform dispersion of SiC as whiskers.

Ex. 1003, Page 288

Table 4

Run No.	Relative Proportions (vol%)						Sintering Conditions				Sinter	
	Coated High-Pressure Boron Nitride Particles				Fibrous Substance							
		Coat Forming Substance			Whisker	Coating Substance	Pressure	Temperature	Time	Apparatus	Density	Hardness
	cBN	TiN-TiB_2	Al_2O_3	PSZ	SiC	TiC	MPa	°C	hr.		%	Hv
2	50	-	50	-	-	-	1000	1200	3	HIP2	95.8	3750
7-1	30	-	70	-	-	-	150	1200	3	HIP1	97.1	2960
7-2	50	-	50	-	-	-	1500	1200	3	PC	98.3	3930
7-3	50	-	50	-	-	-	1500	1300	3	PC	98.8	3970
7-4	60	-	40	-	-	-	1500	1200	6	PC	98.2	4700
7-5	50	5	45	-	-	-	1500	1300	3	PC	99.0	4030
7-6	60	5	35	-	-	-	1500	1300	6	PC	98.5	4780
7-7	60	-	-	40	-	-	1500	1300	6	PC	99.8	2300
7-8	50	5	-	45	-	-	1500	1300	3	PC	99.8	2100
7-9	27	-	63	-	10	-	150	1200	3	HIP1	96.0	2780
7-10	45	-	45	-	10	-	1000	1200	3	HIP2	95.0	3560
7-11	40	-	45	-	10	5	1500	1300	3	PC	98.7	3400

HIP1: HIP apparatus incapable of producing ultrahigh pressures

HIP2: Superhigh pressure HIP apparatus

PC: Piston-cylinder (PC) ultrahigh pressure apparatus

Example 8

Coated diamond particles were prepared by coating the bare diamond particles of Example 1 with various coat forming substances using apparatus of the same type as used in Examples 1 - 3. The names of the coat forming substances used to prepare the coated diamond particles and their coating weights (relative proportions by vol%) are shown in Table 5.

The apparatus used in Runs 8-1, 8-2 and 8-4 were of the same types as used in Examples 1, 2 and 3, respectively; a titanium carbide powder having an average diameter of 2 μm was supplied as a feed of coat forming substance at a rate of 0.4 g/min and the core diamond particles were supplied at a rate of 2.0 g/min; the other conditions in Runs 8-1, 8-2 and 8-4 were generally the same as in Example 1 and Runs 4-1 and 4-2, respectively. In each of those runs, 10% titanium carbide coated diamond particles were obtained. In Runs 8-5 and 8-6, an apparatus of the same type as used in Example 1 was operated under generally the same conditions as in Run 8-1 to produce 10% titanium carbide coated diamond particles; in Runs 8-7 and 8-8, the same procedure was repeated to produce 10% metallic titanium coated diamond particles; in Runs 8-9 and 8-10, the same procedure was repeated to yield 20% titanium carbide coated diamond particles; in Run 8-3, the 10% titanium carbide coated particles prepared in Run 8-2 were used.

The coated diamond particles thus prepared were examined with a scanning electron microscope. All of the particles prepared in Runs 8-1 to 8-4 were uniformly covered with superfine particles (ca. 0.005 μm) of coat forming substances as Fig. 25 shows (particularly for the case of Run 8-1).

The thus prepared powders of coated diamond particles and powders of bonding materials having the various formulations shown in Table 5 were added in the various proportions shown in Table 5 and wet mixed in acetone for 2 h in an alumina ball mill. Thereafter, the mixtures were vacuum dried at 10^{-6} torr and at 200°C to produce dry blends. The bonding material powder used in Runs 8-1 to 8-4 was easily sintrable alumina of high purity (i.e., the alumina powder described in the official gazette of Unexamined Published Japanese Patent Application (kokai) Sho 63-151616); in Runs 8-5 and 8-6, PSZ was used as a bonding material powder; and in Runs 8-7 to 8-10, TiC was used. Take, for example, Run 8-1; in this run, 55 vol% of 10% TiC coated diamond particles were mixed with 43.4 vol% of easily sintrable alumina of high purity as a bonding material powder, and with sintering aids for the alumina powder which were 0.6 vol% and 1.0 vol%, respectively, of magnesia (MgO) and titania (TiOx; x = 1 - 2).

In Runs 8-11 and 8-12, 90 vol% of the mixtures prepared in Runs 8-4 and 8-5, respectively, were mixed with 10 vol% of SiC whiskers and the mixtures thus prepared were used as sinter feeds.

The thus prepared mixtures of coated diamond particles with bonding material powders were stamped into disks having a diameter of 10 mm and a thickness of 8 mm in Runs 8-1, 8-7, 8-8 and 8-10. The disks were buried in a pyrophyllite pressure-transmitting medium surrounded by a h-BN shaped part and the assemblies were set in a piston-cylinder (PC) ultrahigh pressure apparatus, and sintering was performed under the conditions set forth in Table 5. In the other runs, the same mixtures of coated diamond particles with bonding material powders, or mixtures further containing SiC whiskers were stamped into disks having a diameter of 16 mm and a thickness of 5 mm. The disks were placed in a Pyrex glass capsule packed with a h-BN shaped part and the assemblies were degassed to 10^{-6} torr by heating at 400°C for 12 h and sealed. Thereafter, the capsules were set in a HIP or ultrahigh pressure HIP apparatus using an argon gas as a pressure-transmitting medium and sintering was performed under the conditions set forth in Table 5. Subsequently, the temperature was lowered and the pressure reduced for recovering the sinters.

The recovered sinters were measured for their density and Vickers microhardness Hv (0.5/10); the results are shown in Table 5 together with the sintering conditions and the types of sintering apparatus used.

The sinters were also checked for their crystalline phases by powder X-ray diffraction. In Example 8, no diffraction peaks were observed except for the diamond, coat forming substances (coating materials) and the bonding material powders or whisker SiC. Take, for example, Runs 8-1 to 8-4; in these cases, the only diffraction peaks observed were those for diamond, TiC and α-alumina. In the other runs, the only diffraction peaks observed were those for the following: diamond, TiC and PSZ composing monoclinic and tetragonal zirconium oxide (Runs 8-5 and 8-6); diamond, the TiC that was generated by the reaction between the coating metallic titanium and the diamond, as well as TiN (Runs 8-7 and 8-8); diamond and TiC (Run 8-9 and 8-10); diamond, TiC, α-alumina and SiC (Run 8-11); diamond, TiC, the PSZ composing monoclinic and tetragonal zirconium oxide, and SiC (Run 8-12).

The surface of the sinter produced in Run 8-1 was polished with a diamond paste and coated with a gold film by a customary evaporation technique and examined with an electron microscope. The result is shown in the Fig. 26 electron micrograph (X5000), in which the dark area represents the diamond and the light area the alumina-base bonding material.

As one can see from Fig. 26, all the sinters of Example 8 were entirely free from unsintered portions, pores, defects, etc., highly dense, superhard and comprised of highly controlled microstructures in which the diamond particles were surrounded by uniform distributions of the coating materials, the bonding materials (TiC, TiN, α-alumina and PSZ) and the SiC whiskers, if any.

Ex. 1003, Page 290

Table 5

Run No.	Relative Proportions (vol%)										Sintering Conditions				Sinter	
	Diamond	Coating Material			Bonding Material Powder					Whisker	Pressure	Temperature	Time	Apparatus	Density	Hardness
		TiC	TiN	Ti	PSZ	TiN	Al_2O_3	MgO	TiO_x	SiC	MPa	°C	hr.		%	Hv
8-1	50.0	5.0	-	-	-	-	43.4	0.6	1.0	-	1500	1300	3	PC	96.0	4110
8-2	30.0	3.0	-	-	-	-	65.4	0.6	1.0	-	150	1300	3	HIP1	98.8	3150
8-3	40.0	4.0	-	-	-	-	54.4	0.6	1.0	-	1000	1300	3	HIP2	98.8	3580
8-4	20.0	2.0	-	-	-	-	76.4	0.6	1.0	-	150	1300	3	HIP1	97.8	2930
8-5	30.0	3.0	-	-	67.0	-	-	-	-	-	150	1300	3	HIP1	98.5	1810
8-6	45.0	4.5	-	-	50.5	-	-	-	-	-	150	1300	3	HIP1	98.0	1990
8-7	20.0	-	-	2.0	-	78.0	-	-	-	-	1500	1300	3	PC	99.1	2860
8-8	40.0	-	-	4.0	-	56.0	-	-	-	-	1500	1300	3	PC	97.8	3430
8-9	20.0	-	4.0	-	-	76.0	-	-	-	-	1000	1300	3	HIP2	99.0	2840
8-10	40.0	-	8.0	-	-	52.0	-	-	-	-	1500	1300	3	PC	99.7	3400
8-11	18.0	1.8	-	-	-	-	68.8	0.5	0.9	10.0	150	1300	3	HIP1	99.4	2880
8-12	27.0	2.7	-	-	60.3	-	-	-	-	10.0	150	1300	3	HIP1	98.8	1880

HIP1: HIP apparatus incapable of producing ultrahigh pressures

HIP2: Superhigh pressure HIP apparatus

PC: Piston-cylinder (PC) ultrahigh pressure apparatus

Ex. 1003, Page 291

Example 9

Coated cubic boron nitride particles were prepared by coating the bare cubic boron nitride (cBN) particles of Example 1 with various coat forming substances using the apparatus employed in Examples 1 - 3. The names of the coat forming substances used to prepare those coated cubic boron nitride particles and their coating weights (relative proportions by vol%) are shown in Table 6.

The apparatus used in Runs 9-1, 9-2 and 9-3 were of the same types as used in Examples 1, 2 and 3, respectively; a titanium nitride powder having an average diameter of 2 μm was supplied as a feed of coat forming substance at a rate of 0.3 g/min and the cubic boron nitride core particles were supplied at a rate of 1.5 g/min; the other conditions in Runs 9-1, 9-2 and 9-3 were generally the same as in Example 1, Runs 4-2 and 4-2, respectively. In each of those runs, 10% titanium nitride coated cubic boron nitride particles were obtained. In Runs 9-4 and 9-5, an apparatus of the same type as used in Example 1 was operated under generally the same conditions as in Run 9-1 to produce 10% titanium nitride coated cubic boron nitride particles; in Runs 9-6 and 9-7, the same procedure was repeated to produce 10% metallic titanium coated cubic boron nitride particles; and in Runs 9-8 and 9-9, the same procedure was repeated to yield 20% titanium nitride coated cubic boron nitride particles.

The coated cubic boron nitride particles thus prepared were examined with a scanning electron microscope. All of the particles prepared in Runs 9-1 to 9-3 were uniformly covered with superfine particles (ca. 0.005 μm) of coat forming substances as Fig. 27 shows (particularly for the case of Run 9-1).

The thus prepared powders of coated cubic boron nitride particles and powders of bonding materials having the various formulations shown in Table 6 were added in the various proportions shown in Table 6. Those powders were wet mixed in acetone for 2 h in an alumina ball mill as in Example 8. Thereafter, the mixtures were vacuum dried at 10^{-6} torr and at 200°C to produce dry blends. The bonding material powders used in Example 9 were the same as those used in Example 8, i.e., easily sintrable alumina of high purity, PSZ, and TiN. As in Example 8, the use of easily sintrable high-purity alumina with an average particle diameter of 0.2 μm as a bonding material powder was accompanied by the use of sintering aids for the alumina powder which were magnesia (MgO) and titania (TiOx; x = 1 - 2).

In Runs 9-10 and 9-11, 90 vol% of the mixtures prepared in Runs 9-3 and 9-4, respectively, were mixed with 10 vol% of SiC whiskers and the mixtures thus prepared were used as sinter feeds.

The thus prepared mixtures of coated cubic boron nitride particles with bonding material powders were stamped into disks in Runs 9-1, 9-6, 9-7 and 9-9 as in Run 8-1; in the other runs, the same mixtures of coated cubic boron nitride particles with bonding material powders, or mixtures further containing SiC whiskers were stamped into disks as in Run 8-2. The disks were then sintered with the sinter apparatus listed in Table 6 under the sinter conditions also set forth in Table 6. Thereafter, the temperature was lowered and the pressure reduced for recovering the sinters.

The recovered sinters were measured for their density and Vickers microhardness Hv (0.5/10); the results are shown in Table 6 together with the sintering conditions and the types of sintering apparatus used.

The sinters were also checked for their crystalline phases by powder X-ray diffraction. In Example 9, no diffraction peaks were observed except for the cubic boron nitride, coat forming substances (coating materials) and the bonding materials or whisker SiC. Take, for example, Runs 9-1 to 9-3; in these cases, the only diffraction peaks observed were those for cubic boron nitride, TiN and α-alumina. In the other runs, the only diffraction peaks observed were those for the following: cubic boron nitride, TiN and the PSZ composing monoclinic and tetragonal zirconium oxide (Runs 9-4 and 9-5); cubic boron nitride, the TiB_2 that was generated by the reaction between the coating metallic titanium and cubic boron nitride, as well as TiN (Runs 9-6 and 9-7); cubic boron nitride and TiN (Runs 9-8 and 9-9); cubic boron nitride, TiC, α-alumina and SiC (Run 9-10); and cubic boron nitride, TiC, the PSZ composing monoclinic and tetragonal zirconium oxide, and SiC (Run 9-11).

Quantification by X-ray analysis showed that the sinter of Run 9-6 consisted of cubic boron nitride, TiB_2 and TiN in proportions of 19.5%, 1.0% and 79.5%, respectively, by volume whereas the sinter of Run 9-7 had relative volume proportions of 39.1%, 2.0% and 58.9%.

The surface of the sinter produced in Run 9-1 was polished with a diamond paste and coated with a gold film by a customary evaporation technique and examined with an electron microscope. The result is shown in the Fig. 28 electron micrograph (X5000), in which the dark area represents the cubic boron nitride and the light area the alumina base bonding material.

As one can see from Fig. 28, all the sinters of Example 9 were entirely free from unsintered portions, pores, defects, etc., highly dense, superhard and comprised of highly controlled microstructures in which the cubic boron nitride particles were surrounded by uniform distributions of the coating materials, the bonding materials (TiN, TiB_2, α-alumina and PSZ) and the SiC whiskers, if any.

Ex. 1003, Page 292

Table 6

Run No.	Relative Proportions (vol %)									Sintering Conditions				Sinter	
		Coating Material		Bonding Material Powder					Whisker	Pressure	Temperature	Time	Apparatus	Density	Hardness
	cBN	TiN	Ti	PSZ	TiN	Al_2O_3	MgO	TiO_x	SiC	MPa	°C	hr.		%	Hv
9-1	50.0	5.0	-	-	-	43.4	0.6	1.0	-	1500	1300	3	PC	97.5	4020
9-2	40.0	4.0	-	-	-	54.4	0.6	1.0	-	1000	1300	3	HIP2	98.7	3500
9-3	20.0	2.0	-	-	-	76.4	0.6	1.0	-	150	1300	3	HIP1	98.3	2870
9-4	30.0	3.0	-	67.0	-	-	-	-	-	150	1300	3	HIP1	99.8	1830
9-5	45.0	4.5	-	50.5	-	-	-	-	-	150	1300	3	HIP1	99.6	2010
9-6	20.0	-	2.0	-	78.0	-	-	-	-	1500	1300	3	PC	99.0	2890
9-7	40.0	-	4.0	-	56.0	-	-	-	-	1500	1300	3	PC	98.0	3430
9-8	20.0	4.0	-	-	76.0	-	-	-	-	1000	1300	3	HIP2	99.0	2900
9-9	40.0	8.0	-	-	52.0	-	-	-	-	1500	1300	3	PC	99.4	3510
9-10	18.0	1.8	-	-	-	68.8	0.5	0.9	10.0	150	1300	3	HIP1	99.8	2790
9-11	27.0	2.7	-	60.3	-	-	-	-	10.0	150	1300	3	HIP1	99.7	1840

HIP1: HIP apparatus incapable of producing ultrahigh pressures

HIP2: Superhigh pressure HIP apparatus

PC: Piston-cylinder (PC) ultrahigh pressure apparatus

Ex. 1003, Page 293

Examples 10 and 11

Using the apparatus employed in Examples 1 - 3, pseudo-fine diamond particles (Example 10) or pseudo-fine particles in a high-pressure phase boron nitride powder (Example 11) were covered with various coat forming materials. The pseudo-fine diamond particles were the same as those used in Example 3; the pseudo-fine particles in a high-pressure phase boron powder were pseudo-fine cubic boron nitride (cBN) particles that had an average diameter (D_M) of 17 μm and a frequency distribution by volume of ($[D_M/2, 3D_M/2]$, ≧ 90%). The names of the coat forming substances used to prepare the pseudo-fine coated diamond and cubic boron nitride particles and their coating weights (relative proportions by vol%), as well as the specific types of the coating apparatus used and the conditions for their use (generally the same) are shown in Tables 7 and 8 together with the data for Example 3.

The apparatus used in Runs 10-1 and 11-1 was of the same type as used in Example 1; the group of means for high dispersion treatment of particles in this apparatus was to treat the pseudo-fine particles of either a diamond powder or a cubic boron nitride powder, in which the core particles had an average diameter of 17 μm and a frequency distribution by volume of ($[D_M/5, 5D_M]$, ≧ 90%) and, hence, said group of means was designed in such a way that it could realize an output dispersity (β) value of at least 80% (β ≧ 80%) for the core particles in those powders while, at the same time, the coating operation could be started with a dispersity (β) of at least 80% (β ≧ 80%) in the coating start region. A titanium carbide powder (Run 10-1) or a titanium nitride powder (Run 11-1) that were feeds for coat forming substances and which had an average particle diameter of 2 μm were supplied from the feeder 112 at a rate of 0.3 g/min whereas the pseudo-fine diamond or cubic boron nitride core particles were supplied from the feeder 111 at a rate of 3.0 g/min and introduced into the coating chamber after being dispersed to a dispersity (β) of 89%, and the dispersity (β) in the coating start region 3-L1 was 89%. The other conditions in Examples 10 and 11 were the same as in Example 1. The coating weights of titanium carbide and titanium nitride were each 5% by volume.

The apparatus used in Runs 10-2 and 11-2 was of the same type as used in Example 2; the dispersity (β) value attainable in these runs was the same as in Runs 10-1 and 11-1; a metallic titanium powder having an average particle diameter of 25 μm was supplied as a feed for coat forming substance from the feeder 215 at a rate of 0.5 g/min, and the pseudo-fine diamond or cubic boron nitride core particles were supplied from the feeder 214 at a rate of 2.5 g/min; the other conditions in Runs 10-2 and 11-2 were the same as in Example 2. The apparatus used in Run 11-3 was of the same type as used in Example 3; a zirconium powder having an average particle diameter of 25 μm was supplied as a feed for coat forming substance from the feeder 314 at a rate of 0.5 g/min and the pseudo-fine cubic boron nitride core particles were supplied from the feeder 313 at a rate of 2.0 g/min; the other conditions in Run 11-3 were the same as in Example 3. In Runs 10-2, 11-2 and 11-3, the coating weights of metallic titanium and zirconium were each 10% by volume.

The apparatus used in Runs 10-3, 10-4, 11-4 and 11-5 was of the same type as used in Example 1 and operated under generally the same conditions as in Example 1 to prepare the following in the respective runs: pseudo-fine diamond particles first coated with 5 vol% TiC, then with 50 vol% alumina; pseudo-fine diamond particles coated with 10 vol% TiC; pseudo-fine cubic boron nitride particles coated with 50 vol% alumina; and pseudo-fine cubic boron nitride particles coated with 10 vol% TiN.

The coated pseudo-fine diamond and cubic boron nitride particles were examined with a scanning electron microscope. All of the particles prepared in Examples 10 and 11 were uniformly covered with superfine particles (ca. 0.005 μm) of coat forming substances (TiC, TiN, Ti and Zr) as in Example 3 and as Fig. 29 (for the particular case of Run 10-1) and Fig. 30 (for the particular case of Run 11-1) show.

The thus prepared powders of pseudo-fine coated diamond or cubic boron nitride particles, or mixtures thereof with the bonding materials listed in Table 7 or 8 were shaped, set in the various types of sinter apparatus shown in Tables 7 and 8, and sintered under the various sintering conditions also set forth in Tables 7 and 8. Thereafter, the temperature was lowered and the pressure reduced for recovering the sinters. The specific procedures were as follows.

In Runs 10-1 and 10-2, 60% by volume of the powders of pseudo-fine diamond particles coated with TiC and Ti, respectively, were wet mixed in acetone with 40% by volume of bonding materials that were the powders of fine coated diamond particles as prepared by coating fine diamond particles of 0.5 - 2 μm in diameter (average diameter = 1 μm) with 15 vol% of TiC under generally the same conditions. In Runs 11-1, 11-2 and 11-3, 60% by volume of the powders of pseudo-fine cubic boron nitride particles coated with TiN, Ti and Zr, respectively, were wet mixed in acetone with 40% by volume of bonding materials that were the powders of fine coated cubic boron nitride particles as prepared by coating fine cubic boron nitride particles of 0.5 - 2 μm in diameter (average diameter = 1 μm) with 15 vol% of TiC under generally the same conditions. Thereafter, the resulting wet mixtures were shaped, vacuum dried and sintered with a cubic ultrahigh pressure apparatus as in Example 3.

In Runs 10-3 and 11-4, the powder of TiC and alumina coated pseudo-fine diamond particles and the powder of alumina coated fine cubic boron nit ride particles, respectively, were stamped into disks having a diameter of 8 mm and a thickness of 5 mm; the disks were placed in Pyrex glass capsules packed with a h-BN powder. After degassing at 10^{-6} torr by heating at 400°C for 12 h, the capsules were sealed, set in a HIP apparatus using an argon gas as a pressure-transmitting medium and sintered under the conditions set forth in Tables 7 and 8.

Ex. 1003, Page 294

In Runs 10-4 and 11-5, the powder of TiC coated pseudo-fine diamond particles and the powder of TiN coated fine cubic boron nitride particles were respectively wet mixed in 40% by volume as in Run 8-2 with the associated bonding materials that were 53.4%, 0.6% and 1.0% by volume of the high-purity and easily sintrable alumina, magnesia (MgO) and titania (TiOx; x = 1 - 2) which were used in Run 8-1. The mixtures were vacuum dried, shaped into disks, placed into capsules, degassed, sealed, set in a HIP apparatus and sintered under the conditions set forth in Tables 7 and 8.

The sinters thus produced were measured for their density and Vickers microhardness Hv (0.5/10). The results are shown in Tables 7 and 8 together with the sintering conditions and the types of sinter apparatus used.

As for the Vickers microhardness Hv (0.5/10) measurement of the sinters of the coated psudo-fine particles, the hardness values obtained by the measurement were the values of the psudo-fine particles and not the values of the sinters, since particles sizes of the psudo-fine particles were larger than those of the fine particles and the hardness values of the sinters could not be measured. Therefore, in Tables 7 and 8 the Vickers microhardness values of the sinters were not shown.

The sinters were also checked for their crystalline phases by powder X-ray diffraction. In Examples 10 and 11, no diffraction peaks were observed except for the core particles (diamond or cubic boron nitride), coating materials (TiC, TiN and α-alumina), the products of reaction between the core particles and the metallic coating materials (TiC, ZrC, TiN, TiB_2, ZrN and ZrB_2) and the bonding materials (the diamond or cubic boron nitride in the core particles, TiC or TiN and α-alumina as the coating materials). Take, for example, Runs 10-1 and 10-2; in these cases, the only diffraction peaks observed were those for diamond and TiC. In the other runs, the only diffraction peaks observed were those for the following: diamond, TiC and α-alumina (Runs 10-3 and 10-4); cubic boron nitride and TiN (Run 11-1); cubic boron nitride, TiN and TiB_2 (Run 11-2); cubic boron nitride, TiN, ZrN and ZrB_2 (Run 11-3); cubic boron nitride and α-alumina (Run 11-4); and cubic boron nitride, TiN and α-alumina (Run 11-5). Quantification by X-ray analysis showed that the sinter of Run 10-2 consisted of 87% diamond and 13% TiC; the sinter of Run 11-2 consisted of cubic boron nitride, TiN and TiB_2 in proportions of about 87%, 10% and 3%, respectively, by volume; and the sinter of Run 11-3 consisted of cubic boron nitride, ZrN, ZrB_2 and TiN in proportions of about 86%, 2%, 1% and 11%, respectively, by volume.

The polished surface of the sinter of Run 10-1 was coated with a gold film by a customary evaporation technique and examined with an electron microscope. The result is shown in the Fig. 31 electron micrograph (X5000), in which the dark area represents the diamond and the light area TiC. As one can see from Fig. 31, the sinter was entirely free from pores and could be sintered to a relative density of 99%. In addition, it had no portion left unsintered. In areas where pseudo-fine diamond particles and/or fine diamond particles contacted each other via a thin layer of the coat forming substance, the latter was bursted by the contiguous pseudo-fine diamond particles and/or fine diamond particles, which hence were coupled directly as a result of sintering. In all other areas, TiC was distributed uniformly around the pseudo-fine diamond particles, rendering the sinter to be characteristic in that the coated pseudo-fine diamond particles had a dense, uniform and extremely highly controlled distribution. The diamond particles in the sinter were also characterized in that they did not grow at all compared to the particles in the feed powder. It has herefore been impossible to produce sinters having such an extremely highly controlled microstructure. Fig. 32 is an electron micrograph (X5000) showing a polished surface of the sinter of Run 11-1 and this was taken by the same method as used to prepare Fig. 31. Again, this sinter had established therein a microstructure in which pseudo-fine cubic boron nitride particles and/or fine cubic boron nitride particles and TiN as the coat forming substance were distributed uniformly, densely and in an extremely highly controlled manner.

Thus, in spite of the fact that diamonds and cubic boron nitrides are inherently very difficult to sinter, the coated pseudo-fine diamond and high-pressure phase boron nitride particles prepared in accordance with the invention behaved as if they were comparatively easy-to-sinter particles under industrially feasible ultrahigh pressures and elevated temperatures, thereby forming dense, rigid and superhard structures.

The sinters thus produced in Examples 10 and 11 were similar to that produced in Example 3 in that they were sinters or composite sinters that were superhard and which had dense, uniform and highly controlled microstructures with substantially uniform distributions of the bonding materials.

Ex. 1003, Page 295

Table 7

Ex. or Run No.	Relative Proportions (vol %)										Sintering Conditions				Sinter
	Core Particle	Coating Material					Bounding Material Powder				Pressure	Temperature	Time	Sinter Apparatus	Density
	Diamond	TiC	Ti	Zr	Al_2O_3		Core Particle: Diamond	Coating material: TiC	Al_2O_3	MgO/TiOx	GPa	°C	min		%
3	27	-	-	3	-		59.5	10.5	-	-	5.5	1450	30	CB	≥99
10-1	57	3	-	-	-		34	6	-	-	5.5	1450	30	CB	≥99
10-2	54	-	6	-	-		34	6	-	-	5.5	1450	30	CB	≥99
10-3	45	5	-	-	50		-	-	-	-	0.15	1200	180	HIP1	≥99
10-4	36	4	-	-	-		-	-	53.4	0.6/1.0	0.15	1300	180	HIP1	≥99

Table 8

Run. No.	Relative Proportions (vol %)												Sintering Conditions			Sinter
	Core Particle	Coating Material						Bounding Material Powder					Pressure	Temperature	Time	Sinter Apparatus	Density
	CBN	TiN	Ti	Zr	Al_2O_3			Core Particle	Coating material				GPa	°C	min		%
								CBN	TiN	Al_2O_3	MgO/TiOx						
11-1	57	3	-	-	-			34	6	-	-		5.5	1450	30	CB	≥99
11-2	54	-	6	-	-			34	6	-	-		5.5	1450	30	CB	≥99
11-3	27	-	-	3	-			59.5	10.5	-	-		5.5	1450	30	CB	≥99
11-4	50	-	-	-	50			-	-	-	-		0.15	1200	180	HIP1	≥99
11-5	36	4	-	-	-			-	-	53.4	0.6/1.0		0.15	1300	180	HIP1	≥99

Claims

1. A process for producing a diamond or high-pressure phase boron nitride sinter comprising the step of preparing coated diamond or high-pressure phase boron nitride particles and sintering said coated diamond or high-pressure phase boron nitride particles or mixtures containing such particles or mixtures of coated diamond or high-pressure phase boron nitride particles with bonding material,

sintering said coated diamond or high-pressure phase boron nitride particles or said mixtures containing such particles at pressures no less than 2000 MPa and at elevated temperatures; or

sintering said coated diamond or high-pressure phase boron nitride particles or said mixtures containing such particles at pressures of less than 2000 MPa and at temperatures not exceeding 1850°C under such sintering pressure and temperature conditions that diamond or high-pressure phase boron nitride is thermodynamically metastable if not stable; or

sintering said mixtures of coated diamond or high-pressure phase boron nitride particles with a bonding material in a volume ratio of (1 - 90):(99 - 10), said bonding material being sintrable to a density of at least 85% at pressures less than 2000 MPa and at temperatures not exceeding 1850°C under such conditions that diamond and/or high-pressure phase boron nitride particles are thermodynamically metastable, at pressures less than 2000 MPa and at temperatures not exceeding 1850°C under such sintering pressure and temperature conditions that diamond and/or high-pressure phase boron nitride is thermodynamically metastable if not stable.

wherein said coated diamond or high-pressure phase boron nitride particles are prepared by a method comprising the steps of charging a powder of diamond or high-pressure phase boron nitride core particles into a coating space and then permitting a precursor of a coat forming substance which is generated via the vapor phase and/or a precursor of a coat forming substance in a vapor-phase state to contact and/or impinge against the particles in said powder of core particles so that their surfaces are coated with the coat forming substance and

said coated diamond or high-pressure phase boron nitride particles are prepared by coating means comprising:

(A) a dispersing step in which the particles in the powder of core particles are dispersed by a group of means for high dispersion treatment of particles in a gaseous atmosphere to form a mixture of a gas and the particles in a powder of highly dispersed particles, the final treating means in said group of means for high dispersion treatment of particles being selected from between:

(a) dispersing means for dispersing the particles in the powder of core particles in a gaseous atmosphere; and

(b) means for selecting a mixture of a gas and the particles in a powder of highly dispersed particles which comprises:

(b-1) means for selecting a mixture of a gas and the particles in a powder of highly dispersed particles that separates a portion of the powder that is composed of less highly dispersed particles from the mixture of a gas and the particles in the powder of core particles in which the particles in the powder of core particles are dispersed in a gaseous atmosphere, thereby selecting a mixture of a gas and the particles in a powder of highly dispersed core particles in which the particles in the powder of core particles are present in the gaseous atmosphere mainly in a single-particle state; and

(b-2) feedback means by which the portion of the powder that is composed of less highly dispersed particles and which has been separated by said means for selecting a mixture of a gas and the particles in a powder of highly dispersed particles is transported to the final dispersing means among the dispersing means in the group of means for high dispersion treatment of particles and/or treating means upstream of the final dispersing means; and

(B) a coating step in which the particles in the powder of core particles that have been dispersed in the dispersing step (A) start to be coated by being allowed to contact and/or impinge against the precursor of the coat forming substance in the coating start region of the coating space as they are dispersed to satisfy the following conditions:

the dispersity β should be at least 70% if those particles have an average diameter of no more than 10 μm in the frequency distribution by volume;

the dispersity β should be at least 80% if those particles have an average diameter in excess of 10 μm but no more than 20 μm in the frequency distribution by volume;

the dispersity β should be at least 90% if those particles have an average diameter in excess of 20 μm but no more than 50 μm in the frequency distribution by volume;

the dispersity β should be at least 95% if those particles have an average diameter in excess of 50 μm

Ex. 1003, Page 298

but no more than 300 μm in the frequency distribution by volume;

the dispersity β should be at least 97% if those particles have an average diameter in excess of 300 μm but no more than 800 μm in the frequency distribution by volume; and

the dispersity β should be at least 99% if those particles have an average diameter in excess of 800 μm in the frequency distribution by volume, and wherein said sintering step is performed by

sintering said coated diamond or high-pressure phase boron nitride particles or said mixtures containing such particles at pressures no less than 2000 MPa and at elevated temperatures; or

sintering said coated diamond or high-pressure phase boron nitride particles or said mixtures containing such particles at pressures of less than 2000 MPa and at temperatures not exceeding 1850°C under such sintering pressure and temperature conditions that diamond or high-pressure phase boron nitride is thermodynamically metastable if not stable; or

sintering said mixtures of coated diamond or high-pressure phase boron nitride particles with a bonding material in a volume ratio of (1 - 90):(99 - 10), said bonding material being sintrable to a density of at least 85% at pressures less than 2000 MPa and at temperatures not exceeding 1850°C under such conditions that diamond and/or high-pressure phase boron nitride particles are thermodynamically metastable, at pressures less than 2000 MPa and at temperatures not exceeding 1850°C under such sintering pressure and temperature conditions that diamond and/or high-pressure phase boron nitride is thermodynamically metastable if not stable.

2. A process for producing a diamond or high-pressure phase boron nitride sinter according to claim 1 wherein said coated diamond or high-pressure phase boron nitride particles are prepared through the following additional step(s):

a step of disagglomeration or otherwise size reduction in which lumps of the coated diamond and/or high-pressure phase boron nitride particles that form as a result of association of several particles that contact one another via the coat forming substance on the coated particles are disagglomerated and/or otherwise reduced in size; and/or

a step of selective separation in which the associating lumps of the coated diamond or high-pressure phase boron nitride particles are selectively separated from the coated diamond or high-pressure phase boron nitride particles on a primary particle basis.

3. A process for producing a diamond or high-pressure phase boron nitride sinter according to claim 1 wherein the particles in the powder of diamond and/or high-pressure phase boron nitride core particles to be coated with the coat forming substance are coated by a dip method using a molten salt bath with one or more layers of the coat forming substance that derives from the dip method.

4. A process for producing a diamond or high-pressure phase boron nitride sinter according to claim 1, wherein the dispersing step is performed by:

a group of means for high dispersion treatment of particles that has such dispersing performance that a powder of core particles having an average diameter of no more than 10 μm in the frequency distribution by volume is dispersed in a gaseous atmosphere by the final treatment by said group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and that the dispersity β of the particles in said powder of core particles is adjusted to at least 70%;

a group of means for high dispersion treatment of particles that has such dispersing performance that a powder of core particles having an average diameter in excess of 10 μm but no more than 20 μm in the frequency distribution by volume is dispersed in a gaseous atmosphere by the final treatment by said group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and that the dispersity β of the particles in said powder of core particles is adjusted to at least 80%;

a group of means for high dispersion treatment of particles that has such dispersing performance that a powder of core particles having an average diameter in excess of 20 μm but no more than 50 μm in the frequency distribution by volume is dispersed in a gaseous atmosphere by the final treatment by the group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and that the dispersity β of the particles in said powder of core particles is adjusted to at least 90%;

a group of means for high dispersion treatment of particles that has such dispersing performance that a powder of core particles having an average diameter in excess of 50 μm but no more than 300 μm in the frequency distribution by volume is dispersed in a gaseous atmosphere by the final treatment by the group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and that the dispersity β of the particles in said powder of core particles is adjusted to at least 95%;

a group of means for high dispersion treatment of particles that has such dispersing performance that a

Ex. 1003, Page 299

powder of core particles having an average diameter in excess of 300 μm but no more than 800 μm in the frequency distribution by volume is dispersed in a gaseous atmosphere by the final treatment by the group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and that the dispersity β of the particles in said powder of core particles is adjusted to at least 97%; and

a group of means for high dispersion treatment of particles that has such dispersing performance that a powder of core particles having an average diameter in excess of 800 μm in the frequency distribution by volume is dispersed in a gaseous atmosphere by the final treatment by the group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and that the dispersity β of the particles in said powder of core particles is adjusted to at least 99%; and

said coated diamond or high-pressure phase boron nitride particles being prepared by:

a method in which the mixture of a gas and the particles in the powder of highly dispersed core particles as formed by one of the means in said group of means for high dispersion treatment of particles is either discharged directly to the coating step or transported thereto via at least one member indispensable to transport that is selected from the group consisting of a hollow member, an intermediate member composed of a member that forms a hollow portion, and a pipe and which is provided between the dispersing and coating steps, as the mixture emerges from a discharge section that discharges said mixture of a gas and the particles; and/or

a method in which the mixture of a gas and the particles in the powder of highly dispersed core particles as formed by one of the means in said group of means for high dispersion treatment of particles is transported to the coating step via at least one means selected from the group consisting of means for maintaining the dispersion in a gaseous atmosphere of the highly dispersed core particles in the mixture of a gas with the core particles which have been dispersed in the gaseous atmosphere to attain one of the dispersities set forth above, means for enhancing the dispersion in a gaseous atmosphere of the highly dispersed core particles in the mixture of a gas with the core particles which have been dispersed in the gaseous atmosphere to attain one of the dispersities set forth above, and means for separating a mixture of a gas and the particles in a powder of less highly dispersed core particles from said gas-particle mixture so as to select a mixture of a gas and the particles in a powder of highly dispersed core particles in which the core particles are present in the gaseous atmosphere mainly in a single-particle state.

5. A process for producing a diamond or high-pressure phase boron nitride sinter according to claim 1 wherein the dispersing step is performed by:

a group of means for high dispersion treatment of particles that has such dispersing performance that a powder of core particles having an average diameter of no more than 10 μm in the frequency distribution by volume is dispersed in a gaseous atmosphere by the final treatment by said group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and that the dispersity β of the particles in said powder of core particles is adjusted to at least 70%;

a group of means for high dispersion treatment of particles that has such dispersing performance that a powder of core particles having an average diameter in excess of 10 μm but no more than 20 μm in the frequency distribution by volume is dispersed in a gaseous atmosphere by the final treatment by said group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and that the dispersity β of the particles in said powder of core particles is adjusted to at least 80%;

a group of means for high dispersion treatment of particles that has such dispersing performance that a powder of core particles having an average diameter in excess of 20 μm but no more than 50 μm in the frequency distribution by volume is dispersed in a gaseous atmosphere by the final treatment by the group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and that the dispersity β of the particles in said powder of core particles is adjusted to at least 90%;

a group of means for high dispersion treatment of particles that has such dispersing performance that a powder of core particles having an average diameter in excess of 50 μm but no more than 300 μm in the frequency distribution by volume is dispersed in a gaseous atmosphere by the final treatment by the group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and that the dispersity β of the particles in said powder of core particles is adjusted to at least 95%;

a group of means for high dispersion treatment of particles that has such dispersing performance that a powder of core particles having an average diameter in excess of 300 μm but no more than 800 μm in the frequency distribution by volume is dispersed in a gaseous atmosphere by the final treatment by the group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and that the dispersity β of the particles in said powder of core particles is adjusted to at least 97%; and

Ex. 1003, Page 300

a group of means for high dispersion treatment of particles that has such dispersing performance that a powder of core particles having an average diameter in excess of 800 μm in the frequency distribution by volume is dispersed in a gaseous atmosphere by the final treatment by the group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and that the dispersity β of the particles in said powder of core particles is adjusted to at least 99%; and

said coated diamond or high-pressure phase boron nitride particles being prepared by performing at least one part of the dispersing step in such a way that it shares one or more parts of the space with at least one part of the coating step.

6. A process for producing a diamond or high-pressure phase boron nitride sinter according to claim 1 wherein said coated diamond or high-pressure phase boron nitride particles are prepared with the coating start region of the coating space being located in either one of the following space regions that includes planes through which all particles in the powder of highly dispersed core particles as mixed with a gas will pass:

a space region in which a powder of core particles that have an average diameter of no more than 10 μm in the frequency distribution by volume is dispersed in a gaseous atmosphere by the final treatment by said group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and that the dispersity β of the particles in said powder of core particles is adjusted to at least 70%;

a space region in which a powder of core particles that have an average diameter in excess of 10 μm but no more than 20 μm in the frequency distribution by volume is dispersed in a gaseous atmosphere by the final treatment by said group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and that the dispersity β of the particles in said powder of core particles is adjusted to at least 80%;

a space region in which a powder of core particles that have an average diameter in excess of 20 μm but no more than 50 μm in the frequency distribution by volume is dispersed in a gaseous atmosphere by the final treatment by the group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and that the dispersity β of the particles in said powder of core particles is adjusted to at least 90%;

a space region in which a powder of core particles that have an average diameter in excess of 50 μm but no more than 300 μm in the frequency distribution by volume is dispersed in a gaseous atmosphere by the final treatment by the group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and that the dispersity β of the particles in said powder of core particles is adjusted to at least 95%;

a space region in which a powder of core particles that have an average diameter in excess of 300 μm but no more than 800 μm in the frequency distribution by volume is dispersed in a gaseous atmosphere by the final treatment by the group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and that the dispersity β of the particles in said powder of core particles is adjusted to at least 97%; and

a space region in which a powder of core particles that have an average diameter in excess of 800 μm in the frequency distribution by volume is dispersed in a gaseous atmosphere by the final treatment by the group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and that the dispersity β of the particles in said powder of core particles is adjusted to at least 99%, or wherein

said coated diamond or high-pressure phase boron nitride particles are prepared with the coating start region of the coating space being located in either one of the following space regions that includes planes through which all particles to be recovered into the recovery section of the recovery means will pass:

a space region in which a powder of core particles that have an average diameter of no more than 10 μm in the frequency distribution by volume is dispersed in a gaseous atmosphere by the final treatment by said group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and that the dispersity β of the particles in said powder of core particles is adjusted to at least 70%;

a space region in which a powder of core particles that have an average diameter in excess of 10 μm but no more than 20 μm in the frequency distribution by volume is dispersed in a gaseous atmosphere by the final treatment by said group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and that the dispersity β of the particles in said powder of core particles is adjusted to at least 80%;

a space region in which a powder of core particles that have an average diameter in excess of 20 μm but no more than 50 μm in the frequency distribution by volume is dispersed in a gaseous atmosphere by the final treatment by the group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles

Ex. 1003, Page 301

in the powder of highly dispersed core particles and that the dispersity β of the particles in said powder of core particles is adjusted to at least 90%;

a space region in which a powder of core particles that have an average diameter in excess of 50 μm but no more than 300 μm in the frequency distribution by volume is dispersed in a gaseous atmosphere by the final treatment by the group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and that the dispersity β of the particles in said powder of core particles is adjusted to at least 95%;

a space region in which a powder of core particles that have an average diameter in excess of 300 μm but no more than 800 μm in the frequency distribution by volume is dispersed in a gaseous atmosphere by the final treatment by the group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and that the dispersity β of the particles in said powder of core particles is adjusted to at least 97%; and

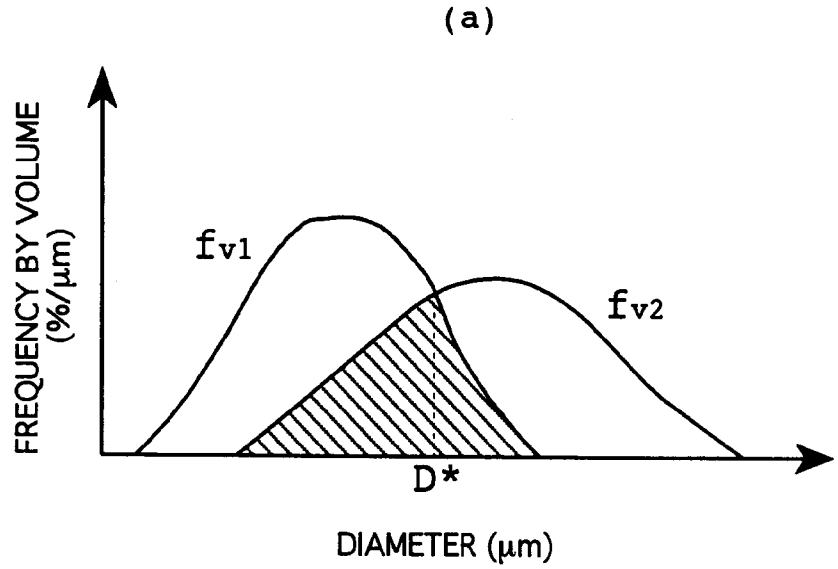
a space region in which a powder of core particles that have an average diameter in excess of 800 μm in the frequency distribution by volume is dispersed in a gaseous atmosphere by the final treatment by the group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and that the dispersity β of the particles in said powder of core particles is adjusted to at least 99%.

7. A process for producing a diamond or high-pressure phase boron nitride sinter according to claim 1 wherein the particles in the powder of core particles to be used have a size distribution of ($[D_M/5, 5D_M]$, $\geqq$ 90%) in terms of frequency distribution by volume, where D_M represents the average particle diameter.

8. A diamond or high-pressure phase boron nitride sinter that is produced by either one of the processes for producing a diamond or high-pressure phase boron nitride sinter as recited in claims 1 - 7.

Ex. 1003, Page 302

FIG.1


(a)
FREQUENCY BY VOLUME (%/μm)
fv1
fv2
D*
DIAMETER (μm)

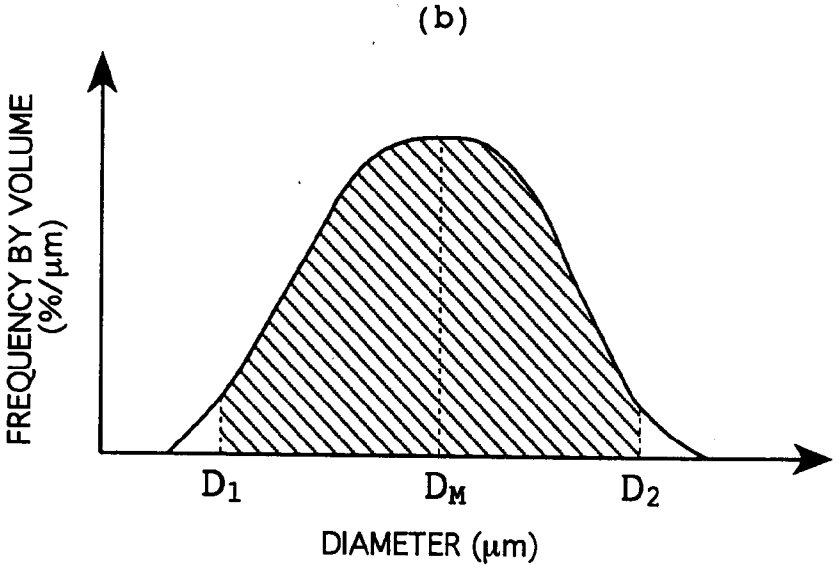
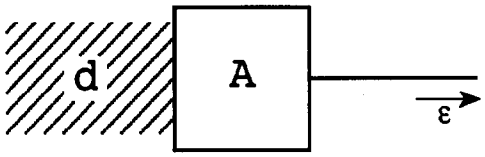
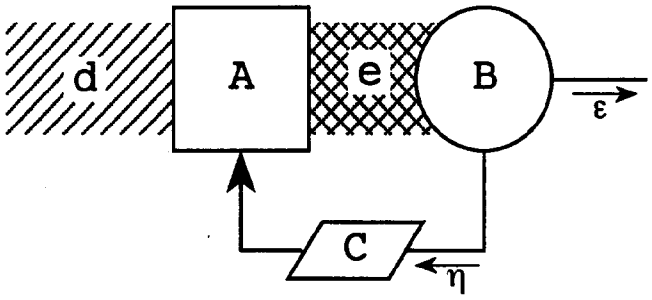

(b)
FREQUENCY BY VOLUME (%/μm)
D1
DM
D2
DIAMETER (μm)

FIG.2

(a)



(b)



(c)

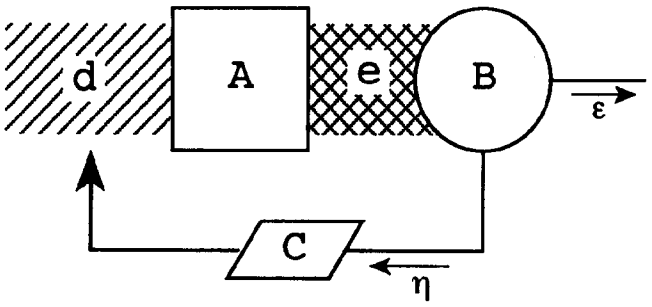


FIG.3

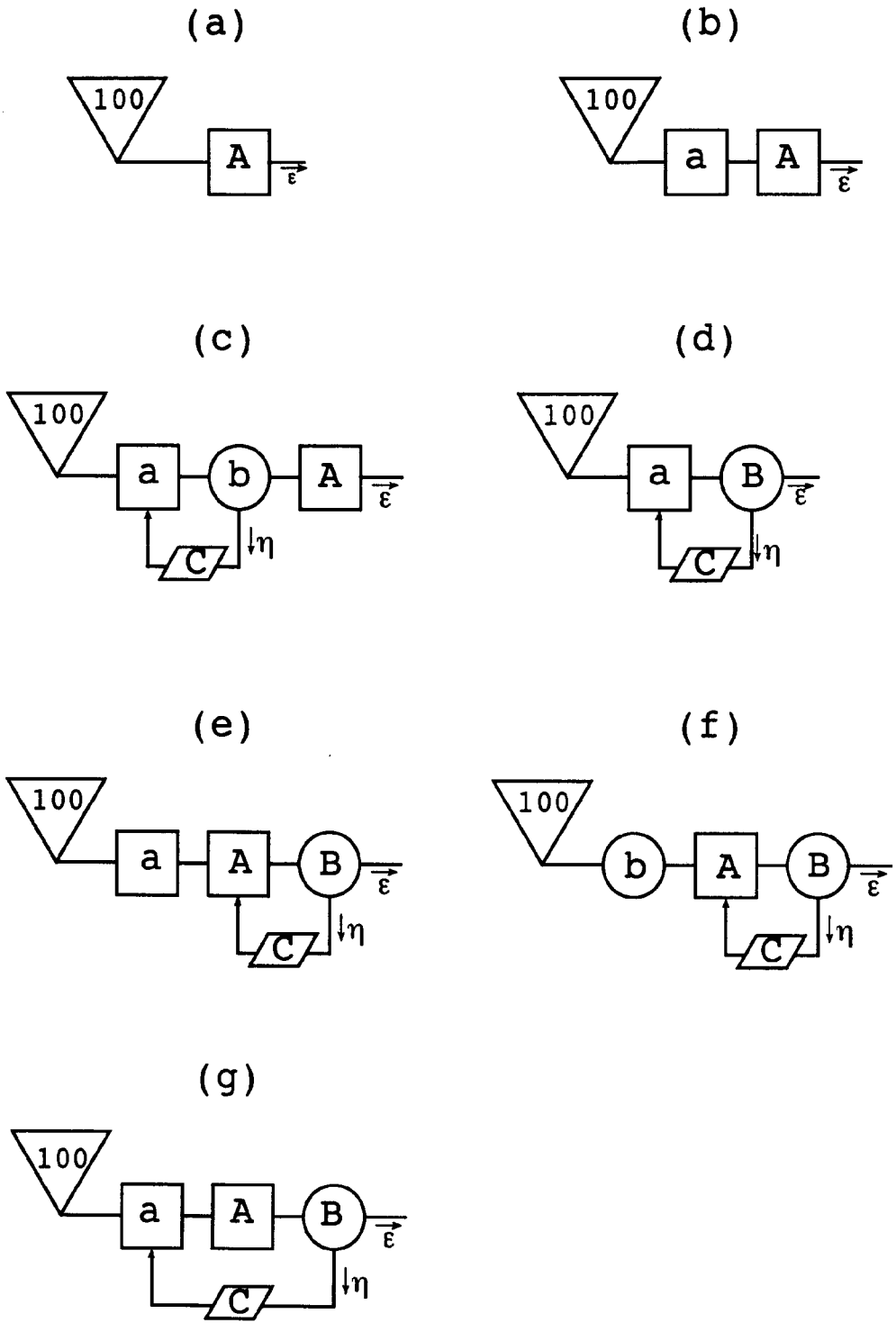

(a)
100
A
ε
(b)
100
a
A
ε
(c)
100
a
b
A
ε
C
η
(d)
100
a
B
ε
C
η
(e)
100
a
A
B
ε
C
η
(f)
100
b
A
B
ε
C
η
(g)
100
a
A
B
ε
C
η

FIG.4

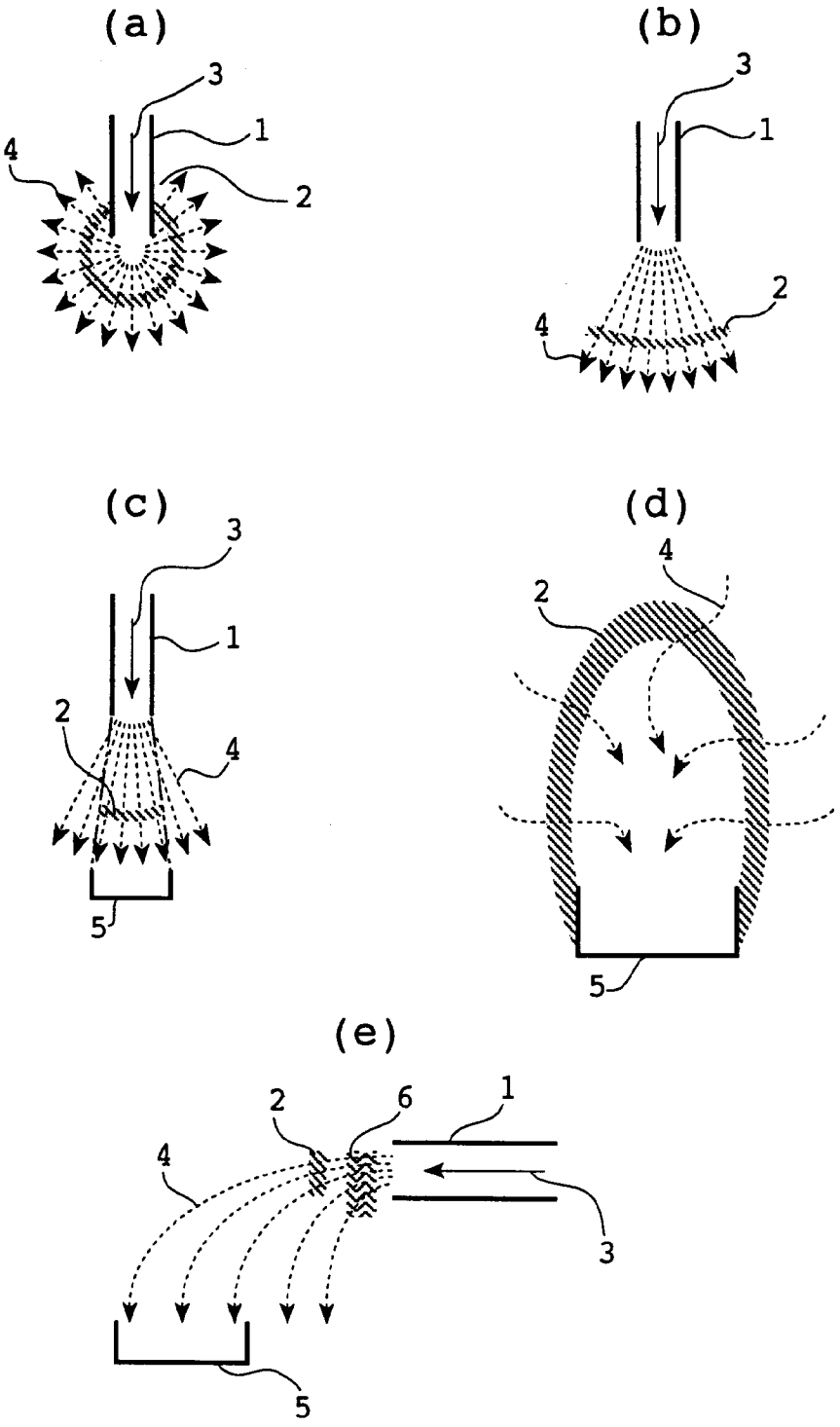
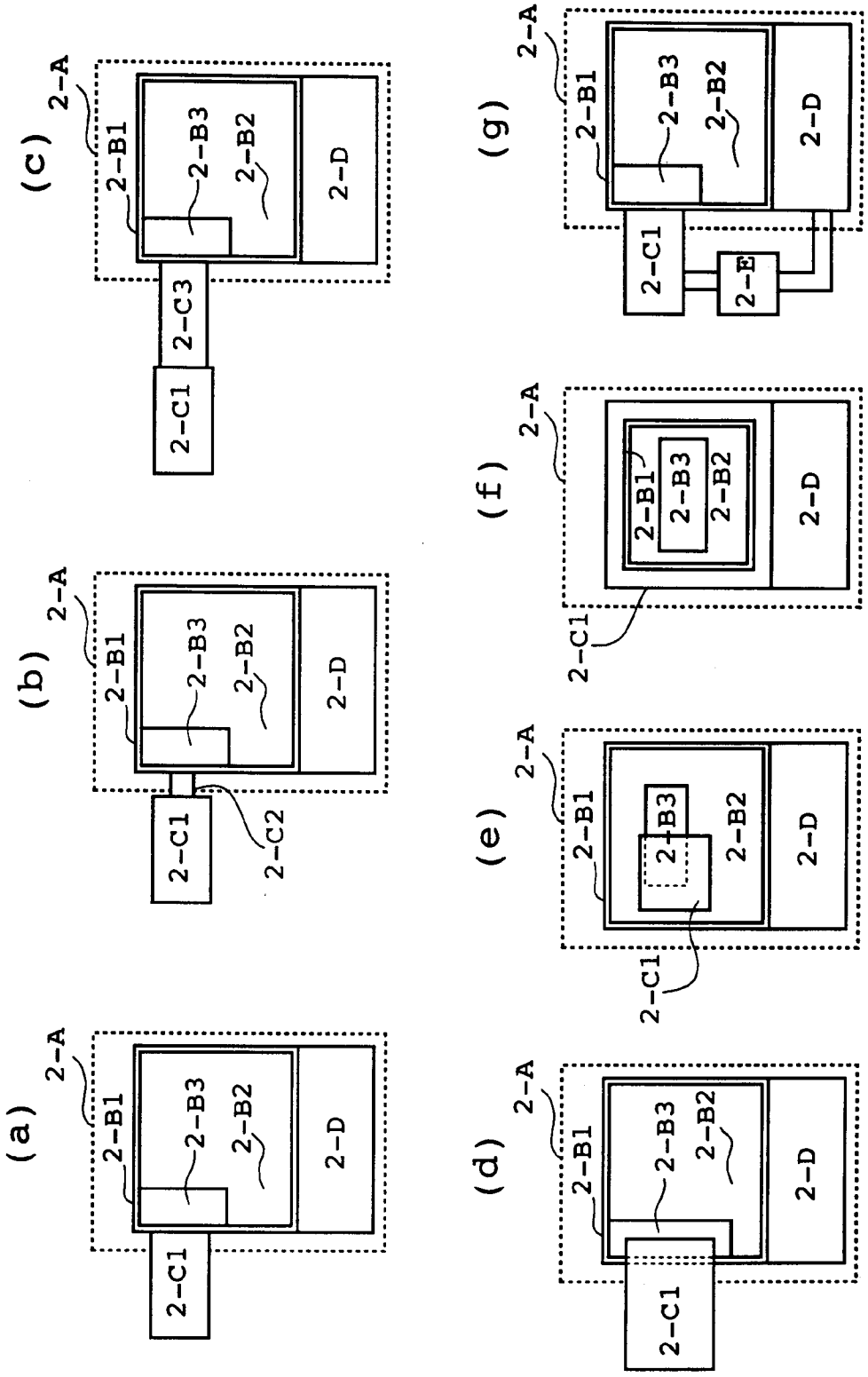


FIG. 5



Ex. 1003, Page 307

FIG.6

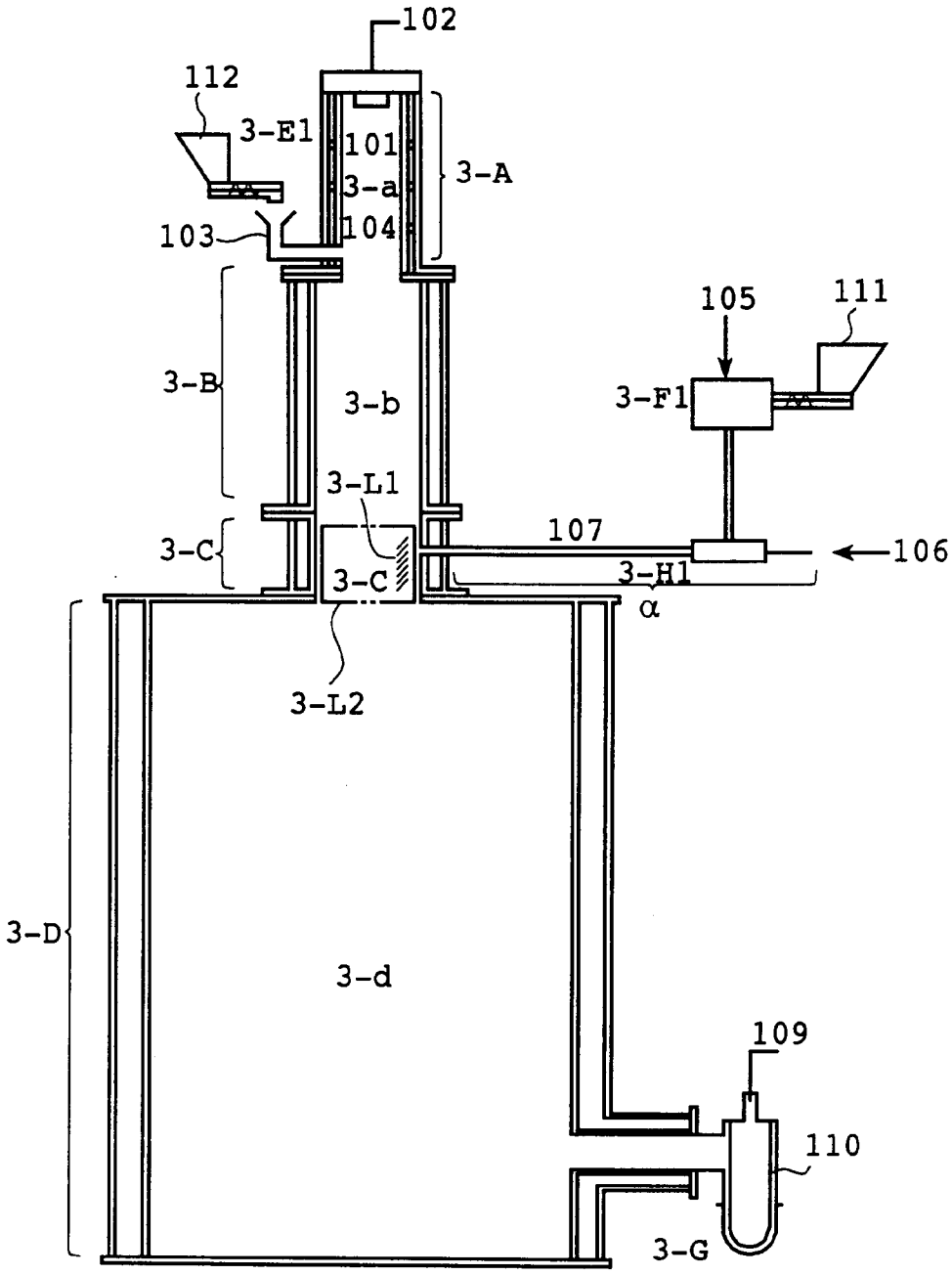

102
112
3-E1
101
3-a
104
3-A
103
105
111
3-B
3-b
3-F1
3-L1
107
106
3-C
3-C
3-H1
α
3-L2
3-D
3-d
109
110
3-G

FIG.7

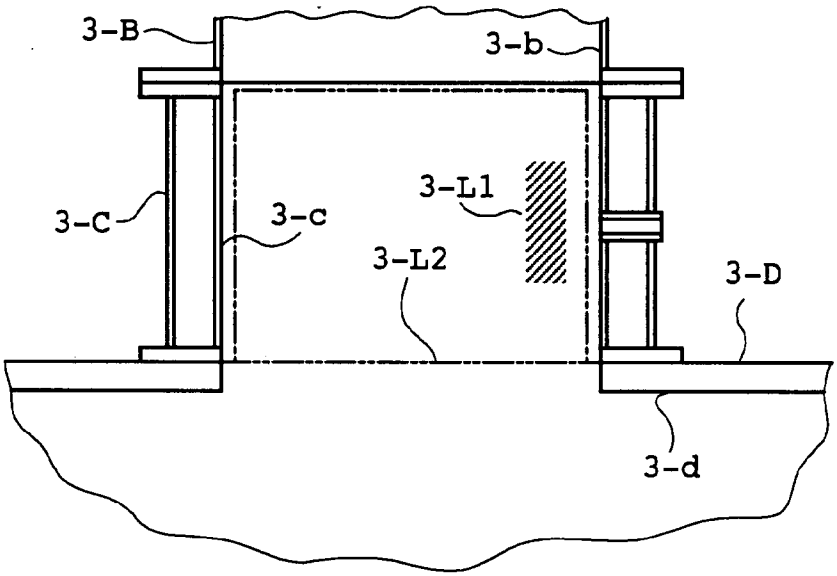


FIG.8



FIG.9

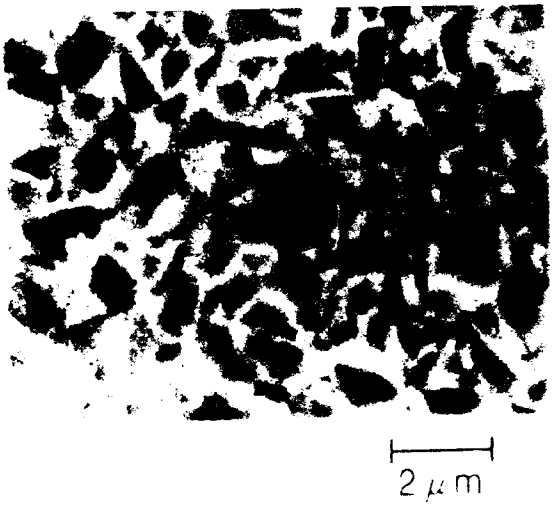


FIG.10

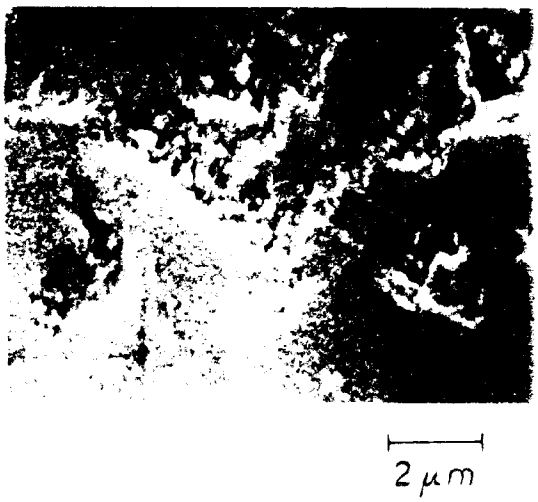


FIG.11

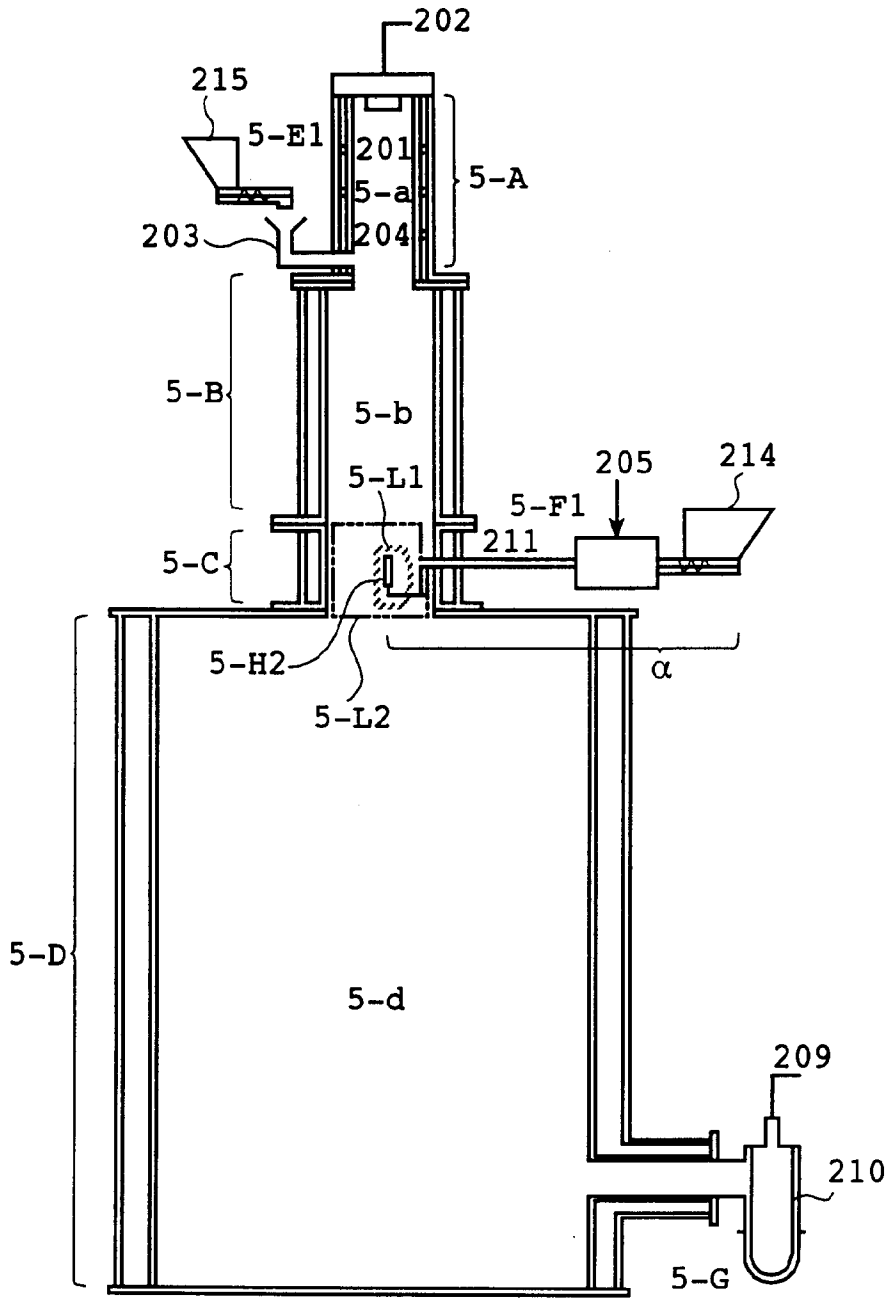

202
215
5-E1
201
5-a
5-A
203
204
5-B
5-b
205
214
5-L1
5-F1
211
5-C
5-H2
α
5-L2
5-D
5-d
209
210
5-G

FIG.12

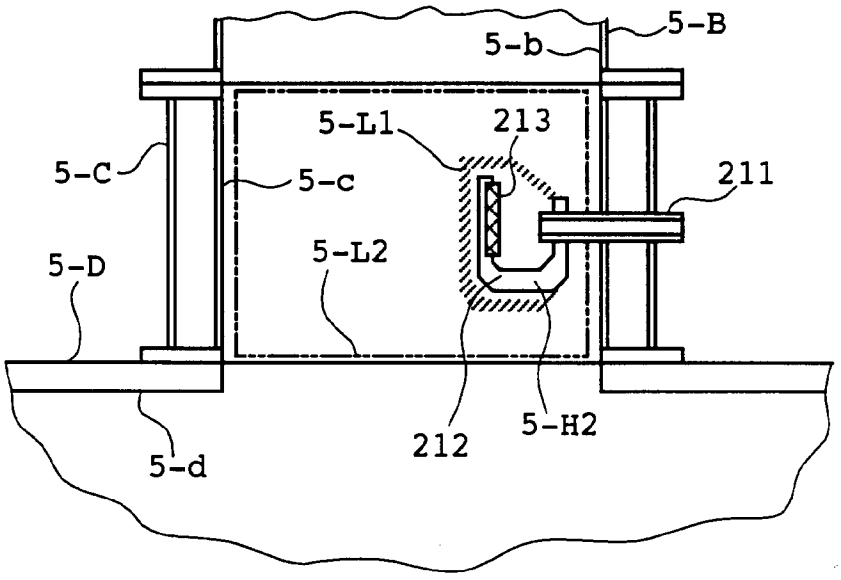


FIG.13

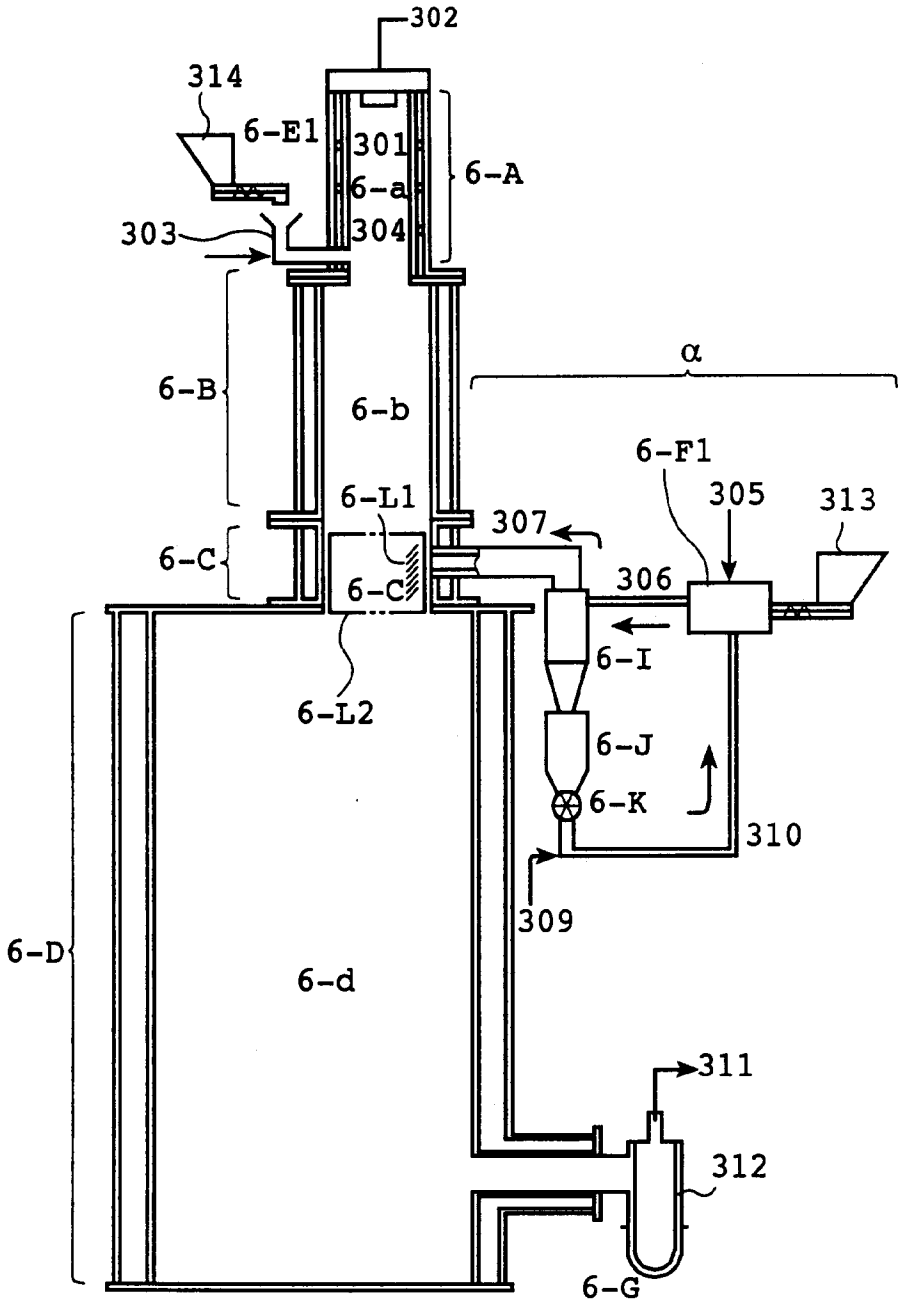

302
314
6-E1
301
6-A
6-a
303
304
α
6-B
6-b
6-F1
305
313
6-L1
307
6-C
6-C
306
6-I
6-L2
6-J
6-K
310
309
6-D
6-d
311
312
6-G

FIG.14

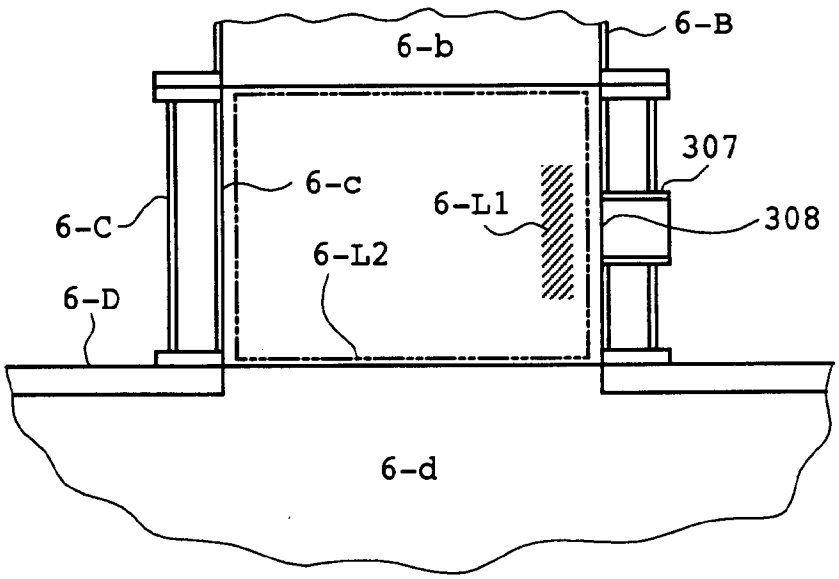


FIG.15



FIG.16

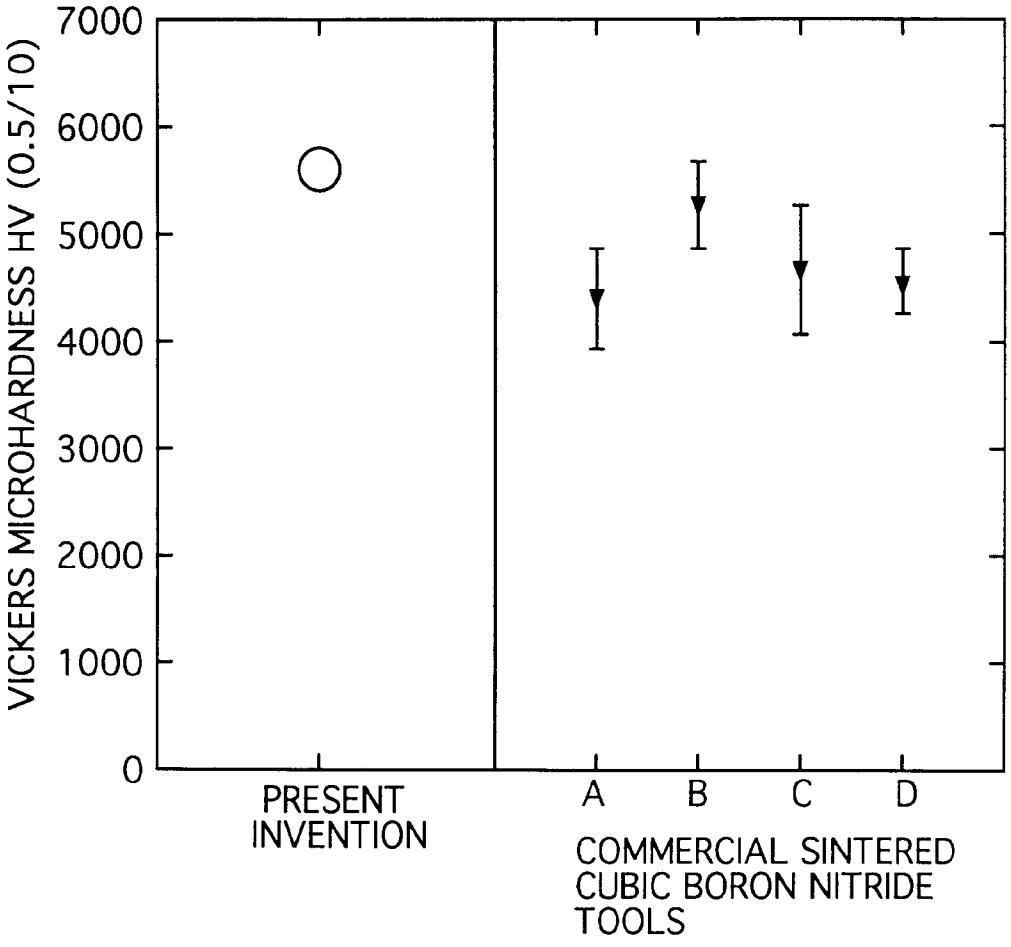

VICKERS MICROHARDNESS HV (0.5/10)
7000
6000
5000
4000
3000
2000
1000
0
PRESENT INVENTION
A
B
C
D
COMMERCIAL SINTERED CUBIC BORON NITRIDE TOOLS

FIG.17



FIG.18

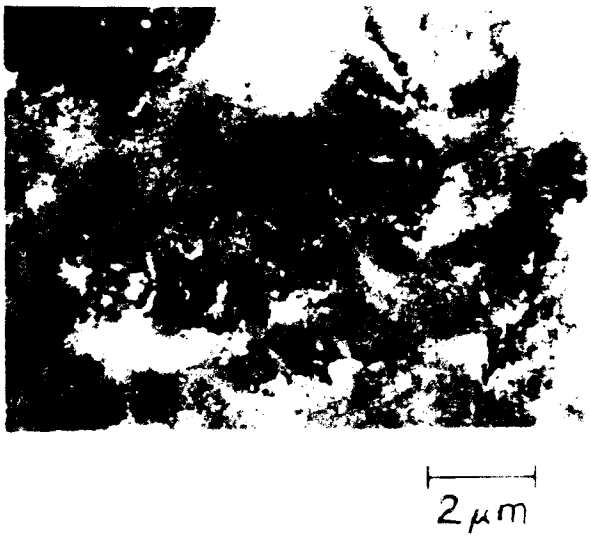


FIG.19



FIG.20

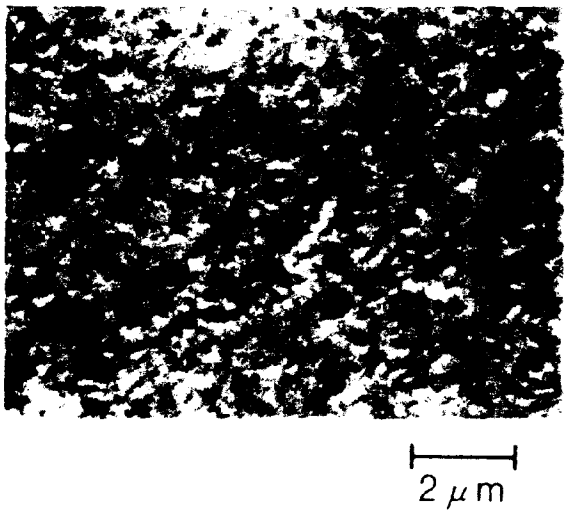


FIG.21

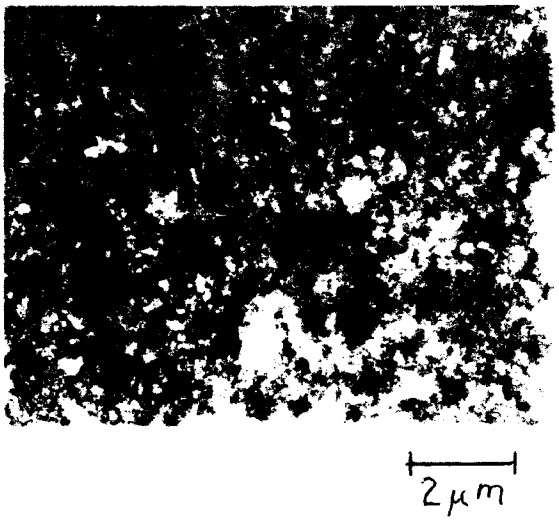


FIG.22



FIG.23

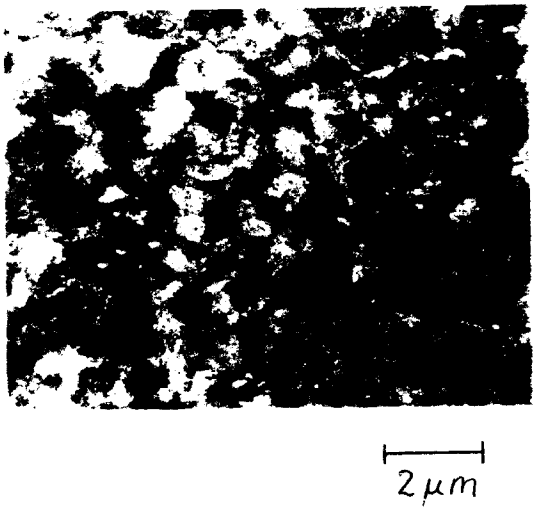


FIG.24

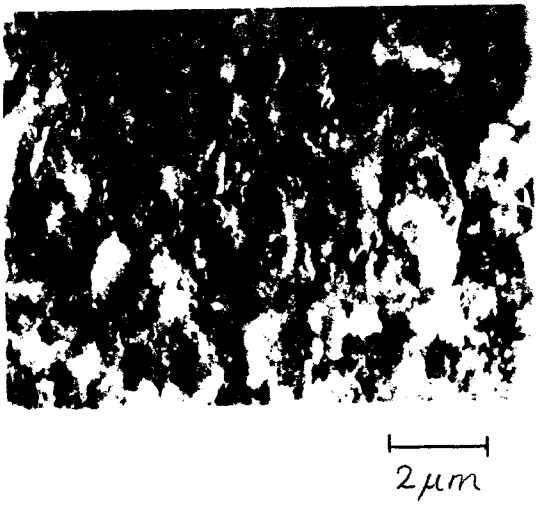


FIG.25

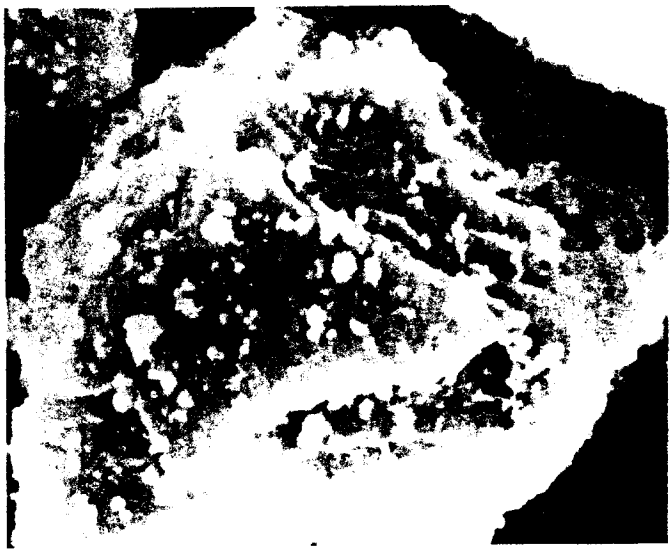


FIG.26

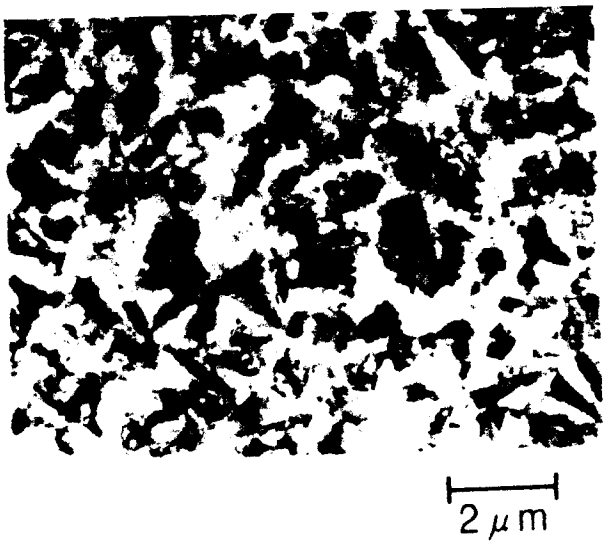


FIG.27



FIG.28

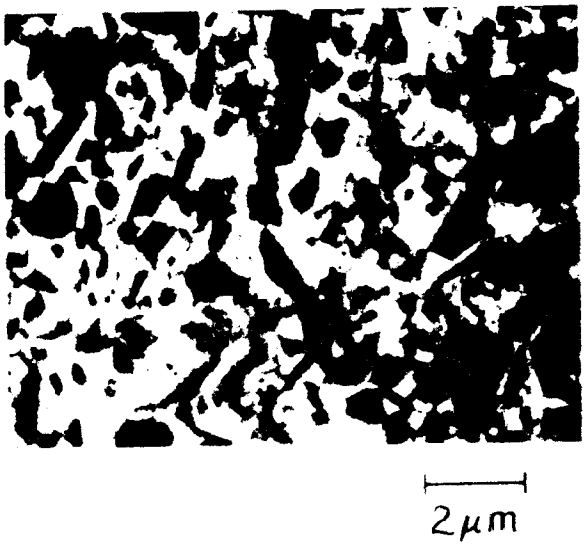


FIG.29



FIG.30

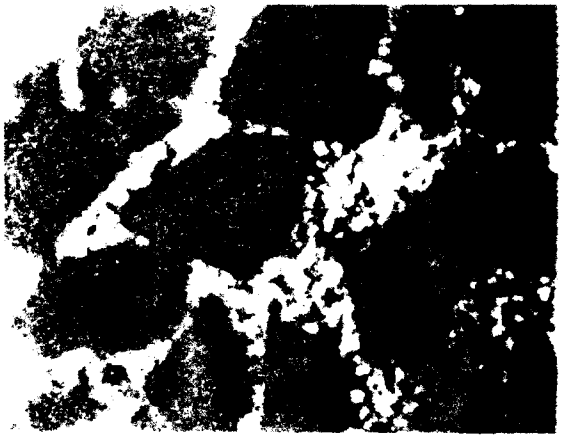


FIG.31



FIG.32

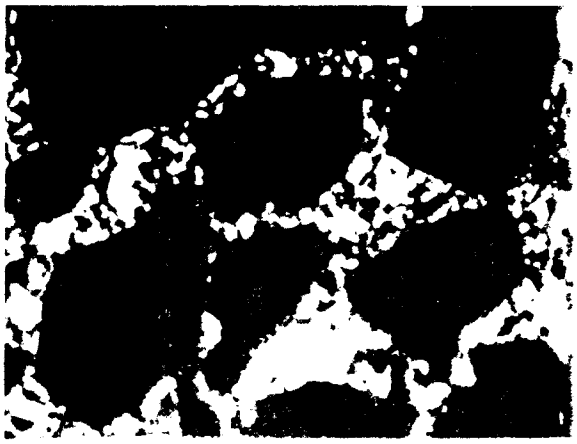


FIG.33a

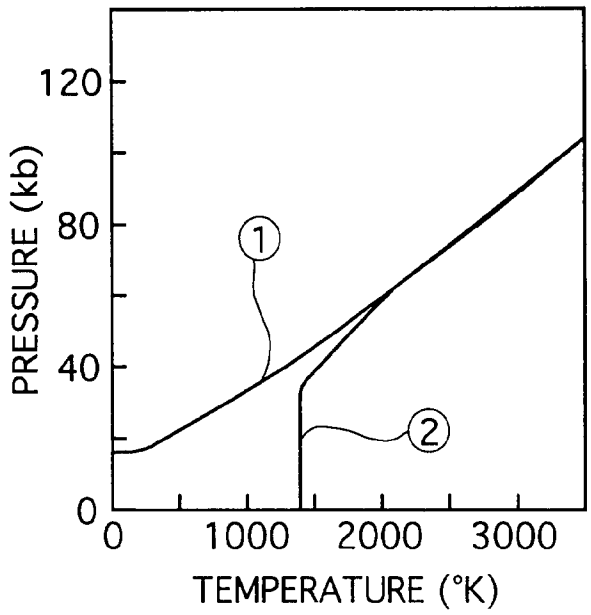
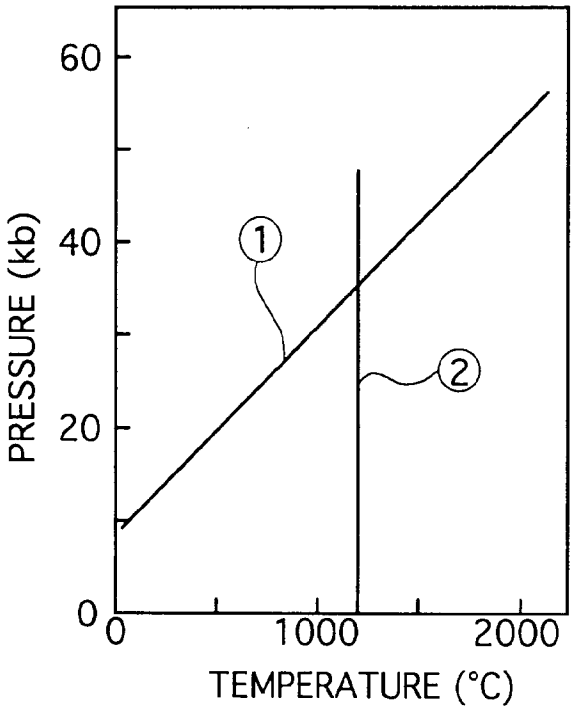


FIG.33b



European Patent Office

EUROPEAN SEARCH REPORT

Application Number
EP 94 11 8222

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int.Cl.6)
X	EP-A-0 273 573 (GA TECHNOLOGIES INC) 6 July 1988 * page 3, line 3 - line 29 *	1,2,4-8	C23C14/22
X	GB-A-2 214 195 (INCO LTD) 31 August 1989 * page 4, line 21 - page 5, line 31 *	1,2,4-8	
A,D	GB-A-2 242 443 (NISSHIN FLOUR MILLING CO) 2 October 1991 * example 4 *	1-8	
A,D	PATENT ABSTRACTS OF JAPAN vol. 014 no. 202 (C-0713) ,25 April 1990 & JP-A-02 043377 (SEIJI MOROOKA;OTHERS: 01) 13 February 1990, * abstract *	1-8	

TECHNICAL FIELDS SEARCHED (Int.Cl.6)

C23C

The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
THE HAGUE	26 April 1995	Schruers, H

CATEGORY OF CITED DOCUMENTS

X : particularly relevant if taken alone
Y : particularly relevant if combined with another document of the same category
A : technological background
O : non-written disclosure
P : intermediate document

T : theory or principle underlying the invention
E : earlier patent document, but published on, or after the filing date
D : document cited in the application
L : document cited for other reasons

& : member of the same patent family, corresponding document

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(54) Single-crystal diamond of very high thermal conductivity

(57) Single-crystal diamond consisting of isotopically pure carbon-12 or carbon-13 has been found to have a thermal conductivity higher than that of any substance previously known, typically at least 40% higher than that of naturally occurring IIA diamond. It may be prepared by a method comprising comminution of diamond of high isotopic purity, such as that obtained by low pressure chemical vapor deposition employing an isotopically pure hydrocarbon in combination with hydrogen, followed by conversion of the comminuted diamond to single-crystal diamond by diffusion under high pressure through a metallic catalyst-solvent material to a region containing a diamond seed crystal.

GB 2 239 011 A

SINGLE-CRYSTAL DIAMOND OF VERY HIGH THERMAL CONDUCTIVITY

This invention relates to the preparation of single crystal diamond of extremely high thermal conductivity.

Diamond substances with high thermal conductivity are known in the art. For example, type IIA natural diamond, characterized by a very high degree of purity, has a thermal conductivity at 25°C (298°K) on the order of 21 watts/cm.°K, the highest of any material known prior to the present invention. Its electrical conductivity, on the other hand, is so low as to be negligible.

These properties make diamond an excellent material for conducting heat away from heat-generating objects or units. It may be used as a heat sink or as a conductor/spreader to conduct heat from the heat-generating object or unit to a heat sink of some other material.

Various areas exist in which heat conductors of very high thermal conductivity are necessary. One example is in repeating stations for fiber optic networks. Signals are transmitted by laser light over the fibers of such networks for very great distances. Since these signals decrease substantially in intensity over several kilometers, it is necessary to construct "repeating stations" periodically along the network, for the purpose of increasing the intensity of the light transmitted along the network. In a typical repeating station of this type, a photodetector is employed to convert the weakened signal transmitted by fiber optics to an electrical signal, which is then magnified, reconverted to a light signal by a light-emitting diode, and transmitted in turn along the next segment of the network.

In order to minimize the requisite number of repeating stations, optimum magnification of the signal in any station is desirable. However, the amount of radiant energy of any kind generated electrically is proportional to the fourth power of the current employed. While a portion of such radiant energy is in the form of light, the remainder thereof is lost as heat. In any individual station, therefore, very large amounts of heat are generated, requiring efficient heat conductors to maintain the operativeness of the repeating station.

The use of natural type IIA diamond as a conductor of heat in these areas has not been foreclosed despite its very high cost since the operative heat-conducting units are very small, typically about 1 mm. on a side. When larger heat-conducting elements are required, expense considerations become important and the use of natural diamond may accordingly be foreclosed.

Conventionally produced high-pressure synthetic diamond of gem quality is lower in cost than natural diamond. However, synthetic diamond of this type having high thermal conductivity cannot effectively be produced directly from graphite because a profound contraction in volume occurs in the conversion and introduces imperfections into the crystalline structure. For the most part, diamond prepared by low-pressure chemical vapor deposition (hereinafter sometimes "CVD") processes are not single crystal diamond and have substantially lower thermal conductivities, typically on the order of 12 watts/cm.°K at about 300°K (hereinafter sometimes "room temperature conductivity").

In U.S. Patent 3,895,313, there are disclosed various diamond materials which allegedly have very high thermal conductivities, and which allegedly are useful as optical elements for very high-power laser beams. In particular, it is stated that synthetic diamond grown from

isotopically pure carbon-12 or carbon-13 would be useful in this way, with room temperature conductivity values in the range of 10-20 watts/cm.°K being mentioned. This is, of course, the order of magnitude of natural type IIA diamond and also of diamond produced by chemical vapor deposition. It is further suggested that at a temperature of 70°K (-203°C), below the temperature of liquid nitrogen, a thermal conductivity exceeding 200 watts/cm.°K may be obtained, perhaps in diamond which has high isotopic purity and "relevant properties which closely approach the limits predicted by the theory of the perfect solid state" (i.e., are in single-crystal form). However, no methods for the preparation of such diamond are suggested, and the state of the art has hitherto been inadequate to put them in the possession of the public.

The present invention provides a method for preparing single-crystal diamond of very high chemical and isotopic purity. The raw material for such diamond is itself diamond, which eliminates the volume contraction encountered in the high-pressure conversion of graphite to diamond. The thermal conductivity of the diamond so produced has been found to be higher than that of any substance presently known including natural type IIA diamond, and also higher than the values in the aforementioned U.S. Patent 3,895,313. Accordingly, it is eminently suitable for use as a conductor of heat and also in numerous other areas.

In one of its aspects, the invention is a method for preparing single-crystal diamond of high isotopic purity which comprises the steps of:

(A) preparing diamond consisting of isotopically pure carbon-12 or carbon-13; and

(B) converting said diamond to single-crystal diamond by diffusion under high pressure through a metallic

catalyst-solvent material to a region containing a diamond seed crystal.

An essential feature of the method of this invention is the employment of isotopically pure carbon-12 or carbon-13. As explained hereinafter, it has been found that the increase in thermal conductivity resulting from the employment of chemically and isotopically pure carbon is vastly greater than would be expected based on theoretical considerations. In general, the isotopic purity of the hydrocarbon should be at least 99.2% by weight; that is, the other isotope should be present in a maximum amount of 8 parts per 1000. An isotopic purity of at least 99.9% by weight is preferred. The hydrocarbon should also have a high degree of chemical purity.

Various methods may be employed in step A for the preparation of diamond in isotopically pure form. For example, a gaseous carbon compound such as carbon monoxide may be separated into carbon-12 and carbon-13 species via differences in diffusivity and then converted to solid carbon by art-recognized means, such as combustion in a reducing flame in the case of carbon monoxide. The carbon thus formed may then be converted to diamond under conventional conditions, including high temperature and high pressure conditions or CVD conditions.

Alternatively, other methods may be employed including shock formation and CVD processes under conditions which produce a mixture of diamond and graphite. In processes of the latter type, the carbon-13 species will concentrate in the diamond phase and the carbon-12 species in the graphite phase. Other diamond precursors which may be employed in enriched form include pyrolytic graphite, amorphous or glassy carbon, liquid hydrocarbons and polymers.

It is usually found that conventional methods of CVD diamond formation are most convenient for the preparation of isotopically pure diamond. In such methods, a layer of diamond is deposited on at least one substrate. Any substrate material suitable for diamond deposition thereon may be employed; examples of such materials are boron, boron nitride, platinum, graphite, molybdenum, copper, aluminum nitride, silver, iron, nickel, silicon, alumina and silica, as well as combinations thereof. Metallic molybdenum substrates are particularly suitable under many conditions and are often preferred.

The method of chemical vapor deposition of diamond on a substrate is known, and the details need not be repeated herein. In brief, it requires high-energy activation of a mixture of hydrogen and a hydrocarbon, typically methane, whereupon the hydrogen gas is converted to atomic hydrogen which reacts with the hydrocarbon to form elemental carbon. Said carbon then deposits on the substrate in the form of diamond. Activation may be achieved by conventional means involving high-energy activation which produces atomic hydrogen from molecular hydrogen; such means include thermal means typically involving heated filaments, flame means, D.C. discharge means and radiation means involving microwave or radio-frequency radiation or the like.

Thermal and especially filament methods, employing one or more resistance heating units including heated wires or filaments, are often preferred for the purposes of this invention. In such methods, the filaments are typically of metallic tungsten, tantalum, molybdenum and rhenium; because of its relatively low cost and particular suitability, tungsten is often preferred. Filament diameters of about 0.2-1.0 mm. are typical, with about 0.8 mm. frequently being preferred. Distances from filaments to substrate(s) are generally on the order of 5-10 mm.

Said filaments are typically heated at temperatures of at least 2000°C and the optimum substrate temperature is in the range of 900-1000°C. The pressure in the deposition vessel is maintained up to about 760 torr, typically on the order of 10 torr. The hydrogen-hydrocarbon mixture generally contains hydrocarbon in an amount up to about 2% by volume based on total gases. For a description of illustrative CVD methods of diamond preparation, reference is made to copending, commonly owned applications U.S. Serial Nos. 07/389,210 and 07/389,212.

Isotopically pure hydrocarbon is employed in the CVD method, when used. In order to avoid contamination thereof, it is essential to employ equipment which does not contain natural carbon as an impurity. For this purpose, the CVD chamber should be constructed of materials substantially incapable of dissolving carbon. Typical materials of this type are quartz and copper.

As between carbon-12 and carbon-13, the former is usually preferred for various reasons. In the first place, carbon-12 is present in nature in much higher proportions than carbon-13, the latter typically occurring in amounts no higher than about 1% by weight; therefore, the use of carbon-12 involves minimum expense. In the second place, thermal conductivity is inversely proportional to the square of the mass number of the isotope, and diamond prepared from carbon-12 can therefore be expected to have a thermal conductivity about 17% greater than those prepared from carbon-13. In some applications, however, carbon-13 is preferred and its preparation and use are part of the invention.

The thickness of the CVD diamond layer deposited on the substrate is not critical. In general, it is convenient to deposit at least as much diamond as will be needed to produce a single crystal of the desired size. Of course, the

production of a larger amount of CVD diamond for use to make several crystals is also contemplated.

It is possible to convert the product of the CVD process directly to diamond of high thermal conductivity by high pressure means, as described hereinafter, employing the same in the form of a slab, sheet or broken pieces thereof. However, the method of this invention is most efficiently conducted if the isotopically pure diamond is first comminuted.

Comminution may be achieved by art-recognized means such as crushing and powdering. The particle size thereof is not critical so long as a sufficient degree of comminution is attained; the form known in the art as "grit diamond" is suitable.

Step B, the production of single crystal diamond, is conventional except that the isotopically pure diamond produced in step A is the raw material employed. Two things are achieved by using diamond rather than graphite or some other allotrope of carbon as the raw material: an easily obtained isotopically pure material may be employed, and the contraction in volume encountered in the conversion of graphite and other allotropes to diamond is avoided, permitting production of a single crystal of regular structure and high quality.

The process for producing single-crystal diamond under high pressure is also known in the art, and a detailed description thereof is not deemed necessary. Reference is made, for example, to Encyclopedia of Physical Science & Technology, vol. 6, pp. 492-506 (Academic Press, Inc., 1987); Strong, The Physics Teacher, January 1975, pp. 7-13; and U.S. Patents 4,073,380 and 4,082,185, for general descriptions of the process. It generally involves diffusion of the carbon employed as a source material through a liquid bath of a metallic catalyst-solvent material, at pressures on the order

of 50,000-60,000 atmospheres and temperatures in the range of about 1300-1500°C. A negative temperature gradient, typically of about 50°C, is preferably maintained between the material being converted and the deposition region, which contains a diamond seed on which crystal growth can begin.

Catalyst-solvent materials useful in step B are known in the art. They include, for example, iron; mixtures thereof with nickel, aluminum, nickel and cobalt, nickel and aluminum, and nickel, cobalt and aluminum; and mixtures of nickel and aluminum. Iron-aluminum mixtures are frequently preferred for the production of single-crystal diamond, with a material consisting of 95% (by weight) iron and 5% aluminum being particularly preferred for the purposes of the invention.

Following preparation of the single-crystal diamond by the method of this invention, it is often preferred to remove the portion attributable to the seed crystal by polishing. This is particularly true if the seed crystal is not isotopically pure.

Studies on single-crystal diamond prepared according to the invention and having various degrees of isotopic purity have shown that at carbon-12 purity levels of 99.2%, 99.5% and 99.9% by weight, the room temperature conductivities are, respectively, 10%, 25% and 40% greater than that of natural type IIA diamond. At lower temperatures, even higher differences in thermal conductivity are to be expected. Such thermal conductivities are higher than those of any material previously known. Single-crystal diamond of this type is another aspect of the invention, as is diamond prepared by the method described herein.

The reasons for the extremely high thermal conductivity of the single-crystal diamond of this invention are not fully understood. It is assumed, however, that the phenomenon is principally a function of the mean free path of

the phonons (i.e., lattice vibration modes) in the diamond crystals. Thermal conductivity is directly proportional to specific heat, sound velocity in and phonon mean free path of the crystal, and isotope effects on specific heat and sound velocity are negligible.

In a simplified calculation, the reciprocal of the phonon mean free path can be considered as equal to the sums of the reciprocals of the mean free paths attributable to phonon-phonon scattering and to isotope effects. It has been calculated that the isotope-related mean free path is 34,000 Angstroms and the phonon scattering-related path is 1900 Angstroms; consequently, the isotope effect would account for a decrease in mean free path on the order of only about 5.2%. One possible reason for the substantially greater observed isotope effect on thermal conductivity is that contrary to theory, the isotopic constitution of the diamond has a direct effect on the mean free path attributable to phonon-phonon scattering. This effect has apparently not been previously recognized.

By reason of the aforementioned high thermal conductivity value and essentially non-existent electrical conductivity, the isotopically pure single crystal diamond of the present invention is singularly useful as a conductor of heat from electronic devices and similar heat-generating sources. Articles comprising such a source in contact with said diamond as a thermal conductor are another aspect of the invention.

A further aspect is abrasive articles comprising said diamond. Such articles can be expected to have extraordinarily long life by reason of their ability to dissipate frictional heat generated during their use. Typical areas of application include abrasive grit, diamond compacts, wire drawing dies, saw blades, scribing tools, drills, tool sharpeners, and polishing tools for optical

items, stone and precious stones including diamonds and articles made therefrom.

Still another aspect is light-filtering articles comprising said diamond with a pinhole aperture therein. They are useful, for example, as spatial filters for laser beams and the like. Articles of this type fabricated from natural diamond are subject to radiation-induced damage, probably thermal in nature. The substantially higher thermal conductivity of the diamond of this invention may be expected to greatly minimize damage of this kind.

The invention is illustrated by an example in which a layer of CVD diamond was first deposited on a molybdenum substrate in a chamber constructed of quartz and copper, neither of which dissolves substantial amounts of carbon. The substrate was vertically disposed in a plane parallel to and 8-9 mm. distant from the plane of a tungsten filament about 0.8 mm. in diameter. The vessel was evacuated to a pressure of about 10 torr, the filament was heated to about 2000°C by passage of an electric current and a mixture of 98.5% (by volume) hydrogen and 1.5% methane was passed into the vessel. The methane employed was substantially impurity-free and 99.9% thereof contained the carbon-12 isotope. Upon removal and mass spectroscopic analysis of the diamond thus obtained, it was found that 99.91% of the carbon therein was carbon-12.

Thermal conductivity of the isotopically pure CVD diamond was measured by the mirage detection of thermal waves generated by a modulated argon-ion beam impinging on the diamond crystals, in accordance with a conventional method. The room temperature conductivity was shown to be about 12 watts/cm.°K. A control sample of similar CVD diamond prepared from methane having the naturally occurring isotope distribution (98.96% C-12, 1.04% C-13) had essentially the same conductivity.

The isotopically pure CVD diamond was crushed and powdered, and was used as a source of carbon for the growth of a single-crystal diamond under high pressure and high temperature conditions. Specifically, a conventional belt apparatus was employed at 52,000 atmospheres and 1400°C, employing a catalyst-solvent mixture of 95% (by weight) iron and 5% aluminum. A small (0.005 carat) single-crystal diamond seed of normal isotopic distribution was used to initiate growth, and a negative temperature gradient of about 50°C was maintained between the CVD diamond and the seed crystal. The process was continued until a single crystal of 0.95 carat had been produced. It was shown by analysis that 99.93% of the carbon therein was the C-12 isotope.

The diamond was polished on a standard diamond scaife to remove the seed crystal, and its room temperature conductivity was compared with those of several other materials, including a control single-crystal diamond prepared from the CVD diamond with normal isotope distribution. The results were as follows, all values being in watts/cm.°K:

Isotopically pure C-12 diamond (this invention)	31.5
Control	21.18
Naturally occurring type IIA diamond	21.2
CVD diamond	12.0
Cubic boron nitride	7.6
Silicon carbide	4.9
Copper	4.0
Beryllium oxide	3.7
Boron phosphide	3.6
Aluminum nitride	3.2
Silicon	1.6
Aluminum oxide	0.2

Thus, the room temperature conductivity of the diamond of this invention is 48.7% greater than that of the control. It is also much higher than the room temperature conductivity of any other diamond or non-diamond material which has been measured.

At 70°K, below the boiling point of liquid nitrogen, the diamond of the present invention is theoretically predicted to have a conductivity of about 2675 watts/cm.°K, more than 13 times the minimum value predicted in the aforementioned U.S. Patent 3,895,313.

CLAIMS

1. A method for preparing single-crystal diamond of high isotopic purity which comprises the steps of:

(A) preparing diamond consisting of isotopically pure carbon-12 or carbon-13; and

(B) converting said diamond to single-crystal diamond by diffusion under high pressure through a metallic catalyst-solvent material to a region containing a diamond seed crystal.

2. A method as claimed in claim 1 wherein the carbon in the hydrocarbon is carbon-12.

3. A method as claimed in claim 1 or claim 2 wherein the product of step A is comminuted.

4. A method as claimed in any of the preceding claims wherein the carbon in the hydrocarbon is at least 99.2% isotopically pure.

5. A method as claimed in any of the preceding claims wherein the diamond of step A is prepared by chemical vapor deposition.

6. A method as claimed in any of the preceding claims wherein the equipment employed in step A is constructed of materials substantially incapable of dissolving carbon.

7. A method as claimed in claim 4 wherein the particle size achieved by comminution is that of grit diamond.

8. A method as claimed in claim 7 or claim 4 wherein step A is conducted at a filament temperature of at least 2000°C, a substrate temperature in the range of 900-1000°C and a pressure of about 10 torr.

9. A method as claimed in claim 7 or claim 8 wherein the filaments are of tungsten and the substrate is molybdenum.

10. A method as claimed in claim 6 wherein the carbon in the hydrocarbon is at least 99.9% isotopically pure.

11. A method asclaimed in claim 10 wherein the catalyst-solvent material employed in step B is an iron-aluminum mixture.

12. A method as claimed in claim 11 wherein a negative temperature gradient is maintained in step B between the material being converted and the deposition region.

13. A method as claimed in claim 12 wherein the catalyst-solvent material employed in step B is a mixture of 95% iron and 5% aluminum by weight.

14. A method as claimed in claim 13 wherein the temperature gradient is about 50°C.

15. A method as claimed in claim 12 wherein the pressure in step B is in the range of 50,000-60,000

atmospheres and the temperature is in the range of 1300-1500°C.

16. A method as claimed in claim 15 wherein the seed crystal is single-crystal diamond of normal isotopic distribution.

17. A method as claimed in claim 16 wherein the portion of the product diamond attributable to the seed crystal is removed by polishing.

18. Single-crystal diamond consisting of at least 99.2% by weight isotopically pure carbon-12 or carbon-13, said diamond having a thermal conductivity at 300°K at least 10% greater than that of natural type IIA diamond.

19. Diamond as claimed in claim 18 which consists of carbon-12.

20. An article comprising a heat-generating source in contact with the diamond of claim 18 as a thermal conductor.

21. An abrasive article comprising the diamond of claim 18.

22. A light-filtering article comprising the diamond of claim 18 with a pinhole aperture therein.

23. Single-crystal diamond consisting of at least 99.5% by weight isotopically pure carbon-12 or carbon-13, said diamond having a thermal conductivity at 300°K at

least 25% greater than that of natural type IIA diamond.

24. Single-crystal diamond consisting of at least 99.9% by weight isotopically pure carbon-12 or carbon-13, said diamond having a thermal conductivity at 300°K at least 40% greater than that of natural type IIA diamond.

25. Single-crystal diamond prepared by the method of claim 1.

26. Single-crystal diamond prepared by the method of claim 2.

27. Single-crystal diamond prepared by the method of claim 4.

28. Single-crystal diamond prepared by the method of claim 10.

29. Single-crystal diamond prepared by the method of claim 17.

30. A method as claimed in claim 1 substantially as hereinbefore described in any one of the examples.

31. A single crystal diamond as claimed in claim 18 substantially as hereinbefore described in any one of the examples.

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PATENT ABSTRACTS OF JAPAN

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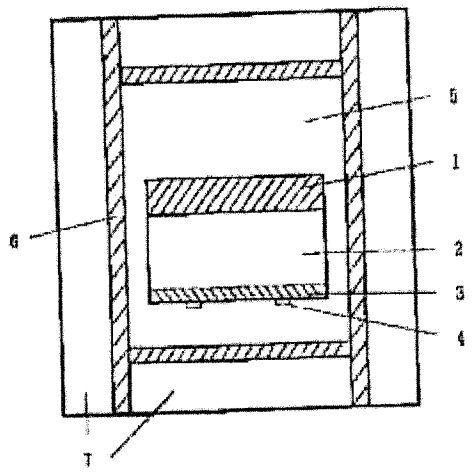
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(54) SYNTHESIS OF SINGLE CRYSTAL DIAMOND

(57)Abstract:
PURPOSE: To advantageously produce a colorless, transparent high-quality single- crystal diamond in the synthesis thereof employing temp. difference process by a method where Ti, etc., are added to a solvent metal as nitrogen getter, a metal plate such as made of Cu is interposed between the solvent metal and seed crystal and, in this condition, the synthesis of the diamond is started.
CONSTITUTION: In the synthesis of a single crystal diamond employing temp. difference process, one or more metals selected as nitrogen getter from among Ti, Zr, Hf, V, Nb and Ta are added to a solvent metal 2, a metal plate 3 consisting of one or more metals selected from among Cu, Ag, Au, Zn and Cd is interposed between the solvent metal 2 and the seed crystal 4 and, in this condition, the diamond synthesis is started. Since, in this way, a colorless, transparent high quality IIa type diamond almost free from inclusion can be synthesized inexpensively and stably, a synthetic diamond useable in accessory or optical part application can be produced advantageously.



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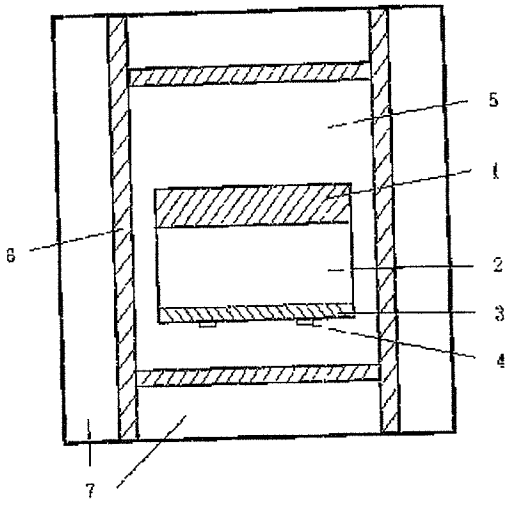
(74)代理人　弁理士　内田　明　(外２名)

(54)【発明の名称】　ダイヤモンド単結晶の合成方法

(57)【要約】

【目的】　装飾用途や光学部品などに用いることができる無色透明で良質なダイヤモンド単結晶の新規な合成方法を提供する。

【構成】　温度差法によるダイヤモンド単結晶合成において、溶媒金属に窒素ゲッターとしてＴｉ，Ｚｒ，Ｈｆ，Ｖ，Ｎｂ，Ｔａから選ばれる一種もしくは二種以上の金属を添加し、かつ、前記溶媒金属と種結晶との間にＣｕ，Ａｇ，Ａｕ，Ｚｎ，Ｃｄから選ばれる一種もしくは二種以上からなる金属板を配置した状態でダイヤモンドの合成を開始することを特徴とする。これにより、無色透明でインクルージョンの殆どない良質なＩＩａ型ダイヤモンド結晶を安価に且つ安定に合成できるので、装飾用途や光学部品に供し得る合成ダイヤモンドの有利な製法である。



1

【特許請求の範囲】

【請求項１】　温度差法によるダイヤモンド結晶合成において、溶媒金属に窒素ゲッターとしてＴｉ，Ｚｒ，Ｈｆ，Ｖ，Ｎｂ，Ｔａから選ばれる一種もしくは二種以上の金属を添加し，かつ，前記溶媒金属と種結晶との間にＣｕ，Ａｇ，Ａｕ，Ｚｎ，Ｃｄから選ばれる一種もしくは二種以上からなる金属板を配置した状態でダイヤモンドの合成を開始することを特徴とするダイヤモンド単結晶の合成方法。

【請求項２】　前記の溶媒金属と種結晶との間に配置される金属板の厚みは０．０１ｍｍ以上．１ｍｍ以下であることを特徴とする請求項１記載のダイヤモンド単結晶の合成方法。

【請求項３】　前記窒素ゲッターとして用いられるＴｉ，Ｚｒ，Ｈｆ，Ｖ，Ｎｂ，Ｔａから選ばれる一種もしくは二種以上の金属の含有量が、溶媒金属に対して０．２～１０重量%であることを特徴とする請求項１または２記載のダイヤモンド単結晶の合成方法。

【請求項４】　前記溶媒金属は、Ｆｅ，Ｃｏ，Ｎｉ，Ｍｎ，Ｃｒの中から選ばれる一種もしくは二種以上からなり，且つ０．１～６．０重量%の炭素を含むことを特徴とする請求項１または２または３記載のダイヤモンド単結晶の合成方法。

【発明の詳細な説明】

【０００１】

【産業上の利用分野】本発明は装飾用途や光学部品などに用いられる無色で透明なダイヤモンド単結晶の合成方法に関するものである。

【０００２】

【従来の技術】現在市販されている装飾用ダイヤモンドは，主に南アフリカ、ロシアより産出されるものの中から，無色透明で内部欠陥の少ないものを選別して用いられている。天然装飾用ダイヤモンドは宝石の中でも最も販売量が多い。また、ダイヤモンドを用いた光学部品として，レーザー窓やＩＲアンビルセルなどがあるが，いずれも天然原石の中から赤外領域に光の吸収のない透明なダイヤモンド（IIa型とよばれる）が選ばれて用いられている。しかし、透明無色な原石の産出は極めて少なく，安定供給や価格に問題がある。一方、人工合成によるダイヤモンドは通常，超高圧高温下で合成する際に溶媒中の窒素が結晶格子内に取り込まれるために黄色く着色してしまうが，溶媒中に窒素ゲッターを添加することで無色透明のダイヤモンドを得ることができる。この窒素ゲッターとしては，たとえば，The Journal of Physical Chemistry, vol.75, No.12 (1971) p1838 に示されているように，Ａｌがよく知られている。具体的には、米国特許第４０３４０６６号明細書には、Ｆｅ溶媒にＡｌを３～５重量%添加することにより宝石級の無色透明なダイヤモンド単結晶が得られると記載されている。Ａｌ以外の窒素ゲッターを用いた例として、たとえば無機

2

材質研究所研究報告書第３９号、ｐ１６～にＴｉやＺｒを溶媒金属に添加することで結晶中の窒素が除去されたという報告がある。

【０００３】

【発明が解決しようとする課題】しかし、特に無色透明の合成ダイヤモンドは合成コストが天然ダイヤモンドよりはるかに高くなるため工業生産は行われていない。この理由は、合成には高価で特殊な装置が必要である上に，Ａｌなどを窒素ゲッターとして添加した場合，その添加量の増加に従って溶媒が結晶中に取り込まれ（以下インクルージョンと呼ぶ）て，不良結晶となることが多くなるため，良質な結晶とするためには成長速度を大幅に下げる必要があるからである。とくにＴｉやＺｒを窒素ゲッターとして用いた場合は合成中に溶媒中に生成したＴｉＣやＺｒＣなどの炭化物が原因でより多くのインクルージョンが結晶中に取り込まれるようになる。

【０００４】本発明者らが行った実験による結果では，窒素ゲッターとしてＡｌを用い、溶媒金属に均一混合した場合、無色透明なダイヤモンド結晶を合成するためには，その添加量は溶媒に対し少なくとも４重量%（約１２体積%）必要であるが，この場合インクルージョンの巻き込みなしに結晶成長させるためには成長速度を１ｍｇ／ｈｒ以下にする必要があった。この成長速度では，たとえば１カラット（２００ｍｇ）の結晶を合成するには２００時間以上の合成時間を要し，製造コストは膨大なものとなる。また、Ｔｉ，Ｚｒなど，Ａｌより窒素との反応性の高い物質を窒素ゲッターとして溶媒に均一添加した場合、添加量は１重量%でも無色透明な結晶となる。しかし、これらは炭化物を形成しやすく、成長速度を大幅に低下させたとしてもＴｉＣやＺｒＣなどの炭化物の影響で、良質な結晶は殆ど得られない。本発明はかかる問題点を解決し、無色透明でインクルージョのほとんどない結晶を，安価にしかも安定して合成できる方法を提供し、人工合成ダイヤモンドの装飾用途又は光学部品用途への使用を可能とするものである。

【０００５】

【課題を解決するための手段】本発明者らは、ＴｉやＺｒを窒素ゲッターとして溶媒に添加して成長させたダイヤモンド結晶を観察したところ、インクルージョンのほとんどは種結晶を起点として連続して結晶中に取り込まれていることがわかった。さらに、詳細に調べると成長初期に種結晶面上に生成したＴｉＣやＺｒＣなどの炭化物がインクルージョンの起点となっていることがわかった。そこで本発明者らは，成長初期に種面上に炭化物生成を防ぐため溶媒と種結晶の間にあらかじめＣｕ，Ａｇ，Ａｕ，Ｚｎ，Ｃｄなど，それ自体炭化物を作らず，加えて、ＴｉＣやＺｒＣやＮｂＣなどのIVa族もしくはＶａ族元素の炭化物を分解する働きのなる金属板を配置したところ、種面上にほとんど炭化物を生成しないことを見いだした。その結果，炭化物を起点としたインクル

ージョンの巻き込みを防ぐことができ．このため．従来の２倍以上の早い成長速度でも、良質なIIa結晶が得られることを確認した。

【０００６】本発明は温度差法によるダイヤモンド結晶合成において．溶媒金属に．窒素ゲッターとしてＴｉ，Ｚｒ、Ｈｆ，Ｖ、Ｎｂ，Ｔａから選ばれる一種もしくは二種以上の金属を添加し．さらに前記溶媒金属と種結晶との間にＣｕ、Ａｇ，Ａｕ、Ｚｎ，Ｃｄから選ばれる一種もしくは二種以上からなる金属板を配置した状態でダイヤモンドの合成を開始することを特徴とするものである。本発明において溶媒金属と種結晶との間に配置される金属板の厚みは０．０１ｍｍ以上．１ｍｍ以下であることが好ましい。また、窒素ゲッターとして用いられるＴｉ．Ｚｒ，Ｈｆ，Ｖ，Ｎｂ，Ｔａから選ばれる一種もしくは二種以上の金属の含有量は、溶媒金属に対して０．２～１０重量％であることが好ましい。窒素ゲッターとしてのこれら金属は．他の金属．例えば、Ａｌ、Ｃｕ．Ｓｉ、Ｓｎ等との合金の形で添加することもできる。また、溶媒金属は、Ｆｅ，Ｃｏ、Ｎｉ，Ｍｎ、Ｃｒの中から選ばれる一種もしくは二種以上からなり．且つ０．１～６．０重量％の炭素を含むことが好ましい。

【０００７】図１は本発明の一具体例であってダイヤモンド結晶合成用の試料室構成を示す概略断面図である。溶媒金属２には窒素ゲッターとしてＴｉ，Ｚｒ、Ｈｆ，Ｖ、Ｎｂ，Ｔａが添加され．この溶媒金属と種結晶４の間にＣｕ，Ａｇ、Ａｕ，Ｚｎ，Ｃｄから選ばれる金属板３が配置される。１は炭素源、５は絶縁体、６は黒鉛ヒーター、７は圧力媒体である。この金属板３の厚みは０．０１ｍｍ未満では効果は不十分で．１ｍｍをこえるとＣｕ，Ａｇ．Ａｕ，Ｚｎ．Ｃｄなどの金属がダイヤモンドの結晶成長に悪影響を及ぼし、結晶が多結晶化したり．埋め残しのある不良結晶となってしまう。また溶媒２に窒素ゲッターとして添加するＴｉ、Ｚｒ，Ｈｆ，Ｖ、Ｎｂ，Ｔａの添加量としては、０．２重量％未満では窒素は十分に除去されず．合成されたダイヤモンドは黄色みを帯びた結晶となる。一方、１０重量％をこえると多結晶化や自然核発生が多くなり．良質なダイヤモンド結晶が得られなくなる。

【０００８】ここで、２の溶媒金属はＦｅ，Ｃｏ、Ｎｉ、Ｍｎ，Ｃｒの中から選ばれる一種もしくは二種以上からなる金属であり、種結晶溶解防止のため０．１～６．０重量％の炭素をあらかじめ添加しておくのが好ましい。炭素添加量が０．１重量％未満もしくは炭素を含まない溶媒金属を用いた場合、種結晶上にＰｔなどの種結晶溶解防止材を配置する必要があるが、このような種結晶溶解防止材を配置することは、多結晶化やインクルージョンの巻き込みの原因となり好ましくない。また、炭素添加量が６重量％をこえると、自然核発生がおこりやすくなり、種結晶以外の部所より結晶成長するため結晶同士が干渉し．良質な結晶が得られなくなる。

【０００９】

【作用】本発明ではＴｉ．Ｚｒ，Ｈｆ、Ｖ，Ｎｂ、Ｔａなど．窒素との反応性の高い元素を窒素ゲッターとするため．少量の添加でほとんど窒素を含まない無色透明なIIa型のダイヤモンド結晶が得られ．また、溶媒と種結晶の間に、炭化物生成防止のために．Ｃｕ，Ａｇ、Ａｕ、Ｚｎ，Ｃｄの金属板をあらかじめ配置した状態で合成を開始する構成になっているため．インクルージョンの混入を大幅に抑えることができる。その結果．従来よりかなり速い成長速度でも良質なIIa型ダイヤモンド結晶が得られるようになる。また、本発明に用いる種結晶．炭素源等はこの種の技術分野で公知のものを用いることができる。また、温度差法による合成の条件等は適宜選択することができる。

【００１０】

【実施例】以下実施例により本発明をさらに詳細に説明する。

実施例１

溶媒の原料として粒径５０～１００ミクロンの高純度Ｆｅ粉末、Ｃｏ粉末、グラファイト粉末を用い、

Ｆｅ：Ｃｏ：Ｃ＝６０：４０：４．５（重量比）

となるように配合した。これに、さらに窒素ゲッターとして平均粒径５０ミクロンのＴｉ粉末を１．５重量％添加し．十分に混合した。この混合粉末を型押し成形し、脱ガス、焼成したもの（直径２０ｍｍ．厚み１０ｍｍ）を溶媒とした。図１に示す試料室構成で炭素源１にはダイヤモンドの粉末、種結晶４には直径５００ミクロンのダイヤモンド結晶３個を用いた。種結晶４と溶媒２の間に配置する金属板３としては厚み０．２ｍｍのＣｕ板を用いた。そして．炭素源１と種結晶４部に約３０℃の温度差がつくように加熱ヒーター６内にセットした。これを超高圧発生装置を用いて．圧力５．５ＧＰａ．温度１３００℃で７０時間保持し．ダイヤモンドの合成を行った。その結果０．７～０．９カラットの無色透明な、インクルージョンのほとんどない良質なIIa型のダイヤモンド結晶が得られた。ＥＳＲにより．結晶中の窒素濃度を測定するといずれも０．１ｐｐｍ以下であった。磁気天秤によりインクルージョン量を測定するといずれも０．３重量％以下であった。

【００１１】実施例２

種結晶４と溶媒２の間に配置する金属板３に厚み０．５ｍｍのＣｕ板を用いた他は実施例１と同様にしてダイヤモンドの合成を行った。その結果、実施例１とほとんど同じ良質なIIa型ダイヤモンド結晶が得られた。

実施例３

種結晶４と溶媒２の間に配置する金属板３に厚み０．２ｍｍのＡｇ板を用いた他は実施例１と同様にしてダイヤモンドの合成を行った。その結果、実施例１とほとんど同じ良質なIIa型ダイヤモンド結晶が得られた。

実施例４

種結晶４と溶媒２の間に配置する金属板３に厚み０．３ｍｍのＡｕ板を用いた他は実施例１と同様にしてダイヤモンドの合成を行った。その結果、実施例１とほとんど同じ良質なIIａ型ダイヤモンド結晶が得られた。

【００１２】実施例５

種結晶４と溶媒２の間に配置する金属板３に厚み０．０５ｍｍのＡｕ板を用いた他は実施例１と同様にしてダイヤモンドの合成を行った。その結果、実施例１とほとんど同じ良質なIIａ型ダイヤモンド結晶が得られた。

実施例６

種結晶４と溶媒２の間に配置する金属板３に厚み０．２ｍｍのＺｎ板を用いた他は実施例１と同様にしてダイヤモンドの合成を行った。その結果、実施例１とほとんど同じ良質なIIａ型ダイヤモンド結晶が得られた。

実施例７

溶媒２に添加する窒素ゲッターに粒径４４ミクロン以下（平均１０ミクロン）のＺｒ粉末を溶媒に対し３重量%添加した他は実施例１と同様にしてダイヤモンドの合成を行った。その結果、実施例１とほとんど同じ良質なIIａ型ダイヤモンド結晶が得られた。

【００１３】実施例８

溶媒２に添加する窒素ゲッターに粒径４４ミクロン以下（平均１０ミクロン）のＡｌＴｉ合金粉末を溶媒に対し３重量%添加した他は実施例１と同様にしてダイヤモンドの合成を行った。その結果、実施例１とほとんど同じ良質なIIａ型ダイヤモンド結晶が得られた。

実施例９

溶媒２に添加する窒素ゲッターとしてＡｌＴｉ合金粉末の代わりに、ＣｕＴｉ、ＳｎＴｉ又はＳｉ、Ｔｉ合金粉末を用いた他は実施例８と同様にしてダイヤモンドの合成を行った。その結果、いずれの場合も、実施例１とほとんど同じ良質なIIａ型ダイヤモンド結晶が得られた。

実施例１０

溶媒の原料として粒径５０～１００ミクロンの高純度Ｆｅ粉末、Ｎｉ粉末、Ｃｏ粉末、グラファイト粉末を用い、

Ｆｅ：Ｎｉ：Ｃｏ：Ｃ＝６０：３０：１０：４．２（重量比）

となるように配合した他は実施例１と同様にしてダイヤモンドの合成を行った。その結果、実施例１とほとんど同じ良質なIIａ型ダイヤモンド結晶が得られた。

【００１４】実施例１１

溶媒の原料として粒径５０～１００ミクロンの高純度Ｆｅ粉末、Ｎｉ粉末、Ｍｎ粉末、グラファイト粉末を用い、

Ｆｅ：Ｎｉ：Ｍｎ：Ｃ＝６０：３０：１０：４（重量比）

となるように配合した他は実施例１と同様にしてダイヤモンドの合成を行った。その結果、実施例１とほとんど

同じ良質なIIａ型ダイヤモンド結晶が得られた。

実施例１２

溶媒の原料として粒径５０～１００ミクロンの高純度Ｆｅ粉末、Ｎｉ粉末、グラファイト粉末を用い、

Ｆｅ：Ｎｉ：Ｃ＝７０：３０：３．５（重量比）

となるように配合した他は実施例１と同様にしてダイヤモンドの合成を行った。その結果、実施例１とほとんど同じ良質なIIａ型ダイヤモンド結晶が得られた。

実施例１３

溶媒の原料として粒径５０～１００ミクロンの高純度Ｃｏ粉末、グラファイト粉末を用い、

Ｃｏ：Ｃ＝１００：４．７（重量比）

となるように配合した他は実施例１と同様にしてダイヤモンドの合成を行った。その結果、実施例１とほとんど同じ良質なIIａ型ダイヤモンド結晶が得られた。

【００１５】実施例１４

溶媒の原料として粒径５０～１００ミクロンの高純度Ｎｉ粉末、グラファイト粉末を用い、

Ｎｉ：Ｃ＝１００：４．２（重量比）

となるように配合した他は実施例１と同様にしてダイヤモンドの合成を行った。その結果、実施例１とほとんど同じ良質なIIａ型ダイヤモンド結晶が得られた。

【００１６】比較例１

溶媒と種結晶の間に金属板を配置せずに、他は実施例１と同様にダイヤモンドの合成を試みた。窒素含有量の少ない（約０．１ｐｐｍ）の結晶が得られたが、インクルージョンの巻き込み量は約１．５重量%と多く、良質な結晶は得られなかった。

実施例１５

溶媒と種結晶の間に配置するＣｕ板の厚みを２ｍｍとした他は実施例１と同様にダイヤモンドの合成を試みた。成長したダイヤモンド結晶は表面に埋め残しの多い骸晶状となった。

実施例１６

窒素ゲッターとして溶媒に添加するＴｉの量を１５重量%とした他は実施例１と同様にダイヤモンドの合成を試みた。種結晶から成長した結晶は多結晶化していた。

【００１７】実施例１７

溶媒の原料として粒径５０～１００ミクロンの高純度Ｆｅ粉末、Ｎｉ粉末、Ｃｏ粉末を用い、

Ｆｅ：Ｎｉ：Ｃｏ＝６０：３０：１０（重量比）

となるように配合し、グラファイトを添加しなかった他は実施例１と同様にしてダイヤモンドの合成を行った。その結果、種結晶は溶媒中に完全に溶解してしまい、ダイヤモンドの成長は不充分であった。

実施例１８

溶媒の原料として粒径５０～１００ミクロンの高純度Ｆｅ粉末、Ｎｉ粉末、Ｃｏ粉末、グラファイト粉末を用い、

Ｆｅ：Ｎｉ：Ｃｏ：Ｃ＝６０：３０：１０：７（重量

http://www4.ipdl.inpit.go.jp/TD/web003/20140816093213520520.gif　8/15/2014
Ex. 1003, Page 347

比）

となるように配合した他は実施例１と同様にしてダイヤモンドの合成を行った。その結果、種結晶以外の所よりダイヤモンドが多数自然核発生し、このため結晶同士が干渉し、良質なダイヤモンド結晶はほとんど得られなかった。

【００１８】

【発明の効果】以上説明したように、本発明によれば、無色透明でインクルージョンのほとんどないダイヤモンド結晶を、安価に安定して合成できる。本方法によって合成ダイヤモンドを装飾用途、光学部品などに利用することが可能なものとなる。

＊【図面の簡単な説明】

【図１】図１は本発明の一具体例であって、結晶合成用の試料室構成を示す概略断面図である。

【符号の説明】

１：炭素源

２：溶媒金属（窒素ゲッターを含む）

３：金属板

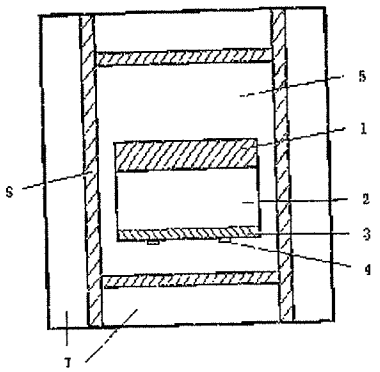
４：種結晶

５：絶縁体

６：黒鉛ヒーター

７：圧力媒体

【図１】



PATENT ABSTRACTS OF JAPAN

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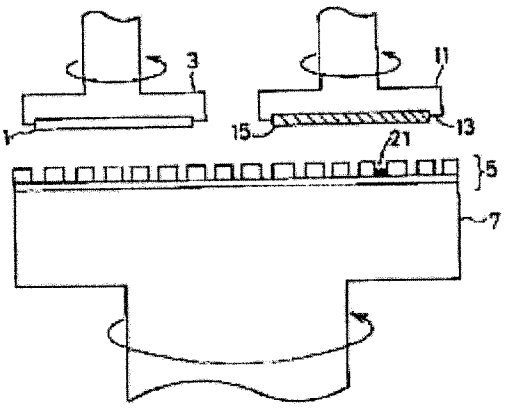
(51)Int.Cl. B24B 37/00
H01L 21/304

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(71)Applicant : TOSHIBA CORP
(72)Inventor : KATO NOBUHIRO
MURAKAMI SATOSHI
WATANABE TOMOJI

(54) CMP DEVICE AND ABRASIVE CLOTH CAPABLE OF BEING USED IN THIS CMP DEVICE

(57)Abstract:
PROBLEM TO BE SOLVED: To provide a CMP device and an abrasive cloth capable of being used in the CMP device, which can suppress deterioration of polishing performance of the abrasive cloth and easily detect a limit of use.
SOLUTION: In a CMP device polishing a semiconductor substrate 1, a diamond dresser 11 is provided with a material 15 capable of mechanically removing a diamond abrasive grain coming off onto an abrasive cloth 5 inside a diamond abrasive grain electrocoating surface 13, and, by the material 15, simultaneously with dressing, the diamond abrasive grain on the cloth 5 is removed. Further, the cloth 5 is provided with a use limit detection part 21 with a work amount of dimple work or the like less than of the other part, and, by polishing the semiconductor substrate 1, the point of time exposing a surface of the use limit detection part 21 is detected as a use limit.



(19)日本国特許庁（ＪＰ）　(12)**公開特許公報**（Ａ）　(11)特許出願公開番号

特開平10－128654

(43)公開日　平成10年(1998)５月19日

(51)Int.Cl.[6]	識別記号	ＦＩ	
Ｂ２４Ｂ　37/00		Ｂ２４Ｂ　37/00	Ａ
Ｈ０１Ｌ　21/304	３２１	Ｈ０１Ｌ　21/304	３２１Ｅ

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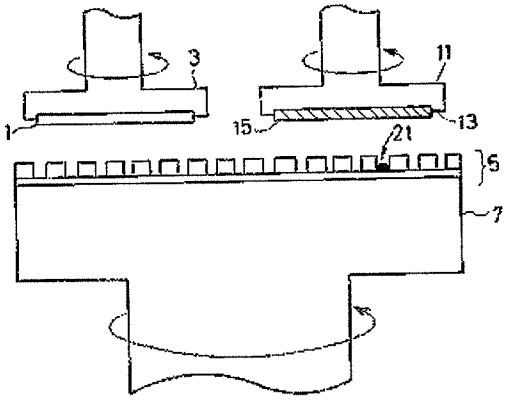
(74)代理人　弁理士　三好　秀和　（外３名）

(54)【発明の名称】　ＣＭＰ装置及び該ＣＭＰ装置に用いることのできる研磨布

(57)【要約】

【課題】　研磨布の研磨性能の劣化を抑制し、かつ、使用限界を容易に検出することができるＣＭＰ装置及び該ＣＭＰ装置に用いることのできる研磨布を提供することを課題とする。

【解決手段】　半導体基板１の研磨を行うＣＭＰ装置で、ダイヤモンドドレッサー１１はダイヤモンド砥粒固着面１３の内側に研磨布５上に脱落したダイヤモンド砥粒を機械的に除去することが可能な材料１５を備え、材料１５によりドレッシングと同時に研磨布５上のダイヤモンド砥粒を除去する。さらに、研磨布５は、ディンプル加工等の加工量が他の部分よりも少ない使用限界検出部２１を備え、半導体基板１の研磨により使用限界検出部２１が表面に露出した時点を使用限界として検出する。



1

【特許請求の範囲】

【請求項１】　底面周辺部に設けられたダイヤモンド砥粒電着面により研磨布表面のドレッシングを行うダイヤモンドドレッサーを有するＣＭＰ装置において．

前記ダイヤモンドドレッサーがドレッシングの際に前記ダイヤモンド砥粒電着面から前記研磨布表面に脱落するダイヤモンド砥粒を除去する手段を具備することを特徴とするＣＭＰ装置。

【請求項２】　底面周辺部に設けられたダイヤモンド砥粒電着面により研磨布表面のドレッシングを行うダイヤモンドドレッサーを有するＣＭＰ装置において．

前記ダイヤモンドドレッサーは、前記ダイヤモンド砥粒電着面の内側に前記研磨布上に脱落したダイヤモンド砥粒を機械的に除去することが可能な材料を備え．前記材料によりドレッシングと同時に該ドレッシングの際に前記ダイヤモンド砥粒電着面から前記研磨布上に脱落した前記ダイヤモンド砥粒を除去することを特徴とするＣＭＰ装置。

【請求項３】　前記材料は．ナイロンブラシであることを特徴とする請求項２記載のＣＭＰ装置。

【請求項４】　前記材料は．スポンジであることを特徴とする請求項２記載のＣＭＰ装置。

【請求項５】　前記材料は．モヘアブラシであることを特徴とする請求項２記載のＣＭＰ装置。

【請求項６】　表面に凹部が形成された研磨布により基板の研磨を行うＣＭＰ装置において．

前記研磨布が該研磨布の使用限界を目視により確認することができる使用限界検出手段を具備することを特徴とするＣＭＰ装置。

【請求項７】　表面に凹部が形成された研磨布により基板の研磨を行うＣＭＰ装置において．

前記研磨布は．前記凹部内に使用限界検出部を備え、前記基板の研磨により前記使用限界検出部が前記研磨布表面に露出した時点を使用限界として検出することを特徴とするＣＭＰ装置。

【請求項８】　前記使用限界検出部は．前記凹部内で他の部分よりも浅く加工された部分であることを特徴とする請求項７記載のＣＭＰ装置。

【請求項９】　前記凹部を通じて砥液が研磨布全面に供給されることを特徴とする請求項６乃至請求項８のいずれか１項に記載のＣＭＰ装置。

【請求項１０】　表面に凹部が形成され、基板の研磨に用いられる研磨布において．

前記凹部内に他の部分よりも浅く加工された部分を使用限界検出部として備え、前記基板の研磨により前記使用限界検出部が前記研磨布表面に露出した時点を使用限界と検出することを特徴とする研磨布。

【請求項１１】　前記凹部は、研磨布全面に砥液を供給するために設けられたものであることを特徴とする請求項１０記載の研磨布。

2

【発明の詳細な説明】

【０００１】

【発明の属する技術分野】本発明はＬＳＩ製造工程における平坦化技術として用いられるＣＭＰ装置及び該ＣＭＰ装置に用いることのできる研磨布に関し、特に．研磨布の研磨性能の劣化を抑制し、かつ．使用限界を容易に検出することができるＣＭＰ装置及び該ＣＭＰ装置に用いることのできる研磨布に関する。

【０００２】

【従来の技術】近年、ＬＳＩ（Large Scale Integrated Circuit）の配線が多層化するのに伴い、層間絶縁膜の平坦化技術が非常に重要となってきているが、その技術として現在最も注目されているものが化学的機械研磨（chemical mechanical polishing ：ＣＭＰ）技術である。

【０００３】図５は、上記ＣＭＰ技術を実現する一般的なＣＭＰ装置の構成を示す図である。同図に示すように．このＣＭＰ装置は半導体基板１を固定し、半導体基板１を回転させるトップリング３と．半導体基板１の表面を研磨する研磨布５と．研磨布５を固定し、研磨布５を回転させるターンテーブル７とを少なくとも有し、所定の製造工程を経て凹凸状態となっている半導体基板１の表面を研磨布５に押し付け、砥液９を供給しながらトップリング３とターンテーブル７をそれぞれ回転させ、化学反応を生じさせながら機械的に半導体基板１表面を研磨布５で研磨することにより半導体基板１表面の平坦化を図るものである。

【０００４】上述したようなＣＭＰ装置においては、研磨布の研磨性能維持のためにドレッシング（dressing）と呼ばれる作業が必要である。ドレッシングとは鈍くなった研磨布の目を再び鋭くする目立のことであり．半導体基板の処理枚数の増加に伴って鈍くなっていく研磨布の目を再び鋭くすることにより、研磨布の研磨性能を維持するのである。このドレッシングは例えば次のようなダイヤモンドドレッサーにより実施される。図６は、ドレッシングを行う際のダイヤモンドドレッサーと研磨布を示す図であり．ドレッシングはダイヤモンドドレッサー１１をターンテーブル７に固定された研磨布５の表面に押し付け、ダイヤモンドドレッサー１１とターンテーブル７それぞれを回転させることにより行われる。また．ダイヤモンドドレッサー１１自体を揺動させる場合もある。図７は．上記ダイヤモンドドレッサー１１の拡大図であり、ダイヤモンドドレッサー１１の底面周辺にはダイヤモンド砥粒を電着して形成したダイヤモンド砥粒電着面１３が設けられ．このダイヤモンド砥粒電着面１３を研磨布表面に当てることにより上記ドレッシングが行われる。なお、ドレッシングは上述した半導体基板の研磨と同時に．または．その研磨前．研磨後に行われる。

【０００５】また、図８は．図５と同様に一般的なＣＭ

Ｐ装置の構成を示す図であるが、図８に示すように、ＣＭＰ装置に用いられる研磨布５の表面は実際には砥液９が研磨布５全面に行き渡るようにディンプル加工や格子状の溝加工が施されている。この研磨布５により上述したように半導体基板１の表面を研磨した場合、半導体基板１の処理枚数の増加に伴って研磨布５は摩耗し薄くなるが、研磨布５の使用限界を越えた場合には研磨速度や研磨量の面内均一性の値が悪化してしまう。そのため、実際に上記ＣＭＰ装置により半導体基板１を研磨する場合には、その直前に別途用意された使用限界検出用のテスト基板を予め研磨し、そのテスト基板を評価することにより研磨速度や研磨量の面内均一性の値を算出し、その算出結果に基づいて研磨布５が使用限界に達しているか否かを判断し、使用限界に達していないと判断した場合にのみ本来の半導体基板１の研磨を行い、使用限界に達していると判断した場合には研磨布５の交換が行われる。

【０００６】

【発明が解決しようとする課題】しかしながら、従来のＣＭＰ装置には次のような問題があった。

【０００７】それは、上述したドレッシングの際に、ダイヤモンド砥粒電着面からダイヤモンド砥粒が研磨布表面に脱落し、その脱落したダイヤモンド砥粒がその後の研磨の時に半導体基板表面にキズをつけてしまうということである。図９は、図７のダイヤモンド砥粒電着面１３の断面図であるが、図９（ａ）に示すように、ダイヤモンド砥粒電着面１３は、ダイヤモンド砥粒１７をニッケル膜１９に埋め込み、露出しているダイヤモンド砥粒１７により研磨布のドレッシングを行うものであるが、同図（ｂ）に示すように、ドレッシング時の研磨布との摩擦によりニッケル膜１９からダイヤモンド砥粒１７が剥離し、研磨布に脱落してしまうのである。さらに、ドレッサー１１の使用と共にニッケル膜１９が膜減りし、新たにニッケル膜１９表面に露出するダイヤモンド砥粒１７やニッケル膜１９への埋まり具合が浅くなってくるものもあり、上述したダイヤモンド砥粒１７の脱落は次々と起こり得るのである。

【０００８】また、研磨布の使用限界を検知するために半導体基板の研磨直前にテスト研磨が行われるが、その時用いられるテスト基板は再利用することはできず、廃棄しなければならない。一方、半導体基板の価格は、そのサイズの拡大と共に徐々に上昇している。従って、将来コストの増大を招くのは必至である。さらに、テスト研磨及びテスト基板の評価に要する時間は量産ラインにおいて生産効率を向上させる上で非常に問題である。

【０００９】本発明は上記事情に鑑みて成されたものであり、その目的は、ドレッシングの際にダイヤモンド砥粒電着面から研磨布表面に脱落したダイヤモンド砥粒をその後の研磨の時までに除去することにより半導体基板表面にキズをつけることのないＣＭＰ装置を提供することにある。また、その他の目的は、研磨布の使用限界をテスト研磨することなく容易に判断することができるＣＭＰ装置及び研磨布を提供することにある。

【００１０】

【課題を解決するための手段】上記の目的を達成するために本発明の第１の特徴は、底面周辺部に設けられたダイヤモンド砥粒電着面により研磨布表面のドレッシングを行うダイヤモンドドレッサーを有するＣＭＰ装置において、前記ダイヤモンドドレッサーがドレッシングの際に前記ダイヤモンド砥粒電着面から前記研磨布表面に脱落するダイヤモンド砥粒を除去する手段を具備することである。

【００１１】ここで、前記手段は、具体的には、前記ダイヤモンド砥粒電着面の内側に配置される前記研磨布上に脱落したダイヤモンド砥粒を機械的に除去することが可能な材料であり、例えば、ナイロンブラシ、スポンジである。また、ナイロンブラシに比べて毛先の柔らかいモヘアブラシを用いれば、押し付け圧にあまり影響されることなく良好な除去効果を得ることができる。なお、上記材料の外径は、少なくともＣＭＰ処理を行う基板の外径以上であることが必要である。さらに、ドレッシングの際にダイヤモンドドレッサー自体を揺動させる場合には、少なくとも、研磨布表面の内で研磨に使用される領域全体に上記材料が接触することが可能な外径であることが必要である。

【００１２】上記構成によれば、ダイヤモンドドレッサーとナイロンブラシ等の材料を一体化させることにより、ドレッシングの際にダイヤモンド砥粒電着面から研磨布上に脱落するダイヤモンド砥粒の除去をドレッシングと同時に行うことができる。従って、その後の研磨においてダイヤモンド砥粒による基板表面のキズを無くすことができる。

【００１３】本発明の第２の特徴は、表面に凹部が形成された研磨布により基板の研磨を行うＣＭＰ装置において、前記研磨布が該研磨布の使用限界を目視により確認することができる使用限界検出手段を具備することである。

【００１４】ここで、前記使用限界検出手段は、好ましくは、前記凹部の加工量を調節してその他の部分よりも浅く加工された部分であり、前記基板の研磨により前記浅く加工された部分が表面に露出した時点を使用限界と検出するものである。なお、前記浅く加工された部分の厚みは所望の基板の研磨速度と研磨量の面内均一性の値が悪化する時期に研磨布表面に露出して目視で確認することができるような値とする。

【００１５】上記構成によれば、使用限界検出手段が研磨布の表面に露出し目視で確認できた時点を研磨布の交換時期とするので、従来のようなテスト研磨を行う必要が無くなり、また、その際無駄に消費されていたテスト基板も不要となる。従って、コストの低減を図ることが

できる。

【００１６】

【発明の実施の形態】以下，本発明の実施の形態について図面を用いて説明する。図１は，本発明の実施の形態に係るＣＭＰ装置の構成を示す図である。なお，従来例と同一部分には同一符号が付してある。

【００１７】同図に示すように，このＣＭＰ装置は半導体基板１を固定し，半導体基板１を回転させるトップリング３と，半導体基板１の表面を研磨する研磨布５と，研磨布５を固定し，研磨布５を回転させるターンテーブル７とを有し，所定の製造工程を経て凹凸状態となっている半導体基板１の表面を研磨布５に押し付け，砥液９を供給しながらトップリング３とターンテーブル７をそれぞれ回転させ，化学反応を生じさせながら機械的に半導体基板１表面を研磨布５で研磨することにより半導体基板１表面の平坦化を図るものである。また，ドレッシングを行うダイヤモンドドレッサー１１を有し，ダイヤモンドドレッサー１１をターンテーブル７に固定された研磨布５の表面に押し付け，ダイヤモンドドレッサー１１とターンテーブル７それぞれを回転させることにより，半導体基板１の処理枚数の増加に伴って鈍くなっていく研磨布５の目を再び鋭くし，その研磨性能を維持するものである。

【００１８】図２は，図１に示すダイヤモンドドレッサー１１の拡大図である。なお，従来例と同一部分には同一符号が付してある。

【００１９】図２に示すように，本実施の形態に係るダイヤモンドドレッサー１１は，ダイヤモンド砥粒電着面１３の内側に，研磨布上に脱落したダイヤモンド砥粒を機械的に除去することが可能な材料，例えば，ナイロンブラシを配置することにより，ドレッシング時に研磨布表面上に脱落するダイヤモンド砥粒を研磨布表面から除去し，あるいは，研磨布表面の内で半導体基板の研磨に使用されない領域へ除外することにより従来では問題となったダイヤモンド砥粒による半導体基板表面のキズを無くすことができる。また，かかるダイヤモンド砥粒を上記ナイロンブラシに取り込むことにより除去することも可能である。

【００２０】さらに，ダイヤモンドドレッサー自体にナイロンブラシ等の材料が内蔵されているので，従来と同じドレッシング時間だけでドレッシングと同時にダイヤモンド砥粒の除去も行うことができる。従って，全体の処理時間を短くすることができる。

【００２１】ここで，研磨布上に脱落したダイヤモンド砥粒を機械的に除去することが可能な材料としては，ナイロンブラシ以外にスポンジでも同様な効果を得ることができる。また，ナイロンブラシに比べて毛先の柔らかいモヘアブラシを用いることにより，押し付け圧にあまり影響されることなく良好な除去効果を得ることができる。また，上記材料の外径は，少なくともＣＭＰ処理を行う半導体基板の外径以上であることが必要である。さらに，ドレッシングの際にダイヤモンドドレッサー自体を揺動させる場合には，少なくとも，研磨布表面の内で研磨に使用される領域全体に上記材料が接触することが可能な外径であることが必要である。

【００２２】図３は，図１に示す研磨布５の拡大図であり，図３（ａ）がその平面図，図３（ｂ）がそのＡ－Ａ′における断面図である。なお，従来例と同一部分には同一符号が付してある。

【００２３】図３に示すように，本実施の形態に係る研磨布５は，例えば従来例と同様にディンプル加工あるいは格子状の溝加工をすることで表面に凹部が形成されているが，さらに，その加工量を調節してその他の部分よりも浅く加工された部分を形成し，その浅く加工された部分を研磨布５の使用限界サイン２１として用いる構成としたものである。このような構成とすることにより，この研磨布５で半導体基板１の表面を研磨した場合に半導体基板１の処理枚数の増加に伴って研磨布５は磨耗し薄くなると使用限界サイン２１が徐々に表面に現れることとなるが，その目視確認によって研磨布５の使用限界を容易に検知することができるのである。従って，従来では研磨布５の使用限界を検知するために必要であったテスト研磨が不要となり，その際無駄に消費されていたテスト基板も必要なくなる。なお，使用限界サイン２１は研磨布５の凹部内に刻設されてもよいが，上述した通り凹部内で浅く加工された部分を使用限界サイン２１として用いることで使用限界サイン２１を研磨布５の基材と同材質から簡易に得ることができ，好ましい。

【００２４】図４は，図３に示す研磨布５を用いて実際に半導体基板を研磨した結果を示す図であり，図４（ａ）が半導体基板が酸化膜を０．６μｍ堆積したシリコン基板である場合，図４（ｂ）がポリシリコン膜を０．５μｍ堆積したシリコン基板である場合である。また，図４（ａ）及び（ｂ）それぞれの横軸は研磨した半導体基板の枚数であり，縦軸は研磨速度及び研磨量の面内均一性である。なお，ここで用いた研磨布５はポリウレタン材料の研磨布であり，その厚みを約１．３μｍ，使用限界サイン２１の厚みを０．５μｍとしたものである。

【００２５】図４（ａ）に示すように，酸化膜を０．６μｍ堆積したシリコン基板を研磨した場合には，約６００枚研磨した辺りで急激に研磨速度は減少すると共に研磨量の面内均一性が悪化していることが分かる。この時，研磨布５表面には使用限界サイン２１が目視で確認され，それにより，容易に研磨布５が交換時期であることを検知することができた。また，図４（ｂ）に示すように，ポリシリコン膜を０．５μｍ堆積したシリコン基板を研磨した場合には，約９００枚研磨した辺りで急激に研磨速度は減少すると共に研磨量の面内均一性が悪化していることが分かる。この時も同様に，研磨布５表面

には使用限界サイン２１が目視で確認され、それにより、容易に研磨布５が交換時期であることを検知することができた。

【００２６】ここで、研磨布５の材料としてポリウレタンを用いて説明したが、ナイロン、レーヨン等の半導体基板を研磨するために用いられる研磨布材料全てに適用することが可能である。また、使用限界サインの厚みは所望の半導体基板の研磨速度と研磨量の面内均一性の値が悪化する時期に研磨布表面に現れて使用限界サインを目視で確認することができるような厚さに適宜設定する。なお、研磨する半導体基板として酸化膜、ポリシリコン膜を堆積したシリコン基板について説明したが、本実施の形態はこれらの研磨に使用されるのみではなく、一般的にＬＳＩ製造工程で用いられる成膜材料、例えば、高融点金属（Ｓｉ、Ｍｏ、Ｗ、Ｔｉ、Ｔａ等）並びにそれら高融点金属の酸化物、窒化物及びシリサイド、金属配線用材料（Ａｌ、Ｃｕ、Ａｌ－Ｓｉ－Ｃｕ、Ａｌ－Ｃｕ等）の研磨においても適用され得る。

【００２７】

【発明の効果】以上説明したように本発明によれば、ドレッシングを行う際同時に研磨布上に脱落したダイヤモンド砥粒を除去することができるので、従来問題であった脱落したダイヤモンド砥粒による基板表面のキズを無くすことができる。また、ダイヤモンドドレッサーに研磨布上に脱落したダイヤモンド砥粒を除去するナイロンブラシ等の材料を一体化させることにより、ドレッシングとダイヤモンド砥粒の除去を同時に行うことができるので、処理時間の低減を図ることができ、さらに、設備の点からコストの低減を行うことができる。

【００２８】また、研磨布の使用限界を目視で容易に検出することができるので、従来必要であったテスト研磨が不要となり、無駄に消費されていたテスト基板を無く＊

＊すことができる。従って、全体の処理時間は短縮され、コストの低減にもつながる。

【図面の簡単な説明】

【図１】本発明の実施の形態に係るＣＭＰ装置の構成を示す図である。

【図２】図１に示すダイヤモンドドレッサーの拡大図である。

【図３】図１に示す研磨布の拡大図である。

【図４】図３に示す研磨布を用いて実際に半導体基板を研磨した結果を示す図である。

【図５】一般的なＣＭＰ装置の構成を示す図である。

【図６】ドレッシングを行う際のダイヤモンドドレッサーと研磨布を示す図である。

【図７】図６に示すダイヤモンドドレッサーの拡大図である。

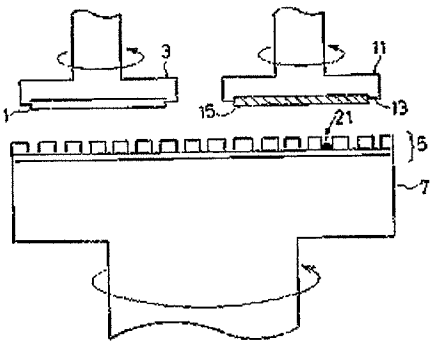
【図８】他の一般的なＣＭＰ装置の構成を示す図である。

【図９】図７に示すダイヤモンド砥粒電着面の断面図である。

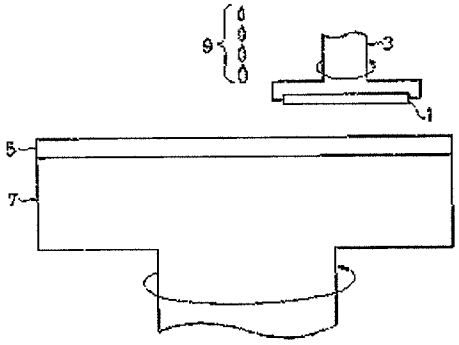
【符号の説明】

１　半導体基板
３　トップリング
５　研磨布
７　ターンテーブル
９　砥液
１１　ダイヤモンドドレッサー
１３　ダイヤモンド砥粒電着面
１５　ナイロンブラシ
１７　ダイヤモンド砥粒
１９　ニッケル膜
２１　使用限界サイン

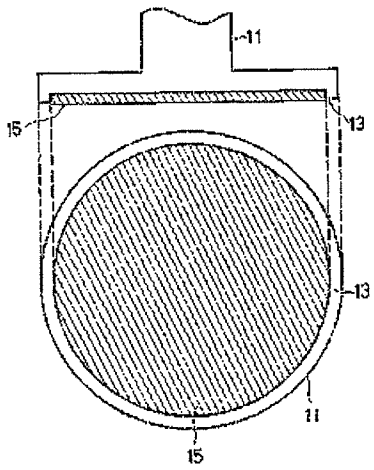
【図１】



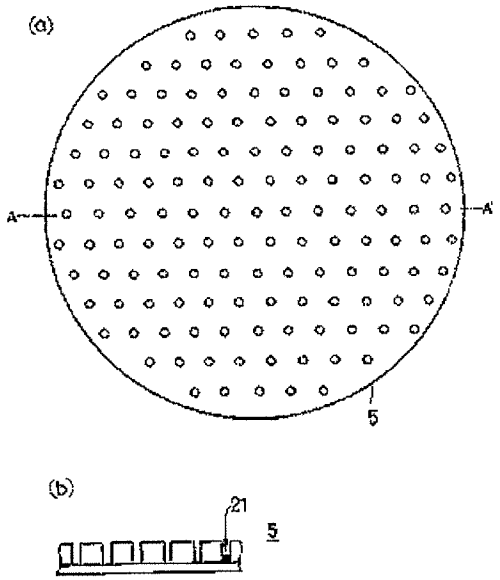
【図５】



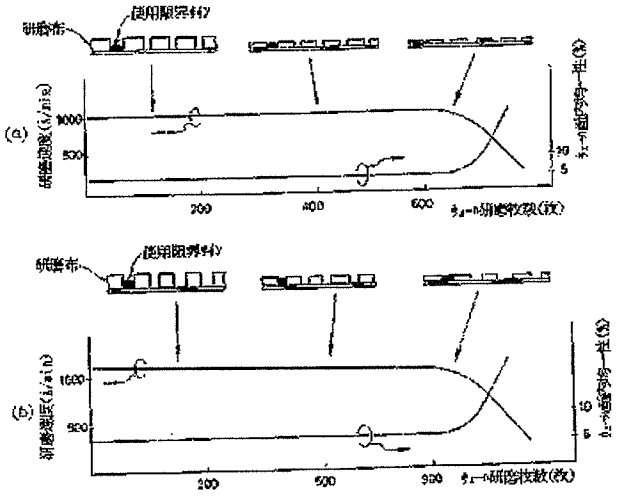
【図2】


11
15
13
13
11
15

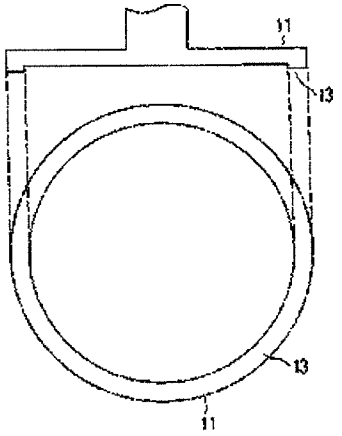
【図3】


(a)
A
A'
5
(b)
21
5

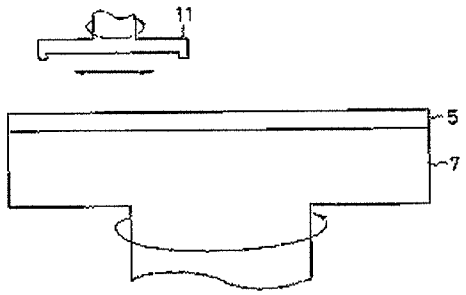
【図4】


研磨布
使用限界47
(a)
1000
500
30
5
200
400
600
研磨枝数(枚)
研磨布
使用限界47
(b)
1000
500
15
5
200
500
900
研磨枝数(枚)

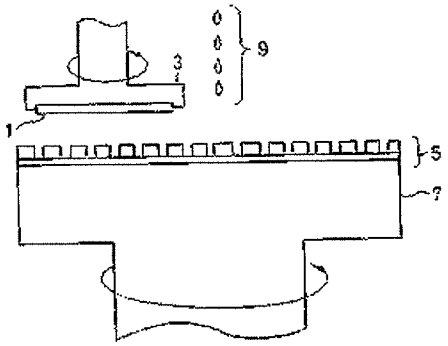
【図7】


11
13
13
11

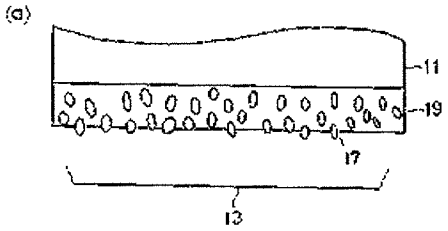
【図6】

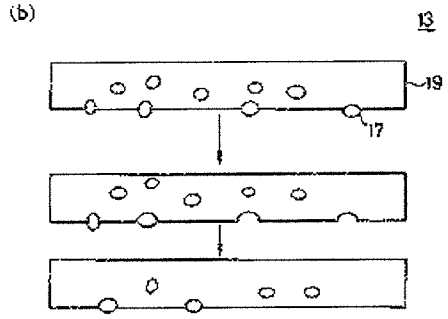


【図8】



【図9】





PATENT ABSTRACTS OF JAPAN

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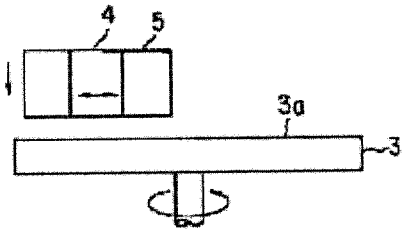
(51)Int.Cl. B24B 37/00
H01L 21/304

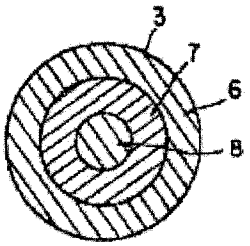
(21)Application number : 08-343567
(22)Date of filing : 24.12.1996

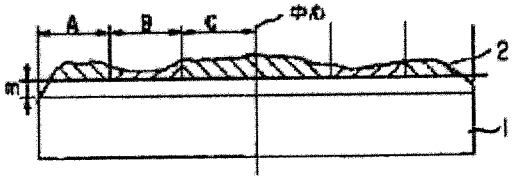
(71)Applicant : NKK CORP
(72)Inventor : TAKEUCHI NOBUYOSHI

(54) GRINDING PAD ADJUSTING METHOD FOR CMP DEVICE

(57)Abstract:
PROBLEM TO BE SOLVED: To maintain the flatness of the device surface of a semiconductor substrate to be ground by dividing the surface of a pad according to the film thickness distribution of the device surface before grinding where the pad surface of the grinding pad is pressed, and adjusting each surface roughness of the pad surface to each different roughness with a plurality of grinding wheels different in hardness.
SOLUTION: The film thickness distribution of a film 2 deposited on the device surface of a semiconductor substrate 1 takes a shape having a doughnut- like recess. According to this film thickness distribution, the inside of a pad surface 3a is divided into, for instance areas A, B and C. A grinding pad 3 needs to adjust surface roughness such that an outer peripheral pad surface part 6 and a center pad surface part 8 for grinding the areas A and C have grinding amounts larger than that of an inner peripheral pad surface part 7 for grinding the area B. Accordingly, a diamond grinding wheel 4 having hardness for reducing a grinding amount more than that of a diamond grinding wheel 5 is loaded, pressed to the pad surface 3a and, then by rotating the grinding pad 3, surface roughness is formed on the pad surface 3a.







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(51)Int.Cl.⁶	識別記号	ＦＩ	
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Ｈ０１Ｌ　21/304	３４１	Ｈ０１Ｌ　21/304	３４１Ｅ

審査請求　未請求　請求項の数３　ＯＬ　（全５頁）

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(22)出願日　平成８年(1996)12月24日

(71)出願人　000004123
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(72)発明者　竹内　信善
東京都千代田区丸の内一丁目１番２号　日本鋼管株式会社内

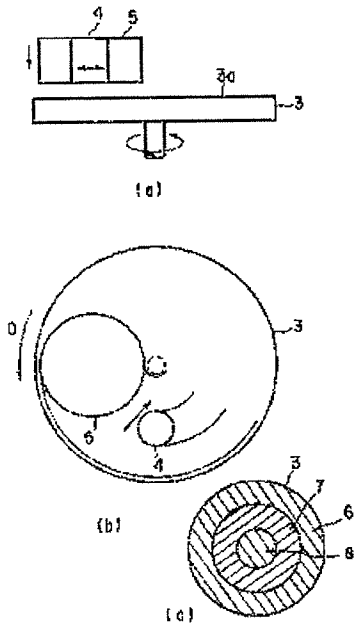
(74)復代理人　弁理士　花輪　義男　（外１名）

(54)【発明の名称】　ＣＭＰ装置の研磨パッドの調整方法

(57)【要約】

【課題】従来の研磨パッドの調整は、パッド面に単一的な表面荒れを形成していたため、半導体装置のデバイス面の凸凹に押し付けられたパッド面が削られ、平坦性がなくなり、表面荒れに研磨の能力の差が生じていた。

【解決手段】本発明は、研磨する半導体基板のデバイス面の膜厚分布を研磨前に測定し、その膜厚分布に対応して、硬度が異なる複数のダイヤモンド砥石を用いて、パッド面内を区分して表面荒れの粗さをそれぞれ調整し、パッド面内での研磨量を変化させることにより、研磨パッドの平坦性を維持させ、研磨される半導体基板のデバイス面の平坦化するＣＭＰ装置の研磨パッドの調整方法である。



1

【特許請求の範囲】

【請求項１】　半導体基板の回路素子を形成するデバイス面を平坦化する化学的機械研磨（ＣＭＰ）装置に用いる研磨パッドの研磨を行うパッド面の調整方法において．

前記研磨パッドのパッド面を研磨前のデバイス面の膜厚分布に応じて．該パッド面内を区分して、硬度の異なる複数の砥石で．該パッド面の表面荒れを異なる粗さにそれぞれ調整することを特徴とするＣＭＰ装置の研磨パッドの調整方法。

【請求項２】　前記ＣＭＰ装置の研磨パッドの調整方法において、

前記デバイス面が平坦化されるまでの研磨量に比例して．そのデバイス面部分に押し付けるパッド面部分の表面荒れが粗くなるように調整することを特徴とする請求項１記載のＣＭＰ装置の研磨パッドの調整方法。

【請求項３】　前記ＣＭＰ装置の研磨パッドの調整方法において、

硬度が異なる砥石を回転する研磨パッドの動径方向にそれぞれ配置して．前記パッド面に押し付け、パッド面を区分してそれぞれ研磨し．異なる粗さの表面荒れに調整することを特徴とする請求項１記載のＣＭＰ装置の研磨パッドの調整方法。

【発明の詳細な説明】

【０００１】

【発明の属する技術分野】本発明は．半導体デバイスの製造工程において、半導体基板表面の平坦化を行う化学的機械研磨（ＣＭＰ：Chemical Mechanical Polishing）技術に係り、特にＣＭＰ装置における研磨パッドの調整方法に関する。

【０００２】

【従来の技術】近年、処理時間の高速化や微細化の要求に伴い半導体デバイスの回路素子は積み上げられて多層化し．配線層も多層化されている。

【０００３】このような回路素子等の多層化は．半導体基板上で回路素子を形成した領域と未形成の領域との間に段差を生じさせ、さらに積み上げる毎にそれらの段差が加算される。この結果．上層側では．フォトリソグラフィ工程のマスク露光の際に、焦点深度を越えたり、エッチング困難な場合が生じる。さらに．例えばビアホールにメタル配線を形成した場合、段差によりビアホールの深さに違いが生じ、ビアホール抵抗のばらつきの原因にもなっている。

【０００４】そこで、各層に回路素子を形成する際に、半導体基板表面の平坦化を図る技術が求められていた。

【０００５】従来の平坦化技術としては、エッチバック技術やＳＯＧ（Spin On Glass）技術が知られている。これらの技術は．段差近傍のローカルな平坦化には有用であった。しかし、半導体デバイスの多層化や大型化が進むと半導体基板全体を平坦化する技術が必要である。

2

【０００６】エッチバック技術による平坦化は．オーバーエッチングの管理が難しく、半導体基板のデバイス面全体を平坦化させるには．容易でなかった。さらに、層間絶縁膜のような同一材料が全表面を覆った面を平坦化するには問題がないが、例えばＣｕ等を含有する配線のようにエッチングされにくい部材を使用した配線等が含まれる構成であれば、エッチングにより平坦化することは難しい。

【０００７】また、ＳＯＧ技術においては、ＳＯＧ膜をスピンコーティングして塗布し、段差の平坦化を実現していたが、ＳＯＧ膜自体のクラック耐性が乏しく．半導体基板全体を平坦化させる膜厚までは形成できない問題があり、さらに塗布により平坦化させるためには．回路素子の表面形状や配置に工夫を施さねばなかった。このため．多層化された回路素子や多層化配線には．十分な平坦化を提供できる技術ではなくなった。

【０００８】そこで新たな平坦化技術として、インゴットからスライスカットされたシリコンウエハの平面研磨を行うポリッシング研磨技術と化学研磨技術の特徴を併せ持たせ、半導体デバイスの平坦化に応用したＣＭＰ技術が注目されている。

【０００９】このＣＭＰ技術は、図４に示すように、ポリッシング研磨における研磨剤として．アルミナやシリカ等の研磨粒子に過酸化水素水等の化学物質を併せた研磨剤（スラリー液）２１を用いて、定盤２２上に軟質若しくは、粘弾性を有する材料からなる研磨パッド２３を取り付け、化学反応を伴う機械研磨によりデバイス面側を微細に研磨して段差を無くし、表面を平坦化するものである。

【００１０】この構成において、揺動可能なテーブル２４に表面側から吸着固定された半導体基板２５は．そのデバイス面２５ａを対向し回転する研磨パッド２３に押しつけられ、研磨剤２１が添加されて研磨され．デバイス面が平坦化される。このＣＭＰ技術は、研磨レートが小さいため微細な研磨が可能であり．研磨された面に加工変質を極めてわずかしか与えない特徴がある。

【００１１】

【発明が解決しようとする課題】前述したＣＭＰ技術において、研磨に用いられる研磨パッドの表面は．砥石例えば．ダイヤモンド砥石で削られ、適当な表面荒れが設けられている。この研磨パッドの表面荒れにより．デバイス面上に積層形成された絶縁膜又は金属膜を研磨を行っているが、ダイヤモンド砥石自らの表面も削られるため．表面荒れが削り取られて研磨能力が劣化する。

【００１２】そこで、研磨パッドは．予め定めた処理回数（削った量）毎に、その表面を再度．ダイヤモンド砥石で削り直し．適当な表面荒れが保たれるように調整を行っている。

【００１３】通常、研磨前のデバイス面は、いわゆるａｓ depoの状態にあり、その膜厚分布は下地の平坦さによ

る影響と、成膜装置の特性に依存する面内膜厚分布の凸凹形状があり．ＣＭＰ技術において研磨パッドによる研磨を行った場合．研磨後の研磨パッドもこの凸凹形状に依存して平坦な表面の状態が変化する。つまり．膜なりに削れていくのである。

【００１４】例えば、図５に示すようなデバイス面では．凸形状の部分２５ｂに押しつけられた研磨パッド部分２３ｂは多く削れ、表面荒れの部分も削り取られる。また．凹形状の部分２５ｃを押し付けられた研磨パッド部分２３ｃはあまり削れないため、表面荒れの部分も残っている。

【００１５】この削れ具合の差により．研磨後の研磨パッド表面は、偏った凸凹形状に変化し．且つ研磨パッド内で研磨の能力の差が生じている。従って、このように表面形状が変化していく研磨パッドで．引き続きデバイス面を研磨すると、半導体基板の表面も平坦にならない場合が生じる。

【００１６】また従来の研磨パッドの調整は、研磨パッド表面の平坦化維持を考慮したものではなく、単一的な表面荒れを形成すること目的としているため、研磨パッドの表面全体を１つのダイヤモンド砥石で均一的に削るだけであった。

【００１７】よって、研磨パッドの表面荒れの均一性を持たせる管理を厳しくして．研磨パッドのパッド面の平坦性を維持させ．研磨された半導体基板のデバイス面の平坦化を維持させるために．研磨パッドの交換回数や調整回数が多くなり、作業効率の低下を招き、結果的にコストアップとなる。

【００１８】そこで本発明は、半導体基板のデバイス面の膜厚分布に応じて、研磨パッドのパッド面内を区分して表面荒れの粗さをそれぞれ調整し．研磨面の平坦性と研磨された半導体基板のデバイス面の平坦化を維持させるＣＭＰ研磨パッドの調整方法を提供することを目的とする。

【００１９】

【課題を解決するための手段】本発明は上記目的を達成するために、半導体基板の回路素子を形成するデバイス面を平坦化する化学的機械研磨（ＣＭＰ）装置に用いる研磨パッドの研磨を行うパッド面の調整方法において、前記研磨パッドのパッド面を押し付ける研磨前のデバイス面の膜厚分布に応じて．該パッド面内を区分して、硬度の異なる複数の砥石で．該パッド面の表面荒れを異なる粗さにそれぞれ調整するＣＭＰ装置の研磨パッドの調整方法を提供する。

【００２０】以上のようなＣＭＰ研磨パッドの調整方法は．研磨前の半導体基板のデバイス面の膜厚分布に対応して．硬度が異なる複数の砥石により．デバイス面の膜の研磨量に比例して、そのデバイス面に押し付けられるパッド面の表面荒れが粗くなるように研磨して．パッド面内での研磨量を変化させることにより、研磨される半導体基板のデバイス面の平坦化を維持する。

【００２１】

【発明の実施の形態】以下．図面を参照して本発明の実施形態について詳細に説明する。

【００２２】まず、本発明によるＣＭＰの研磨パッドの調整方法の概要について説明する。図２（ａ）には、半導体基板上に回路素子を形成するための絶縁膜や金属膜をＣＶＤ（Chemical Vaper Deposition）装置やスパッタリング装置を用いて半導体基板上に形成した場合の膜厚分布の一例を示す。

【００２３】この半導体基板１のデバイス面上に堆積された堆積膜２の膜厚分布の形状は、同心円状で．半導体基板１の中心で厚く、エッヂ方向に向かい一旦薄くなるが．次第に厚くなり、再度エッヂで薄くなる。すなわち．ドーナツ形状の凹みを有した形状である。

【００２４】このような膜厚分布を持つ半導体基板をＣＭＰ技術により平坦化させる場合、図２（ｂ）に示すように．斜線で示すＡ領域及びＣ領域の膜厚が近似し、仮に膜厚ｍに平坦化するものとすると．Ａ領域及びＣ領域は．研磨量を多くし、Ｂ領域は、研磨量を少なくすることにより、効率的に平坦化できる。

【００２５】従って、半導体基板１のデバイス面に押し付けられ研磨を行う研磨パッドのパッド面（研磨面）は．膜厚分布と逆の削れ具合の調整が必要となる。つまり後述する図１（ｃ）に示すように．堆積膜の厚いＡ領域及びＣ領域に押し付けられるパッド面部分６及びパッド面部分８は．表面荒れを粗くして研磨レートを上げるように調整し．堆積膜が薄く研磨量が少ないＢ領域に押し付けられるパッド面部分７は、その表面荒れを研磨パッド部分６に比べて、粗くせずに研磨レートを下げるように調整する。

【００２６】このような調整を行うためには研磨パッドのパッド面内を区分して．それぞれの表面荒れの粗さに調整する必要がある。

【００２７】図１（ａ）、（ｂ）に示すように．図２（ｂ）に示したような膜厚分布に対応する、少なくとも硬度が異なる２つのダイヤモンド砥石４、５を研磨パッド３の動径方向Ｏに配置して、研磨パッドのパッド面３ａの表面荒れを調整する。少なくともダイヤモンド砥石４は移動可能に備えられている。

【００２８】この膜厚分布は、予め成膜装置毎に膜厚分布の形状に関するデータを取って、パーソナルコンピュータ等に記録させておき．研磨の際に読み出して利用してもよいし、その都度、半導体基板のデバイス面の膜厚分布を計測してもよい。

【００２９】本実施形態では、図２（ｂ）に示した堆積膜の膜厚分布を例とすると．研磨パッド３は、図１（ｃ）に示すように半導体基板１のＡ領域及びＣ領域を研磨する外周部のパッド面部分６及び中心部のパッド面部分８が、Ｂ領域を研磨する内周部のパッド面部分７よ

りも研磨量が多くなるように、表面荒れの粗さを調整する必要がある。

【００３０】その調整方法は、ダイヤモンド砥石５に比べて、研磨量が異る硬度のダイヤモンド砥石４を装着してパッド面３ａに押し付け、研磨パッド３を回転させて、パッド面３ａに表面荒れを形成する。

【００３１】本実施形態の研磨パッドの調整方法によれば、ＣＭＰ装置に装着して、半導体基板のデバイス面を平坦化する研磨パッド内の表面荒れの粗さを、デバイス面の膜厚分布（研磨すべき量）に基づき、研磨量の多い部分ほど粗くなるように調整することにより、パッド面の平坦化を維持し、且つ同じ膜厚分布の傾向を持つデバイス面を研磨する処理回数（研磨量）を増やすことができる。

【００３２】また、表面荒れの粗さは、研磨量が研磨剤や研磨機構及び研磨を行う堆積膜の材質や成膜方法により異なるため、経験的に求めて決定する。

【００３３】次に図３を参照して、本発明による第２の実施形態としてのＣＭＰ研磨パッドの調整方法について説明する。

【００３４】前述した第１の実施形態では、硬度が異なる２つのダイヤモンド砥石により、研磨パッドを調整したため、２種類の粗さの表面荒れが形成されたが、図２（ｂ）に示した堆積膜２の膜厚分布で、例えば、Ｃ領域の膜厚がＡ領域の膜厚より、さらに厚かった場合には、Ｃ領域の堆積膜の研磨量をＡ領域よりも多くする必要がある。そこで、図３（ａ），（ｂ）に示すように、硬度が異なる３つのダイヤモンド砥石１１，１２，１３を研磨パッド３の動径方向に配置し、研磨パッド３の表面荒れの調整を行う。このダイヤモンド砥石１１，１２，１３の取り付け機構においては、砥石の交換や研磨パッドに対する研磨位置の移動が可能な構成になっているものとする。

【００３５】この調整は、図３（ｃ）に示すような半導体基板１の膜厚分布において、堆積膜２を膜厚ｎに平坦化する際に、Ｃ’領域の研磨量ｑがＡ’領域の研磨量ｐよりも２倍程度ある場合など、半導体基板の外周側と中心部の膜厚に差があり、前述した第１の実施形態による硬度が２種類のダイヤモンド砥石では、十分な平坦化が実現されない膜厚分布に好適する。このような調整において、砥石の配置は、ダイヤモンド砥石１２、ダイヤモンド砥石１１、ダイヤモンド砥石１３の順に研磨量が多くなる硬度ものを配置する。

【００３６】以上のことから本実施形態は、第１の実施形態に比べて、パッド面内の表面荒れの粗さをさらに微

調整でき、研磨する堆積膜の膜厚分布に依らず、平坦化が実現でき、第１の実施形態と同様な効果が得られる。

【００３７】本実施形態では、硬度が異なる２若しくは３個のダイヤモンド砥石を用いて説明したが、これらに限定されるものではなく、半導体基板の径やデバイス面の膜厚分布、膜の材質により、自由に硬度の選択と配置をしてもよく、また、砥石側を回転させてパッド面を押し付け研磨してもよい。

【００３８】尚、従来、ＣＭＰ装置の研磨機構では研磨パッドだけでなく、研磨を施す半導体基板側も回転させ、且つ半導体基板支持体を動径方向に揺動する研磨機構もあり、半導体基板のエッヂ側周辺部は、研磨パッドの外周側と内周側とで研磨されるが半導体基板中心部は揺動中心近くで研磨されている。このような研磨機構では、半導体基板の外周側が中心部分よりも、研磨量が多くなっていたが、本実施形態の調整方法を利用して、研磨パッドの表面荒れを調整し、中心部分の研磨量を多くすることにより、この問題を解決できる。

【００３９】

【発明の効果】以上詳述したように本発明によれば、半導体基板のデバイス面の膜厚分布に応じて、研磨パッドのパッド面内を区分して表面荒れの粗さをそれぞれ調整し、研磨面の平坦性と研磨された半導体基板のデバイス面の平坦化を維持させるＣＭＰ研磨パッドの調整方法を提供することができる。

【図面の簡単な説明】

【図１】本発明による第１の実施形態としてのＣＭＰ研磨パッドの調整方法を実施するための研磨パッドと砥石の配置の構成例を示す図である。

【図２】成膜装置を用いて形成した時の堆積膜の膜厚分布の一例を示す図である。

【図３】本発明による第２の実施形態としてのＣＭＰ研磨パッドの調整方法を実施するための研磨パッドと砥石の配置の構成例を示す図である。

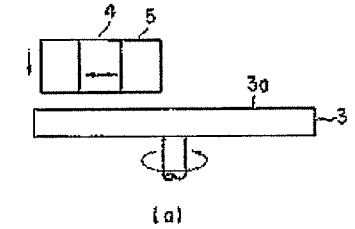
【図４】ＣＭＰ装置の概略的な構成を示す図である。

【図５】従来の半導体基板と研磨パッドの研磨状態を説明するための図である。

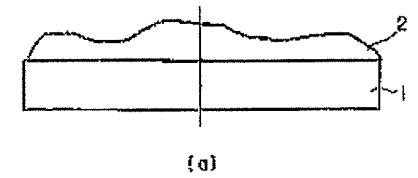
【符号の説明】

１…半導体基板
２…堆積膜
３…研磨パッド
３ａ…パッド面
４，５…ダイヤモンド砥石
６，７，８…研磨パッド部分

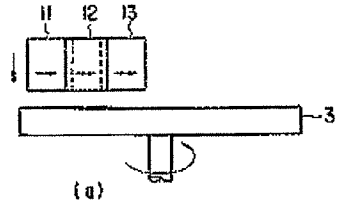
【図1】


4
5
3a
3
(a)
3
0
5
4
(b)
3
7
6
8
(c)

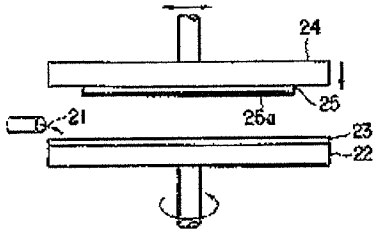
【図2】


2
1
(a)
A
B
C
中心
2
1
(b)

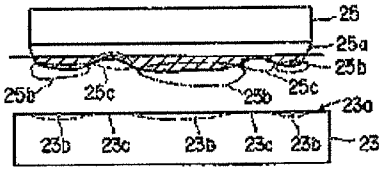
【図3】

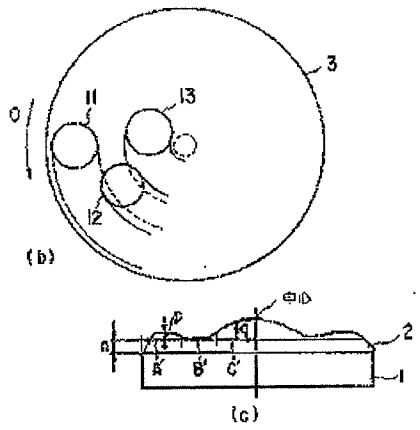

11
12
13
3
(a)

【図4】


24
25
26a
21
23
22

【図5】


25
25a
25b
25b
25c
25c
25b
23a
23b
23c
23b
23c
23b
23


3
0
11
13
12
(b)
中心
2
1
A'
B'
C'
(c)

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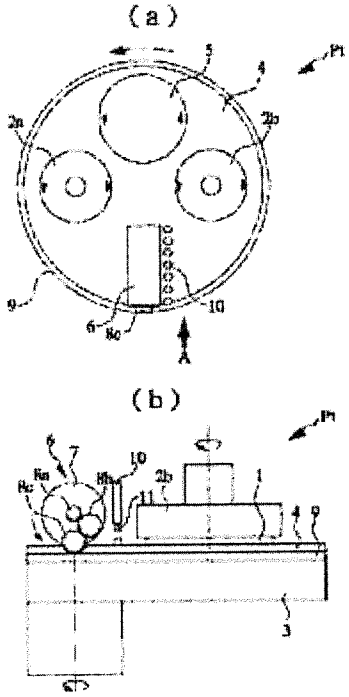
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(54) CHEMICAL-MECHANICAL POLISHING DEVICE, AND MANUFACTURE OF SEMICONDUCTOR INTEGRATED CIRCUIT DEVICE USING SAME

(57)Abstract:
PROBLEM TO BE SOLVED: To provide a polishing pad of long life in a chemical-mechanical polishing(CMP) technique, as well as to polish uniformly a whole surface of a polished film on a semiconductor wafer at stable polishing speed.
SOLUTION: After a surface of a polishing pad 4 is ground by the first dressor 5 with embedded diamond particles to achieve flatness, a surface of a polished film on a semiconductor wafer 1 is polished, and a sodding grain on a surface of the polishing pad 4 is set up simultaneously by the second dressor 6 composed of a cylindrical brush 7 while supplying a polishing abrasive solution through a supplying nozzle 10 so as to restore an original coarse sodding grain.



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Ｈ０１Ｌ　21/304	３２１	Ｈ０１Ｌ　21/304	３２１Ｅ

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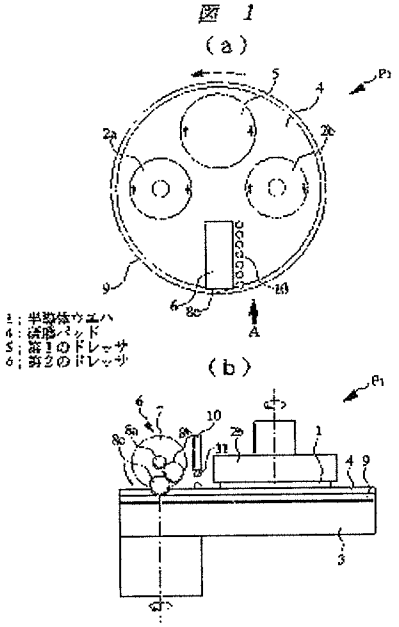
(54)【発明の名称】　化学的機械研磨装置およびこれを用いた半導体集積回路装置の製造方法

(57)【要約】

【課題】　ＣＭＰ技術において、寿命の長い研磨パッドを得ると同時に、安定した研磨速度で半導体ウエハ上の被研磨膜の全面を均一に研磨する。

【解決手段】　ダイヤモンド粒子が埋め込まれた第１のドレッサ５で研磨パッド４の表面を切削して平坦度を出した後、半導体ウエハ１上の被研磨膜の表面を研磨すると同時に、供給ノズル１０から研磨砥粒溶液１１を供給しながら円筒状のブラシ７によって構成される第２のドレッサ６で研磨パッド４の表面の芝めを立たせて、元の荒い芝めを復元する。

図　1



1

【特許請求の範囲】

【請求項１】　研磨定盤上に貼り付けられた研磨パッドに半導体ウエハを押し付けて前記半導体ウエハ上に設けられた各種膜の表面を研磨する化学的機械研磨装置であって、前記研磨パッドの表面を整形するドレッサが、少なくとも２種類設けられていることを特徴とする化学的機械研磨装置。

【請求項２】　請求項１記載の化学的機械研磨装置において、前記研磨パッドの表面を整形するドレッサとして、少なくとも前記研磨パッドの表面を平坦化する第１のドレッサと、前記研磨パッドの表面の荒さを復元させる第２のドレッサとが設けられていることを特徴とする化学的機械研磨装置。

【請求項３】　請求項２記載の化学的機械研磨装置において、前記第１のドレッサはダイヤモンド粒子が埋め込まれたドレッサであり、前記第２のドレッサはポリマ繊維からなるブラシによって構成されたドレッサであることを特徴とする化学的機械研磨装置。

【請求項４】　請求項３記載の化学的機械研磨装置において、前記半導体ウエハ上に設けられた各種膜の表面を研磨すると同時に、前記研磨パッドの表面に研磨砥粒溶液を供給しながら、前記第２のドレッサを前記研磨パッドの表面に押さえ付けて断続的または連続的に摺動させることを特徴とした化学的機械研磨装置。

【請求項５】　請求項４記載の化学的機械研磨装置において、前記第２のドレッサの前記研磨パッドに対する摺動方向が、少なくとも前記半導体ウエハの摺動方向とほぼ逆方向の成分を有していることを特徴とする化学的機械研磨装置。

【請求項６】　請求項４記載の化学的機械研磨装置において、前記研磨砥粒溶液の供給口が前記第２のドレッサに内蔵されていることを特徴とする化学的機械研磨装置。

【請求項７】　請求項２～６のいずれか１項に記載の化学的機械研磨装置において、前記第２のドレッサが前記研磨パッドの研磨面に対して平行な回転軸を有する円筒状のドレッサであることを特徴とする化学的機械研磨装置。

【請求項８】　請求項７記載の化学的機械研磨装置において、前記第２のドレッサが強制的に回転されることを特徴とする化学的研磨装置。

【請求項９】　請求項７項に記載の化学的機械研磨装置において、前記第２のドレッサの円周速度は、前記研磨パッドが貼り付けられた研磨定盤の最外周部の円周速度よりも速いことを特徴とする化学的機械研磨装置。

【請求項１０】　請求項１～９のいずれか１項に記載の化学的機械研磨装置を用いた半導体集積回路装置装置の製造方法であって、前記第１のドレッサおよび前記第２のドレッサで整形される前記研磨パッドによって、半導体ウエハ上に設けられた各種膜の表面を平坦に加工する工程を有することを特徴とする半導体集積回路装置の製造方法。

2

【発明の詳細な説明】

【０００１】

【発明の属する技術分野】本発明は、半導体集積回路装置の製造技術に関し、特に、半導体ウエハ上に堆積された絶縁膜または金属膜の表面の凹凸を化学的機械研磨（Chemcal Mechanical Polishing；ＣＭＰ）装置を用いて平坦に加工する技術に関するものである。

【０００２】

【従来の技術】従来のＣＭＰ装置Ｐ₂の要部断面の模式図を図５に示す。図において、５１は半導体ウエハ、５２は剛性板、５３は低剛性の合成樹脂から成る加圧用パッド、５４は剛性板５２にその厚み方向に複数個設けられた通気孔、５５は剛性板５２に設けられた通気孔５４と一致する如く加圧用パッド５３に配列された通気孔、５６は加圧ヘッド、５７は研磨パッド、５８は研磨パッドが貼り付けられた研磨定盤、５９は半導体ウエハ５１が研磨中に加圧ヘッド５６から外れないように設けられたストッパである。

【０００３】半導体ウエハ５１は、剛性板５２および加圧用パッド５３にそれぞれ設けられた通気孔５４、５５からの真空吸引により加圧ヘッド５６に装着された後、研磨パッド５７上に押し付けられる。研磨時の圧力は加圧ヘッド５６、剛性板５２、加圧用パッド５３を介して半導体ウエハ５１に加えられる。半導体ウエハ５１は加圧ヘッド５６と共に回転し、同じく回転する研磨定盤５８に貼り付けられた研磨パッド５７に押さえつけながら研磨される。

【０００４】次に、前記ＣＭＰ装置Ｐ₂を用い、半導体ウエハ上に堆積された絶縁膜または金属膜の表面の凹凸を平坦化する製造工程の一例を図６（ａ）～（ｆ）を用いて説明する。

【０００５】これらの図において、６０は半導体基板、６１は第１層目の配線、６２は層間絶縁膜、６３は層間絶縁膜６２に開孔されたスルーホール、６４は金属膜、６５は第２層目の配線である。

【０００６】まず、半導体基板６０上に、例えばトランジスタ等の半導体素子、電荷蓄積用容量素子または抵抗素子（いずれも図示せず）などを形成した後、これらを互いに電気的に接続する第１層目の配線６１を形成する（工程（ａ））。この際、半導体基板６０の表面には第１層目の配線６１の厚み分の凸部が生ずる。

【０００７】次に、ＣＶＤ（Chemical Vapor Deposition）法を用いて酸化シリコン膜または窒化シリコン膜などによって構成される層間絶縁膜６２を半導体基板６０上に堆積させて、上記第１層目の配線６１を層間絶縁膜６２で覆う（工程（ｂ））。この際、ほぼ前記第１層目の配線６１の厚み分の段差が層間絶縁膜６２の表面に残留する。次いで、前記ＣＭＰ装置Ｐ₂を用いて、層間絶

縁膜６２の表面を研磨して平坦に加工する（工程（ｃ））。

【０００８】次に、第１層目の配線６１に達するスルーホール６３を層間絶縁膜６２に設けた後、ＣＶＤ法またはスパッタリング法などによって半導体基板６０上に金属膜６４を堆積し（工程（ｄ））、次いで、前記ＣＭＰ装置Ｐ₁を用いて層間絶縁膜６２の表面に積層された金属膜６４を研磨除去する。これによって、スルーホール６３に金属膜６４を埋め込む（工程（ｅ））。

【０００９】次に、半導体基板６０上に金属膜（図示せず）を堆積した後、この金属膜をパターニングすることによって第２層目の配線６５を形成する（工程（ｆ））。第１層目の配線６１と第２層目の配線６５とはスルーホール６３に埋め込まれた金属膜６４を介して電気的に接続される。また、上記工程（ａ）～（ｆ）を同様に繰り返すことにより所望の多層配線を有する半導体集積回路装置を形成することができる。

【００１０】ところで、上記工程（ｃ）での層間絶縁膜６２の表面の平坦化および上記工程（ｅ）での金属膜６４の表面の平坦化で要求される研磨技術の基本的性能としては、被研磨膜の凹凸の平坦度、研磨量の均一性、スループットおよび研磨速度の安定性が挙げられる。これらのうち、被研磨膜の凹凸の平坦度は使用する研磨パッドの機械的材料、研磨量の均一性はウエハ加圧方式によってほぼ決定されるのに対し、スループットおよび研磨速度の安定性は、研磨砥粒の材質、加圧力または揺動速度などの研磨条件に加えて、研磨パッドの表面状態に強く依存する。

【００１１】特に、本発明者の実験により、研磨速度の安定性は研磨パッドの表面の荒さの変化と強い相関関係があることが明らかとなった。図７は、本発明者の実験結果の一例であり、半導体ウエハの処理枚数に対する半導体ウエハ上の被研磨膜の研磨速度の変化および研磨パッドの表面の荒さの変化を示すグラフ図である。

【００１２】図に示すように、半導体ウエハの処理枚数が増加するに従い、研磨速度は初期に急激に低下し、その後、なだらかに低下した後、ほぼ一定の速度に安定する。また、研磨速度の変化と同様に、研磨パッドの表面の荒さも半導体ウエハの処理枚数が増加するに従い減少した後、ほぼ一定の荒さに落ち着く。

【００１３】この現象は、研磨パッドの表面の荒さが減少すると研磨砥粒液の保持力が低下して研磨パッドの表面に供給された研磨砥粒液が半導体ウエハの揺動により半導体ウエハの外周端で掻き出されてしまい、半導体ウエハ上の研磨面と研磨パッド面との間に存在する研磨砥粒の量が減少して、研磨速度が低下するものと考えられる。なお、研磨パッドの表面の荒さが減少する理由は、研磨パッドの表面の芝め構造が、研磨中に繰り返し受ける半導体ウエハの研磨圧力によって倒れてしまうためである。

【００１４】そこで、研磨中における研磨パッドの表面状態の経時変化を防ぎ、半導体ウエハの表面の研磨速度を一定に保つために、半導体ウエハ上の被研磨膜を研磨する加圧ヘッドとは別に、ダイヤモンド粒子を埋め込んだ円盤（以下、ドレッサと称す）を研磨作業後または研磨作業中に研磨パッド上に摺動させて、研磨パッドの表面を整形するドレッシングが研磨パッドに施されている。

【００１５】図８および図９に従来の研磨パッドのドレッシング方法の概略図を示す。図８に示すドレッシング方法は、大型のドレッサ６６に回転と揺動を加えながら研磨パッド５７の表面を整形する方法であり、図９に示すドレッシング方法は、アーム機構６７を用いて小型のドレッサ６８を研磨パッド５７の全面に可変速で揺動させて、研磨パッド５７の表面を整形する方法である。これらのドレッシング方法には、半導体ウエハ上の被研磨膜の研磨と研磨パッドの表面のドレッシングとを同時に行う同時ドレッシング、または半導体ウエハ上の被研磨膜の研磨前後の半導体ウエハを搬送する空き時間に研磨パッドの表面のドレッシングを行う間欠ドレッシングがある。

【００１６】なお、ＣＭＰ装置のドレッシング方法については、例えばソリッド・ステート・テクノロジ（Solid State Technology, 日本版,Dec. 1994, Iqbal Ali et al.,"層間絶縁膜の化学的機械研磨に関する概観"）などに記載されている。

【００１７】

【発明が解決しようとする課題】しかしながら、前記ドレッシング方法には、以下の問題点があることを本発明者は見いだした。

【００１８】すなわち、まず、同時ドレッシングでは、研磨パッドの表面が常にドレッシングされるので安定な研磨速度が得られるが、研磨パッドの表面が常にドレッサによって削り取られるため研磨パッドの寿命が著しく低下する。また、間欠ドレッシングでは、研磨パッドの寿命は長いが、半導体ウエハ上の被研磨膜の研磨中に研磨速度が低下して安定な研磨速度が得られず、さらに、ドレッシングに要する時間が単独で必要なためＣＭＰ装置のスループットが低下する。

【００１９】これらの問題を解決する方法の一つとして、研磨パッドに対するドレッサの押圧力を低下させる方法がある。この方法を採用すれば、研磨速度をある程度安定化させ、研磨パッドの寿命を長くすることができる。

【００２０】しかし、ドレッシングには、研磨パッドの表面の荒さを一定に保ち研磨速度を安定化させる目的の他に、研磨パッドの全面をドレッシングでの切削によって整形し、研磨パッドの表面の平坦度を得ることにより半導体ウエハ上の被研磨膜の全面の均一性を確保する目的があり、研磨パッドに対するドレッサの押圧力を低下

させると研磨パッドの表面の平坦度を確保することが難しく、半導体ウエハ上の被研磨膜の全面を均一に研磨することができない問題がある。

【００２１】本発明の目的は、ＣＭＰ技術において、寿命の長い研磨パッドを得ると同時に、安定した研磨速度で半導体ウエハ上の被研磨膜の全面を均一に研磨することができる技術を提供することにある。

【００２２】本発明の前記ならびにその他の目的と新規な特徴は、本明細書の記述および添付図面から明らかになるであろう。

【００２３】

【課題を解決するための手段】本願において開示される発明のうち、代表的なものの概要を簡単に説明すれば、次のとおりである。

【００２４】すなわち、本発明のＣＭＰ装置は、研磨定盤上に貼り付けられた研磨パッドの表面を整形するドレッサとして、少なくとも、ダイヤモンド粒子が埋め込まれ、研磨パッドの表面を平坦化する第１のドレッサと、ポリマ繊維からなるブラシによって構成され、研磨パッドの表面の荒さを復元させる第２のドレッサとを有しており、上記第１のドレッサにより研磨パッドの表面を切削した後、第１のドレッサを後退させ、次いで、加圧ヘッドに装着された半導体ウエハを研磨パッドの表面に押さえ付けて半導体ウエハ上の被研磨膜の表面を研磨すると同時に、研磨パッドの表面に研磨砥粒溶液を供給しながら上記第２のドレッサを断続的または連続的に研磨パッドの表面に押さえ付けて、研磨パッドの表面の芝めを立たせる方向に摺動させるものである。

【００２５】上記した手段によれば、第２のドレッサによるドレッシングによって、研磨パッドの表面を切削することなく、半導体ウエハ上の被研磨膜の表面を研磨することによって倒れた研磨パッドの表面の芝めを立たせて、比較的容易に元の荒い芝めに復元できるので、研磨パッドの寿命を大幅に長くできると同時に、半導体ウエハ上の被研磨膜の研磨速度を安定化することができる。さらに、研磨パッドに対する第１のドレッサの押圧力を低下させる必要がないので、研磨パッドの表面の平坦度が確保できて、半導体ウエハ上の被研磨膜の全面を均一に研磨することができる。

【００２６】

【発明の実施の形態】以下、本発明の実施の形態を図面に基づいて詳細に説明する。

【００２７】なお、実施の形態を説明するための全図において同一機能を有するものは同一の符号を付し、その繰り返しの説明は省略する。

【００２８】本発明の一実施の形態であるＣＭＰ装置Ｐ，が有する研磨パッドのドレッシング方法を図１に示す模式図を用いて説明する。図１（ａ）は、ＣＭＰ装置Ｐ，の上面図であり、図１（ｂ）は、図１（ａ）中に記載のＡ方向から見たＣＭＰ装置Ｐ，の側面図である。１は

半導体ウエハ、２ａ，２ｂは加圧ヘッド、３は研磨定盤、４は研磨パッド、５は第１のドレッサ、６は第２のドレッサ、７はブラシ、８ａ，８ｂ，８ｃは歯車、９はギア、１０は供給ノズル、１１は研磨砥粒溶液である。

【００２９】被研磨材料である半導体ウエハ１は、加圧ヘッド２ａ，２ｂに装着され、回転する研磨定盤３上に張り付けられた研磨パッド４の表面に加圧ヘッド２ａ，２ｂを介して押さえ付けられて、その表面は研磨される。なお、研磨時の加圧ヘッド２ａ、２ｂおよび研磨定盤３の回転数は、例えば共に３０回／分であり、半導体ウエハ１および研磨定盤３の直径は、例えばそれぞれ８インチおよび６００ｎｍである。半導体ウエハ１は、例えば３００ｇｒ／ｃｍ² の圧力で研磨パッド４の表面に押さえ付けられている。

【００３０】研磨パッド４の表面は、第１のドレッサ５および第２のドレッサ６を用いてドレッシングされる。第１のドレッサ５にはダイヤモンド粒子が埋め込まれており、第１のドレッサ５は研磨パッド４の表面を切削して平坦度を出すために用いられる。第２のドレッサ６は研磨パッドの研磨面に対して平行な回転軸を有する円筒状のブラシ７と歯車８ａ，８ｂ，８ｃとによって構成されており、研磨パッド４の表面の芝めを立たせて元の荒い芝めを復元するために用いられる。

【００３１】第２のドレッサ６の歯車８ｃを研磨定盤３の外周壁面に形成したギア９と噛み合わせて研磨定盤３を回転させることにより、歯車８ｂを介して歯車８ａと一体のブラシ７を強制的に回転駆動させる。この際、第２のドレッサ６の研磨パッド４に対する摺動方向は、半導体ウエハ１の摺動方向とほぼ逆方向の成分を有している。また、ブラシ７の円周速度は研磨定盤３の最外周部の円周速度より約1.5倍速くなるように、歯車８ｃのギア比とブラシ７の径は選ばれる。ブラシ７には、例えば線径１００μｍ、長さ５ｍｍのナイロンブラシを使用した。

【００３２】まず、第１のドレッサ５を用いて研磨パッド４の表面を切削した後、第１のドレッサ５を後退させ、次いで、半導体ウエハ１上の被研磨膜の表面を研磨すると同時に、第２のドレッサ６を断続的または連続的に研磨パッド４上で摺動させて、研磨パッド４の表面を整形する。この際、第２のドレッサ６の近傍に設置した供給ノズル１０から研磨砥粒溶液１１が約２００ｍｌ／分の速度で研磨パッド４上に供給される。研磨砥粒溶液１１の供給口は第２のドレッサ６に内蔵してもよい。

【００３３】図２に、本実施の形態である前記ＣＭＰ装置Ｐ，を用いて、半導体ウエハ上にプラズマＣＶＤ法で堆積された酸化シリコン膜の表面を研磨した際の酸化シリコン膜の研磨速度と半導体ウエハの研磨枚数との関係を示す。図において、四角印は前記本実施の形態のドレッシング、白丸印は従来の同時ドレッシング、黒丸印は従来の間欠ドレッシングを採用した際のそれぞれの酸化

シリコン膜の研磨速度を示す。

【００３４】いずれの場合も一つの研磨定盤に対して２つの加圧ヘッドを有し、２枚の半導体ウエハを同時に研磨した結果であり、図中には２枚の半導体ウエハで得られたそれぞれの酸化シリコン膜の研磨速度の平均値を１点で示してある。半導体ウエハ上の酸化シリコン膜の研磨時間は５分である。

【００３５】本実施の形態のドレッシングでは、半導体ウエハ１上の酸化シリコン膜の研磨前の第１のドレッサ５によるドレッシング時間は３分であり、半導体ウエハ１上の酸化シリコン膜の研磨中は第２のドレッサ６でドレッシングを行っている。研磨パッドには厚さが1.2ｍｍの発泡ポリウレタン性のＩＣ１０００（米国ロデール社）を用いた。なお、従来の間欠ドレッシングのドレッシング時間は２分である。

【００３６】図から明らかなように、従来の間欠ドレッシングの研磨速度は他のドレッシングの研磨速度と比べて約１０％低く、また、±５％以上の研磨速度の変動がある。しかし、本実施の形態のドレッシングでは半導体ウエハの研磨枚数が２５枚まで従来の同時ドレッシングとほぼ同じ研磨速度が得られ、また、±２％以下の研磨量の均一性（図示せず）が得られる。

【００３７】図３に、本実施の形態である前記ＣＭＰ装置P_1を用いて、半導体ウエハ上に堆積された酸化シリコン膜を研磨した際の研磨パッドの厚さと半導体ウエハの研磨枚数との関係を示す。図において、四角印は前記本実施の形態のドレッシング、白丸印は従来の同時ドレッシング、黒丸印は従来の間欠ドレッシングを採用した際のそれぞれの研磨パッドの厚さを示す。

【００３８】なお、本発明者の実験から、研磨パッドをその厚さが初期の厚さ（例えば1.2ｍｍ）の約半分になるまで研磨すると、半導体ウエハ上の被研磨膜の研磨速度の安定性または半導体ウエハ上の被研磨膜の研磨量均一性に顕著な劣化が認められることが明らかになっており、このことから、研磨パッドの厚さが半分となった時点を研磨パッドの寿命と称す。

【００３９】図に示すように、従来の同時ドレッシングでは約１００枚の半導体ウエハを研磨するだけで研磨パッドは寿命に達し、従来の間欠ドレッシングでも約２５０枚の半導体ウエハを研磨すると研磨パッドは寿命に達する。これに対し、本実施の形態のドレッシングでは、約４０００枚の半導体ウエハを研磨すると研磨パッドが寿命に達する。

【００４０】次に、本実施の形態の前記ＣＭＰ装置P_1を用いたＤＲＡＭ（Dynamic RandomAccess Memory）の多層配線の製造方法を図４を用いて説明する。なお、ＤＲＡＭの周辺回路部はｎチャネル型ＭＩＳＦＥＴ（Meta l Insulator SemiconductorField Effect Transistor）Q_sの製造方法のみを記載する。

【００４１】まず、p^-型シリコン単結晶からなる半導体基板１２の主面上に周知の方法でｐ型ウエル１３、フィールド絶縁膜１４およびゲート絶縁膜１５を順次形成する。

【００４２】次に、図示はしないが、半導体基板１２上に堆積された窒化シリコン膜、タングステンシリサイド（$WS_{1.x}$）膜および多結晶シリコン膜からなる積層膜を順次エッチングすることにより、$WS_{1.x}$膜および多結晶シリコン膜からなるメモリセルのメモリセル選択用ＭＩＳＦＥＴのゲート電極１６および周辺回路部のｎチャネル型ＭＩＳＦＥＴQ_sのゲート電極１６を形成する。

【００４３】なお、上記ゲート電極１６を構成するメタルシリサイド膜に$WS_{1.x}$膜を用いたが、その他のメタルシリサイド膜、例えばモリブデンシリサイド（$MoS_{1.x}$）膜、チタンシリサイド（$TiS_{1.x}$）膜、タンタルシリサイド（$TaS_{1.x}$）膜などを用いてもよい。

【００４４】次に、レジストパターンならびに上記窒化シリコン膜、$WS_{1.x}$膜および多結晶シリコン膜からなる積層膜をマスクにして、周辺回路部のｐ型ウエル１３にｎ型不純物、例えばリン（Ｐ）をイオン注入し、ｎチャネル型ＭＩＳＦＥＴQ_sのｎ型半導体領域（ソース領域、ドレイン領域）１７を、ゲート電極１６に対して自己整合で形成する。

【００４５】その後、半導体基板１２上に堆積された窒化シリコン膜をＲＩＥ（Reactive Ion Etching）法などの異方性エッチングで加工することによって、ゲート電極１６の側壁にサイドウォールスペーサを形成し、ゲート電極１６を窒化シリコン膜からなる絶縁膜１８で覆う。

【００４６】なお、上記サイドウォールスペーサを形成した後、周辺回路部のｐ型ウエル１３に高濃度のｎ型不純物、例えば砒素（Ａｓ）をイオン注入することにより、ｎチャネル型ＭＩＳＦＥＴQ_sのソース領域、ドレイン領域をＬＤＤ（Lightly Doped Drain）構造としてもよい。

【００４７】次に、半導体基板１２上に酸化シリコン膜１９および第１のＢＰＳＧ（Boron-doped Phospho Silicate Glass）膜２０をＣＶＤ法によって堆積した後、９００～９５０℃のリフロー処理により上記第１のＢＰＳＧ膜２０の表面を平坦化する。

【００４８】その後、レジストパターンをマスクにして第１のＢＰＳＧ膜２０、酸化シリコン膜１９およびゲート絶縁膜１５と同一層の絶縁膜を順次エッチングすることにより、メモリセル選択用ＭＩＳＦＥＴの一方の後に形成されるｎ型半導体領域２１上に第１のコンタクトホール２２を形成する。

【００４９】次いで、上記第１のコンタクトホール２２内にＰが導入された多結晶シリコン膜２３からなる第１プラグ電極を形成する。なお、この多結晶シリコン膜２３に導入されたＰの拡散によってメモリセル選択用ＭＩ

ＳＦＥＴの一方のｎ型半導体領域２１が形成される。

【００５０】次に、半導体基板１２上に酸化シリコン膜２４をＣＶＤ法によって堆積する。次いで、図示はしないが、レジストパターンをマスクにして酸化シリコン膜２３、第１のＢＰＳＧ膜２０、酸化シリコン膜１９およびゲート絶縁膜１５と同一層の絶縁膜を順次エッチングすることにより、メモリセル選択用ＭＩＳＦＥＴの他方の後に形成されるｎ型半導体領域上に第２のコンタクトホールを形成する。この際、後に形成されるビット線を周辺回路部に延在し、周辺回路部の半導体基板１２に接続するための第２のコンタクトホール（周辺回路部）２５を形成してもよい。

【００５１】次いで、半導体基板１２上にＰが導入された多結晶シリコン膜２６およびWSi_x膜２７をＣＶＤ法によって順次堆積した後、レジストパターンをマスクにしてWSi_x膜２７および多結晶シリコン膜２６を順次エッチングすることにより、WSi_x膜２７および多結晶シリコン膜２６からなるビット線を形成する。

【００５２】また、多結晶シリコン膜２６に導入されたＰの拡散によってメモリセル選択用ＭＩＳＦＥＴの他方のｎ型半導体領域（図示せず）は形成され、ビット線は第２のコンタクトホールを通して、このメモリセル選択用ＭＩＳＦＥＴの他方のｎ型半導体領域に接続される。この際、多結晶シリコン膜２６に導入されたＰの拡散によって周辺回路部のｐ型ウエル１３にもｎ型半導体領域（周辺回路部）２８が形成され、第２のコンタクトホール（周辺回路部）２５を通して、ビット線が上記ｎ型半導体領域（周辺回路部）２８に接続される。

【００５３】次に、半導体基板１２上に酸化シリコン膜２９、窒化シリコン膜３０および第２のＢＰＳＧ膜（図示せず）をＣＶＤ法によって順次堆積した後、９００～９５０℃のリフロー処理により上記第２のＢＰＳＧ膜の表面を平坦化する。

【００５４】次に、半導体基板１２上にＰが導入された多結晶シリコン膜３１をＣＶＤ法によって堆積した後、レジストパターンをマスクにして多結晶シリコン膜３１をエッチングする。次いで、半導体基板１２上にＣＶＤ法によって堆積されたＰが導入された多結晶シリコン膜３２をＲＩＥ法などの異方性エッチングによって加工し、上記多結晶シリコン膜３１の側壁に多結晶シリコン膜３２からなるサイドウォールスペーサを形成する。

【００５５】次いで、レジストパターンをマスクにしてメモリセルの第２のＢＰＳＧ膜、窒化シリコン膜３０、酸化シリコン膜２９および酸化シリコン膜２４を順次エッチングすることにより、第１のコンタクトホール２２内に設けられた第１プラグ電極上に第３のコンタクトホール３３を形成した後、半導体基板１２上にＰが導入された多結晶シリコン膜３４および第３のＢＰＳＧ膜（図示せず）をＣＶＤ法によって順次堆積する。

【００５６】次に、レジストパターンをマスクにして上記第３のＢＰＳＧ膜および多結晶シリコン膜３４，３１を順次エッチングした後、半導体基板１２上にＰが導入された多結晶シリコン膜３５をＣＶＤ法によって堆積する。次いで、この多結晶シリコン膜３５をＲＩＥ法などの異方性エッチングによって加工し、メモリセルの第３のＢＰＳＧ膜および多結晶シリコン膜３４，３１の側壁に多結晶シリコン膜３５を残す。

【００５７】次に、例えば、フッ酸溶液を用いたウエットエッチングによって、第３のＢＰＳＧ膜および第２のＢＰＳＧ膜を除去し、メモリセルに多結晶シリコン膜３１，３２，３４，３５からなる円筒型の蓄積電極を形成する。

【００５８】次に、半導体基板１２上に厚さ約２ｎｍの窒化シリコン膜（図示せず）をＣＶＤ法によって堆積し、続いて、厚さ約３０ｎｍの非晶質の酸化タンタル（Ta_2O_5）膜（図示せず）をＣＶＤ法によって堆積した後、半導体基板１２に熱酸化処理を施すことによって、上記Ta_2O_5膜を結晶化する。その後、半導体基板１２上にＴｉＮ膜３６をＣＶＤ法によって堆積し、次いで、フォトレジストをマスクにしてこのＴｉＮ膜３６をエッチングすることにより、ＴｉＮ膜３６からなるプレート電極を形成する。

【００５９】なお、容量絶縁膜にTa_2O_5膜を用いたが、その他の酸化メタル膜（例えば、（Ｂａ，Ｓｒ）ＴｉＯ膜またはＰｂ（Ｚｒ，Ｔｉ）O_3膜）などを用いてもよく、また、上記プレート電極にＴｉＮ膜を用いたが、その他のメタルナイトライド膜（例えば、ＷＮ膜）またはメタル膜（例えば、Ｗ膜）などを用いてもよい。

【００６０】次に、半導体基板１２上に酸化シリコン膜３７および第４のＢＰＳＧ膜３８をＣＶＤ法によって順次堆積した後、９００～９５０℃のリフロー処理により上記第４のＢＰＳＧ膜３８の表面を平坦化する。

【００６１】次いで、レジストパターンをマスクにして第４のＢＰＳＧ膜３８および酸化シリコン膜３７を順次エッチングすることにより、ＴｉＮ膜３６からなるプレート電極上に第４のコンタクトホール３９ａを形成し、同時に、第４のＢＰＳＧ膜３８、酸化シリコン膜３７および酸化シリコン膜２９を順次エッチングすることにより、WSi_x膜２７および多結晶シリコン膜２６からなるビット線上に第４のコンタクトホール３９ｂを形成する。

【００６２】さらに、同時に、第４のＢＰＳＧ膜３８、酸化シリコン膜３７、酸化シリコン膜２９、酸化シリコン膜２４、第１のＢＰＳＧ膜２０、酸化シリコン膜１９およびゲート絶縁膜１５と同一層の絶縁膜を順次エッチングすることにより、ｎチャネル型ＭＩＳＦＥＴQ_sのｎ型半導体領域１７上に第４のコンタクトホール３９ｃを形成する。

【００６３】次に、半導体基板１２上に金属膜（図示せず）を堆積した後、レジストパターンをマスクにして上

記金属膜をエッチングすることにより．第１層目のメタル配線M_1が形成される。次いで、半導体基板１２上にＥＣＲ（Electron CyclotronResonance：電子サイクロトロン共鳴）プラズマＣＶＤ法によって酸化シリコン膜を堆積した後．この酸化シリコン膜の表面を本実施の形態である前記ＣＭＰ装置P_1を用いて研磨することによって．その表面が平坦化された酸化シリコン膜によって構成される第１の層間絶縁膜４０を設ける。

【００６４】次に、レジストパターンをマスクにして上記第１の層間絶縁膜４０をエッチングすることにより、第１層目のメタル配線M_1に達するスルーホール４１を形成した後、半導体基板１２上に金属膜（図示せず）を堆積し、次いで．この金属膜をレジストパターンをマスクにしてエッチングすることにより．第２層目のメタル配線M_2を形成する。

【００６５】ここで、第１の層間絶縁膜４０の表面が平坦化されているので、第２層目のメタル配線M_2を形成する際のフォトリソグラフィ工程において、マスクパターン転写における焦点深度に余裕が生じ、高解像度のレジストパターンが形成される。これによって、レジストパターンをマスクとして加工、形成される第２層目のメタル配線M_2の欠損または隣接する第２層目のメタル配線M_2間の接触を防ぐことができる。

【００６６】さらに、上記第１の層間絶縁膜４０と同様に．半導体基板１２上にＥＣＲプラズマＣＶＤ法によって酸化シリコン膜を堆積した後、この酸化シリコン膜の表面を本実施の形態である前記ＣＭＰ装置P_1を用いて研磨することによって、その表面が平坦化された酸化シリコン膜によって構成される第２の層間絶縁膜４２を設ける。

【００６７】次に、レジストパターンをマスクにして上記第２の層間絶縁膜４２をエッチングすることにより、第２層目のメタル配線M_2に達するスルーホール４２を形成した後、半導体基板１２上に金属膜（図示せず）を堆積し、次いで．この金属膜をレジストパターンをマスクにしてエッチングすることにより．第３層目のメタル配線M_3を形成する。

【００６８】最後に、半導体基板１２の表面をパッシベーション（図示せず）で被覆することにより、本実施の形態のＣＭＰ装置P_1を用いて加工された第１の層間絶縁膜４０および第２の層間絶縁膜４２を有するＤＲＡＭが完成する。

【００６９】このように．本実施の形態では、研磨パッド４の寿命が．従来の同時ドレッシングを採用した研磨パッドの寿命の約４０倍以上、また．従来の間欠ドレッシングを採用した研磨パッドの寿命の約１６倍以上長くなり．例えば．従来の同時ドレッシングでは４～５時間毎に必要があった研磨パッドの交換を週に１度の頻度とすることができる。さらに．第２のドレッサ６のブラシ７を研磨パッド４の表面の芝めを立たせる方向に摺動させることによって、研磨パッド４の表面の倒れた芝めを立たせて、比較的容易に元の荒い芝めに復元することができるので、研磨速度を安定化することができる。さらに．研磨パッド４に対する第１のドレッサ５の押圧力を低下させる必要がないので．研磨パッド４の表面の平坦度が確保できて．半導体ウエハ１上の被研磨膜の全面を均一に研磨することができる。

【００７０】以上、本発明者によってなされた発明を発明の実施の形態に基づき具体的に説明したが、本発明は前記実施の形態に限定されるものではなく、その要旨を逸脱しない範囲で種々変更可能であることはいうまでもない。

【００７１】

【発明の効果】本願によって開示される発明のうち、代表的なものによって得られる効果を簡単に説明すれば、以下のとおりである。

【００７２】本発明によれば、ブラシによって構成された第２のドレッサによるドレッシングによって．研磨パッドの表面を切削することなく、比較的容易に研磨パッドの表面の芝めを荒い芝めに復元することができるので．研磨パッドの寿命を大幅に長くできると同時に、半導体ウエハ上の被研磨膜の表面の研磨速度を安定化することができ、さらに、ダイヤモンド粒子を埋め込んだ第１のドレッサの研磨パッドに対する押圧力を低下させる必要がないので．研磨パッドの表面の平坦度が確保できて．半導体ウエハ上の被研磨膜の全面を均一に研磨することができる。

【００７３】また、本発明によれば．研磨パッドの寿命が長くなるので．半導体製造装置としての生産能力、稼働率が大幅に向上し、また．高価な研磨パッドの消費量が減少する。

【図面の簡単な説明】

【図１】本発明の一実施の形態であるＣＭＰ装置の模式図であり、（ａ）は上面図．（ｂ）は要部側面図である。

【図２】本発明の一実施の形態であるドレッシング、従来の同時ドレッシングまたは従来の間欠ドレッシングをそれぞれ採用した研磨パッドを有するＣＭＰ装置を用いて．半導体ウエハ上の酸化シリコン膜の表面を研磨した際の酸化シリコン膜の研磨速度の経時変化を示すグラフ図である。

【図３】本発明の一実施の形態であるドレッシング、従来の同時ドレッシングまたは従来の間欠ドレッシングをそれぞれ採用した研磨パッドを有するＣＭＰ装置を用いて．半導体ウエハ上の酸化シリコン膜の表面を研磨した際の研磨パッドの厚さの経時変化を示すグラフ図である。

【図４】本発明の一実施の形態であるドレッシングを採用した研磨パッドを有するＣＭＰ装置を用いて平坦化される層間絶縁膜を有するＤＲＡＭを示す半導体基板の要

部断面図である。

【図５】従来のＣＭＰ装置の要部側面の模式図である。

【図６】（ａ）～（ｆ）はそれぞれ従来のドレッシングを採用した研磨パッドを有するＣＭＰ装置を用いて平坦化される絶縁膜または金属膜を有する半導体基板の要部断面図である。

【図７】従来のドレッシングを採用した研磨パッドを有するＣＭＰ装置を用いて研磨された半導体ウエハ上の被研磨膜の研磨速度と研磨パッドの荒さの経時変化を示すグラフ図である。

【図８】従来の研磨パッドのドレッシング方法を説明するための概略図である。

【図９】従来の研磨パッドのドレッシング方法を説明するための概略図である。

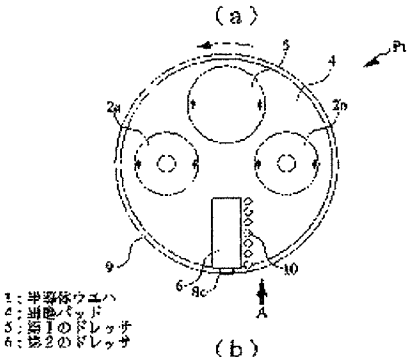
【符号の説明】

１　半導体ウエハ
２ａ　加圧ヘッド
２ｂ　加圧ヘッド
３　研磨定盤
４　研磨パッド
５　第１のドレッサ
６　第２のドレッサ
７　ブラシ
８ａ　歯車
８ｂ　歯車
８ｃ　歯車
９　ギア
１０　供給ノズル
１１　研磨砥粒溶液
１２　半導体基板
１３　ｐ型ウエル
１４　フィールド絶縁膜
１５　ゲート絶縁膜
１６　ゲート電極
１７　ｎ型半導体領域（ソース領域，ドレイン領域）
１８　絶縁膜
１９　酸化シリコン膜
２０　第１のＢＰＳＧ膜
２１　ｎ型半導体領域
２２　第１のコンタクトホール
２３　多結晶シリコン膜
２４　酸化シリコン膜
２５　第２のコンタクトホール（周辺回路部）
２６　多結晶シリコン膜

２７　タングステンシリサイド膜
２８　ｎ型半導体領域（周辺回路部）
２９　酸化シリコン膜
３０　窒化シリコン膜
３１　多結晶シリコン膜
３２　多結晶シリコン膜
３３　第３のコンタクトホール
３４　多結晶シリコン膜
３５　多結晶シリコン膜
３６　窒化チタン膜
３７　酸化シリコン膜
３８　第４のＢＰＳＧ膜
３９ａ　第４のコンタクトホール
３９ｂ　第４のコンタクトホール
３９ｃ　第４のコンタクトホール
４０　第１の層間絶縁膜
４１　スルーホール
４２　第２の層間絶縁膜
４３　スルーホール
５１　半導体ウエハ
５２　剛性板
５３　加圧用パッド
５４　通気孔
５５　通気孔
５６　加圧ヘッド
５７　研磨パッド
５８　研磨定盤
５９　ストッパ
６０　半導体基板
６１　第１層目の配線
６２　層間絶縁膜
６３　スルーホール
６４　金属膜
６５　第２層目の配線
６６　大型のドレッサ
６７　アーム機構
６８　小型のドレッサ
P_1　本発明の実施の形態のＣＭＰ装置
P_2　従来のＣＭＰ装置
Q_5　ｎチャネル型ＭＩＳＦＥＴ
M_1　第１層目のメタル配線
M_2　第２層目のメタル配線
M_3　第３層目のメタル配線

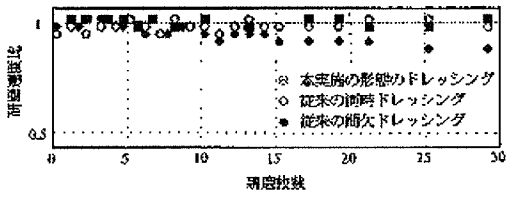
【図１】

図 1



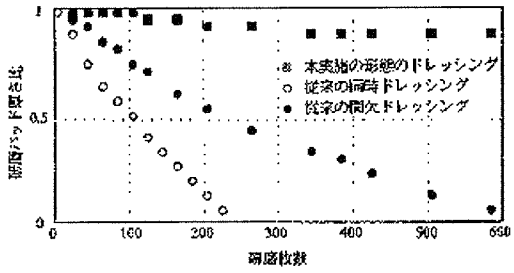
【図２】

図 2



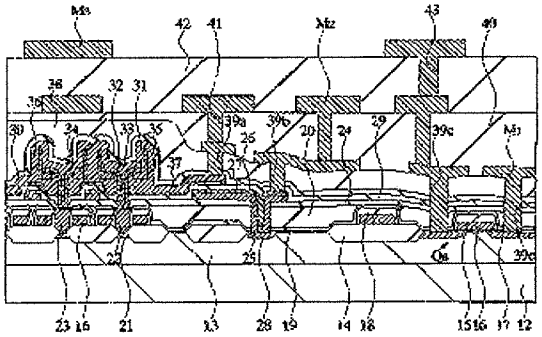
【図３】

図 3



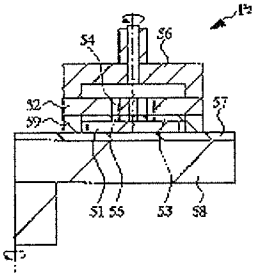
【図４】

図 4



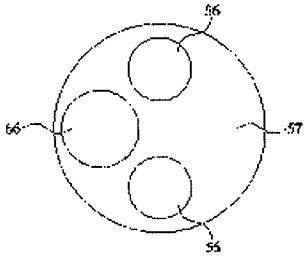
【図５】

図 5



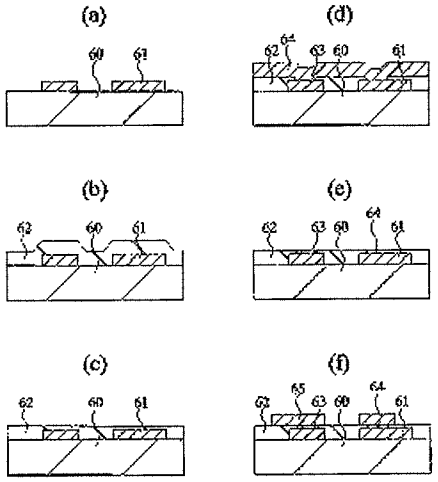
【図８】

図 8



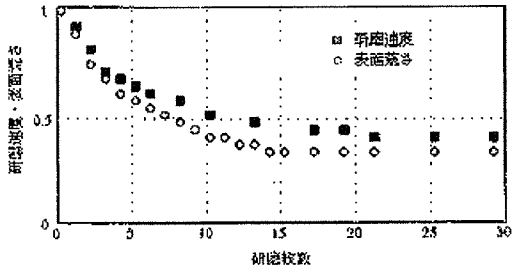
【図６】

図 6



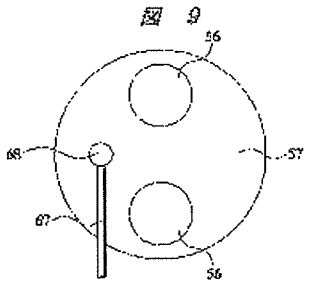
【図７】

図 7



【図９】

図 9



フロントページの続き

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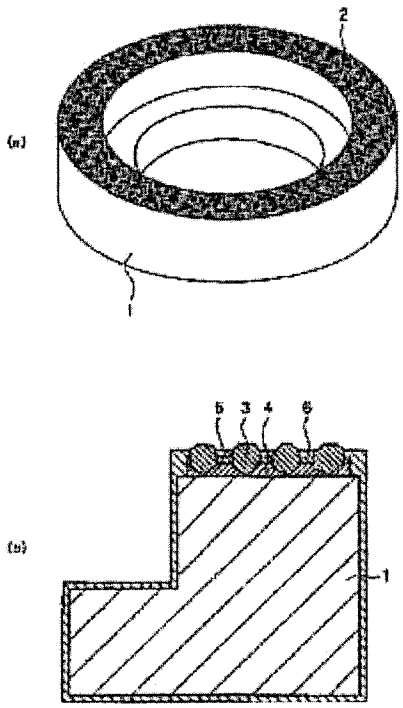
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(71)Applicant : ASAHI DIAMOND IND CO LTD
(72)Inventor : HARA TOMOYOSHI

(54) CONDITIONER FOR CMP AND ITS MANUFACTURE

(57)Abstract:
PROBLEM TO BE SOLVED: To prevent a plated layer fixing diamond abrasive grains from being corroded even by highly acidic polishing liquid and to prevent contamination caused by metallic elution, and falling off of diamond abrasive grains.
SOLUTION: This CPM conditioner has diamond abrasive grains 3 fixed by a plated layer 4, and is applied with nitric acid resistant plating to the plated layer 4; coated its surface with a synthetic resin, and having action tip parts of the diamond abrasive grains projected out of the synthetic resin layer 6. The CPM conditioner is manufactured by fixing the diamond abrasive grains 3 to an action surface by an electrodeposition or a reversible electroforming followed by nitric acid resistant plating 5 and covering with synthetic resin its action surface.



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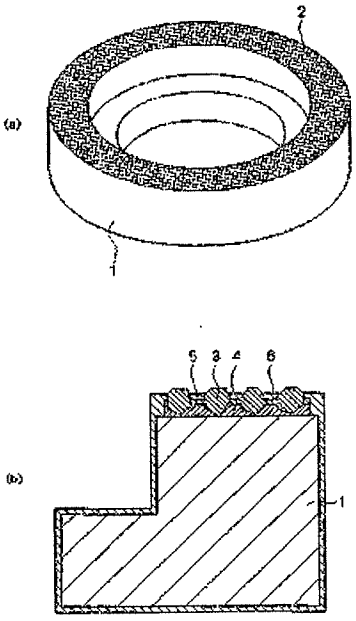
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(54)【発明の名称】　ＣＭＰ用コンディショナ及びその製造方法

(57)【要約】
【課題】半導体ウェーハ研磨用のポリッシングマットのコンディショニングにおいて、酸性の高い研磨液によってもダイヤモンド砥粒を固着するメッキ層が侵されるおそれがなく、金属の溶出によるコンタミネーションやダイヤモンド砥粒の脱落を生じないＣＭＰ用コンディショナ及びその製造方法を提供する。
【解決手段】ダイヤモンド砥粒がメッキ層により固着されたコンディショナにおいて、メッキ層上に耐硝酸性のメッキが施され、その表面がさらに合成樹脂により被覆され、ダイヤモンド砥粒の先端作用部が合成樹脂層より突出してなることを特徴とするＣＭＰ用コンディショナ、及び、作用面にダイヤモンド砥粒を電着法又は反転電鋳法によって固着したのち、さらに耐硝酸性のメッキを施し、その表面を合成樹脂により被覆することを特徴とする該ＣＭＰ用コンディショナの製造方法。



1

【特許請求の範囲】

【請求項１】ダイヤモンド砥粒がメッキ層により固着されたコンディショナにおいて、メッキ層上に耐硝酸性のメッキが施され、その表面がさらに合成樹脂により被覆され、ダイヤモンド砥粒の先端作用部が合成樹脂層より突出してなることを特徴とするＣＭＰ用コンディショナ。

【請求項２】耐硝酸性のメッキが、アルミニウム、クロム、金、ロジウム、錫又はこれらの合金のメッキである請求項１記載のＣＭＰ用コンディショナ。

【請求項３】作用面にダイヤモンド砥粒を電着法又は反転電鋳法によって固着したのち、さらに耐硝酸性のメッキを施し、その表面を合成樹脂により被覆することを特徴とする請求項１又は請求項２記載のＣＭＰ用コンディショナの製造方法。

【発明の詳細な説明】

【０００１】

【発明の属する技術分野】本発明は、ＣＭＰ用コンディショナ及びその製造方法に関する。さらに詳しくは、本発明は、半導体ウェーハ研磨用のポリッシングマットのコンディショニングにおいて、酸性の高い研磨液によってもダイヤモンド砥粒を固着するメッキ層が侵され、ダイヤモンド砥粒が脱落するおそれのないＣＭＰ用コンディショナ及びその製造方法に関する。

【０００２】

【従来の技術】一般に半導体ウェーハの表面を研磨するウェーハ加工装置では、円盤状の定盤の上に研磨用ポリッシングマットを貼り付け、定盤上面に１枚又は複数枚のウェーハを載置し、これらウェーハをポリッシングマット上でキャリアにより強制回転させつつポリッシングマットとウェーハの間に微細な研磨粒子と研磨液を供給して、界面の化学的機械的作用によりケミカルメカニカルポリッシング（ＣＭＰ）を行っている。ポリッシングマットとしては、ポリエステル不織布にポリウレタン樹脂を含浸させたベロアタイプマット、ポリエステル不織布を基材としてその上に発泡ポリウレタン層を形成したスエードタイプマット、あるいは独立気泡を有する発泡ポリウレタンのマットなどや、さらにこれらの多層構造体などが使用されている。また、研磨粒子としては、酸化鉄、アルミナ、炭酸バリウム、酸化セリウム、コロイダルシリカなどが用いられ、研磨液には水酸化カリウム溶液、希塩酸、過酸化水素水、硝酸鉄水溶液など、それぞれの場合に応じて使い分けられる。このようなウェーハの研磨を繰り返すうちに、研磨粒子や研磨屑などがポリッシングマットの微細な孔に入り込んで目詰まりを起こしたり、研磨粒子と研磨液の化学反応熱によってポリッシングマットの表面が鏡面化して、研磨速度が低下してしまう。このため、ポリッシングマットの表面を再生して研磨速度を回復させる、いわゆるコンディショニングと呼ばれる操作を常時又は定期的に行う必要があり、

2

このような操作にはＣＭＰ用コンディショナと呼ばれる工具が使用される。ダイヤモンド砥粒は優れたコンディショニング材料であり、ダイヤモンド砥粒を利用した半導体ウェーハ研磨用のポリッシングマットのコンディショニングが検討されている。例えば、特開昭６４－７１６６１号公報には、ダイヤモンド砥粒と合金粉末を混合し、加熱焼結したダイヤモンドペレットを端面に貼り付けるか、あるいは、端面にダイヤモンド砥粒を均一に分布するように載せて電着した修正リングを用い、ポリッシングマットと修正リングを相対移動させることによりポリッシングマットの表面を研削して平坦度を高める方法が提案されている。しかし、合金粉末を用いて焼結したダイヤモンドペレットからは、コンディショニングの際に合金が溶出してウェーハの研磨時にウェーハを汚染するおそれがある。また、特開平４－３６４７３０号公報には、ウェーハ研磨装置の定盤に貼り付けられたポリッシングマットのコンディショニングに、ダイヤモンド砥粒をエポキシ樹脂に電着したペレットを用いる方法が提案されている。しかし、ペレットがメタルボンドの場合、金属が研磨液によって溶解され、半導体ウェーハに残存して悪影響を及ぼすおそれがある。特に、一般にメタルボンドの主成分として使用される銅は悪影響が著しい。本発明者らは、先に半導体デバイスの層間絶縁膜及び金属配線などのＣＭＰ用のポリッシングマットのコンディショニングに際して、金属分による汚染を生じないコンディショナとして、台金作用面にメッキにより固着されたダイヤモンド砥粒層を有し、メッキされた金属が合成樹脂層により被覆され、ダイヤモンド砥粒の先端作用部が合成樹脂層より突出してなるコンディショナを提案した。このコンディショナは、ＣＭＰ用ポリッシングマットのコンディショニングに好適に使用されているが、さらに強い酸性条件下でのＣＭＰ加工においても、金属の溶出やダイヤモンド砥粒の脱落のおそれのないＣＭＰ用コンディショナが求められるようになった。

【０００３】

【発明が解決しようとする課題】本発明は、半導体ウェーハ研磨用のポリッシングマットのコンディショニングにおいて、酸性の高い研磨液によってもダイヤモンド砥粒を固着するメッキ層が侵されるおそれがなく、金属の溶出によるコンタミネーションやダイヤモンド砥粒の脱落を生じないＣＭＰ用コンディショナ及びその製造方法を提供することを目的としてなされたものである。

【０００４】

【課題を解決するための手段】本発明者は、上記の課題を解決すべく鋭意研究を重ねた結果、ダイヤモンド砥粒がメッキ層により固着されたコンディショナにおいて、メッキ層上に耐硝酸性のメッキを施し、さらにその表面を合成樹脂で被覆することにより、強い酸性条件下でのＣＭＰ加工の際にも、金属の溶出によるコンタミネーションやダイヤモンド砥粒の脱落を生ずることがなく、ま

た．このようなコンディショナは、作用面にダイヤモンド砥粒を電着法又は反転電鋳法によって固着したのち、その表面に耐硝酸性のメッキを施し．さらに合成樹脂で被覆することにより、容易に製造し得ることを見いだし．この知見に基づいて本発明を完成するに至った。すなわち、本発明は、（１）ダイヤモンド砥粒がメッキ層により固着されたコンディショナにおいて、メッキ層上に耐硝酸性のメッキが施され、その表面がさらに合成樹脂により被覆され、ダイヤモンド砥粒の先端作用部が合成樹脂層より突出してなることを特徴とするＣＭＰ用コンディショナ．（２）耐硝酸性のメッキが、アルミニウム．クロム、金．ロジウム．錫又はこれらの合金のメッキである第(１)項記載のＣＭＰ用コンディショナ．及び．（３）作用面にダイヤモンド砥粒を電着法又は反転電鋳法によって固着したのち、さらに耐硝酸性のメッキを施し、その表面を合成樹脂により被覆することを特徴とする第(１)項又は第(２)項記載のＣＭＰ用コンディショナの製造方法．を提供するものである。さらに．本発明の好ましい態様として．（４）耐硝酸性のメッキが、金メッキである第(２)項記載のＣＭＰ用コンディショナ．（５）金メッキの厚さが２～１０μｍである第(４)項記載のＣＭＰ用コンディショナ．を挙げることができる。

【０００５】

【発明の実施の形態】本発明のＣＭＰ用コンディショナは．台金作用面にダイヤモンド砥粒がメッキ層により固着され、メッキ層上に耐硝酸性のメッキが施され．その表面がさらに合成樹脂層により被覆され、ダイヤモンド砥粒の先端作用部が合成樹脂層より突出してなるものである。図１(ａ)は、本発明のＣＭＰ用コンディショナの一態様の斜視図であり、図１(ｂ)は．図１(ａ)のコンディショナを中心軸を通る平面で切断したときの切断部端面図である。図１に示すＣＭＰ用コンディショナは、カップ型の台金１の作用面にダイヤモンド砥粒がメッキにより固着されて．ダイヤモンド砥粒層２を形成している。ダイヤモンド砥粒３は．メッキ層４により台金１に固着され、メッキ層上に耐硝酸性のメッキ５が施され、その表面が合成樹脂層６により被覆され、ダイヤモンド砥粒の先端作用部が合成樹脂層より突出している。本発明において、耐硝酸性のメッキとは．２０㎜×２０㎜×０．２～２㎜の金属試験片を５重量%硝酸５０mlに２４時間浸漬したのち、ＪＩＳ Ｋ ０１２１にしたがって硝酸をフレーム原子吸光分析して、金属の溶出量が５μｇ／ml以下である金属のメッキをいう。このような金属としては、例えば．アルミニウム、クロム、金、ロジウム．錫などや．これらの金属の合金を挙げることができる。本発明のコンディショナは、ダイヤモンド砥粒がすべてメッキにより台金に強固に固着され、メッキ層上に耐硝酸性のメッキが施され．その表面がさらに合成樹脂層により被覆されているので、ｐＨが２以下であるような強い酸性の研磨液を用いた場合でも．研磨液が合成樹脂層に浸透してダイヤモンド砥粒を固着しているメッキ層と直接接触することがなく．金属分の溶出やダイヤモンド砥粒の脱落を生ずるおそれがない。そのため．本発明のＣＭＰ用コンディショナは、半導体デバイスの層間絶縁膜及び金属配線などのＣＭＰ用のポリッシングマットのコンディショニングに使用したとき．脱落したダイヤモンド砥粒によるウェーハの損傷を生ずることがなく、ＣＭＰ加工後のウェーハの洗浄工程を簡略化することができる。

【０００６】図２及び図３は、本発明のＣＭＰ用コンディショナの製造方法の一態様の説明図である。本態様においては、台金作用面にダイヤモンド砥粒をメッキにより仮固定し、次いでダイヤモンド砥粒をメッキ層により固着してダイヤモンド砥粒層を形成し．メッキ層上に耐硝酸性のメッキを施し、その表面をさらに合成樹脂により被覆し、ダイヤモンド砥粒の先端作用部を突出させる。ダイヤモンド砥粒をメッキにより仮固定する場合は．図２(ａ)に示すように．台金１のダイヤモンド砥粒固定面７を残して、絶縁性のマスキング材８で被覆する。台金をメッキ浴に浸漬し、ダイヤモンド砥粒固定面にダイヤモンド砥粒３を載置し、台金に陰極を接続し、メッキ液に陽極を接続して．電気メッキを行う。使用するダイヤモンド砥粒に特に制限はないが、ＪＩＳ Ｂ ４１３０に規定する粒度１００／１２０～３０／４０の粒径を有する砥粒であることが好ましく．粒度６０／８０程度の粒径を有する砥粒であることがより好ましい。ダイヤモンド砥粒の粒径が．粒度１００／１２０に相当する粒径未満であると、被覆する合成樹脂層の厚さが薄くなって、耐硝酸性のメッキの表面を完全に被覆しきれなくなるおそれがある。ダイヤモンド砥粒の粒径が．粒度３０／４０に相当する粒径を超えると．ダイヤモンド砥粒が高価になって経済的に不利であるばかりでなく、コンディショニングの際にポリッシングマットが組面化するおそれがある。メッキする金属は．ダイヤモンド砥粒を仮固定することができるものであれば特に制限はなく．例えば、ニッケル、クロムなどを好適に使用することができる。ダイヤモンド砥粒が仮固定され、ダイヤモンド砥粒固定面から脱落しない状態になれば、余剰のダイヤモンド砥粒を除去する。図２(ｂ)は、ダイヤモンド砥粒が、ダイヤモンド砥粒固定面に仮固定された状態を示す断面図である。ダイヤモンド砥粒を仮固定したとき．大部分のダイヤモンド砥粒はその一部が台金のダイヤモンド砥粒固定面に接した状態で仮固定されるが、台金のダイヤモンド砥粒固定面に接しない状態で付着し、浮き石９となっているダイヤモンド砥粒が存在する場合もあるので、仮固定を終了したのち．ダイヤモンド砥粒層面の表面をアルミナ砥石．シリコンカーバイド砥石などを用いて軽く研磨することにより．浮き石を除去することが好ましい。図２(ｃ)は、浮き石を除去した状態を

示す断面図である。

【０００７】浮き石を除去したのち、台金をふたたびメッキ浴に浸漬し、台金に陰極を接続し、メッキ液に陽極を接続して、ダイヤモンド砥粒固定面のメッキを行い、ダイヤモンド砥粒をメッキ層４により固着してダイヤモンド砥粒層を形成し、図２(d)に示す状態とする。メッキする金属は、ダイヤモンド砥粒を固定することができるものであれば特に制限はなく、例えば、ニッケル、クロムなどを使用することができるが、特にニッケルが好ましい。ニッケルメッキによりダイヤモンド砥粒を固着する場合、添加剤を加えたスルファミン酸ニッケル浴を使用すると、ニッケルの硬度はＨＶ４００～６００、伸び率は１～５％となり、ニッケル固着層は十分な靭性を有する。ダイヤモンド砥粒を固着するメッキ層の厚さは、ダイヤモンド砥粒の粒径の４０％以上であることが好ましく、５０％以上であることがより好ましい。メッキ層の厚さがダイヤモンド砥粒の粒径の４０％未満であると、ダイヤモンド砥粒を保持する力が不足するおそれがある。ダイヤモンド砥粒をメッキ層により固着してダイヤモンド砥粒層を形成したのち、さらにメッキ層上に耐硝酸性のメッキを施す。耐硝酸性のメッキを施す方法には特に制限はなく、メッキする金属に応じてそれぞれ公知の方法を適宜選択することができる。例えば、耐硝酸性のメッキが金メッキである場合は、金シアン化カリウムを用いるシアン化物メッキ浴法や塩化金を用いるリン酸塩浴法などを挙げることができる。金は最も貴な金属であるので、台金をメッキ浴に浸漬するだけで置換によって表面に金が析出する。このような析出物は素地金属との密着力が弱く剥離しやすいので、台金に陰極を空気中で接続し、通電を始めてから手早くメッキ浴に浸漬することが好ましい。使用する陽極の材質には特に制限はなく、例えば、硬質炭素、ステンレス鋼、ニクロム鋼などを使用することができる。金のメッキを続けると、時間の経過にしたがって析出結晶が粗雑になるので、時々台金をメッキ浴から取り出して刷毛で摩擦し、ふたたびメッキを繰り返すことにより、平滑なメッキ面を得ることができる。金メッキの厚さは、２～１０μｍであることが好ましく、３～６μｍであることがより好ましい。金メッキの厚さが２μｍ未満であると、強い酸性条件下でダイヤモンド砥粒を固着するメッキ層が侵されるおそれがある。金メッキの厚さは１０μｍで十分であり、１０μｍを超える金メッキは通常は必要はない。図３(a)は、メッキ層上に耐硝酸性のメッキ５が施された状態を示す断面図である。メッキ層上に耐硝酸性のメッキを施したのち、台金よりマスキング材を外して洗浄、乾燥し、合成樹脂層により耐硝酸性のメッキの表面を被覆する。使用する合成樹脂には特に制限はなく、熱硬化性樹脂及び熱可塑性樹脂のいずれをも使用することができる。熱硬化性樹脂としては、例えば、エポキシ樹脂、ポリウレタン樹脂、不飽和ポリエステル樹脂、フッ素樹脂などを挙げることができる。熱可塑性樹脂としては、例えば、フッ素樹脂、ポリアミド樹脂、ポリイミド樹脂などを挙げることができる。また、本発明においては、樹脂の機械的強度を向上させるために、非導電性の無機化合物粒子やファイバーを加えることもできる。熱硬化性樹脂としては、加熱硬化型のほかに常温硬化型の樹脂も使用することができ、さらに、熱硬化性樹脂に代えて光硬化性樹脂も使用することができる。

【０００８】図３(b)は、耐硝酸性のメッキの表面を、さらに合成樹脂層で被覆した状態を示す断面図である。合成樹脂層６で被覆したのち合成樹脂層の表面部分を除去することにより、ダイヤモンド砥粒３の先端作用部を突出させる。ダイヤモンド砥粒の先端作用部を突出させる方法に特に制限はなく、例えば、ダイヤモンド砥粒の最突出部が見えるまで研削盤などを用いて合成樹脂層を除去したのち、一般砥石などによるドレッシング、鋳鉄などの定盤上でのシリコンカーバイドやアルミナなどの遊離砥粒を用いたドレッシング、ショットブラストなどにより、ダイヤモンド砥粒の突出量を容易に所定の値とすることができる。ダイヤモンド砥粒は、メッキによって強固に台金に固着されているので、ダイヤモンド砥粒に強い力がかかっても、コンディショナからダイヤモンド砥粒が脱落するおそれがない。図３(c)は、合成樹脂層の表面部分を除去してダイヤモンド砥粒の先端作用部が合成樹脂層６より突出し、かつ耐硝酸性のメッキの表面が合成樹脂層に覆われて露出しない状態を示す断面図である。

【０００９】図４及び図５は、本発明のＣＭＰ用コンディショナの製造方法の他の態様の説明図である。本態様においては、反転型のダイヤモンド砥粒固定面にダイヤモンド砥粒をメッキにより仮固定し、さらにダイヤモンド砥粒をメッキ層により埋め込んだのち、必要に応じてメッキ層を平坦化し台金の作用面に接合する。その後、反転型を除去し、メッキ層の表面部分を除去してダイヤモンド砥粒を露出させ、メッキ層上に耐硝酸性のメッキを施し、その表面をさらに合成樹脂により被覆し、ダイヤモンド砥粒の先端作用部を突出させる。ダイヤモンド砥粒をメッキにより仮固定する場合は、図４(a)に示すように、反転型１０のダイヤモンド砥粒固定面？を残して、絶縁性のマスキング材８で被覆する。反転型をメッキ浴に浸漬し、ダイヤモンド砥粒固定面にダイヤモンド砥粒３を載置し、反転型に陰極を接続し、メッキ液に陽極を接続して、電気メッキを行う。使用するダイヤモンド砥粒に特に制限はないが、ＪＩＳ Ｂ ４１３０に規定する粒度１００／１２０～３０／４０の粒径を有する砥粒であることが好ましく、粒度６０／８０程度の粒径を有する砥粒であることがより好ましい。ダイヤモンド砥粒の粒径が、粒度１００／１２０に相当する粒径より小さいと、被覆する合成樹脂層の厚さが薄くなって、耐硝酸性のメッキの表面を完全に被覆しきれなくなるおそれ

がある。ダイヤモンド砥粒の粒径が、粒度３０／４０に相当する粒径より大きいと、ダイヤモンド砥粒が高価になって経済的に不利であるばかりでなく、コンディショニングの際にポリッシングマットが組面化するおそれがある。メッキする金属は、ダイヤモンド砥粒を仮固定することができるものであれば特に制限はなく、例えば、ニッケル、クロムなどを好適に使用することができる。ダイヤモンド砥粒が仮固定され、ダイヤモンド砥粒固定面から脱落しない状態になれば、余剰のダイヤモンド砥粒を除去する。図４（ｂ）は、ダイヤモンド砥粒が、ダイヤモンド砥粒固定面に仮固定された状態を示す断面図である。

【００１０】余剰のダイヤモンド砥粒を除去したのち、反転型をふたたびメッキ浴に浸漬し、反転型に陰極を接続し、メッキ液に陽極を接続して、ダイヤモンド砥粒固定面のメッキを行い、ダイヤモンド砥粒をメッキ層４により埋め込んでダイヤモンド砥粒層を形成し、図４（ｃ）に示す状態とする。メッキする金属は、ダイヤモンド砥粒を埋め込んで固着することができるものであれば特に制限はなく、例えば、ニッケル、クロムなどを使用することができるが、特にニッケルが好ましい。ニッケルメッキによりダイヤモンド砥粒を埋め込む場合、添加剤を加えたスルファミン酸ニッケル浴を使用すると、ニッケルの硬度はＨＶ４００～６００、伸び率は１～５％となり、ニッケル固着層は十分な靭性を有する。ダイヤモンド砥粒をメッキにより埋め込んでダイヤモンド砥粒層を形成した反転型は、マスキング材を外して洗浄、乾燥し、必要に応じて、反転型上のメッキ層の表面を平坦に加工する。次いで、図４（ｄ）に示すように、反転型１０上のメッキ層４を台金１に接合する。反転型上のメッキ層４若しくは台金１のダイヤモンド砥粒層接合面又はその双方に接着剤を塗付し、メッキ層とダイヤモンド砥粒層接合面を合わせて固定し、メッキ層を台金に接合する。使用する接着剤は、コンディショナの使用に十分な強度を有するものであれば特に制限はなく、例えば、エポキシ接着剤などを好適に使用することができる。必要に応じて、接着剤に無機充填材、例えば、アルミニウム粉末などを配合することができる。メッキ層４を台金１に接合したのち、反転型１０を除去する。反転型を除去する方法には特に制限はなく、例えば、旋盤加工、フライス盤加工などを挙げることができるが、旋盤加工を特に好適に使用することができる。図５（ａ）は、メッキ層４を台金１に接合し、反転型を除去した状態を示す。

【００１１】台金に接合したメッキ層は、金属の表面部分を除去してダイヤモンド砥粒を露出させる。ダイヤモンド砥粒を露出させる方法には特に制限はなく、例えば、一般砥石などによるドレッシング、鋳鉄などの定盤上でのシリコンカーバイドやアルミナなどの遊離砥粒によるドレッシング、ショットブラスト、金属剥離剤による化学エッチング、電解エッチングなどにより行うことができる。化学エッチング処理は、台金及びダイヤモンド砥粒層を形成する材料のうち、メッキされた金属のみを溶解する薬剤に浸漬することにより行うものである。このような薬剤としては、金属がニッケルである場合はエンストリップＮＰ［メルテックス(株)製］、金属がクロムである場合はエンストリップＣＲ－５［メルテックス(株)製］などの市販されている薬剤を使用することができる。図５（ｂ）は、メッキ層４の表面部分を除去して、ダイヤモンド砥粒３を露出させた状態を示す。ダイヤモンド砥粒を露出させたメッキ層は、次いでその上に耐硝酸性のメッキを施す。耐硝酸性のメッキを必要としない台金の部分は、あらかじめマスキング材により保護することができる。耐硝酸性のメッキを施す方法には特に制限はなく、メッキする金属に応じてそれぞれ公知の方法を適宜選択することができる。例えば、耐硝酸性のメッキが金メッキである場合は、金シアン化カリウムを用いるシアン化物メッキ浴法や塩化金を用いるリン酸塩浴法などを挙げることができる。金は最も貴な金属であるので、台金をメッキ浴に浸漬するだけで置換によって表面に金が析出する。このような析出物は素地金属との密着力が弱く剥離しやすいので、台金に陰極を空気中で接続し、通電を始めてから手早くメッキ浴に浸漬することが好ましい。使用する陽極の材質には特に制限はなく、例えば、硬質炭素、ステンレス鋼、ニクロム鋼などを使用することができる。金のメッキを続けると、時間の経過にしたがって析出結晶が粗雑になるので、時々台金をメッキ浴から取り出して刷毛で摩擦し、ふたたびメッキを繰り返すことにより、平滑なメッキ面を得ることができる。金メッキの厚さは、２～１０μｍであることが好ましく、３～６μｍであることがより好ましい。金メッキの厚さが２μｍ未満であると、強い酸性条件下でダイヤモンド砥粒を固着するメッキ層が侵されるおそれがある。金メッキの厚さは１０μｍで十分であり、１０μｍを超える金メッキは通常は必要はない。図５（ｃ）は、メッキ層４上に耐硝酸性のメッキ５が施された状態を示す断面図である。メッキ層上に耐硝酸性のメッキを施したのち、洗浄、乾燥し、合成樹脂層により耐硝酸性のメッキの表面を被覆する。使用する合成樹脂には特に制限はなく、熱硬化性樹脂及び熱可塑性樹脂のいずれをも使用することができる。熱硬化性樹脂としては、例えば、エポキシ樹脂、ポリウレタン樹脂、不飽和ポリエステル樹脂、フッ素樹脂などを挙げることができる。熱可塑性樹脂としては、例えば、フッ素樹脂、ポリアミド樹脂、ポリイミド樹脂などを挙げることができる。また、本発明においては、樹脂の機械的強度を向上させるために、非導電性の無機化合物粒子やファイバーを加えることもできる。熱硬化性樹脂としては、加熱硬化型のほかに常温硬化型の樹脂も使用することができ、さらに、熱硬化性樹脂に代えて光硬化性樹脂も使用することができる。

【００１２】図５(ｄ)は、耐硝酸性のメッキの表面を、さらに合成樹脂層で被覆した状態を示す断面図である。合成樹脂層６で被覆したのち合成樹脂層の表面部分を除去することにより、ダイヤモンド砥粒の先端作用部を突出させる。ダイヤモンド砥粒の先端作用部を突出させる方法に特に制限はなく、例えば、ダイヤモンド砥粒の最突出部が見えるまで研削盤などを用いて合成樹脂層を除去したのち、一般砥石などによるドレッシング、鋳鉄などの定盤上でのシリコンカーバイドやアルミナなどの遊離砥粒を用いたドレッシング、ショットブラストなどにより、ダイヤモンド砥粒の突出量を容易に所定の値とすることができる。ダイヤモンド砥粒は、メッキによって強固に固着されているので、ダイヤモンド砥粒に強い力がかかっても、コンディショナからダイヤモンド砥粒が脱落するおそれがない。図５(ｅ)は、合成樹脂層の表面部分を除去してダイヤモンド砥粒の先端作用部が合成樹脂層より突出し、かつ耐硝酸性のメッキの表面が合成樹脂層に覆われて露出しない状態を示す断面図である。本態様により製造されたコンディショナは、ダイヤモンド砥粒の仮固定の際に、ダイヤモンド砥粒が反転型のダイヤモンド砥粒固定面に接して固定され、ダイヤモンド砥粒層を台金に接合したとき、ダイヤモンド砥粒の最突出部は反転型のダイヤモンド砥粒固定面に接した部分であるので、コンディショナにおいて、各ダイヤモンド砥粒の最突出部が完全に同一平面上に存在する構造となる。また、ダイヤモンド砥粒層と台金の接合後の基準面転写により、ダイヤモンド砥粒層面の振れ精度は数μｍである。ニッケルの電鋳によりダイヤモンド砥粒を固着する場合、添加剤を加えたスルファミン酸ニッケル浴を使用すると、ニッケルの硬度はＨＶ４００～６００、伸び率は１～５％となり、ニッケル固着層は十分な靭性を有する。このために、ポリッシングマットのコンディショニングを精度よく能率よく行うことができ、ダイヤモンド砥粒の脱落が長期にわたって生じない。

【００１３】合成樹脂によりダイヤモンド砥粒層を覆う方法によれば、数ミリ程度の厚さの合成樹脂層も短時間で形成することができ、研削盤や遊離砥粒による合成樹脂の除去も短時間で精度よく行うことができる。本発明のＣＭＰ用コンディショナにおいて、ダイヤモンド砥粒の突出量は、ダイヤモンド砥粒の平均粒径の５～５０％であることが好ましく、平均粒径の２０～３０％であることがより好ましい。ダイヤモンド砥粒の突出量が平均粒径の５％未満であると、コンディショナのコンディショニング作用が微弱になるおそれがある。ダイヤモンド砥粒の突出量が平均粒径の５０％を超えると、コンディショニング作用が強すぎてポリッシングマットを損傷し、ダイヤモンド砥粒が脱落するおそれがある。本発明方法においては、必要に応じてさらにラップ加工を行うことができる。ラップ加工は、ダイヤモンド砥石を用いて突出したダイヤモンド砥粒の先端部をカットするものであり、ラップ加工を行うことにより、コンディショナに、使用当初から安定した優れた性能を発揮させることができる。本発明のＣＭＰ用コンディショナによれば、耐硝酸性のメッキがバリアとなって、ｐＨの低い酸性の強い研磨液を用いても、メッキ層の金属と研磨液が接触することがないので、ダイヤモンド砥粒が脱落してウェーハを傷つけたり、金属分が溶出してウェーハを汚染することがない。さらに、本発明のコンディショナはダイヤモンド砥粒の最突出部の平坦性に優れるので、ポリッシングマットの平坦度が向上し、ポリッシングマットの切れ味が向上する。

【００１４】

【実施例】以下に、実施例を挙げて本発明をさらに詳細に説明するが、本発明はこれらの実施例によりなんら限定されるものではない。

実施例１

寸法が１２７Ｄ－６Ｗ－２０Ｔ－３０Ｈの台金を、ステンレス鋼（ＳＵＳ３０４）を旋盤加工することにより作製した。次に、ダイヤモンド砥粒固定面を残して、表面を絶縁テープ及び塗料によりマスキングした。台金のアルカリ脱脂処理を行い、塩化ニッケル２４０ｇ／リットル及び塩酸１００ｇ／リットルを含有する前処理液に浸漬し、電流密度１０Ａ／ｄｍ²で常温にて陽極側に台金をセットし２分間電解エッチングしたのち、陰極側に台金をセットしてストライクメッキを３分間行った。次いで、スルファミン酸ニッケルメッキ浴で電流密度１Ａ／ｄｍ²で１５分間メッキを行い、下地メッキ層を３μｍ形成した。次に、ダイヤモンド砥粒固定面に粒度＃６０／８０、平均粒径２５０μｍのダイヤモンド砥粒を載置し、メッキ応力と硬度調節のための添加剤を加えたスルファミン酸ニッケルメッキ浴を用い、電流密度０．５Ａ／ｄｍ²で３時間メッキを行い、ダイヤモンド砥粒一層分を仮固定した。余剰のダイヤモンド砥粒を払い落とし、電流密度１Ａ／ｄｍ²で１時間埋め込みメッキを行ったのち、＃１００アルミナ砥石を当て、浮き石となっているダイヤモンド砥粒を除去した。次いで、塩化ニッケル２４０ｇ／リットル及び塩酸１００ｇ／リットルを含有する前処理液に浸漬して陽極側に台金をセットし、電流密度１０Ａ／ｄｍ²で常温にて３０秒電解エッチングしたのち、陰極側に台金をセットしてストライクメッキを２分間行った。次に、スルファミン酸ニッケルメッキ浴で電流密度１Ａ／ｄｍ²で１５分間メッキを行い、さらに同じスルファミン酸ニッケルメッキ浴で、電流密度１Ａ／ｄｍ²で５時間メッキを行い、ダイヤモンド砥粒を固着するよう厚さ約１２５μｍのメッキを施した。ダイヤモンド砥粒固定面及び台金を洗浄したのち、シアン化金２．５ｇ／リットル、シアン化カリウム３０．０ｇ／リットル、第二リン酸ナトリウム１５．０ｇ／リットル及びシアン化コバルト１０．０ｇ／リットルを含むメッキ浴に浸漬し、ステンレス鋼を陽極とし、浴温５０℃、電流密

度０．２Ａ／dm²．電圧２Ｖの条件で２０時間メッキを行い．厚さ４μｍの金合金層を形成した。金合金の組成は．金９９．７重量%．コバルト０．３重量%であった。マスキングをすべて外し．ダイヤモンド砥粒層が形成された台金を洗浄．乾燥したのち、フッ素樹脂塗料［日建塗装工業(株)．ＰＦＡ］を．塗膜厚さが１００μｍとなるように塗付して、３３０℃で３０分間焼き付けた。その後．アルミナ砥粒＃８００を用いてスエードタイプの研磨マットで．ダイヤモンド砥粒先端部が５０μｍ露出するまでラップ加工を行った。得られたＣＭＰ用コンディショナを５重量%硝酸に２４時間浸漬したが．金属分の溶出は全く認められなかった。また．このＣＭＰ用コンディショナと研磨マット（ｉＣ１０００）を用い、スラリー（硝酸鉄＋硝酸＋アルミナ）でＣＭＰ加工を行ったところ、金属膜ウェーハの研磨レート１，８７０Å／分という良好な結果が得られた。

実施例２

寸法が２８０Ｄ－６Ｗ－２０Ｔ－１４０Ｈの反転型（Ｓ４５Ｃ）を、旋盤加工により作製した。ダイヤモンド砥粒固定面をマスキングテープで囲い．さらにその他のダイヤモンド砥粒層非形成部分のマスキング処理を行った。アルカリ脱脂処理したのち、反転型を、メッキ応力と硬度調節のため添加剤を加えたスルファミン酸ニッケル浴に浸め、粒度＃６０／８０のダイヤモンド砥粒をダイヤモンド砥粒固定面に載置した。電流密度１Ａ／dm²で２時間メッキを行い、ダイヤモンド砥粒の仮固定を行ったのち、余剰のダイヤモンド砥粒を除去し、さらに電流密度２Ａ／dm²で９６時間メッキを行い．３mmの厚みのニッケル固着層を形成してダイヤモンド砥粒を完全に埋め込んだ。次いで、反転型上のニッケル固着層面を、旋盤加工により平坦に加工した。寸法２８６Ｄ－１２Ｗ－３５Ｔ－１６０Ｈの台金（ＳＵＳ３０４）を．旋盤加工により作製した。２液型エポキシ接着剤１０重量部にアルミニウム粉末５重量部を混合し．反転型上のニッケル固着層面と台金のダイヤモンド砥粒層接着部分に塗布し接合した。接合層が十分に硬化したのち、旋盤で反転型を正にして台金の裏面を切削して基準面転写した。さらに．台金を旋盤にくわえ．反転型を切除した。その後．ニッケル剥離剤エンストリップＮＰ［メルテックス(株)］を用いて．ダイヤモンド砥粒層面のニッケルを約１２５μｍ溶解除去した。ダイヤモンド砥粒層が形成された台金を洗浄．乾燥したのち、ダイヤモンド砥粒層以外の部分をマスキング処理し、シアン化金２．５ｇ／リットル、シアン化カリウム３０．０ｇ／リットル、第二リン酸ナトリウム１５．０ｇ／リットル及びシアン化コバルト１０．０ｇ／リットルを含むメッキ浴に浸漬し．ステンレス鋼を陽極とし．浴温５０℃．電流密度０．２Ａ／dm²．電圧２Ｖの条件で２５時間メッキを行い、厚さ５μｍの金合金層を形成した。金合金の組成は．金９９．７重量%、コバルト０．３重量%であった。次いで、エポキシ塗料を塗膜厚さが１１０μｍとなるように塗付し．焼き付けた。その後．シリコンカーバイド＃８００を用いてエポキシ樹脂層約５０μｍを削り出し．ダイヤモンド砥粒の先端作用部をエポキシ樹脂層から突出させて．ＣＭＰ用コンディショナを得た。得られたＣＭＰ用コンディショナを５重量%硝酸に２４時間浸漬したが、金属分の溶出は全く認められなかった。また、このＣＭＰ用コンディショナを用いて、ポリエステル不織布を基剤とし、その上に発泡ポリウレタン層を形成したスエードタイプのポリッシングマットのコンデショニングを行った。平坦性に優れたコンデショニングを、効率よく行うことができた。

【００１５】

【発明の効果】本発明のＣＭＰ用コンディショナは、ｐＨ２以下のような強い酸性条件下でのＣＭＰ加工に使用しても．メッキ層から金属が溶出したり．ダイヤモンド砥粒が脱落するおそれがなく．安定してコンディショニングを行うことができる。

【図面の簡単な説明】

【図１】図１は．本発明のＣＭＰ用コンディショナの一態様の斜視図及び切断部端面図である。

【図２】図２は．本発明のＣＭＰ用コンディショナの製造方法の一態様の説明図である。

【図３】図３は．本発明のＣＭＰ用コンディショナの製造方法の一態様の説明図である。

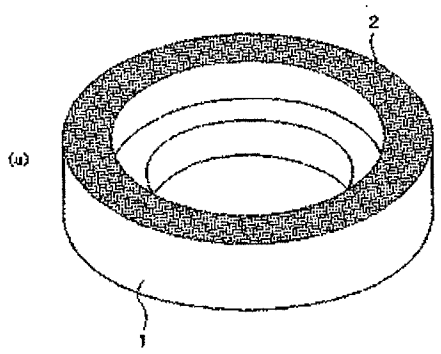
【図４】図４は．本発明のＣＭＰ用コンディショナの製造方法の他の態様の説明図である。

【図５】図５は．本発明のＣＭＰ用コンディショナの製造方法の他の態様の説明図である。

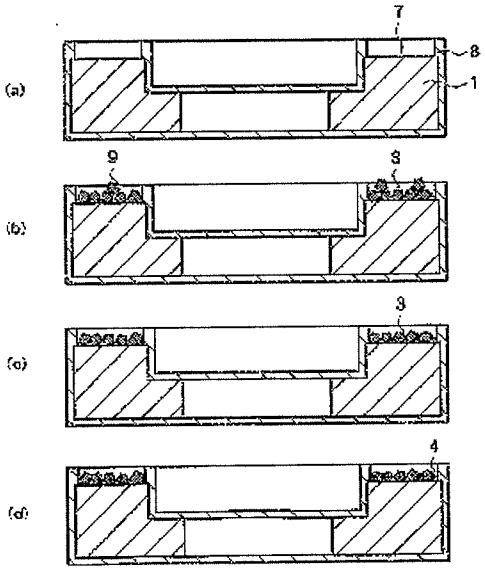
【符号の説明】

１　台金
２　ダイヤモンド砥粒層
３　ダイヤモンド砥粒
４　メッキ層
５　耐硝酸性のメッキ
６　合成樹脂層
７　ダイヤモンド砥粒固定面
８　マスキング材
９　浮き石
１０　反転型

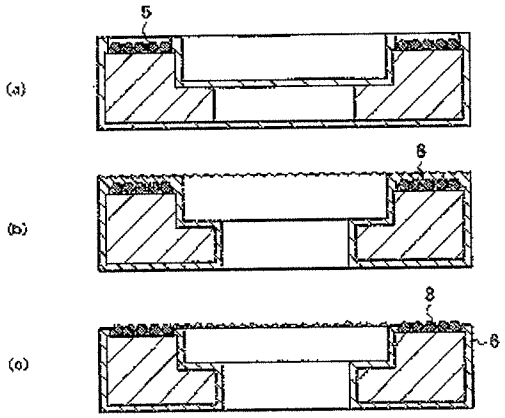
【図1】



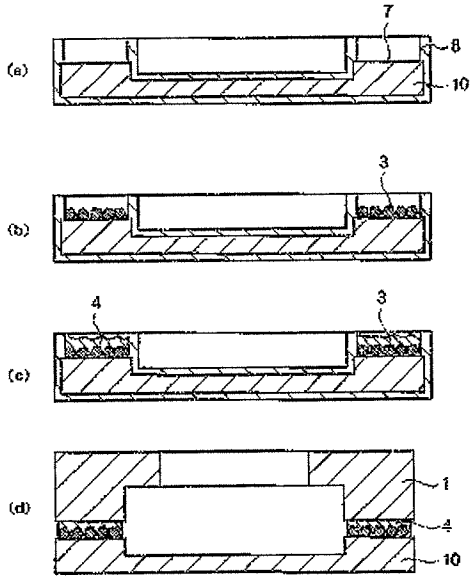
【図2】



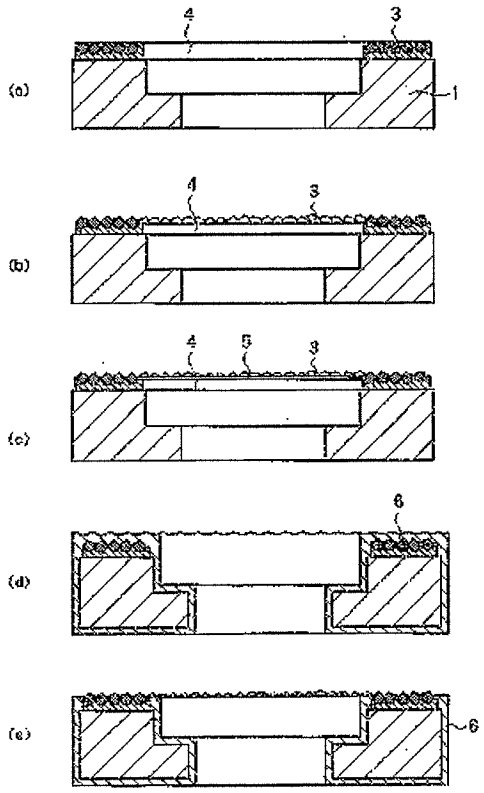
【図3】



【図4】



【図5】



PATENT ABSTRACTS OF JAPAN

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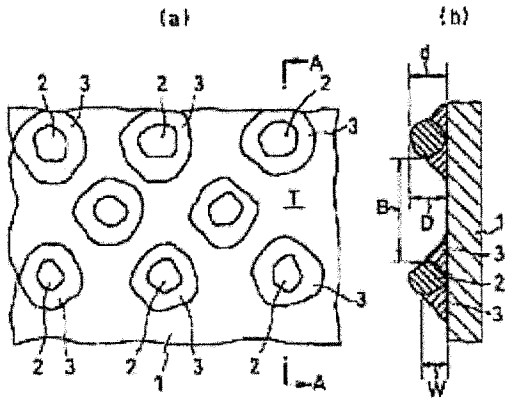
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(54) GRINDING WHEEL AND MANUFACTURE THEREOF

(57)Abstract:
PROBLEM TO BE SOLVED: To obtain long-term stable cutting quality through the excellent supply of a polishing solution and discharge of chips while obtaining the holding force and necessary protruding height of abrasive grains by setting wax material thickness to be specific times as large as an abrasive grain diameter, and forming a chip pocket with base metal matrix exposed with a width specific times as large as the abrasive grain diameter between the abrasive grains.
SOLUTION: In arranging abrasive grains 2 in one layer on a base metal 2 for brazing, the abrasive grains 2 are held with a wax material thickness W of about 1/3-1/2 times as large as an abrasive grain diameter(d),and a chip pocket T with the matrix of the base metal 1 exposed with a width about 2/3-2.5 times as large as the abrasive grain diameter(d) between the adjacent abrasive grains 2 is regularly formed. Such a wax material thickness W can ensure the protruding height of the abrasive grains 2 required to exhibit excellent cutting quality while ensuring the holding force of the abrasive grains 2, and the regular formation of the chip pocket T with the exposed matrix of the base metal 1 permits the uniform distribution of the abrasive grains 2 and excellent supply of a polishing solution and discharge of chips.



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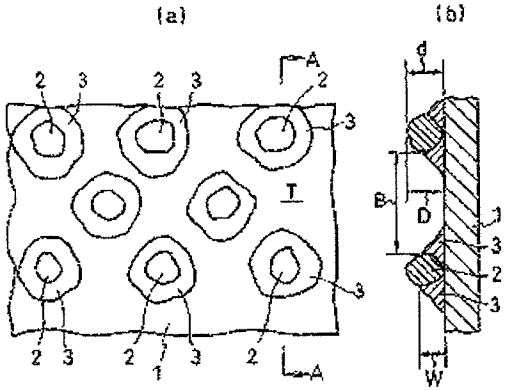
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(54)【発明の名称】　研削砥石およびその製造方法

(57)【要約】
【課題】　超砥粒を台金に一層配列してろう付けした研削砥石において，砥粒層の全面に砥粒を均一に分布させ，良好な切れ味と切粉の排出を安定的に維持する。
【解決手段】　砥粒粒径ｄの１／３～１／２倍の厚さのろう材３で砥粒２を保持するとともに，隣り合う砥粒２との間に砥粒粒径の２／３～２．５倍の幅で台金１の素地を露出させた部分を含むチップポケットＴを規則的に形成した。



1

【特許請求の範囲】

【請求項１】　ダイヤモンド、ＣＢＮなどの超砥粒を台金に一層配列してろう付けした研削砥石において．砥粒粒径の１／３～１／２倍のろう材厚さで砥粒を保持するとともに、隣り合う砥粒との間に砥粒粒径の２／３～２．５倍の幅で台金素地を露出させた部分を含むチップポケットを規則的に形成した研削砥石。

【請求項２】　前記チップポケットの深さを砥粒粒径の２／３～１倍の深さとした請求項１記載の研削砥石。

【請求項３】　ダイヤモンド、ＣＢＮなどの超砥粒を台金に一層配列してろう付けする研削砥石の製造方法において．所定の間隔で規則的に孔を開けたスクリーンを用いて台金上に有機接着剤を塗布し、この有機接着剤の上に砥粒を配置した状態で乾燥炉中で乾燥させた後．接着部にのみろう材とバインダーの混合物を塗布し加熱、ろう付けすることにより、砥粒を台金上に所定の間隔で配列させることを特徴とする研削砥石の製造方法。

【発明の詳細な説明】

【０００１】

【発明の属する技術分野】本発明はダイヤモンド．ＣＢＮなどの超砥粒を用いた研削砥石にかかり、とくに砥石の切れ味を向上させた研削砥石およびその製造方法に関する。

【０００２】

【従来の技術】従来より．ダイヤモンド砥粒やＣＢＮ砥粒などの超砥粒をろう付け法により単層に固着した研削砥石が研削・研磨用砥石として使用されている。このような研削砥石には、切れ味に優れ、しかもその切れ味が長期にわたって安定的に持続することが要求される。ところが、ダイヤモンド砥粒にしろ、ＣＢＮ砥粒にしろ、研削作業の進行に伴い切れ味と寿命が低下する。

【０００３】その主な原因として、台金への砥粒の固着状態が不安定で砥粒が脱落しやすいこと、チップポケットが小さいこと．砥粒間隔のコントロールが不十分なために研削作業中に切粉によって目詰まりが生じることが挙げられる。とくに砥粒間隔に関しては、台金の表面に砥粒を均一に分散させるのが難しく．砥粒どうしが近接した状態にある部分においては、研削液が充分に供給されにくく、また切粉が排出されにくく．切れ味の低下につながる。

【０００４】ろう付け法による砥石の製造において、砥粒を均一に分散させるための方策として、特開平９－１９８６７号公報には、金属被覆したダイヤモンド砥粒を砥石台金と金型の間隙に充填し、砥石台金と金型を加熱しながら銀ろうを間隙に溶浸させる砥石の製造方法が開示されている。また特開平１０－３４５４０号公報には．台金の表面に複数本のＶ溝を平行にかつ等間隔に設け．このＶ溝に砥粒をろう付けする方法が開示されている。

【０００５】

2

【発明が解決しようとする課題】上記の公開公報に記載の方法によれば．砥粒が所定の間隔をもって一様に分布した砥石が得られる。しかし、特開平９－１９８６７号公報に記載の方法により製造された砥石は、台金と金型の間隙によって決まる一定厚さの砥粒層が形成され、砥粒と砥粒の間に砥粒の金属被覆成分と銀ろうとが充填されたかたちであるので、いわゆるチップポケットが形成されず、充分な切れ味と切粉の排出が得られないという欠点がある。

【０００６】また特開平１０－３４５４０号公報に記載の方法により製造された砥石は、砥粒の一部がＶ溝に埋設されたかたちであるので．砥粒の突出量が小さく、切れ味が不十分となり、また．Ｖ溝内に配列される個々の砥粒どうしの間隔をコントロールすることができないため．Ｖ溝内の砥粒間隔が小さい部分では部分的に目詰まりが生じるという欠点がある。

【０００７】本発明が解決すべき課題は、超砥粒を台金に一層配列してろう付けした研削砥石において．砥粒層の全面に砥粒を均一に分布させ、良好な切れ味と切粉の排出を安定的に維持することにある。

【０００８】

【課題を解決するための手段】本発明の研削砥石は、ダイヤモンド、ＣＢＮなどの超砥粒を台金に一層配列してろう付けした研削砥石であって、砥粒粒径の１／３～１／２倍のろう材厚さで砥粒を保持するとともに．隣り合う砥粒との間に砥粒粒径の２／３～２．５倍の幅で台金素地を露出させた部分を含むチップポケットを規則的に形成したことを特徴とする。

【０００９】本発明の研削砥石においては、ろう材の厚さを砥粒粒径の１／３～１／２倍とすることによって、砥粒の保持力を確保したうえで、良好な切れ味を発揮するのに必要な砥粒の突出高さを確保することができる。ろう材の厚さが砥粒粒径の１／３倍より小さいと．ろう材による砥粒の保持力が低下し、砥粒が脱落しやすくなる。砥粒粒径の１／２倍より大きくなると、砥粒の切削に寄与する部分が小さいため切削量が少なく、切れ味が悪くなり、また．切粉が排出されにくく、ろう材が摩耗しやすくなる。ろう材の厚さが上記の範囲内にあるとき．砥粒が被研削材に十分に食い込んで研削され．また．切粉が効率よく排出され、目詰まりや焼付きが発生せず．安定した切れ味が得られる。

【００１０】また、個々の砥粒どうしの間に砥粒粒径の２／３～２．５倍の幅で台金素地を露出させた部分を含むチップポケットを規則的に形成させることによって、砥粒層の全面にわたって砥粒の分布を均一にするとともに．研削液の供給と切粉の排出を良好にして切れ味を向上させることができる。チップポケットの幅が砥粒粒径の２／３倍より小さいと．研削液の供給と切粉の排出が不十分となり．砥粒粒径の２．５倍より大きくすると、砥粒の絶対量が少なくなって砥石全体としての研削能力が

低下し、砥石の寿命も短くなる。

【００１１】ここで、前記チップポケットの深さを砥粒粒径の２／３～１倍の深さとするのが望ましい。チップポケットの深さをこの範囲とすることにより、研削液の供給と切粉の排出がより良好となり、切れ味がより向上する。

【００１２】上記のように個々の砥粒どうしの間に所定の範囲内の幅のチップポケットが形成されるように砥粒を配列させた砥石は、つぎのいずれかの方法により製造することができる。第１の方法は、所定の間隔で規則的に孔を開けたスクリーンを用いて、台金上に有機接着剤を塗布し、この有機接着剤の上に砥粒を配置した状態で乾燥炉中で乾燥させて砥粒を仮固定した後、接着部にのみろう材とバインダーの混合物を塗布し、加熱．ろう付けして本固定することにより、砥粒を台金上に所定の間隔で配列させる方法である。第２の方法は、スクリーンを用いてろう材とバインダーの混合体を塗布し、砥粒を配置した状態で加熱することにより．仮固定なしで本固定する方法である。砥粒の確実な保持と所定の範囲内の幅のチップポケットの形成のためにろう材とバインダーの混合物の塗布量をコントロールするという点からは、砥粒の仮固定後に砥粒一粒ずつに対して確実に塗布することのできる前記第１の方法が適している。

【００１３】

【発明の実施の形態】図１は本発明の実施形態における研削砥石の台金上の砥粒の配列を示す模式図であり、（ａ）は平面図、（ｂ）はＡ－Ａ線断面図である。

【００１４】本実施形態は．ＡＤＣ（アルミダイキャスト）合金、鋳鉄などの粗研削用の砥石に本発明を適用した例である。図１は砥石の砥粒層形成部分の一部を示す＊図であり、１は台金、２は砥粒、３はろう材である。この砥石の製造手順はつぎの通りである。

①台金１として外径１００ｍｍ、厚さ１０ｍｍの鉄製台金を準備する。

②砥粒２として平均粒径約４００μｍのダイヤモンド砥粒を準備する。

③直径０．２ｍｍの孔を１．２ｍｍ間隔で千鳥状に配列したスクリーンを用いて．台金１上に有機接着剤を塗布する。

④この有機接着剤の上に砥粒を配置する。この状態で砥粒は台金上に１．２ｍｍ間隔で千鳥状に配列されたかたちとなる。

⑤これを乾燥炉中で１２０℃、１時間乾燥させ．砥粒を仮固定する。

⑥三次元移動が可能なアプリケータ（吐出機）を用いて．接着部にろう材とバインダーの混合物を砥粒粒径の１／２の高さに塗布する。

⑦これを非酸化性雰囲気中で１０００℃、１時間加熱し．砥粒を台金に本固定する。

【００１５】このようにして、ろう材３の厚さＷが砥粒粒径ｄ（４００μｍ）の１／２で、砥粒２のまわりに幅Ｂ．深さＤともに砥粒粒径ｄに相当するチップポケットＴを形成した研削砥石を製造した。この砥石（発明品）と．同じ砥粒と台金を用いて前述した特開平１０－３４５４０号公報記載の方法によって製造した砥石（比較品）について研削試験を行った。両砥石の仕様のうち数値の異なる項目を表１に示す。

【００１６】

【表１】

	発明品	比較品
砥粒ピッチ	１．２ｍｍ	溝間隔０．８ｍｍ、溝内ピッチ不定
砥粒突出高さ	０．４ｍｍ	０．２ｍｍ
チップポケット幅	０．４ｍｍ	０～０．４ｍｍ
チップポケット深さ	０．４ｍｍ	０．２ｍｍ

【００１７】〔試験条件〕

研削機械　：工具研削盤

砥石周速度：１２００ｍ／ｍｉｎ

被研削材　：ハイシリコンアルミ（ＡＤＣ－１４）

切り込み量：０．１ｍｍ／ｐａｓｓ

【００１８】図２に試験結果を示す。同図は、比較品の砥石の切断速度および砥石寿命を１００としたときの本発明品の切断速度および砥石寿命を示すものである。同図からわかるように、Ｖ溝に砥粒を配列した比較品の砥石に比べて、全面に砥粒を一様に分布させ、かつ適正なチップポケットを形成した発明品は．切断速度は１．５倍程度、砥石寿命は１．７倍程度向上している。

【００１９】

【発明の効果】本発明によって以下の効果を奏する。

【００２０】（１）ろう材の厚さを砥粒粒径の１／３～１／２倍とし．さらに個々の砥粒どうしの間に砥粒粒径の２／３～２．５倍の幅で台金素地を露出させた部分を含むチップポケットを形成させることによって．砥粒の保持力を確保したうえで．良好な切れ味を発揮するのに必要な砥粒の突出高さを確保することができ、また、研削液の供給と切粉の排出を良好にして目詰まりや縞付きが発生せず、長期にわたって安定した切れ味が得られ、砥石の寿命も延長する。

【００２１】（２）チップポケットの深さを砥粒粒径の２／３～１倍の深さとすることにより．研削液の供給と切粉の排出がより良好となり、切れ味がより向上する。

【００２２】（３）所定の間隔で規則的に孔を開けたスクリーンを用いて台金上に有機接着剤を塗布し．この有

機接着剤の上に砥粒を配置した状態で乾燥炉中で乾燥させた後、接着部にのみろう材とバインダーの混合物を塗布し加熱、ろう付けすることにより、砥粒を台金上に所定の間隔で配列させることができる。

【図面の簡単な説明】

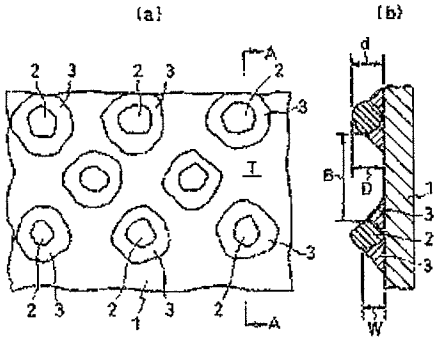
【図１】　発明の実施形態における研削砥石の台金上の砥粒の配列を示す模式図であり、（ａ）は平面図、（ｂ）はＡ－Ａ線断面図である。

【図２】　研削試験結果を示すグラフである。

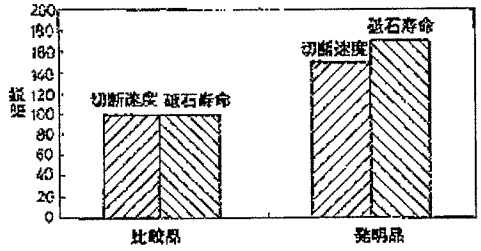
＊【符号の説明】

1　台金
2　砥粒
3　ろう材
d　砥粒粒径
W　ろう材厚さ
T　チップポケット
B　チップポケット幅
D　チップポケット深さ

【図１】



【図２】



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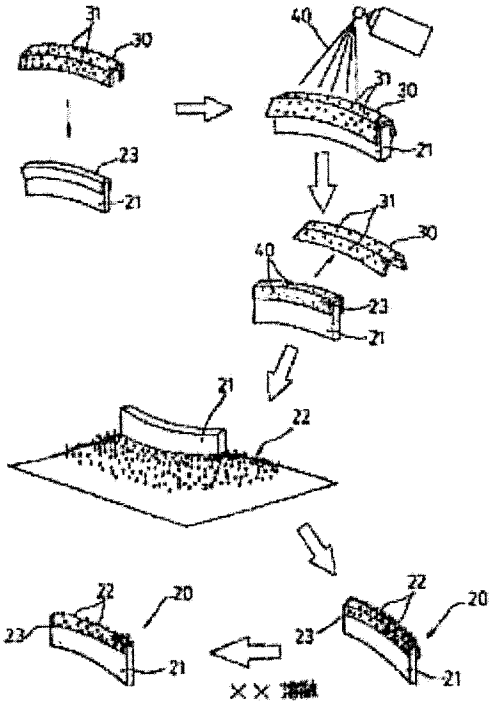
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(54) MANUFACTURE OF DIAMOND CUTTER AND DIAMOND CUTTER AND DIAMOND CUTTER MANUFACTURING JIG

(57)Abstract:
PROBLEM TO BE SOLVED: To increase fixation strength of diamond particles to prevent diamond particles from falling out by fixing diamond particles forming a diamond layer on the diamond layer at a predetermined interval mutually.
SOLUTION: A manufacturing method of a diamond cutter includes a bond paste fixation process in which bond (registered trademark) paste in which binder and bond powders are blended is attached on a substrate, a screen covering process in which a screen 30 in which holes are drilled to a desired manner after the bond paste fixation process is covered in a bond paste fixation scope, an application process in which viscous substance 40 is applied from above the screen 30 on which the bond paste is covered, a screen removing process in which the screen 30 is removed after the application process, and a diamond abrasive grain adhesion process in which diamond abrasive grains 22 come into contact with a bond paste face on which the viscous substance 40 adheres from a hole drilled part 31 of the screen 30 to form the substrate and the diamond layer integrally with each other by melting the bond after binder removing treatment.



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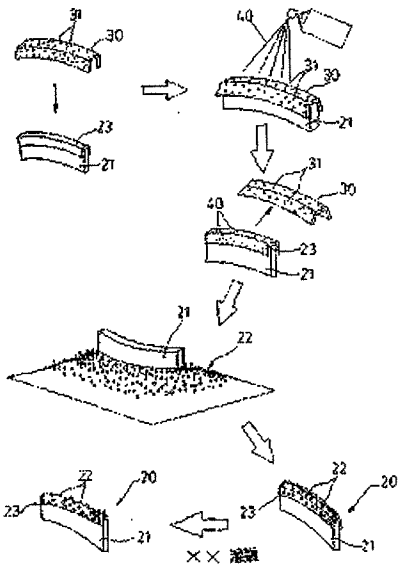
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最終頁に続く

(54)【発明の名称】　ダイヤモンドカッターの製造方法及びダイヤモンドカッター並びにダイヤモンドカッター製造治具

(57)【要約】
【課題】　本発明の目的はダイヤモンド層を形成するダイヤモンド粒子が互いに所定間隔をもってダイヤモンド層に固着され、ダイヤモンド粒子の固着強度を高めてダイヤモンド粒子の脱落を防止できるダイヤモンドカッターの製造方法及びダイヤモンドカッター並びにダイヤモンドカッター製造治具を提供する。
【解決手段】　ダイヤモンドカッターＣの製造方法は、基板１０にバインダとボンド粉とを混練したボンド（登録商標）ペーストを取着させるボンドペースト取着工程と、ボンドペースト取着工程の後で所望に穿孔されたスクリーン３０をボンドペースト取着範囲に被覆するスクリーン被覆工程と、ボンドペーストを被覆したスクリーン３０上から粘性物質４０を塗布する塗布工程と、塗布工程の後でスクリーン３０を取り除くスクリーン除去工程と、スクリーン３０の穿孔部分３１から粘性物質４０が付着したボンドペースト面にダイヤモンド砥粒２２を接触させるダイヤモンド砥粒付着工程と、脱バインダ処理後ボンドを溶融させることによって基板１０とダイヤモンド層を一体に形成する。



1

【特許請求の範囲】

【請求項１】　取付部が形成された基板と、該基板の外周端面に取着されたダイヤモンド層と、を備えたダイヤモンドカッターの製造方法において、前記基板にバインダとボンド粉とを混練したボンドペーストを取着させるボンドペースト取着工程と、該ボンドペースト取着工程の後で所望に穿孔されたスクリーンを前記ボンドペースト取着範囲に被覆するスクリーン被覆工程と、前記ボンドペーストを被覆したスクリーン上から粘性物質を塗布する塗布工程と、該塗布工程の後で前記スクリーンを取り除くスクリーン除去工程と、前記スクリーンの穿孔部分から粘性物質が付着した前記ボンドペースト面にダイヤモンド砥粒を接触させるダイヤモンド砥粒付着工程と、脱バインダ処理後前記ボンドを溶融させることによって基板とダイヤモンド層を一体に形成してなることを特徴とするダイヤモンドカッターの製造方法。

【請求項２】　取付部が形成された基板と、該基板の外周端面に取着されたダイヤモンド層と、を備えたダイヤモンドカッターであって、前記ダイヤモンド層はボンド材料により所定間隔でダイヤモンド砥粒が配列されて前記基板に固定されていることを特徴とするダイヤモンドカッター。

【請求項３】　取付部が形成された基板と、該基板の外周端面に取着されたダイヤモンド層と、を備えたダイヤモンドカッターの製造過程で用いられるダイヤモンドカッター製造治具であって、ダイヤモンド層となる部分を被覆するカバー部材と、該カバー部材に所望間隔で形成された多数の小孔とから形成されていることを特徴とするダイヤモンドカッター製造治具。

【発明の詳細な説明】

【０００１】

【発明の属する技術分野】本発明はダイヤモンドカッターの製造方法及びダイヤモンドカッター並びにダイヤモンドカッター製造治具に係り、特に石材、コンクリートなどの切断に用いるのに好適なダイヤモンドカッターの製造方法及びダイヤモンドカッター並びにダイヤモンドカッター製造治具に関するものである。

【０００２】

【従来の技術】従来、取り付け部を有する基板の外周にダイヤモンド層が形成されたダイヤモンドカッターは公知である。そして、このようなダイヤモンドカッターとしては、例えば特公昭５５－２２１９４号公報で示されているように、「一時的な結合剤を使用して金属基材表面にダイヤモンド結晶とろう付け用合金の粒子を附着させて組立てたものを造る工程と、該組合せを還元性雰囲気または真空中で約１３００℃以下の温度迄に加熱してろう付け用合金を熔融する工程、該組合せを冷却して該ろう付け用合金を固化させダイヤモンドを基材に結合する工程よりなり、ここに該ろう付け用合金はニッケルおよびコバルトからなる群から選ばれた１種または２種の

2

金属の少なくとも５０重量%、クロムの２～２６重量%、および硼素、珪素、錫より成る群より選ばれた１種または２種以上の元素の合計１２重量%以下よりなる、金属基材にダイヤモンド結晶を結合する」技術が知られている。

【０００３】また特開平９－２７２０６０号公報で示されているように、「化学的または物理的に表面処理された前記砥石刃取付部に、有機溶剤と結合材として自溶合金粉末の混合物を塗布し、その後乾燥させ、さらに有機溶剤を塗布し、砥粒を散布し、乾燥させ次いで、非酸化性雰囲気中において９００～１１５０°Ｃの加熱温度に加熱し、前記自溶合金の融着により前記砥石刃取付部に前記砥粒を固定し、前記加熱温度から徐々に冷却する」技術が知られている。

【０００４】上記従来技術では、基板等に取着させたボンドペーストにダイヤモンド砥粒を附着させるときに篩を利用したり、直接散布することにより、ダイヤモンド層を形成している。

【０００５】

【発明が解決しようとする課題】上述のように、ダイヤモンド砥粒を篩や散布することによっているため、ダイヤモンド層を形成するボンドペーストにダイヤモンド粒子が、一部分に団塊となって附着したり、ダイヤモンド粒子同士が隣接して結合することになり、ダイヤモンド粒子の固着が弱くなるという不都合があった。すなわち、このようにダイヤモンド粒子が団塊になったり、ダイヤモンド粒子が隣接した状態は、ダイヤモンド粒子を固定するためのボンドペーストが、ダイヤモンド粒子の周りに少ない状態となり、ダイヤモンド粒子の固着力の低下を意味するものである。

【０００６】本発明の目的は、ダイヤモンド層を形成するダイヤモンド粒子が互いに所定間隔をもってダイヤモンド層に固着され、ダイヤモンド粒子の固着強度を高めてダイヤモンド粒子の脱落を防止できるダイヤモンドカッターの製造方法及びダイヤモンドカッター並びにダイヤモンドカッター製造治具を提供することにある。

【０００７】

【課題を解決するための手段】上記課題は、請求項１のダイヤモンドカッターの製造方法によれば、取付部が形成された基板と、該基板の外周端面に取着されたダイヤモンド層と、を備えたダイヤモンドカッターの製造方法において、前記基板にバインダとボンド粉とを混練したボンドペーストを取着させるボンドペースト取着工程と、該ボンドペースト取着工程の後で所望に穿孔されたスクリーンを前記ボンドペースト取着範囲に被覆するスクリーン被覆工程と、前記ボンドペーストを被覆したスクリーン上から粘性物質を塗布する塗布工程と、該塗布工程の後で前記スクリーンを取り除くスクリーン除去工程と、前記スクリーンの穿孔部分から粘性物質が付着した前記ボンドペースト面にダイヤモンド砥粒を接触させ

るダイヤモンド砥粒付着工程と、脱バインダ処理後前記ボンドを溶融させることによって基板とダイヤモンド層を一体に形成してなる構成とすることにより、解決される。

【０００８】上記のように、ボンドペースト取着工程の後で所望に穿孔されたスクリーンを前記ボンドペースト取着箇所に被覆するスクリーン被覆工程と、前記ボンドペーストを被覆したスクリーン上から粘性物質を塗布する塗布工程を行なうことにより、ボンドペーストに塗布される粘性物質は、スクリーンを除去すると、スクリーンの小孔が形成された範囲だけとなる。したがって、スクリーンを除去した後でダイヤモンド砥粒を接触させると、ダイヤモンド粒子は、粘性物質が塗布された個所、すなわちスクリーンに形成された小孔の位置に付着することになる。このようにしてスクリーンに形成された小孔にしたがって、ダイヤモンド粒子を所定間隔で付着させたダイヤモンド層を形成することができる。

【０００９】また、上記課題は、請求項２に係るダイヤモンドカッターによれば、取付部が形成された基板と、該基板の外周端面に取着されたダイヤモンド層と、を備えたダイヤモンドカッターであって、前記ダイヤモンド層はボンドペーストにより所定間隔でダイヤモンド砥粒が配列されて前記基板に固定されている構成とすることにより、解決される。

【００１０】このようなダイヤモンド層はボンドペーストにより所定間隔でダイヤモンド砥粒が配列されて前記基板に固定されているダイヤモンドカッターによれば、ダイヤモンド粒子が所定間隔で配置されているために、ダイヤモンド粒子の固着強度が高まる。そして、ダイヤモンド層に均一にダイヤモンド粒子を配置して固着されているために、被切削材に対して均一な切り口となり、切削がスムーズに行なわれることになる。

【００１１】さらに、上記課題は、請求項３に係るダイヤモンドカッター製造治具によれば、取付部が形成された基板と、該基板の外周端面に取着されたダイヤモンド層と、を備えたダイヤモンドカッターの製造過程で用いられるダイヤモンドカッター製造治具であって、ダイヤモンド層となる部分を被覆するスクリーン部材と、該スクリーン部材に所望間隔で形成された多数の小孔とから形成されている構成とすることにより、解決される。

【００１２】このようなスクリーン部材によれば、スクリーン部材に形成する小孔の間隔や数を調整することにより、ダイヤモンド粒子を一定間隔で所望数配置することが可能となり、ダイヤモンドカッターにおけるダイヤモンド粒子の配置調整が可能となる。またダイヤモンド粒子が団塊になったり、隣接して配置されることがないために、各ダイヤモンド粒子が溶融ボンドによって確実に基板に固定できることになる。

【００１３】

【発明の実施の形態】ここでは、ダイヤモンドカッター

としてダイヤモンドチップを用いた例を示すが、これに限定されるものではなく、基板に直接ダイヤモンド層を形成してもよい。本発明のダイヤモンドカッターＣは、基板１０にダイヤモンドチップ２０を取着しており、このダイヤモンドチップは、基台にバインダペーストとボンド粉とを混練したボンドペーストを取着させ、ボンドペースト取着箇所に所望に穿孔されたスクリーンを被覆し、このスクリーン上から粘性物質を塗布し、塗布工程の後でスクリーンを取り除き、スクリーンの穿孔部分から粘性物質が付着したボンドペースト面にダイヤモンド砥粒を接触させ、脱バインダ処理後ボンドを溶融させることによって基板とダイヤモンド層を一体に形成することにより製造することが可能となる。

【００１４】以上のように、ダイヤモンド砥粒のばらつきがなく、均等に配置することが可能となり、ダイヤモンド層の全ての部分で切削が均等に行うことが可能となる。

【００１５】

【実施例】以下、本発明の一実施形態について、図を参照して説明する。なお、以下に説明する部材、配置等は、本発明を限定するものではなく、本発明の趣旨に沿って各種改変することができることは勿論である。

【００１６】図１乃至図６は本発明に係る実施例を示すものであり、図１はダイヤモンドカッターの一例としてダイヤモンドチップとして適用したときの製造工程を示す説明図、図２乃至図４は製造工程の拡大説明図、図５は製造工程のブロック図、図６はダイヤモンドカッターの一例を示す正面図である。また図７はダイヤモンドカッター製造治具としてのスクリーンの例を示す説明図である。

【００１７】本例のダイヤモンドカッターＣは、図６で示すように、基板１０と、ダイヤモンドチップ２０と、ダイヤモンド粒子（砥粒）２２と、を主要構成要素としており、ダイヤモンドチップ２０は、基台２１とダイヤモンド粒子（砥粒）２２とを固定するボンドペースト層２３から構成されている。そして上記円形基板１０の外周に、ダイヤモンド粒子（砥粒）２２が取着されたダイヤモンドチップ２０を焼結等により固着している。

【００１８】本例の基板１０は、鋼製であり、この鋼製基板１０は中心に取付孔１１が形成され、円形形状として形成されている。本例の基板１０は、冷間プレスで成形されている。また、本例の基板１０は、外周端面が同一円の面に形成され、所定間隔で冷却用溝等が形成されているものを用いている。このとき、基板１０の外周端面に若干の凹凸等が形成されている場合には、基台２１を基板外周端面と整合するような形状に成形する。しかし、外周端面に若干の凹凸等が形成されたり、溝を設けていないものを用いても良い。

【００１９】次に本発明に係るダイヤモンドカッターＣの製造方法について、図１乃至５を参照して説明する。

本発明のダイヤモンドカッターＣは、基板１０にダイヤモンドチップ２０を取着しており、このダイヤモンドチップ２０は、基台２１にバインダペーストとボンド粉とを混練したボンドペースト２３を取着させ、ボンドペースト取着範囲に所望に穿孔されたスクリーン３０を被覆し、このスクリーン３０上から粘性物質４０を塗布し、塗布工程の後でスクリーン３０を取り除き、スクリーン３０の穿孔部分から粘性物質４０が付着したボンドペースト面にダイヤモンド砥粒２２を接触させ、少なくともボンドペースト２３中のバインダを除去した後、ダイヤモンド砥粒２２の周辺部分のボンドを溶融させて基板１０とダイヤモンド層を一体に形成することにより製造する。これをさらに詳細に説明する。

【００２０】はじめにペースト用バインダとボンド粉とを混練してボンドペースト２３を作成する。ボンドペースト２３を構成するペースト用バインダとボンド粉は、公知の材料を使用するものであり、本例で使用するボンドペースト２３としては、ダイヤとの化学的親和力の高いＴｉ、Ｃｒ等を含むボンドであり、ダイヤに対するボンドの濡れが良く、ダイヤ保持力がかなり強化されている。基台２１（基板１０）との接着力は、ボンドと基台２１（基板１０）が化学的に結合され、溶融に伴う拡散もあるので、接着力は比較的強固となる。具体的にはＣｕ－Ｓｎ－Ｔｉ系や、Ｎｉロウ系のボンドと、各種のバインダ用ペーストを用いてボンドペーストを形成している。

【００２１】本例で用いる粘性物質４０としては、例えば、市販の水溶性メチルセルロースを約３．３倍量の水で希釈した有機糊剤、その他糊材等の粘着のある物質を使用する。

【００２２】なお溶融のボンド合金化は９４０℃～９８０℃が適正温度範囲であり、ボンドとダイヤの濡れ具合が良好となる。また適正脱脂条件について説明すると、脱脂の昇温速度が速すぎると急激なバインダの分解ガスでボンドとダイヤの飛散が生じる。そこで、真空脱脂はバインダ熱分解温度まで５℃／ｍｉｎで昇温し２０ｍｉｎ保持の条件が有効である。使用金属粉としては、ブロンズ粉が溶融温度が低めで溶融状態が安定している。またＣｕＰの添加をすることにより、濡れ性を改善することができる。

【００２３】次に図１に示すように、上記混練されたペースト用バインダとボンド粉からなるボンドペースト２３を、ディスペンサなどの射出機や刷毛を使用して、ダイヤモンドチップ２０を構成する基台２１に塗布等により取着する。そして、次に述べるスクリーン３０を固着させないように、若干の所定時間を経過させて、乾燥させる。

【００２４】次いで、基台２１のボンドペースト２３の取着範囲にスクリーン３０を被覆する。本例のスクリーン３０は、断面コ字状の枠体であり、ボンドペースト２３が取着された基台端部に嵌合可能に形成されている。スクリーン３０はボンドペースト２３の取り付けを考慮して、基台２１より若干大き目で基台２１と略同形状に形成されている。そして、基台２１にスクリーン３０を被覆するように嵌合させる。

【００２５】なおスクリーン３０は基台端部を連続して被覆するように形成しても良く、或いは所定長さに形成し、この所定長さに形成された複数のスクリーン３０を用いて基台端部を被覆するように構成しても良い。スクリーン３０には、所望箇所に多数の小孔３１が穿孔されており、この小孔３１を介して、粘性物質４０が基台２１に塗布されるように構成されている。

【００２６】本例のスクリーン３０は、アルミ等の金属から形成されている。このようにスクリーン３０を金属製とすることにより、表面や小孔３１に付着した粘性物質４０を取り除いて再使用することが可能となり、低コスト化を実現するとともに、廃棄物を減少させて環境への影響を抑えることができる。またスクリーン３０を単一金属で形成することにより、リサイクルが可能となり資源の有効利用を図ることが可能となる。

【００２７】上記スクリーン３０の形状は、上記形状に限定されるものではなく、基台２１（基板１０）の端部形状に合わせて、各種形成するものである。例えば図７（ａ）に示すスクリーン３０は、端部側が薄く中心部に向けて厚みが増した形状の基台２１（基板１０）に用いられるものであり、図７（ｂ）に示すスクリーン３０は、端部がアール形状の基台２１（基板１０）に用いられるものである。さらに図７（ｃ）に示すスクリーン３０は端部側が薄く、且つ端部がアール形状の基台２１（基板１０）に用いられるものである。

【００２８】また、スクリーン３０は上記金属製に限らず、紙製或いは樹脂フィルム製とすることもできる。紙製や樹脂フィルム製のスクリーン３０とすれば、製造時において、各基台２１（基板１０）に常に新しいスクリーン３０を使用することが可能となり、生産効率の向上を図ることができる。

【００２９】そして基台２１に配設されたスクリーン３０に、粘性物質４０をディスペンサなどの射出機や刷毛を使用して塗布する。塗布された粘性物質４０は、スクリーン３０の小孔３１を介して基台２１に付着する。

【００３０】粘性物質４０を塗布後、基台２１からスクリーン３０を取り外す。スクリーン３０が取り外された基台２１表面には、図３に示すように、スクリーン３０の小孔３１と同様のパターンで粘性物質４０が付着している。

【００３１】次に基台２１にダイヤモンド砥粒２２を付着させる。ダイヤモンド砥粒２２としては通常４０～５０メッシュのダイヤモンド粒子を使用する。ダイヤモンド砥粒２２は、図１及び図３に示すように、予め作業台等の上に所定量が載せられており、このダイヤモンド砥

粒２２中に前記基台２１の粘性物質４０が付着した側を接触させ、基台２１にダイヤモンド砥粒２２を付着させる。

【００３２】次に基台２１にダイヤモンド砥粒２２を一体に形成するため、加熱炉の炉内にセットし、排気を行う。処理は、乾燥、昇温、減圧脱脂、溶融、炉冷（真空冷却）、取り出しの順で行なう。なお製造における加熱条件としては、温度１２０℃で乾燥し、５℃／ｍｉｎで昇温し、脱脂は、１８０℃２０ｍｉｎ保持～３８０℃２０ｍｉｎ保持で行なうと好適である。次に溶融について昇温を３０℃／ｍｉｎで行い、９００℃～１１００℃で１５ｍｉｎ保持する。次に、炉冷（真空冷却）を行なうが、約７℃／ｍｉｎで行なう。さらに外熱炉体移動を６００℃で行ない、その後取り出す。なお上記では、溶融接合時の炉内真空度１０－５～１０－６Ｔｏｒｒとしている。

【００３３】上記製造工程により、基台２１にダイヤモンド粒子を単一層に保持したダイヤモンドチップ２０を得ることができる。そしてこのダイヤモンドチップ２０を基板１０に取着してダイヤモンドカッターＣが形成される。なお上記実施例では複数の基台２１にダイヤモンド砥粒２２を取着させ、基台２１を基板１０に貼着した構成例を示したが、基板１０に直接ダイヤモンド砥粒２２を取着させた構成としても良い。またダイヤモンドカッターＣとしては、円形基板の外周にダイヤモンド層が形成されたダイヤモンドホイール、筒状の一方端面側にダイヤモンドチップを取りつけたコアビット、皿状のダイヤモンドカッター等各種形態のものとすることが可能である。

【００３４】上記各種形態のダイヤモンドカッターを製造するときも、スクリーンとしては、上記各ダイヤモンド層を形成する構成に合わせて、各種形成するものである。スクリーンの形状をダイヤモンドチップの形状に合わせて構成することにより、所望形状のダイヤモンド砥粒層を取着することができる。

【００３５】

【発明の効果】以上のように本発明に係るダイヤモンドカッターの製造方法によれば、ボンドペースト取着工程の後で所望に穿孔されたスクリーンを前記ボンドペースト取着範囲に被覆するスクリーン被覆工程と、前記ボンドペーストを被覆したスクリーン上から粘性物質を塗布する塗布工程を行なうことにより、ボンドペーストに塗布される粘性物質は、スクリーンを除去すると、スクリーンの小孔が形成された範囲だけとなる。したがって、

スクリーンを除去した後でダイヤモンド砥粒を接触させると、ダイヤモンド粒子は、粘性物質が塗布された個所、すなわちスクリーンに形成された小孔の位置に付着することになる。このようにしてスクリーンに形成された小孔にしたがって、ダイヤモンド粒子を所定間隔で付着させたダイヤモンド層を形成することができる。

【００３６】また、本発明のダイヤモンドカッターによれば、ダイヤモンド粒子が所定間隔で配置されているために、ダイヤモンド粒子の固着強度が高まる。そして、ダイヤモンド層に均一にダイヤモンド粒子を配置して固着されているために、被切削材に対して均一な切り口となり、切削がスムーズに行なわれることになる。

【００３７】さらに、本発明のダイヤモンドカッター製造治具としてのスクリーン部材によれば、スクリーン部材に形成する小孔の間隔や数を調整することにより、ダイヤモンド粒子を一定間隔で所望数配置することが可能となり、ダイヤモンドカッターにおけるダイヤモンド粒子の配置調整が可能となる。またダイヤモンド粒子が団塊になったり、隣接して配置されることがないために、各ダイヤモンド粒子が溶融ボンドによって確実に基板に固定できることになる。

【図面の簡単な説明】

【図１】本発明をダイヤモンドカッターの一例としてダイヤモンドチップとして適用したときの製造工程を示す説明図である。

【図２】図１の製造工程の拡大説明図である。

【図３】図１の製造工程の拡大説明図である。

【図４】図１の製造工程の拡大説明図である。

【図５】製造工程のブロック図である。

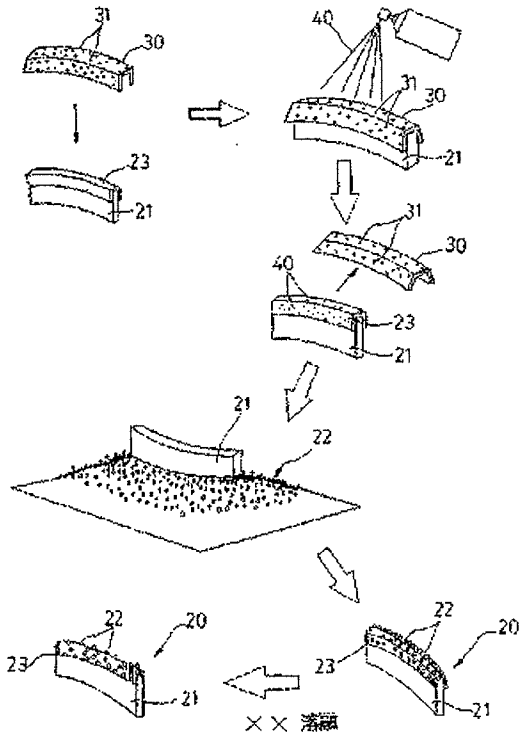
【図６】ダイヤモンドカッターの一例を示す正面図である。

【図７】ダイヤモンドカッター製造治具としてのスクリーンの例を示す説明図である。

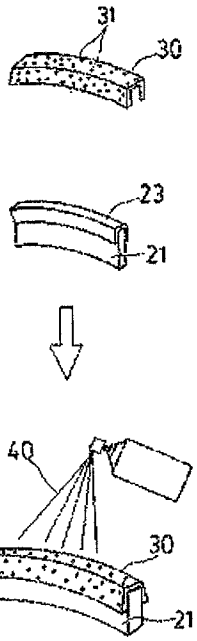
【符号の説明】

１０　基板
１１　取付孔
２０　ダイヤモンドチップ
２１　基台
２２　ダイヤモンド砥粒
２３　ボンドペースト
３０　スクリーン
３１　小孔
４０　粘性物質
Ｃ　ダイヤモンドカッター

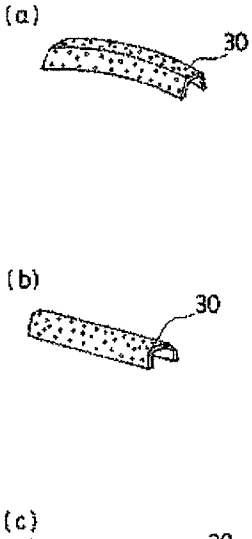
【図１】


31
30
40
23
21
22
20
××溶融

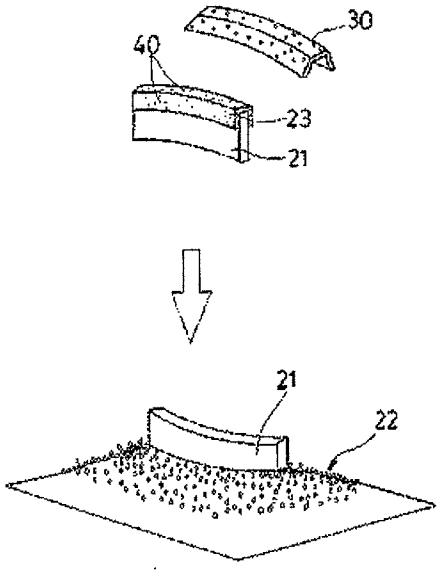
【図２】


31
30
23
21
40

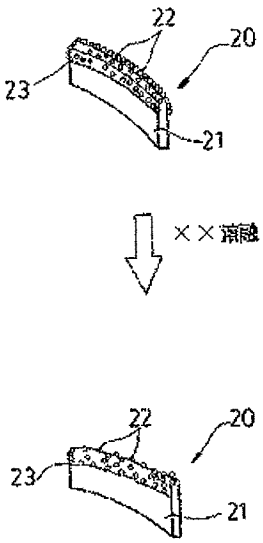
【図７】


(a)
30
(b)
30
(c)
30

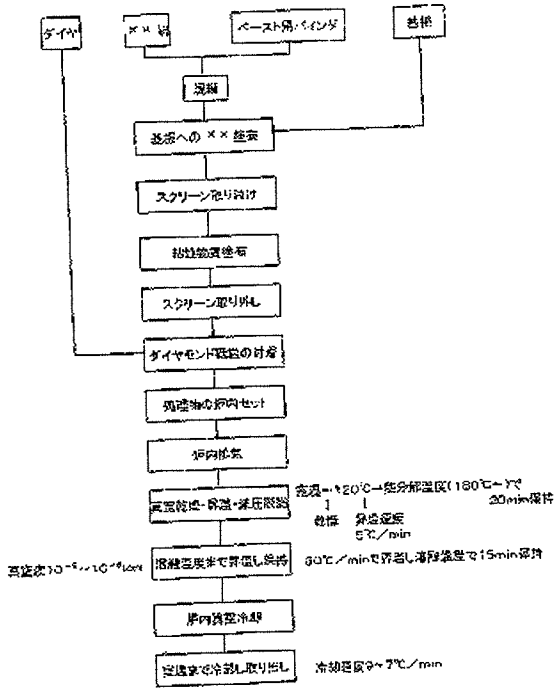
【図３】


30
40
23
21
22

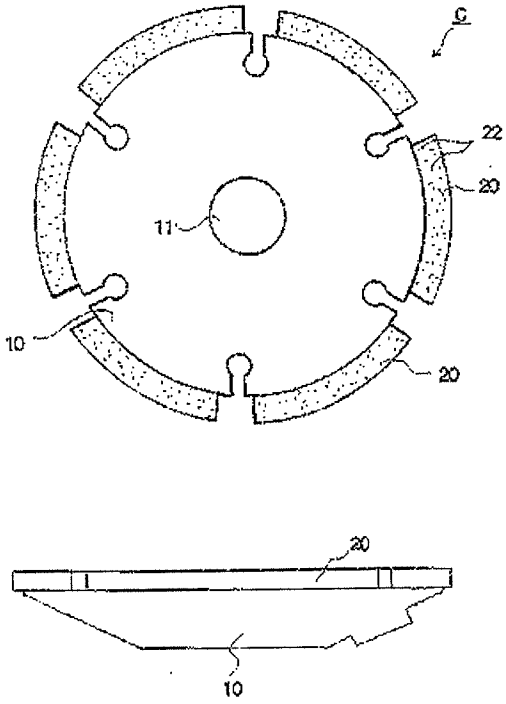
【図４】


22
20
23
21
××溶融

【図５】


ダイヤ
××粉
ペースト用バインダ
基板
混練
基板への××塗布
スクリーン抜り設け
粘性物質塗布
スクリーン取り外し
ダイヤモンド粒子の付着
処理物の炉内セット
炉内排気
減圧乾燥・昇温・減圧脱脂
1
1
数時
昇温速度
5℃／min
20min保持
真空度10⁻³～10⁻⁴Torr
溶融温度以上で昇温し保持
30℃／minで所定の溶融温度で15min保持
炉内降温冷却
室温まで冷却し取り出し
冷却速度3～7℃／min

【図６】



フロントページの続き

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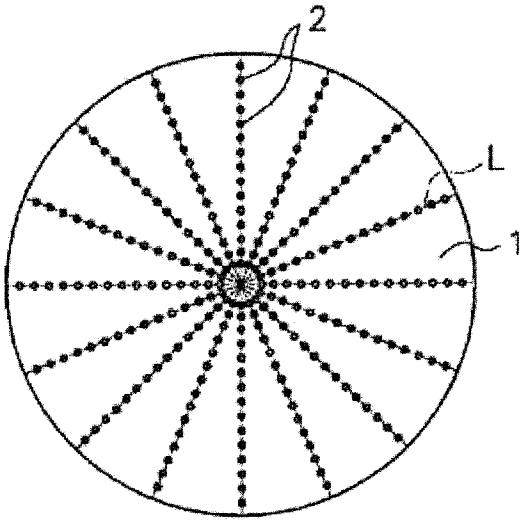
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(54) CMP CONDITIONER, METHOD FOR ARRANGING HARD ABRASIVE GRAIN USED IN CMP CONDITIONER AND METHOD FOR MANUFACTURING CMP CONDITIONER

(57)Abstract:
PROBLEM TO BE SOLVED: To provide stable CMP conditioner characteristics and to relieve slurry or the like in polishing without forming relief groove or the like on a support member.
SOLUTION: This CMP conditioner of polishing cloth for a semiconductor substrate is composed of the support member 1 and plural diamond grains 2 brazed on a surface of the supporting member 1. Plural straight lines or curved lines radially extending from a center of the support member 1 are laid out on the surface of the disk-shaped support member 1 and the diamond grains 2 are arranged on the straight lines or the curved lines. The diamond grains 2 are arranged in such a manner that density of the diamond grains 2 at edge parts is smaller than that at a center par of the support member 1. Since area where no diamond grain 2 exists is secured in radial shape on the surface of the support member 1, slurry can be relieved outward from the support member 1 in polishing.



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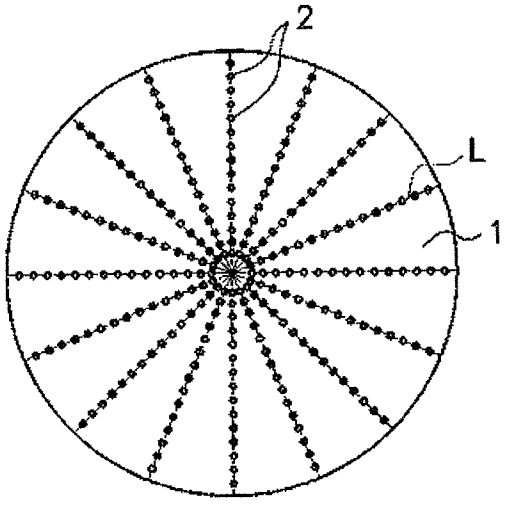
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最終頁に続く

(54)【発明の名称】　ＣＭＰコンディショナー，ＣＭＰコンディショナーに使用する硬質砥粒の配列方法、及びＣＭＰコンディショナー製造方法

(57)【要約】
【課題】　安定したＣＭＰコンディショナー特性が得られるとともに，支持部材に逃し溝等を形成しなくとも研磨時にスラリー等を逃すことができるようにする。
【解決手段】　半導体基盤用研磨布のＣＭＰコンディショナーは，支持部材１と，支持部材１の面上にろう付けされた複数のダイヤモンド粒２とからなり，円板状の支持部材１の表面において，支持部材１の中心から放射状に伸びる複数の直線或いは曲線を考え，それら直線或いは曲線上にダイヤモンド粒２を配置するようにしている。このようにしたＣＭＰコンディショナーでは，ダイヤモンド粒２が支持部材１の中央部分に比べて縁部分における密度が小さくなるよう配列されており，支持部材１の表面上には，ダイヤモンド粒２が存在しない領域が放射状に確保されることになるので，研磨時にスラリーを支持部材１の外側に向けて逃すことができる。



1

【特許請求の範囲】

【請求項１】　支持部材と、前記支持部材の面上に設けられた複数の硬質砥粒とを備えたＣＭＰコンディショナーであって、

前記支持部材の面上に、前記複数の硬質砥粒を規則的に、かつ、前記支持部材の内側から外側にかけて密度が減少するように配列させたことを特徴とするＣＭＰコンディショナー。

【請求項２】　前記硬質砥粒を、前記支持部材の中心から略放射状に配列させていることを特徴とする請求項１に記載のＣＭＰコンディショナー。

【請求項３】　支持部材と、前記支持部材の面上に設けられた複数の硬質砥粒とを備えたＣＭＰコンディショナーであって、

前記支持部材の面上に、前記複数の硬質砥粒が存在しない領域を略放射状に確保していることを特徴とするＣＭＰコンディショナー。

【請求項４】　前記硬質砥粒はダイヤモンド粒であることを特徴とする請求項１～３のいずれか１項に記載のＣＭＰコンディショナー。

【請求項５】　チタン、クロム、又はジルコニウムより選ばれた１種以上を０．５～２０ｗｔ％含む融点６５０℃～１２００℃の合金を用いて、前記ダイヤモンド粒を金属及び／又は合金からなる前記支持部材に、単層、ろう付けし、前記ダイヤモンド粒と前記合金との界面にチタン、クロム、又はジルコニウムより選ばれた金属の炭化物層が形成されていることを特徴とする請求項４に記載のＣＭＰコンディショナー。

【請求項６】　前記融点６５０℃～１２００℃の合金がニッケル基合金であることを特徴とする請求項５に記載のＣＭＰコンディショナー。

【請求項７】　規則的に、かつ、内側から外側にかけて密度が減少するように配列させた複数の貫通穴が形成された薄板状の配列部材を被配列面上に位置させる手順と、

前記配列部材の各貫通穴に硬質砥粒を入れ込む手順とを有することを特徴とするＣＭＰコンディショナーに使用する硬質砥粒の配列方法。

【請求項８】　複数の貫通穴の存在しない領域が略放射状に確保された薄板状の配列部材を被配列面上に位置させる手順と、

前記配列部材の各貫通穴に硬質砥粒を入れ込む手順とを有することを特徴とするＣＭＰコンディショナーに使用する硬質砥粒の配列方法。

【請求項９】　前記被配列面は、ＣＭＰコンディショナーを構成する支持部材の表面であることを特徴とする請求項７又は８に記載のＣＭＰコンディショナーに使用する硬質砥粒の配列方法。

【請求項１０】　複数の硬質砥粒を規則的に、かつ、内側から外側にかけて密度が減少するように配列させた状態で保持部材に保持する手順と、

前記保持部材により保持された硬質砥粒を、ＣＭＰコンディショナーを構成する支持部材の表面に転写する手順とを有することを特徴とするＣＭＰコンディショナーに使用する硬質砥粒の配列方法。

【請求項１１】　複数の硬質砥粒の存在しない領域が略放射状に確保された状態で前記複数の硬質砥粒を保持部材に保持する手順と、

前記保持部材により保持された硬質砥粒を、ＣＭＰコンディショナーを構成する支持部材の表面に転写する手順とを有することを特徴とするＣＭＰコンディショナーに使用する硬質砥粒の配列方法。

【請求項１２】　前記保持部材には前記硬質砥粒を保持するための第１の接着手段を設け、前記支持部材の表面には第２の接着手段を設け、これら第１、２の接着手段の性質に差を持たせたことを特徴とする請求項１０又は１１に記載のＣＭＰコンディショナーに使用する硬質砥粒の配列方法。

【請求項１３】　請求項７～１２のいずれか１項に記載のＣＭＰコンディショナーに使用する硬質砥粒の配列方法を利用して前記硬質砥粒を前記支持部材の表面上に配列させた後、前記硬質砥粒を前記支持部材の表面に固着することを特徴とするＣＭＰコンディショナー製造方法。

【発明の詳細な説明】

【０００１】

【発明の属する技術分野】本発明は、半導体基板用の研磨布の目詰まりを解消し、異物を除去するのに使用されるＣＭＰコンディショナー、ＣＭＰコンディショナーに使用する硬質砥粒の配列方法、及びＣＭＰコンディショナー製造方法に関する。

【０００２】

【従来の技術】ウェハのポリッシングにおいては、ＣＭＰ(Chemical Mechanical Polishing)と呼ばれる研磨方法が提案されている。ＣＭＰは、機械的研磨作用に化学的研磨作用を重畳して働かせることにより、研磨速度の確保と被研磨材が無欠陥であることの両立を可能としたものであり、シリコンウェハの仕上げポリッシング工程で広く使用されている。

【０００３】また、近年ではデバイスの高集積化に伴い、集積回路を製造する所定の段階で、ウェハ表面やウェハ表面に導電体・誘電体層が形成された半導体基板表面を研磨することが必要になってきた。半導体基板は、研磨されて、高い隆起、引っかき傷、粗さ等の表面欠陥が除去される。通常、この工程は、ウェハ上に種々の素子及び集積回路を形成する間に行われる。この研磨工程では、シリコンウェハの仕上げポリッシング工程と同様に、研磨速度と無欠陥であることの両立が必要である。化学スラリーを導入することにより、半導体表面により大きな研磨除去速度及び無欠陥性が与えられる化学的か

つ機械的平坦化が行われる。

【０００４】ＣＭＰ工程の一例としては、図８に示すように、例えば５～３００ｎｍ程度の粒径を有するシリカ粒子を苛性ソーダ、アンモニア及びアミン等のアルカリ溶液に懸濁させてＰＨ９～１２程度に化学スラリー１０１と、ポリウレタン樹脂等からなる研磨布１０２とが用いられる。研磨時には、化学スラリー１０１を流布しながら、半導体基板１０３を研磨布１０２に適当な圧力で当接させ、同図の矢印に示すように相対回転させることにより研磨が行われる。

【０００５】そして、前記研磨布１０２のコンディショニング法としては、研磨布１０２に水又は化学スラリー１０１を流しながら、ＣＭＰコンディショナーを用いたコンディショニングを行って、研磨布１０２の目詰まりを解消し、異物を除去していた。ＣＭＰコンディショナーを用いたコンディショニングは、半導体基板１０３の研磨が終わった後に、ＣＭＰコンディショナーを研磨布１０２に当接させるか、或いは、半導体基板１０３の研磨と同時に、半導体基板１０３が当接する位置とは別の位置でＣＭＰコンディショナーを研磨布１０２に当接させるかして行われる。

【０００６】

【発明が解決しようとする課題】従来の研磨布のコンディショニングに用いられるＣＭＰコンディショナーでは、図９に示すように、円板状の支持部材２０１の表面に、硬質砥粒としてダイヤモンド粒２０２を人手で撒く等して適当に均一に分布させた後、これらダイヤモンド粒２０２を固着させていた。しかし、この場合、いかに丁寧にダイヤモンド粒２０２を散布したとしても、ダイヤモンド粒２０２は不均一に分布し、ＣＭＰコンディショナー個体間での相違の原因となり、安定したＣＭＰコンディショナー特性の発現が妨げられていた。

【０００７】また、従来のＣＭＰコンディショナーでは、スラリーの逃げが悪いため、マイクロスクラッチが多くなった。また、スラリーの逃げを改良するためには、図１０に示すように、支持部材２０１に化学スラリー１０１を逃すための逃し溝２０３等を形成しておき、研磨時に、この逃し溝２０３を介して化学スラリー１０１を逃すことがなされていた。しかし、支持部材２０１に逃し溝２０３を形成するのでは、ＣＭＰコンディショナー特性に悪影響を与えるおそれがあり、また、その逃し溝の加工に手間がかかり、コストアップの要因となってしまう。

【０００８】本発明は前記のような点に鑑みてなされたものであり、安定したＣＭＰコンディショナー特性が得られるとともに、逃し溝等を形成しなくとも研磨時にスラリー等を逃すことができ、マイクロスクラッチを減らすようにすることを目的とする。

【０００９】

【課題を解決するための手段】本発明のＣＭＰコンディショナーは、支持部材と、前記支持部材の面上に設けられた複数の硬質砥粒とを備えたＣＭＰコンディショナーであって、前記支持部材の面上に、前記複数の硬質砥粒を規則的に、かつ、前記支持部材の内側から外側にかけて密度が減少するように配列させた点に特徴を有する。

【００１０】本発明の他のＣＭＰコンディショナーは、支持部材と、前記支持部材の面上に設けられた複数の硬質砥粒とを備えたＣＭＰコンディショナーであって、前記支持部材の面上に、前記複数の硬質砥粒が存在しない領域を略放射状に確保している点に特徴を有する。

【００１１】本発明のＣＭＰコンディショナーに使用する硬質砥粒の配列方法は、規則的に、かつ、内側から外側にかけて密度が減少するように配列させた複数の貫通穴が形成された薄板状の配列部材を被配列面上に位置させる手順と、前記配列部材の各貫通穴に硬質砥粒を入れ込む手順とを有する点に特徴を有する。

【００１２】本発明の他のＣＭＰコンディショナーに使用する硬質砥粒の配列方法は、複数の貫通穴の存在しない領域が略放射状に確保された薄板状の配列部材を被配列面上に位置させる手順と、前記配列部材の各貫通穴に硬質砥粒を入れ込む手順とを有する点に特徴を有する。

【００１３】本発明の他のＣＭＰコンディショナーに使用する硬質砥粒の配列方法は、複数の硬質砥粒を規則的に、かつ、内側から外側にかけて密度が減少するように配列させた状態で保持部材に保持する手順と、前記保持部材により保持された硬質砥粒を、ＣＭＰコンディショナーを構成する支持部材の表面に転写する手順とを有する点に特徴を有する。

【００１４】本発明の他のＣＭＰコンディショナーに使用する硬質砥粒の配列方法は、複数の硬質砥粒の存在しない領域が略放射状に確保された状態で前記複数の硬質砥粒を保持部材に保持する手順と、前記保持部材により保持された硬質砥粒を、ＣＭＰコンディショナーを構成する支持部材の表面に転写する手順とを有する点に特徴を有する。

【００１５】本発明のＣＭＰコンディショナー製造方法は、前記ＣＭＰコンディショナーに使用する硬質砥粒の配列方法を利用して前記硬質砥粒を前記支持部材の表面上に配列させた後、前記硬質砥粒を前記支持部材の表面に固着する点に特徴を有する。

【００１６】

【発明の実施の形態】以下、図面を参照して、本発明の半導体基板用研磨布のＣＭＰコンディショナー、半導体基板用研磨布のＣＭＰコンディショナーに使用する硬質砥粒の配列方法、及びＣＭＰコンディショナー製造方法の実施の形態について説明する。

【００１７】図１を用いて、ＣＭＰコンディショナーについて説明する。同図に示すように、ステンレス鋼等からなる円板状の支持部材１の表面には、硬質砥粒としてダイヤモンド粒２が固着されている。

【００１８】図２、３には、支持部材１の表面におけるダイヤモンド粒２の配列の概要を示す。図２に示す例は、円板状の支持部材１の中心から放射状に伸びる複数の直線（一点鎖線Ｌ）を考え、それら直線上にダイヤモンド粒２を配置したものである。このようにしたＣＭＰコンディショナーでは、ダイヤモンド粒２が支持部材１の内側から外側にかけて密度が小さくなるように配列されており、支持部材１の表面上には、ダイヤモンド粒２の存在しない領域が放射状に確保されることになる。

【００１９】また、図３に示す例は、円板状の支持部材１の中心から放射状に伸びる複数の曲線（一点鎖線Ｌ）を考え、それら曲線上にダイヤモンド粒２を配置したものである。このようにしたＣＭＰコンディショナーでは、ダイヤモンド粒２が支持部材１の内側から外側にかけて密度が小さくなるように配列されており、支持部材１の表面上には、ダイヤモンド粒２が存在しない領域が放射状に確保されることになる。本発明でいう略放射状とは、図２に示すように直線的に放射する場合だけでなく、図３に示すように曲線的に放射する場合も含むものとする。

【００２０】なお、実際のダイヤモンド粒２は、支持部材１に比べて非常に小さなものであるが、図１や後述する図２、３では、説明を簡単にするためダイヤモンド粒２を大きく図示する。また、直線や曲線の数についても、より密な状態で放射させるようにするが、図２、３では簡単に図示する。

【００２１】以下、図４～７を参照して、ダイヤモンド粒２の配列方法について説明する。本実施の形態では、次の２通りの方法により、ダイヤモンド粒２を配列させている。

【００２２】第１の方法では、図４に示すように、ろう材３が設けられた支持部材１の表面に、接着剤４を塗布しておく。そして、接着剤４を塗布した支持部材１の表面上に配列板５を載置して、マスキングする。

【００２３】図５に示すように、配列板５には、ダイヤモンド粒２を配列させるための貫通穴６が形成されている。すなわち、配列板５には、図２や図３に示す配列と同様に貫通穴６を配列させている。貫通穴６の口径Ｘは、ダイヤモンド粒２のサイズＤに対して、１．０Ｄ＜Ｘ＜２．０Ｄとなっており、１つの貫通穴６に１個以上のダイヤモンド粒２が同時に入り込まないようにしている。なお、配列板５の周囲には、飛散防止用壁５ａが設けられている。

【００２４】図４に示すように、前記配列板５を支持部材１の表面に載置した状態で、配列板５上にダイヤモンド粒２を散布する。このとき、配列板５に適当な振動を加える等して、ダイヤモンド粒２が全ての貫通穴６に入り込むようにする。全ての貫通穴６にダイヤモンド粒２が入り込んだならば、配列板５上の余分なダイヤモンド粒２をはけ等を用いて取り除く。その後、配列板５を支持部材１の表面から取り外せば、ダイヤモンド粒２は、図２や図３に示すように配列された状態で支持部材１の表面上に残ることになる。

【００２５】以上述べたようにして支持部材１の表面にダイヤモンド粒２を配列させたならば、単層、ろう付けを行い、ダイヤモンド粒２を固定する。このろう付けの際に、支持部材１の表面に塗布された接着剤４はろう材３への加熱によって昇華し、支持部材１の表面上に残留しない。

【００２６】なお、第１の方法において、配列板５の代わりに、ワイヤで編まれたメッシュを用いてもよい。すなわち、メッシュの各開口部分を配列板５でいう貫通穴６として使用し、該開口部分にダイヤモンド粒２を入れ込んで、支持部材１の表面に配列させる。

【００２７】第２の方法では、前記第１の方法のようにダイヤモンド粒２を支持部材１の表面に直接的に配列するのではなく、粘着シート等の保持部材にいったん配列させてから、支持部材１の表面に転写するようにしている。

【００２８】図６（ａ）に示すように、配列板７には、ダイヤモンド粒２を配列させるための凹部８が形成されている。すなわち、配列板７には、図２や図３に示す配列と同様に凹部８を配列させている。なお、凹部８の口径Ｘを、ダイヤモンド粒サイズＤに対して、１．０Ｄ＜Ｘ＜２．０Ｄとすることは、前記第１の方法で述べた貫通穴６と同じである。

【００２９】前記配列板７上にダイヤモンド粒２を散布する。このときも、前記第１の方法で説明したように、配列板７に適当な振動を加える等して、ダイヤモンド粒２が全ての凹部８に入り込むようにする。全ての凹部８にダイヤモンド粒２が入り込んだならば、配列板７上の余分なダイヤモンド粒２をはけ９等を用いて取り除く。

【００３０】次に、配列板７の凹部８が開口する面に粘着シート１０を貼り付ける。そして、図６（ｂ）に示すように、配列板７の上下を逆にする等して、粘着シート１０を剥がすと、粘着シート１０にダイヤモンド粒２が配列された状態で保持されることになる。

【００３１】前記粘着シート１０のダイヤモンド粒２を保持する粘着面を、接着剤４が塗布された支持部材１の表面に貼り合わせるようにする。したがって、図７に示すように、ダイヤモンド粒２は、一端が粘着シート１０側で、他端が支持部材１の表面側で支持された状態となる。その後、支持部材１の表面側にダイヤモンド粒２を残し、粘着シート１０だけを取り除けば、ダイヤモンド粒２を支持部材１の表面上に配列させることができる。

【００３２】粘着シート１０だけを取り除く手法としては、例えば、粘着シート１０の接着材の溶解性と、支持部材１側の接着剤４の溶解性とに差を持たせておけばよい。この場合、図７に示す状態で粘着シート１０の接着剤が溶けるような環境にすれば、支持部材１側の接着剤

４は保持力を維持したまま、粘着シート１０の接着材だけを溶かし、粘着シート１０だけを取り除くことができる。

【００３３】以上述べたようにして支持部材１の表面にダイヤモンド粒２を配列させたならば、単層、ろう付けを行い、ダイヤモンド粒２を固定する。このろう付けの際に、支持部材１の表面に塗布された接着剤４はろう材３への加熱によって昇華し、支持部材１の表面上に残留しない。

【００３４】なお、第２の方法では、配列板７に凹部８を形成するようにしたが、貫通穴としてよい。この場合、図４に示す支持部材１を粘着シート１０に変更すれば、粘着シート１０にダイヤモンド粒を配列させることができるので、それを支持部材１の表面に転写すればよい。

【００３５】以上述べたように本実施の形態によれば、ダイヤモンド粒２を規則的に配列させているので、ＣＭＰコンディショナー間での個体差がなくなり、安定したＣＭＰコンディショナー特性を得ることができる。また、ダイヤモンド粒２を、支持部材１の中心から略放射状に配列させることにより、支持部材１の内側から外側にかけて密度が小さくなるよう配列するようにし、また、ダイヤモンド粒２が存在しない領域を放射状に確保するようにしたので、研磨時にスラリーを支持部材１の外側に向けて逃すことができ、マイクロスクラッチが減少する。そして、スラリーを逃すための特別な加工を支持部材１に施す必要がなくなるので、加工の手間やコストを軽減させることができる。

【００３６】なお、本実施の形態では、本発明でいう硬質砥粒としてダイヤモンド粒２を用いたが、その他の材質、例えば立方晶窒化ホウ素、炭化ホウ素、炭化珪素又は酸化アルミニウム等からなるものであってもよい。

【００３７】また、ダイヤモンド粒２の支持部材１への固着方法としては、ろう付け以外の方法、例えばニッケル電着等により固着させてもよい。

【００３８】ここで、好適な一例として、ダイヤモンド粒をろう付けにより固着する方法について説明すると、ろう材として、チタン、クロム、又はジルコニウムより選ばれた１種以上を０．５～２０ｗｔ％含む融点６５０℃～１２００℃の合金を用いることにより、ダイヤモンド粒とろう付け合金との界面に当該金属の炭化物層が形成される。ろう材に含まれるチタン、クロム、又はジルコニウムより選ばれた１種以上を０．５～２０ｗｔ％とするのは、０．５ｗｔ％より少ない含有量ではダイヤモンド－ろう付け合金の界面に当該金属の炭化物層が形成されないためであり、２０ｗｔ％添加すれば十分な接合強度を示す炭化物層が形成されるためである。

【００３９】ろう付け合金を融点６５０℃～１２００℃の合金とするのは、６５０℃未満のろう付け温度では、接合強度が得られず、１２００℃超のろう付け温度では、ダイヤモンドの劣化が起こるので好ましくないからである。ろう付け合金としては、例えば、上記成分を含有するニッケル（Ｎｉ）基合金等が好適に用いることができる。

【００４０】ろう付け合金の厚さは、ダイヤモンド粒の０．２～１．５倍の厚さが適当である。薄すぎると、ダイヤモンドとろう付け合金との接合強度が低くなり、厚すぎると、ろう材と支持部材との剥離が起こりやすくなるためである。

【００４１】ダイヤモンド粒の径は、５０μｍ～３００μｍとすることが好ましい。５０μｍ未満の微粒ダイヤモンド粒では、十分な研磨速度が得られず、また、凝集しやすい傾向があり、脱落しやすくなるためである。また、３００μｍ超の粗粒のダイヤモンド粒では、研磨時の応力集中が大きくなり、脱落しやすくなるためである。

【００４２】

【発明の効果】以上述べたように本発明によれば、ＣＭＰコンディショナー間での個体差がなくなり、安定したＣＭＰコンディショナー特性を得ることができるので、安定した量産ＣＭＰプロセスを実現することが可能となる。また、研磨時にスラリーを逃すことができ、マイクロスクラッチを減らすことができ、しかも、そのための特別な加工を支持部材に施す必要がなくなるので、加工の手間やコストを軽減させることができる。

【図面の簡単な説明】

【図１】ＣＭＰコンディショナーについて説明するための図である。

【図２】ダイヤモンド粒２の配列の一例を示す図である。

【図３】ダイヤモンド粒２の配列の一例を示す図である。

【図４】第１の方法によるダイヤモンド粒２の配列方法を説明するための図である。

【図５】配列板５を説明するための図である。

【図６】第２の方法によるダイヤモンド粒２の配列方法を説明するための図である。

【図７】第２の方法によるダイヤモンド粒２の配列方法を説明するための図である。

【図８】ＣＭＰ工程を説明するための図である。

【図９】従来のＣＭＰコンディショナーについて説明するための図である。

【図１０】逃し溝２０３を形成したＣＭＰコンディショナーを示す模式図である。

【符号の説明】

１　支持部材
２　ダイヤモンド粒
３　ろう材
４　接着剤
５　配列板

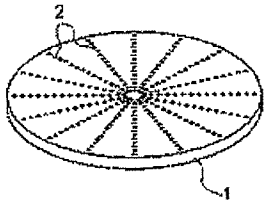
9　　10

5 a　飛散防止用壁
6　貫通穴
7　配列板
8　凹部
9　はけ

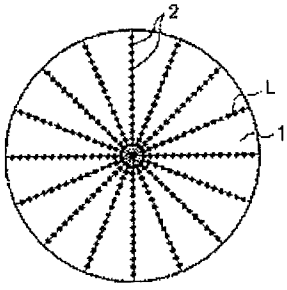
＊１０　粘着シート
１０１　化学スラリー
１０２　研磨布
１０３　半導体基板

＊

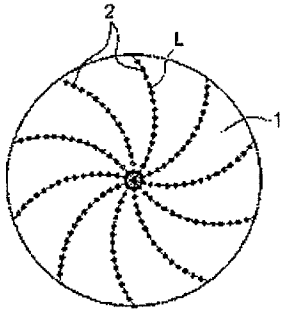
【図１】



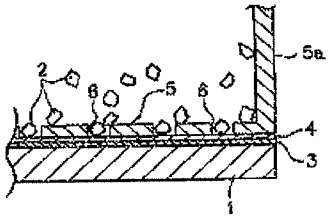
【図２】



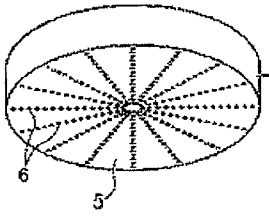
【図３】



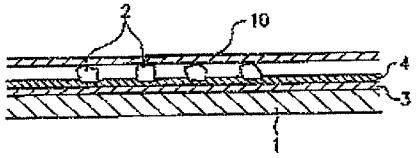
【図４】



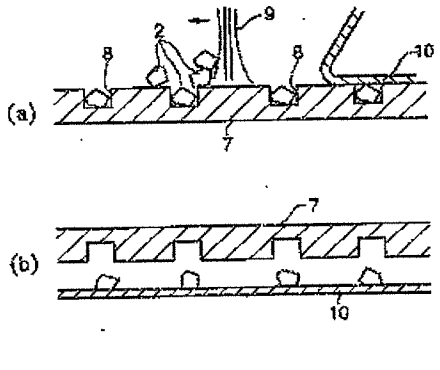
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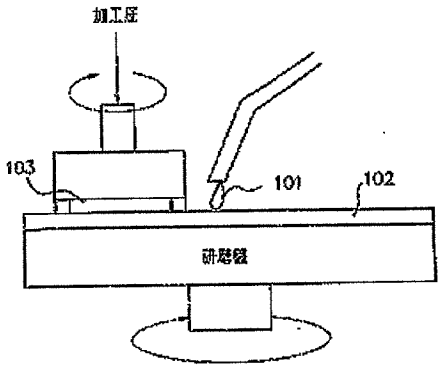
【図７】



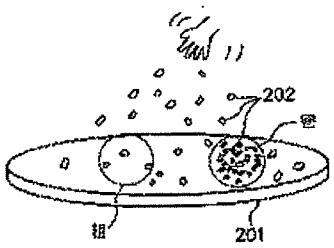
【図６】



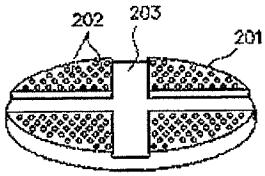
【図８】



【図９】



【図１０】



フロントページの続き

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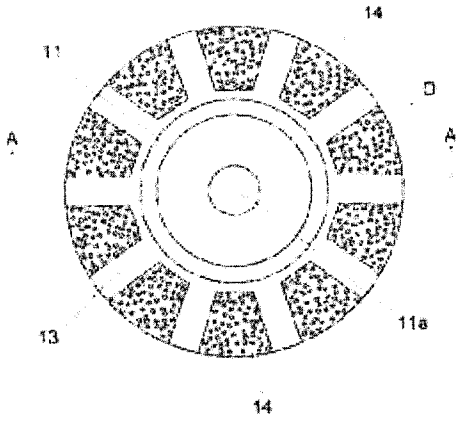
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(54) DISC-SHAPED DIAMOND GRINDING WHEEL

(57)Abstract:
PROBLEM TO BE SOLVED: To provide a disc-shaped diamond grinding wheel hardly causing loading and having flexibility.
SOLUTION: A base plate 11 of the disc diamond grinding wheel is made into a cup form of 10 cm in diameter having flexibility, and a jig mounting hole 11a to mount the base plate 11 on a grinder, etc. is provided on a central part of the base plate 11. Additionally, a rubber sheet 13 is adhered on a do-nut shaped part on a flat surface of a peripheral part of the base plate 11 , and a plurality of polishing chips 14 are adhered along a circumference under a deposition method coating diamond abrasive grains D on its upper layer. As they are supported by the base plate 11 excellent in flexibility, strength, etc. and the rubber sheet 13 transmits its elasticity to the polishing chips 14, the polishing chips 14 respectively and independently polish a polishing article along its surface and also polish the small area part and the curved surface.



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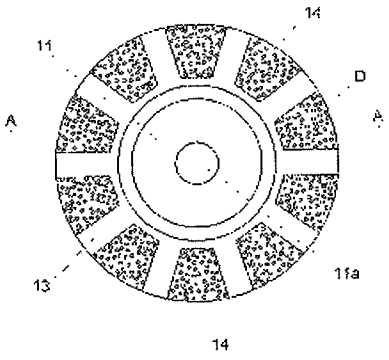
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(54)【発明の名称】ディスク状ダイヤモンド砥石

(57)【要約】
【課題】目詰まりの起こし難く、可撓性を有するディスク状ダイヤモンド砥石を得る。
【解決手段】ディスク状ダイヤモンド砥石の基板１１は可撓性を有する直径１０ｃｍのカップ状とされ、基板１１の中心部には基板１１をグラインダ等に取り付けるための治具取付孔１１ａが設けられている。また、基板１１の周辺部の平面上のドーナツ状部分にはゴムシート１３が接着され、更にその上層にダイヤモンド砥粒Ｄを塗布した溶着法による複数個の研磨チップ１４が円周に沿って接着されている。可撓性、強度等に優れた基板１１に支持され、ゴムシート１３がその弾力性を研磨チップ１４に伝えるため、研磨チップ１４はそれぞれ独立に被研磨物の面に沿って研磨でき、小面積部や曲面も研磨することができる。
【選択図】　図１



【特許請求の範囲】
【請求項１】
ダイヤモンド砥粒を塗布した複数個の金属片から成る研磨チップを可撓性材料から成るディスク状基板上の外周平坦部表面に配列したことを特徴とするディスク状ダイヤモンド砥石。
【請求項２】
前記研磨チップは前記ディスク状基板の円周に沿って等間隔に配列したことを特徴とするディスク状ダイヤモンド砥石。
【請求項３】
前記研磨チップは前記ディスク状基板上に積層したゴム層上に接着した請求項１に記載のディスク状ダイヤモンド砥石。
【請求項４】
前記ダイヤモンド砥粒は溶着法により前記研磨チップに塗布した請求項１に記載のディスク状ダイヤモンド砥石。
【発明の詳細な説明】
【０００１】
【発明の属する技術分野】
本発明は、グラインダ等の回転工具に取り付け、硬質材料表面に塗装された表面塗膜を剥離したり、金属表面の錆を除去したり、或いは研磨をするディスク状ダイヤモンド砥石に関するものである。
【０００２】
【従来の技術】
例えば、既設の橋梁や工場の煙突、タンク等の保守に際して、表面塗膜の補修をする場合において、それらの表面に塗装されている重防食系塗料の剥離はなかなか困難であり、二度、三度の手間を要する難作業である。
【０００３】
通常では、グラインダ等の電動工具の回転により金属片を表面塗膜に打ち付けることにより面を荒らし、その後にディスク状サンドペーパによって塗膜を剥離する。
【０００４】
【発明が解決しようとする課題】
しかしながら上述の従来例においては、金属片を打ち付ける際に、その母材の鉄やコンクリートの打撃音や振動が激しく、母材を痛めることもある。また、ディスク状サンドペーパも重防食塗料により短時間で目詰まりを起こしてしまい、交換頻度も多く、作業効率も極めて悪い。
【０００５】
通常の建物の外壁塗膜の剥離においても、塗料の違いこそあれ、目詰まりによる交換損失を生ずる。目詰まりさえしなければ摩耗し難い砥粒が好適であることは明白であるが、実際には砥粒は摩耗しておらず研磨性能は十分あっても、目詰まりで砥石が使用できなくなるという問題がある。
【０００６】
現在、ダイヤモンド砥石を使用した研磨材としては、図４に模式的に示すような金属粉末を用いてダイヤモンド砥粒を混入して固めたメタルボンドと称される研磨剤１と、図５に示すように表面にメッキによる電着法により製造された研磨剤２が知られている。
【０００７】
しかしながら、前述のメタルボンド製の研磨剤１は、ダイヤモンド砥粒Ｄの突き出しが少なく、目詰まりするため砥膜剥離には不適である。後者の電着製の研磨剤２は砥粒の突き出しは多いが、ダイヤモンド砥粒Ｄとメッキの間に隙間Ｓが発生してメッキが剥離し易く、ダイヤモンド砥粒Ｄの脱落が多く目詰まりを生じ易い。
【０００８】
また、現状のディスク状ダイヤモンド砥石としては、カップ型のものが多く用いられてい

るが、金属製の椀形基板の平坦部にメタルボンド製ダイヤモンドを下駄歯状のブロックにして取り付けたものが多く、騒音、振動共に非常に激しい。

【０００９】

本発明の目的は、上述の問題点を解消し、金属片を打ち付ける作業を不要とし、目詰まりが少なく長時間の作業に耐え、作業音や振動が静かな可撓性を有し、小面積部や曲面をも研磨することのできるディスク状ダイヤモンド砥石を提供することにある。

【００１０】

【課題を解決するための手段】

上記目的を達成するための本発明に係るディスク状ダイヤモンド砥石は、ダイヤモンド砥粒を塗布した複数個の金属片から成る研磨チップを可撓性材料から成るディスク状基板上の外周平坦部表面に配列したことを特徴とする。

【００１１】

【発明の実施の形態】

本発明を図１～図３に図示の実施の形態に基づいて詳細に説明する。

図１は本実施の形態におけるディスク状ダイヤモンド砥石の平面図、図２は図１におけるＡ－Ａ線に沿った断面図を示している。基板１１は例えばエポキシ樹脂材に繊維質材としてガラスファイバ材料を混合し、例えば直径１０ｃｍの可撓性を有するカップ状とされ、基板１１の中心部には基板１１をグラインダ等に取り付けるための治具取付孔１１ａが設けられている。また、基板１１の周辺部の平面状のドーナツ状部分１２にはゴムシート１３が接着され、更にその上層にダイヤモンド砥粒Ｄを塗布した例えば１０個の研磨チップ１４が接着されている。

【００１２】

研磨チップ１４としては、特に溶着方法を用いてダイヤモンド砥粒Ｄを塗布したたものが好適である。溶着方法は自溶性合金タイプとブロンズタイプの２種があり、自溶性合金タイプはＮｉ－Ｃｒ系合金粉末、ブロンズタイプはブロンズ系粉末及びＴｉ等の活性金属粉末を、市販の真空用バインダと混合することにより、先ずペーストを作製する。続いて、所定の形状、例えば本実施の形態では、周辺の基板１１と同曲率にして扇状に打ち抜いた金属片上に、厚みが一定になるようにすり切り治具を使用して上述のペーストを塗布する。

【００１３】

ペーストを塗布すり切りした後に、塗布したペースト上に振動機等を用い、ダイヤモンド砥粒を散布する。なお、自溶性合金タイプの場合には市販の真空用バインダを金属片上に均一に塗布し、ダイヤモンド砥粒Ｄを散布した後に、Ｎｉ－Ｃｒ系合金粉末を振りかける方法でも同様なものが作製できる。

【００１４】

次に、ダイヤモンド砥粒Ｄを散布した金属片を乾燥機に入れ、２４時間程乾燥することにより、バインダに含まれている油脂成分を十分に除去する。続いて、この乾燥処理を行った金属片を真空炉にセットし、真空度が10^{-2}以上の高真空とした後に、自溶性合金タイプは１０００～１１００℃、ブロンズタイプは８００～９５０℃の温度で焼成を行う。これらの温度で焼成を行うことによりダイヤモンド砥粒ＤをＮｉ－Ｃｒ系金属、活性金属含有ブロンズ系金属でそれぞれ良好に溶着することができる。

【００１５】

溶着されたダイヤモンド砥粒Ｄは、図３の模式図に示すように活性金属と反応層Ｒを形成し強固な金属結合することにより、研磨チップ１４上からのダイヤモンド砥粒Ｄの突き出しが大きい。そして、研磨チップ１４は真空炉内の温度が室温に下った後に取り出す。

【００１６】

なお、研磨チップ１４のダイヤモンド砥粒Ｄの溶着や電着等の塗布方式では、被研磨物によって耐久性上問題があることもあり、基板１１にダイヤモンド砥粒Ｄを混入したメタルボンド製のブロックを、円形や扇状等に成形し貼り付けたものでも、振動や騒音を格段に解消することができる。

【００１７】
ディスク状ダイヤモンド砥石を被研磨物の研磨に用いる際には、基板１１をグラインダ等の回転工具に取り付けて回転させ、被研磨物に研磨チップ１４を押し当てる。この際に、基板１１は柔軟性、屈曲性、強度等に優れた基板１１に支持されつつ、ゴムシート１３がその弾力性を研磨チップ１４に伝えるため、研磨チップ１４はそれぞれ独立に被研磨物の面に沿って研磨できるため、小面積部や曲面においても、優れた研磨を行うことができる。

【００１８】
なお、本実施の形態においては研磨チップ１４はゴム層１３を介して取り付けたが、ゴム層１３を省略して基板１１に直接取り付けてもよい。また、研磨チップ１４は扇状の金属片上にダイヤモンドを散布したが、研磨チップ１４の形状は扇状に限らず、円形やその他の形状でも支障はない。

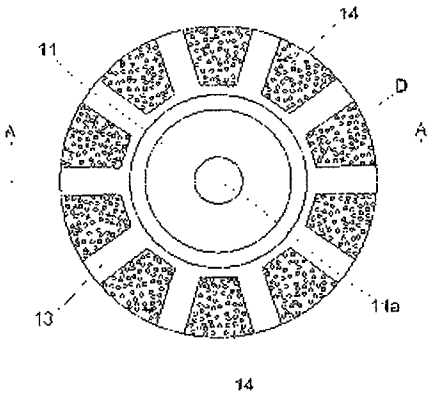
【００１９】
【発明の効果】
以上説明したように本発明に係るディスク状ダイヤモンド砥石は、特に、金属の錆取りや硬質樹脂類、塗料等の研磨・剥離に好適であり、均一な広い面或いは狭い面をも容易に得ることができると共に、打撃音や振動を押さえ、作業者の労務上も、周囲への騒音問題についても、問題が発生することは少ない。

【００２０】
また、可撓性を有するため研磨チップが被研磨面に良く密着し、効率良く剥離作業をうことができる。更に、ダイヤモンド砥粒を溶着法により塗布すれば、電着法のように切れ味があり、メタルボンド法に近い耐久性のあるものとなる。

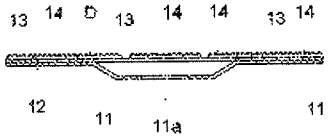
【図面の簡単な説明】
【図１】ディスク状ダイヤモンド砥石の平面図である。
【図２】図１のＡ－Ａ線に沿った断面図である。
【図３】溶着による研磨チップの模式的断面図である。
【図４】メタルボンドによる研磨チップの模式的断面図である。
【図５】電着法による研磨チップの模式的断面図である。

【符号の説明】
１１　基板
１２　ドーナツ状部分
１３　ゴムシート
１４　研磨チップ
Ｄ　ダイヤモンド砥粒
Ｒ　反応層

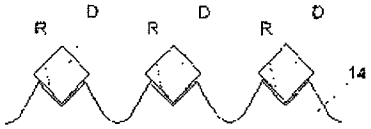
【図１】


14
11
D
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13
11a
14

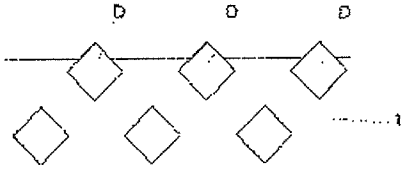
【図２】


13 14 D 13 14 14 13 14
12
11
11a
11

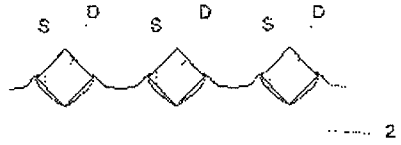
【図３】


R D R D R D
14

【図４】


D D D
1

【図５】


S D S D S D
2

Espacenet

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요약

본 고안은 화학적-기계적-연마(CMP)용 폴리싱 패드의 평탄화를 향상시킴과 동시에, 이 패드의 컨디셔닝을 효과적으로 행하는 기능을 가지고, 더구나 컨디셔닝 성능이 우수하고, 수명이 길며, 컨디셔너의 제조가 용이하여 제조시의 불량률을 현저히 감소시킬 수 있는 CMP 장치용 컨디셔너를 제공함에 그 목적이 있다.

이러한 목적을 달성하기 위한 본 고안은, 샹크(3)의 표면에 구멍 형상의 비마스킹부(1)를 가지는 마스킹 시트(2)를 점착하고, 하나의 비마스킹부(1)에 하나의 다이아몬드 입자(4) 만을 예비 고정한 후에 마스킹 시트(2)를 박리시키고, 이어서 도금액 안에 넣어서 다이아몬드 입자 직경의 40~60% 정도의 두께를 가지도록 Ni 도금층(6)을 석출시키며, 더욱이 1개 구멍 형상의 비마스킹부(1)의 면적이 다이아몬드 입자(4)의 투영 면적의 110~160%배로 하고, 상기한 마스킹 시트(2)의 비마스킹부(1)의 전면적이 바스킹 시트(2)의 전면적의 0.04~0.2배의 범위로 하는 것이 특징으로 된 화학적-기계적-연마 패드용 다이아몬드 전착 컨디셔너를 제공한다.

본 고안에 의해 치수 정밀도가 엄격한 패턴 형상을 재현성 높게 용이하게 형성시킬 수 있고, 마스킹 시트(2)의 샹크(3)에 대한 밀착력이 높으며, 또한 샹크(3)에 전착되는 80~120메쉬의 다이아몬드 입자(4)가, 구멍형의 비마스킹부(1)에 한 개씩 분산된다. 따라서 CMP용 폴리싱 패드의 평탄화를 향상시킴과 동시에, 이 패드의 컨디셔닝을 효과적으로 행하는 기능을 가지고, 더구나 컨디셔닝 성능이 우수하고, 수명이 길며, 컨디셔너의 제조가 용이하기 때문에 제조시의 불량률을 현저히 감소시킬 수 있다.

대표도

도 1

색인어

화학적-기계적-연마, 패드, 컨디셔너, 폴리싱, 다이아몬드

명세서

도면의 간단한 설명

도 1은 마스킹 시트를 점착한 샹크의 부분 평면도

도 2는 다이아몬드 입자가 예비 고정된 샹크의 부분 평면도

도 3은 본 고안 컨디셔너의 연삭면의 부분 단면도

도 4는 본 고안에 의한 일실시예인 컨디셔너의 작용면의 평면도

도 5는 본 고안에 의한 다른 실시예인 컨디셔너의 작용면의 평면도

〈도면의 주요부분에 대한 부호의 설명〉

1 : 구멍형상의 비마스킹부 2 : 마스킹 시트

3 : 샹크 4 : 다이아몬드 입자

5 : 예비 도금층 6 : Ni 도금층

7 : 컨디셔너 8 : 비전착 영역

고안의 상세한 설명

고안의 목적

고안이 속하는 기술 및 그 분야의 종래기술

본 고안은 반도체의 웨이퍼 가공에 사용되는 CMP(Chemical mechanical planarization) 장치의 패드 컨디셔너에 관한 것이다. 보다 상세하게는, 본 고안은 CMP용 폴리싱 패드의 평탄화를 향상시킴과 동시에, 이 패드의 컨디셔닝을 효과적으로 행하는 기능을 가지고, 더구나 컨디셔닝 성능이 우수하고, 수명이 긴 CMP 장치용 컨디셔너에 관한 것이다.

반도체 웨이퍼의 연마를 반복하기 위하여, 연마 입자나 연마칩 등이 폴리싱 패드의 미세한 구멍에 들어가서 눈메움이 일어난다든지, 연마 입자와 웨이퍼와의 화학 반응열에 의해 폴리싱 패드의 표면이 경면화하여 연마 속도를 저하시키게 된다. 이 때문에 폴리싱 패드의 표면을 재생시켜 연마 속도를 회복시키는 컨디셔너라고 불리는 공구가 사용된다. 다이아몬드 입자는 우수한 컨디셔닝 재료이며, 다이아몬드 입자를 이용한 컨디셔닝이 실용화되어 있다. 이와 같은 CMP용 폴리싱 패드의 컨디셔너에 대해서는, 폴리싱 패드의 마멸을 가급적 억제할 수 있으며, 더욱이 패드의 표면 상태를 항상 소정의 연마속도가 얻어지도록 일정하게 유지되는 것과, 다이아몬드 입자의 탈락이 없고, 패드면에 눈메움을 일으키지 않는 것이 요구된다.

다이아몬드 입자를 컨디셔너 본체의 작용면에 금속 결합제를 사용하여 고착하는 방법에는 여러 가지가 있으나, 전착법은 비교적 용이하고 더욱이 다이아몬드 입자를 컨디셔너 본체의 작용면에 확실하게 고착시킬 수 있으므로 많이 사용되고 있다. 전착법에 의해 다이아몬드 입자를 고착하는 경우는, 컨디셔너 본체의 작용면에 다이아몬드 입자의 고정면을 남겨 두고서, 절연성의 마스킹 재료로 피복한다. 컨디셔너의 본체를 도금액에 담그고 입자 고정면에 입자를 놓고서 컨디셔너의 본체에 음극을 접속하고, 도금액에 양극을 접속하여 전기 도금을 행하여 다이아몬드 입자를 예비 고정한다. 다이아몬드 입자를 예비 고정한 후에 대부분의 입자는 그 일 부가 본체의 입자 고정면에 접한 상태에서 예비 고정되지만, 본체의 입자 고정면에 접하지 않은 상태에 부착하여, 부유하는 입자가 존재하는 경우도 있다. 컨디셔너의 본체를 다시 도금액에 넣어서, 본체에 음극을 접속하고 도금액에 양극을 접속하여 다이아몬드 입자 고정면의 도금을 행한다.

그러나 종래의 전착 숫돌의 제조에 있어서, 컨디셔너 본체의 작용면에 다이아몬드 입자의 고정면을 남겨 두고서, 절연성의 마스킹 재료로 피복하는 경우에 미세한 크기의 적절한 치수 정밀도로 피복하는 것이 곤란하여 마스킹 되어 있지 않은 곳에 두 개 이상의 다이아몬드 입자가 동시에 들어가는 문제가 발생하였고, 마스킹 위치가 어긋나서 동일한 형상으로 재현하는 것이 곤란하였고, 피복층이 평활하게 되지않고 요철(凹凸)이 심하였으며, 피복층의 밀착력이 약하여 이후에 공정에서 벗겨지는 경우가 있었기 때문에, CMP 패드를 컨디셔닝 하는데 있어서 불량률이 매우 높았다. 또한 종래의 컨디셔너는, 패드의 표면 상태를 항상 소정의 연마속도가 얻어지도록 일정하게 유지시키는 것이 곤란하였으며, 다이아몬드 입자의 탈락이 많고, 패드면에 눈메움을 일으키는 경우가 많았으며, 종래의 컨디셔너를 사용하여 패드를 컨디셔닝하면, 웨이퍼의 표면거칠기 값 및 스크래치 개수가 높은 것이 큰 문제로 되어왔다.

고안이 이루고자 하는 기술적 과제

본 고안은 CMP용 폴리싱 패드의 평탄화를 향상시킴과 동시에, 이 패드의 컨디셔닝을 효과적으로 행하는 기능을 가지고, 더구나 컨디셔닝 성능이 우수하고, 수명이 길며, 컨디셔너의 제조가 용이하여 제조시의 불량률을 현저히 감소시킬 수 있 는 CMP 장치용 컨디셔너를 제공함에 그 목적이 있다.

고안의 구성 및 작용

본 고안자들은, 상기의 과제를 해결하기 위하여 실험적인 연구를 거듭한 결과, 전착 컨디셔너의 연삭면에 있어서, 하나의 비마스킹부에 한 개씩의 다이아몬드 입자를 분산시키는 것에 의해, 연삭 저항을 감소시키고 컨디셔너의 수명이 증가되며, 연마속도비와 컨디셔닝 속도가 높아지며, 웨이퍼의 표면거칠기 값이 낮아지며, 웨이퍼의 스크래치 개수가 감소됨을 확인하였다. 즉, 본 고안은, 샹크(3)의 표면에 구멍 형상의 비마스킹부(1)를 가지는 마스킹 시트(2)를 점착하고, 하나의 비마스킹부(1)에 하나의 다이아몬드 입자(4) 만을 예비 고정한 후에 마스킹 시트(2)를 박리시키고, 이어서 도금액 안에 넣어서 다이아몬드 입자 직경의 40~60% 정도의 두께를 가지도록 Ni 도금층을 석출시키며, 더욱이 1개의 구멍 형상의 비마스킹부(1)의 면적이 다이아몬드 입자(4)의 투영 면적의 110~160%배로 하고, 상기한 마스킹 시트(2)의 비마스킹부(1)의 전면적이 마스킹 시트(2)의 전면적의 0.04~0.2배의 범위로 하는 것이 특징으로 된 화학적-기계적-연마 패드 컨디셔너를 제공하는 것이다.

아래에, 도면을 참고하여 본 고안을 상세히 설명한다. 그림 1은 마스킹 시트(2)를 점착한 샹크의 부분 평면도이다. 그림 1에서 원으로 표시되는 부분이 비마스킹부(1)로서, 한 개의 비마스킹부(1)에 1개의 다이아몬드 입자(4)가 고착된다. 1개의 비마스킹부(1)에 고착되는 다이아몬드 입자(4)가 2개를 넘으면, 패드에 의한 연마에서 웨이퍼의 표면거칠기 값이 낮아지며, 웨이퍼의 스크래치 개수가 감소되기 쉽다. 그림 1에서 비마스킹부(1)의 전면적이 다이아몬드 입자(4)를 고착한 연삭면 적의 0.04~0.2배이다. 비마스킹부(1)의 전면적이 다이아몬드 입자(4)를 고착한 연삭면의 전면적의 0.04배 미만이면, 컨디셔너(7)로서의 연삭력이 부족한 문제가 있으며, 컨디셔너(7)의 수명이 짧아지게 된다. 또한 비마스킹부(1)의 전면적이 다이아몬드 입자(4)를 고착한 연삭면의 전면적의 0.3배 이상이면 패드에 의한 연마에서 웨이퍼의 표면거칠기 값이 낮아지며, 웨이퍼의 스크래치 개수가 감소되기 쉽다. 본 고안의 컨디셔너의 샹크(3) 재료로는, 스테인레스 강재인 STS 304를 사용하는 것이 바람직하나, 경우에 따라서는 예를 들면 알루미늄 합금 등이 사용될 수 있다.

본 고안의 컨디셔너를 제조하기 위해서는, 우선 샹크(3)의 탈지를 행하는 것이 바람직하다. 탈지를 종료한 샹크면에, 구멍 형상의 비마스킹부(1)를 가지는 마스킹 시트(2)를 점착한다. 그림 1에서, 원형의 구멍 형상의 비마스킹부(1)를 가지는 마스킹 시트(2)가 샹크(3)에 점착되고, 샹크(3)의 일부가 비마스킹부(1)의 형상으로 노출되어 있다. 본 고안에서는, 마스킹 시트(2)를 점착한 상태에서, 컨디셔너의 연삭면으로 되는 샹크면에 예비 도금층(5)을 형성시키는 것이 바람직하다. 예비 도금을 실시하는 것에 의해, 다이아몬드 입자(4)와 샹크(3)가 직접 접촉하는 것이 아니고, 다이아몬드 입자(4)의 샹크(3)에 대한 고착성이 개량된다. 예비 도금하는 금속으로는 니켈 및 니켈 합금을 사용할 수가 있다. 본 고안에서는, 예비 도금을 실시한 샹크를, 마스킹 시트(2)를 점착한 상태에서 도금 용기에 넣고, 도금 용기 중에서 샹크의 마스킹 시트(2)를 점착한 면의 부분에 다이아몬드 입자(4)를 충진한다. 이어서, 샹크에 음극을 접속하고, 도금액에 양극을 접속하여, 예비 도금층(5)의 형성을 위한 전기 도금을 실시한다. 예비 도금을 위한 금속은 다이아몬드 입자(4)를 샹크(3)에 예비 고정할 수 있는 것이라면 제한이 없으나, 니켈 및 니켈 합금을 사용할 수가 있다. 각 다이아몬드 입자(4)의 일부분이 비마스킹부(1)에서 예비 도금층(5)을 사이에 두고 예비고정되며, 샹크 표면에서 탈락하지 않는 상태로 되면, 예비 고정을 위한 도금을 종료하고, 샹크를 도금 용기로부터 꺼낸다. 그림 2는, 다이아몬드 입자가 예비 고정된 샹크의 부분 단면도이다. 본 그림에서, 샹크(3)에 마스킹 시트(2)가 점착되고, 마스킹 시트의 구멍 형상의 비마스킹부(1)에 다이아몬드 입자(4)가 예비 도금층(5)에 의해 예비 고정되어 있다.

이어서 샹크(3)의 표면으로부터 마스킹 시트(2)를 박리시키고, 더욱이 마스킹부의 탈지와 활성화 처리를 행한 후에, 다시 샹크(3)를 도금 용기에 담그서 음극을 접속하고, 도금액에 양극을 접속하여 도금을 계속하며, 다이아몬드 입자(4)를 샹크(3)에 전착하여 연삭면을 형성하고, 컨디셔너를 완성하게 된다. 도금에 사용하는 금속은, 예비 고정의 경우

와 마찬가지로 다이아몬드 입자(4)를 샹크(3)에 고착할 수 있는 것이면 제한이 없고, 예를 들면 니켈 및 니켈 합금을 사용할 수 있다. 그림 3은 본 고안의 컨디셔너의 연삭면의 부분 단면도이다. 본 그림에서 다이아몬드 입자(2)는 샹크(3)에 예비 도금층(5)에 의해 예비 고정되며, 더욱이 도금에 의해 형성된 Ni 도금층(6)에 의해 완전히 고착되어 있다. 본 고안에서 이용하는 마스킹 시트(2)는, 도금 조건에 견딜 수 있는 것이면 제한은 없으며, 예를 들면 폴리 염화비닐 등의 수지 시트의 일면에 아크릴계 또는 비닐 에테르계 등의 점착제를 도포한 뒤에 박리시트를 적층시키고, 또한 구멍 형상의 비마스킹부(1)의 가공을 실시한 것을 용이하게 사용할 수가 있다. 마스킹 시트(2)의 두께란, 수지 시트 및 점착 제 층의 두께의 합이 되며 이러한 마스킹 시트의 두께는 다이아몬드 입자 직경의 60%~140%의 범위로 하는 것이 바람직하다. 또한 더욱이 1개의 구멍 형상의 비마스킹부(1)의 면적이 다이아몬드 입자의 투영 면적의 110~160%배의 범위로 되어야 한다. 마스킹 시트의 두께를 다이아몬드 입자 직경의 60% 미만으로 한다든지, 또는 한 개의 비마스킹부의 면적이 다이아몬드 입자의 투영 면적의 160%를 초과하면, 한 개의 비마스킹부에 2개 이상의 다이아몬드 입자가 고착되어서 균등한 분산이 이루어지지 않는다. 또한 비마스킹부의 면적이 다이아몬드 입자의 투영 면적의 110% 미만에서는, 다이아몬드 입자의 샹크에 대한 부착이 곤란하게 된다. 따라서 마스킹의 두께를 전착할 다이아몬드 입자경의 60~140%의 범위로 하고, 마스킹하지 않는 구멍의 직경을 전착할 입자경의 110~160%의 범위로 하는 것에 의해, 컨디셔너 금속에 전착되는 다이아몬드 입자(4)가, 비마스킹부(1)에 한 개씩 분산되도록 된다. 다이아몬드 입자의 평균 투영 면적은, 다이아몬드 입자가 분산된 상태에서 확대 사진을 촬영하는 것에 의해 구할 수 있다. 본 고안에서는, 상기한 마스킹 시트의 비마스킹부의 전면적이 마스킹 시트의 전면적의 0.04~0.2배의 범위이다. 마스킹 시트의 비마스킹부의 전면적은, 본 고안의 컨디셔너의 연삭면에서 다이아몬드 입자가 고착된 부분의 전면적에 대응한다. 마스킹 시트의 비마스킹부의 전면적이 마스킹 시트의 전면적의 0.04배 미만이면, 구멍 형상의 비마스킹부의 전면적이 다이아몬드 입자를 고착한 연삭면의 전면적의 0.04배 미만으로 되어, 컨디셔너로서의 연삭력이 부족할 염려가 있다. 마스킹 시트의 비마스킹부의 전면적이 마스킹 시트의 전면적의 0.2배를 초과하면, 구멍 형상의 비마스킹부의 전면적이 다이아몬드 입자를 고착 한 연삭면의 전면적의 0.2배를 초과하여, 연삭시에 눈메움이 발생할 염려가 있다.

〈실시예〉

컨디셔너 샹크(3)의 연삭면으로 되는 작용면을 탈지하고, 마스킹 시트를 점착한 후에, 니켈에 의한 예비 도금층(5)을 형성하였다. 마스킹 시트(2)의 두께를 전착할 #120 메쉬 다이아몬드 입자경의 80%로 하였으며, 더욱이 다이아몬드 입자(4)를 전착시키기 위해 비마스킹부(1)의 직경을 전착할 다이아몬드 입자경의 140%로 하였다. 구멍 형상의 비마스킹부(1)의 전면적을 마스킹 시트(2)의 전면적의 0.1배가 되도록 하였다. 그 후에 컨디셔너의 본체를 도금액에 담그고 입자 고정면에 다이아몬드 입자(4)를 놓고서 컨디셔너의 본체에 음극을 접속하고, 도금액에 양극을 접속하여 전기도금을 행하여 다이아몬드 입자(4)를 예비 고정하였다. 샹크를 도금액으로부터 빼내고, 마스킹 시트(2)를 박리시키며, 탈지를 행한 후에 컨디셔너의 본체를 다시 도금액에 담그며, 본체에 음극을 접속하고 도금액에 양극을 접속하여 다이아몬드 입자 고정면의 최종 Ni 도금을 행함에 의해 다이아몬드 입자(4)를 샹크의 연삭면에 고착하였다.

컨디셔너의 형상은 실시예 1에서는 도 4와 같은 다이아몬드 입자의 분포를 가지도록 하였으며, 실시예 2에서는 도 5와 같이 전착부가 스파이럴 형상이 되도록 하였고, 실시예 2에는 비전착 영역(8)이 형성되도록 하였다. 실시예 1 및 실시예 2는 컨디셔너의 형상을 제외하고는 다른 모든 제조 조건은 동일하였다. 비교예는 실시예 1 및 실시예 2의 컨디셔너의 성능과 비교를 행하기 위하여 당사에서 종래의 제조방법으로 제조되어 시중에서 판매되는 것으로, 비교예 1은 실시예 1과 동일 형 상이며, 비교예 2는 실시예 2와 동일 형상의 컨디셔너를 사용하였다. 실시예 1, 실시예 2, 비교예 1 및 비교예 2의 컨디셔너를 사용하여, 반도체의 웨이퍼 가공에 사용되는 화학-기계-연마 장치의 패드 컨디셔닝을 행한 결과를 표 1에 나타낸다.

[표 1]

	실시예 1	실시예 2	비교예 1	비교예 2
연마속도비	1.38	1.35	1	0.9
컨디셔닝속도(㎛/h)	35	34	12	11
컨디셔너수명(hr)	600	590	160	155
웨이퍼표면거칠기(nm)	0.33	0.34	0.43	0.44
웨이퍼의 스크래치 개수	1	1	7	8
패드의 표면상태	연삭흔 거의 없음	연삭흔 거의 없음	연삭흔 많음	연삭흔 많음

실시예 1 및 실시예 2의 패드 연마속도비, 컨디셔닝 속도 및 컨디셔너 수명이 비교예에 비해 현저히 높으며, 웨이퍼의 표면거칠기 및 스크래치 개수의 경우에서도 실시예 1과 실시예 2가 비교예에 비해 현저히 낮은 값을 보이며, 실시예 1 및 실시예 2의 컨디셔너를 사용한 경우 패드의 연삭흔이 거의 나타나지 않았으나, 비교예의 컨디셔너를 사용한 경

우는 패드의 표면에 연삭흔이 많이 나타났다.

고안의 효과

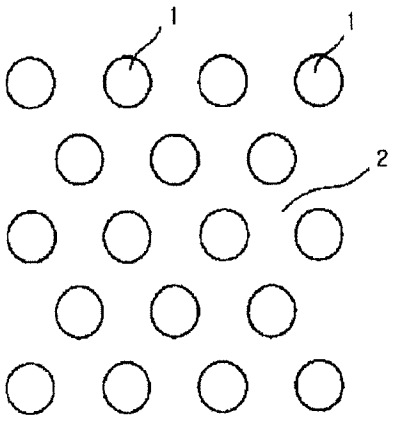
이상에서 서술한 바와 같이 본 고안은, 마스킹 시트(2)의 점착에 의해 마스킹을 행하므로 치수 정밀도가 엄격한 패턴 형상을 재현성 높게 용이하게 형성시킬 수 있으며, 마스킹 시트(2)의 컨디셔너 금속에 대한 밀착력이 높으며, 또한 컨디셔너 금속에 전착되는 80~120메쉬의 다이아몬드 입자(4)가, 비마스킹부(1)에 한 개 씩 분산된다. 따라서 CMP용 폴리싱 패드의 평탄화를 향상시킴과 동시에, 이 패드의 컨디셔닝을 효과적으로 행하는 기능을 가지고, 더구나 컨디셔닝 성능이 우수하고, 수명이 길며, 컨디셔너의 제조가 용이하기 때문에 제조시의 불량률을 현저히 감소시킬 수 있다.

(57) 청구의 범위

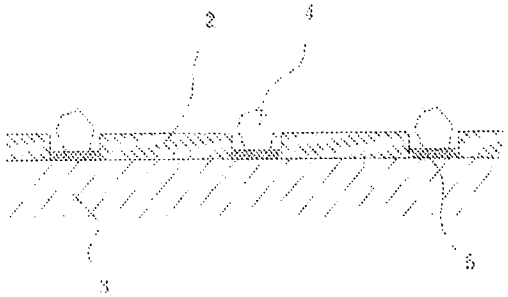
청구항 1.
Ni 도금층이 다이아몬드 입자 직경의 40~60% 정도의 두께를 가지며, 샹크의 표면에 있어서, 1개의 구멍 형상의 비마스킹부의 면적이 다이아몬드 입자의 투영 면적의 110~160%배로 하고, 마스킹 시트의 비마스킹부의 전면적이 마스킹 시트의 전면적의 0.04~0.2배의 범위로 하는 것이 특징으로 된 화학적-기계적-연마 패드 컨디셔너.

도면

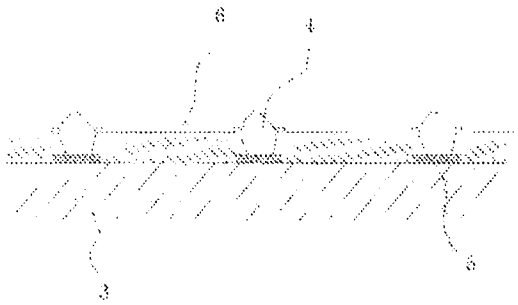
도면1



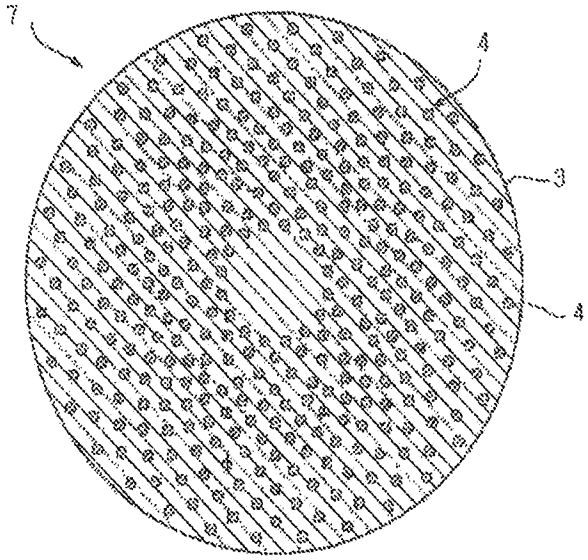
도면2



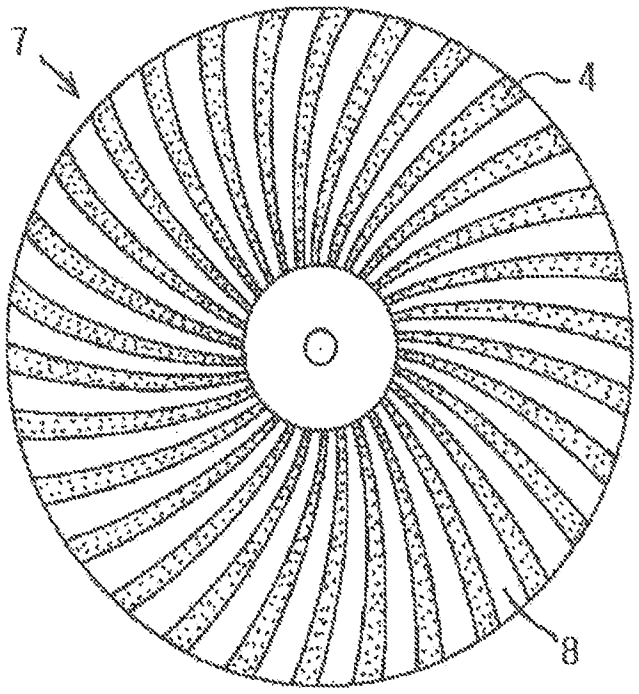
도면3



도면4



도면5



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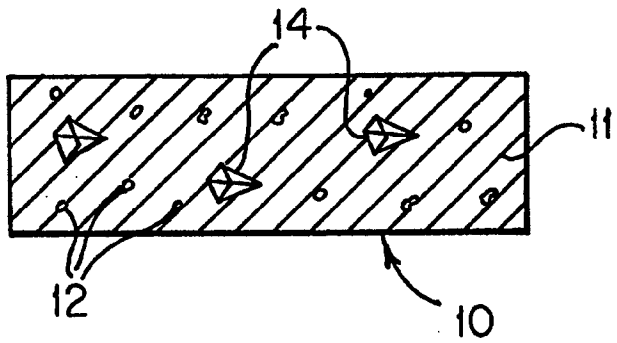
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(54) Title: METHOD FOR MAKING POWDER PREFORM AND ABRASIVE ARTICLES MADE THEREFROM

(57) Abstract

A method for making abrasive articles uses a soft, easily deformable and flexible preform having a high binder content. The binder gives the preform its integrity, and is present in greater quantity than the retaining powder. The preform can have superabrasive particles mixed therein, or added later. The preform allows even distribution of a small quantity of retaining powder for thin superabrasive articles. A porous layer may be added to the assembly for making an abrasive article, the porous layer absoring the liquid binder, supporting the retaining powder and superabrasive particles to prevent lateral movement, and perhaps giving strength to the preform. The final assembly to be heated or sintered (preferably under pressure) for making the abrasive article, which may include any number of layers of superabrasive particles, porous layers and preforms.



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Description

METHOD FOR MAKING POWDER PREFORM AND ABRASIVE ARTICLES MADE THEREFROM

Technical Field

This invention relates generally to the making of abrasive articles and the like, and is more particularly concerned with the use of soft, flexible and easily deformable powdered pieces as preforms for the manufacture of abrasive articles having superabrasive particles therein.

Background Art

Powdered preforms are widely used in the manufacture of abrasive articles that include a plurality of superabrasive particles such as diamond, cubic boron nitride and the like. Such powdered preforms are conventionally manufactured by compacting powder mixtures of retaining compositions and superabrasives particles in cold presses or roll compactors. Compacting pressure ranges from 300 to 10,000 kg/sq. cm, resulting in 20-50% relative density of the green compacts. Such green compacts are hard, stiff and brittle. The green compacts are sintered, either with or without pressure, and with or without impregnation.

There is a method of making abrasive articles wherein a non-compacted mixture of the powdered retaining composition, with the plurality of superabrasive particles therein, is placed directly into a sinter mold, then compacted and sintered in the sinter mold. This method requires a lot of adjustments in attempts to spread the powder evenly within the sinter mold. The required adjustments slow the manufacturing process, so the method does not fit well with mass production requirements.

In all the above mentioned methods, the powdered mixture can contain some binders, but the conventional green compacts are held together, not by the binder, but primarily by interaction among the particles of the powder, e.g. by mechanical interlocking of the particles. The above mentioned methods are widely used to produce traditional

cutting, drilling, and grinding abrasive tools and elements of abrasive tool, such as segments for saws and the like.

There are powdered preforms formed by spraying powder onto a substrate, and fixing the powder to itself and to the substrate by an adhesive, for example by an adhesive spray. Such preforms are flexible, but may experience loss of some powder when flexed. Also, such a method must deal with air borne particles and aerosol sprays that, because of environmental concerns, put serious limitations on the implementation of the method. This method has been used by the present inventor to make articles disclosed in U.S. Patents Nos. 4,925,457, 5,049,165, 5,092,910 and 5,190,568, as well as in U.S. patent Application Ser. No. 08/066,475 titled "patterned Abrasive Material and Method" and Ser. No. 08/024,649 titled "Abrasive Cutting Tool".

Soft and flexible preforms of powders and/or fibers, including both metallic and non-metallic materials, are also known; but, to the knowledge of the present inventor, such preforms are not known in the art of manufacturing articles that include superabrasive particles. Presently, the soft and flexible preforms are made by casting, or extruding a composition of brazing filler metal, or ceramic components, or hard facing compositions including metallic components and non-metallic abrasive components such as tungsten carbide particles. Such soft and flexible preforms can be bent more than 90°, and can be cut by scissors or the like.

The earlier known soft and flexible preforms comprise a high content of various binders, up to 95% by volume, and up to 20% by weight. It is the binder that makes such preforms soft and flexible; but, even with the high content of binders, the preforms are flimsy and must be handled with care. This is especially true for the very thin preforms, around .005--.010", or 0.10--0.25 mm.

It is important to distinguish between the soft and flexible preforms and the products of roll compacting of powders, even in the presence of a binder. When a roll compacted product includes a binder, the binder is in a much

smaller quantity than in a flexible preform. The roll compacted product is held together, not by the binder, but by the mechanical interlocking of particles, which makes the roll compacted product much less flexible than the soft and flexible preforms.

Soft and flexible preforms made of brazing filler metal compositions are used to put some parts together through brazing, mostly through furnace brazing. Soft and flexible preforms made for hard facing compositions are used to repair worn parts. For this purpose, the preforms are applied to a worn spot on the part.

The brazing process using the soft and flexible preforms made of brazing filler metal has a significant time duration because of the necessity for removal of the substantial quantity of binder. The time for removal of the binder is called the "dewaxing" cycle, and it allows the binder to melt, evaporate, or run out from the preform. It has been found that, if the dewaxing time is shortened or omitted, the powder of the soft and flexible preform can be literally washed out by the liquefied binder.

In attempting to use the known soft and flexible preform to hold a plurality of superabrasive particles in order to produce abrasive articles, it will be recognized that:

1. Compositions of the brazing filler preform do not correspond to the desired matrix compositions to hold superabrasive particles;

2. Soft and flexible preforms are not produced with superabrasive particles on, or within, the preforms;

3. Soft and flexible preforms are quite flimsy and not as strong as desired for production of abrasive articles, especially for mass production requirements of abrasive articles requiring thin (.005--.020", or 0.1-- 0.5 mm) flexible preforms;

4. De-waxing time must be severely reduced to meet production rates, especially for mass production; and,

5. Heating and/or brazing processes alone do not provide the most reliable matrix for retaining superabrasive particles.

Disclosure of the Invention

The present invention provides a method for manufacturing abrasive articles and wear resistant parts, such articles or parts comprising a plurality of superabrasive particles such as diamond, cubic boron nitride or the like randomly or systematically distributed in a retaining matrix. Specifically, the method of the present invention includes the preparation and utilization of powdered preforms in the form of soft, easily deformable flexible (SEDF) bodies that may include a plurality of superabrasive particles.

In making the SEDF preforms, the powdered compositions will be chosen based on criteria related to the holding necessary for the superabrasive particles to be included. Any number of matrix materials, or powdered compositions may be used, with any number of binders. The binder will be selected to provide the desired integrity of the product, while maintaining the flexibility and processability. In any case it must be remembered that the concentration of powdered composition and abrasive particles (if included) is low, and the volume of the binder is high. The volume of the binder phase substantially exceeds the volume of the powdered composition and the abrasive particles within the SEDF preform.

In one preferred form of the present invention, a porous layer will be placed against the SEDF preform. The purpose of the porous layer is to hold the abrasive particles in place during subsequent processing of the material. Successful material can be made without the porous layer, but the porous layer provides a better quality product than is obtained without the porous layer.

Final processing of the SEDF preform of the present invention includes sintering or other heat treating. The result is a high quality abrasive material, with or without a

porous layer therein, which can be used for numerous cutting or abrasive tools and the like.

Brief Description of the Drawings

These and other features and advantages of the present invention will become apparent form consideration of the following specification when taken in conjunction with the accompanying drawings in which:

Fig. 1 is a cross-sectional view showing one form of preform made in accordance with the present invention, the preform having some superabrasive particles therein;

Fig. 2 is a cross-sectional view of another preform made in accordance with the present invention, the preform being formed on a substrate which may be a porous material;

Fig. 3 is a view similar to Fig. 1 but showing the superabrasive particles on the surface of the preform;

Fig. 4 is a view similar to Fig. 3 wherein the superabrasive particles are held by a carrier which is placed against the preform;

Fig. 5 is a cross-sectional view illustrating a continuous process for forming the preform, and placing superabrasive particles on one surface of the preform;

Fig. 6 is a view similar to Fig. 5, but showing the preform being formed on a substrate having superabrasive particles thereon;

Fig. 7 is a view similar to Fig. 1 and showing a comparison between the thickness of the preform and the size of the superabrasive particles;

Fig. 8 is a view similar to Fig. 7, but showing the preform after sintering;

Fig. 9 is a cross-sectional view illustrating a method and apparatus for casting preforms according to the present invention;

Fig. 10 is a cross-sectional view showing a process of sintering under pressure;

Fig. 10A is a view similar to Fig. 10 but showing a plurality of preforms within the mold;

Fig. 11 is a cross-sectional view illustrating an assembly of a preform with porous layers in accordance with the present invention;

Figs. 12--17 are similar to Fig. 11 and show various modifications thereof;

Fig. 18 is a cross-sectional view illustrating extrusion of the preform into openings of a porous layer;

Fig. 19 is a cross-sectional view showing a continuous process for assembling a preform in accordance with the present invention using rolls;

Fig. 19A is a cross-sectional view illustrating the casting of a profiled preform on a substrate;

Fig. 19B is a view similar to Fig. 19A but showing a preform being cast between two substrates;

Fig. 19C is a cross-sectional view showing the deformation of a flat preform;

Fig. 20 is an exploded, cross-sectional view showing an be mixed assembly for producing an abrasive article;

Fig. 20A is a view similar to Fig. 20 but showing a modification thereof;

Fig. 21 is a cross-sectional view showing the assembly of Fig. 20 after assembly and sintering;

Figs. 22 and 23 are similar to Figs 20 and 21 but showing a modification thereof;

Figs. 24 and 25 and Figs. 26 and 27 are similar to Figs 20 and 21 but showing additional modifications thereof; and,

Fig. 28 is a side elevational view showing the assembly of a cutting tool in accordance with the present invention.

Best Mode for Carrying Out the Invention

Referring now more particularly to the drawings, and to those embodiments of the invention here presented by way of illustration, the invention has two major parts: preparation of soft, easily deformed flexible (SEDF) preforms; and, utilization of SEDF preforms for making abrasive articles.

Preparation of Preform

The preform is prepared by mixing a binder with a powder composition in the required proportions. The mixture may or may not include a plurality of superabrasive particles. Thus, depending on the particular portions chosen, one may produce the binder-powder mixture in the form of a slurry, granulated powder, or a paste.

If the granulated powder is used, a plurality of superabrasive particles can be placed onto the granules by a supplementary process. The granules may be mixed with a plurality of superabrasive particles, and the superabrasive particles may stick to the granules either because of the binder in the granules, or because of an additional binder covering the granules and/or the superabrasive particles.

Mixing the binder with the retaining powder can be performed on a variety of standard equipment, including virtually any equipment suitable for mixing powder and liquid together. Thus, no detail discussion of the equipment is necessary herein.

There is a variety of materials that can be used as the binder for the preform. The binder may be organic or inorganic, but should be selected to carry the particles of the powder, keep the particles suspended, and provide integrity and flexibility to the final preform. It is preferable to choose a binder that allows air, a low vacuum, heat, or a combination of these, to evaporate at least some of the volatile components of the binder for at least partial curing of the binder. Such binders include water soluble binders.

It is well recognized that the prior art powder technology requires that a person mix powders and superabrasive particles. Such powders and superabrasive particles become air borne, and are deleterious to the health of workers. Safety masks and the like are available, but are uncomfortable to wear, and of course are not totally effective. The present invention overcomes this difficulty with the prior art in that the powders and superabrasive

particles can be handled by machines, appropriately covered to minimize the escape of particles. The material is available to be manipulated by people only after mixing powdered components with the binder, so there is no longer a hazard of air borne particles.

Those skilled in the art will understand that many materials will be acceptable as binders, depending on the precise characteristics desired. However, by way of example, the following have been found to be suitable binders: Sanford's Rubber Cement (commercially available from Sanford Corporation, Bellwood IL) in a combination with Carter's Rubber Cement Thinner (commercially available from Dennison Carter's Division, Dennison Manufacturing Company, Framington, MA); Nicrocoat Cements (available from Wall Colmanoy Company, Madison Heights, MI) in a combination with Exosen No. 40 (available from Smithkline Beckman Company, Lewistown, PA).

In the binder-powder composition, the binder is usually 3 to 20% by weight of the composition, but the ratio can be extended. By volume, the percentage of the powder within the binder-powder composition is usually from 1 to 5%, but it can be extended to a range of 0.3 to 10%. One successful preform has from 5.0 to 8.5% by weight of rubber cement and thinner. The retaining powder is dispersed in the binder and held thereby. Superabrasive particles may also be dispersed within the binder, and also held therein.

Referring to Fig. 1 of the drawings, it will be seen that the SEDF preform 10, comprises mostly binder 11. There is a plurality of particles 12 of a retaining powder distributed in the binder 11, and there are superabrasive particles 14 also distributed in the binder. From the above discussion it will be understood that the superabrasive particles 14 may or may not be included. This will also be discussed in more detail below.

Looking at Fig. 2, it will be seen that the SEDF preform 10 includes the binder 11 and retaining powder 12. Superabrasive particles 15 are here shown as fixed to a

substrate 16, the substrate 16 then being placed against the SEDF preform 10. The substrate 16 may take many forms, including a film having a low melting point or the like, but it is preferably a porous material, which will be discussed in more detail hereinafter.

Fig. 3 shows a modification of Fig. 2, the SEDF preform 10 being substantially the same. The superabrasive particles 18 in Fig. 3, however, are placed on the upper surface of the preform 10. The superabrasive particles may be pressed into the preform 10, or may be held by an adhesive. The adhesive may be the binder 11, or may be a separately applied adhesive. Similarly, Fig. 4 shows the arrangement of Fig. 3, but with a carrier 19 having the abrasive particles 20 adhered thereto. The particles 20 on the carrier 19 can therefore be brought into contact with the preform 10 when desired.

In accordance with the present invention, SEDF preforms can be formed by spreading a binder-powder composition on a substrate. The composition may be powdered slurry, granules, or a paste. The composition is then cured, e.g. dried, on the substrate, and one may use applied heat or pressure if desired.

When dealing with granules, an external pressure in the range of 50 to 200 kg/cm^2 is helpful to apply a binder to the granules while the granules are on the substrate. The binder, which may be spread on the substrate, may penetrate between the granules and assists in integrating the granules into a preform.

In some cases, especially when the superabrasive particles are substantially larger than the particles of the retaining powder, or the viscosity of the liquid is not balanced to suspend the superabrasive particles, some measure must be taken to prevent separation or sedimentation of the superabrasive particles after mixing has stopped. One might therefore pour immediately after mixing, or combine continuous mixing with simultaneous pouring or coating.

The superabrasive particles in the SEDF preforms are not surrounded by closely packed particles of a retaining powder as in the traditional green compacts. Rather, the superabrasive particles in the SEDF preform are suspended predominantly by the binder, and in contact with a very few particles of the retaining powder. This is illustrated in Figs. 1--4 of the drawings.

Superabrasive particles can be added to the SEDF preform during the process of forming or curing the preform. By way of example, attention is directed to Fig. 5 of the drawings. A binder- powder composition 21 is dispensed onto a substrate 22, and doctored to a uniform thickness by a doctor blade 24. After the doctor blade 24,. superabrasive particles 25 are dispensed onto the surface of the preform 26. It will be understood that the composition 21 is not cured at the time the superabrasive particles 25 are placed onto the preform 26, so the particles will be adhered thereto. If desired, or necessary due to the viscosity of the preform and the weight of the particles 25, pressure can be applied to assist in urging the superabrasive particles 25 at least partially into the preform 26. Also, additional adhesives or the like can be applied as needed.

Fig. 6 illustrates a modification of the arrangement shown in Fig. 5. In Fig. 5, the binder-powder composition 21 is dispensed onto the substrate 22 and doctored to the desired thickness by doctor blade 24. In Fig. 6, however, the substrate 22 carries a plurality of superabrasive particles 28, and the binder-powder composition is dispensed onto the particles 28. The superabrasive particles 28 may be completely covered, or only partially covered by the binder-powder composition as desired.

The difference between the thickness of the SEDF preform and the size of the superabrasive particles can vary considerably; but, it will be realized that the difference will change significantly in sintering. Fig. 7 shows an SEDF preform 10 having superabrasive particles 14 therein. At this stage, the thickness t of the SEDF preform may be equal

to 3d to 10d, where d is the dimension of the superabrasive particles in the direction of the thickness of the SEDF preform. After sintering, the same preform is shown in Fig. 8. It will of course be realized that the superabrasive particles 14 will not change in size during sintering, but the preform 10 will be significantly condensed. After sintering, the preferable ratio is that the thickness t is approximately equal to the dimension d, the desirable range being t = 0.3--2d. It should be mentioned that superabrasive particles of all sizes are suitable for use with the technology disclosed herein, but the preferable sizes are from 18 to 324 mesh (about 1.0 mm to about 0.035 mm).

The weight of the dry retaining powder per unit volume of the SEDF preform (grams of powder per cubic centimeter of preform) determines the thickness of the sintered abrasive material, it being realized that the binder will run off, or evaporate, during sintering or other heat processing. For example, the density of cobalt is 8.9 g/cm^3, and a cobalt preform contains 0.8 g/cm^2 of the dry cobalt powder; therefore, the thickness of the fully densified, sintered product will be about 0.9 mm, which is found by dividing 0.8 g/cm^2 by 8.9 g/cm^3. It will be noted that the thickness of the SEDF preform is not in the calculation, this being irrelevant. The important consideration is the quantity of the dry powder per unit area of the preform.

One technique for production of SEDF preforms of the present invention is illustrated in Fig. 9. Essentially, a plurality of trays 29 is moved under a hopper 30 which dispenses the binder-powder composition. Each tray 29 will receive a predetermined quantity of the composition to provide SEDF preforms of predetermined weight. As shown in Fig. 9, the trays 29 can be placed on a conveyor 31, or may be part of a conveyor 31 which can move continuously, or intermittently, and timed so the binder in the composition will be cured before the preforms are removed from the trays 29. In the system illustrated, the preforms are received by another conveyor 32 which will carry the preforms to the next

processing step. It should be understood that the conveyor 31 can take various geometrical arrangements, including a zig-zag shape in the horizontal plane and a stepped shape in vertical plane.

In using the system shown in Fig. 9, if a substrate is desired on the preform, the substrate, with or without superabrasive particles thereon, can be placed in the bottom of the trays 29. Also, superabrasive particles, with or without a substrate, can be placed on top of the preform after the tray 29 is filled to the desired extent.

It will therefore be realized that the SEDF preform may be made in the form of discrete plates as shown in Fig. 9, or may be made in the form of continuous tapes as shown in Figs. 5 and 6. Either form can then be cut easily with scissors, paper cutter, die cutting or the like.

Preparation of Abrasive Articles

Fig. 10 of the drawings shows the preferred means and method for heating an SEDF preform and condensing the preform. Fig. 10 illustrates a generally conventional sinter fixture for sintering under pressure. It will be seen that there is a bottom punch 34 and a top punch 35, the space between the punches 34 and 35 being closed by the side plates 36. Within the cavity so defined, there is an SEDF preform 38, here shown as having superabrasive particles 39 distributed therein, and a plurality of superabrasive particles 40 on the top side of the preform 38.

Those skilled in the art will understand that the punches 34 and 35 will be urged towards each other as indicated by the arrows, and an electric current will be passed through the sinter fixture and/or the preform to heat the preform. An important feature of the present method is that the side plates 36 will tend to restrain lateral movement of the SEDF preform during sintering, even though there may be a flow of liquid as the binder and/or retaining matrix melt and run.

A further advantage of the SEDF preform in a sintering fixture as shown in Fig. 10 is that the softness of the preform makes redistribution of material quite easy. As a result, variations in thickness and stress can be made uniform simply through the usual pressure on the preform during sintering. The preform therefore has less sensitivity to various non- uniformities, and tends to reduce damage to the sinter molds. The inventor has experienced a 50-fold reduction in consumption of graphite mold parts since using the technique disclosed herein. It should be noted that, because of the softness and deformability of the SEDF preform, abrasive articles with a corrugated shape can be mass produced without significant consumption of corrugated (hence expensive) punches, e.g. graphite or metal punches.

It should be understood that the sinter mold can be loaded with several assemblies of SEDF preforms, the assemblies being separated from one another by punches and/or separators as disclosed in U. S. Patent No. 5,203,880, "Method and Apparatus for Making Abrasive Tools", by the present inventor. Such sintering "in stock" is illustrated in Fig. 10A. The unique uniformity, softness and deformability of the SEDF preform make sintering in stock acceptable for mass production technology.

While the heating of the SEDF preforms under pressure has many advantages, there is one severe disadvantage: the heating melts and vaporizes the binder, which runs; and, the liquid or vaporized binder, intensified by the applied pressure, tends to carry the retaining powder and superabrasive particles out of the mold. If most of the retaining powder is washed out of the mold, there will of course be practically no matrix material to hold the left over superabrasive particles in place. Also, melted binder and/or melted or moved retaining matrix of SEDF preform will catch the superabrasive particles, which can be washed out of the mold.

To solve the problem of the loss of retaining powder and superabrasive particles, it has been found that a porous

layer can be placed against the SEDF preform to prevent lateral movement of the particles. The porous layer may take many forms, but will not be held together by a binder as used in the SEDF preform. Rather, the porous layer may be screen wire, a conventional compacted preform, egg-crate or reticulated metal structures or the like.

Looking at Fig. 11, it will be noticed that the superabrasive particles 41 are larger than the openings in the porous layer 42. Under pressure, the particles 41 may cut into the porous layer 42. The particles 44 of the retaining powder are smaller than the opening in the layer 42, so these particles will pass easily into the openings of the layer 42.

As shown in Fig. 11, there is a second porous layer 45 on the opposite side of the SEDF preform; and, the assembly shown in Fig. 11 will be urged together and heated under pressure. The porous layers 42 and 45 will support the superabrasive particles and prevent lateral movement (perpendicular to the direction of the applied compaction force), and will provide additional volume to receive the SEDF preform, and restrain lateral motion of the particles of retaining powder in the SEDF preform. The porous layers will also temporarily absorb liquid binder to reduce the flow of binder and thereby help prevent washout of retaining powder and superabrasive particles.

The porous layer, or layers, can be placed in various positions relative to the SEDF preform and other layers of an assembly to be sintered. By way of example, and not by way of limitation, Fig. 12 shows the SEDF preform 46 having a porous layer 48 on one side, and a layer of abrasive particles 49 on the opposite side of the porous layer 48, a substrate, or carrier 50 holding the particles 49 in place. Fig. 13 shows the same arrangement, but the substrate 50 is between the particles 49 and the porous layer 48.

Fig. 14 shows the SEDF preform 46 in the middle with the porous layer 48 on one side, and the superabrasive particles 49 and substrate 50 on the opposite side. Fig. 15 shows the