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Application Data Sheet 37 CFR 1.76	Attorney Docket Number	02074-32836.US
	Application Number	
Title of Invention	COMPOSITE CONDITIONER 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.
This document may be completed electronically and submitted to the Office in electronic format using the Electronic Filing System (EFS) or the document may be printed and included in a paper filed application.

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
	Chien-Min		Sung	

Residence Information (Select One) ○ US Residency ◉ Non US Residency ○ Active US Military Service

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Postal Code	251	**Country**	TW

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

Correspondence Information:

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

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Email Address		Add Email	Remove Email

Application Data Sheet 37 CFR 1.76	Attorney Docket Number	02074-32836.US
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Title of Invention	COMPOSITE CONDITIONER AND ASSOCIATED METHODS	

Application Information:

Title of the Invention	COMPOSITE CONDITIONER AND ASSOCIATED METHODS		
Attorney Docket Number	02074-32836.US	**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)	9	**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☐ **Request Not to Publish.** I hereby request that the attached application not be published under 35 U.S.C. 122(b) and certify that the invention disclosed in the attached application **has not and will not** be the subject of an application filed in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	12726786	2010-03-18
Prior Application Status	Patented		Remove

Application Number	Continuity Type	Prior Application Number	Filing Date (YYYY-MM-DD)	Patent Number	Issue Date (YYYY-MM-DD)
12726786	Continuation in part of	12255823	2008-10-22	8393934	2013-03-12

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Prior Application Status	Patented				Remove
Application Number	Continuity Type	Prior Application Number	Filing Date (YYYY-MM-DD)	Patent Number	Issue Date (YYYY-MM-DD)
12255823	Continuation in part of	12168110	2008-07-05	8398466	2013-03-19
Prior Application Status	Expired				Remove
Application Number	Continuity Type	Prior Application Number	Filing Date (YYYY-MM-DD)		
12168110	non provisional of	60976198	2007-09-28		
Prior Application Status	Patented				Remove
Application Number	Continuity Type	Prior Application Number	Filing Date (YYYY-MM-DD)	Patent Number	Issue Date (YYYY-MM-DD)
12168110	Continuation in part of	11560817	2006-11-16	7762872	2010-07-27
Prior Application Status	Abandoned				Remove
Application Number	Continuity Type	Prior Application Number	Filing Date (YYYY-MM-DD)		
11560817	Continuation in part of	11357713	2006-02-17		
Prior Application Status	Expired				Remove
Application Number	Continuity Type	Prior Application Number	Filing Date (YYYY-MM-DD)		
11357713	non provisional of	60681798	2005-05-16		
Prior Application Status	Patented				Remove
Application Number	Continuity Type	Prior Application Number	Filing Date (YYYY-MM-DD)	Patent Number	Issue Date (YYYY-MM-DD)
	Continuation in part of	12628859	2009-12-01	7901272	2011-03-08
Prior Application Status	Patented				Remove
Application Number	Continuity Type	Prior Application Number	Filing Date (YYYY-MM-DD)	Patent Number	Issue Date (YYYY-MM-DD)
12628859	Continuation in part of	11804221	2007-05-16	7651386	2010-01-26
Prior Application Status	Abandoned				Remove
Application Number	Continuity Type	Prior Application Number	Filing Date (YYYY-MM-DD)		
11804221	Continuation in part of	11223786	2005-09-09		
Prior Application Status	Pending				Remove
Application Number	Continuity Type	Prior Application Number	Filing Date (YYYY-MM-DD)		
	Continuation in part of	12850747	2010-08-05		
Prior Application Status	Expired				Remove
Application Number	Continuity Type	Prior Application Number	Filing Date (YYYY-MM-DD)		
12850747	non provisional of	61246816	2009-09-29		
Prior Application Status	Pending				Remove
Application Number	Continuity Type	Prior Application Number	Filing Date (YYYY-MM-DD)		
	Continuation in part of	13034213	2011-02-24		
Prior Application Status	Expired				Remove

Application Data Sheet 37 CFR 1.76	Attorney Docket Number	02074-32836.US
	Application Number	
Title of Invention	COMPOSITE CONDITIONER AND ASSOCIATED METHODS	

Application Number	Continuity Type	Prior Application Number	Filing Date (YYYY-MM-DD)
13034213	non provisional of	61333162	2010-05-10

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
101112625	TW	2012-04-10	(•) Yes ( ) No

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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.

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◯ 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)	2013-03-12
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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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.

COMPOSITE CONDITIONER AND ASSOCIATED METHODS

PRIORITY DATA

This application is a continuation-in-part of U.S. Patent Application Serial No. 12/726,786, filed on March 18, 2010, which is a is a continuation-in-part of U.S. Patent Application Serial No. 12/255,823, filed October 22, 2008, which is a continuation-in-part of U.S. Patent Application Serial No. 12/168,110, filed on July 5, 2008, which claims the benefit of U.S. Provisional Patent Application Serial No. 60/976,198, filed September 28, 2007, and which is also a continuation-in-part of U.S. Patent Application Serial No. 11/560,817, filed November 16, 2006, which is a continuation-in-part of U.S. Patent Application Serial No. 11/357,713, filed February 17, 2006, which claims the benefit of U.S. Provisional Patent Application Serial No. 60/681,798, filed May 16, 2005. This application is additionally a continuation-in-part of U.S. Patent Application Serial No. 12/628,859, filed December 1, 2009, which is a continuation of U.S. Patent Application Serial No. 11/804,221, filed May 16, 2007, which is a continuation of U.S. Patent Application Serial No. 11/223,786, filed September 9, 2005. This application is also a continuation-in-part of U.S. Patent Application Serial No. 12/850,747, filed August 5, 2010, which claims the benefit of United States Provisional Patent Application Serial No. 61/246,816, filed on September 29, 2009. This application is further a continuation-in-part of U.S. Patent Application Serial No. 13/034,213, filed February 24, 2011, which claims the benefit of U.S. Provisional Patent Application Serial No. 61/333,162, filed on May 10, 2010. Each of these applications is incorporated herein by reference. This application also claims the benefit of Taiwan Patent Application Serial No. 101112625, filed on April 10, 2012, which is incorporated herein by reference.

BACKGROUND OF THE INVENTION

For the tiny copper circuit or the interlayer tungsten circuit on the silicon wafer surface, even for the oxide film dielectric layer of the insulated circuit, making the surface flat through the planarization process to facilitate the subsequent process steps is necessary. Currently, with the process of manufacture integrated circuits (IC) on the

semiconductor wafer, the most outstanding planarization technology is the chemical-mechanical planarization (CMP), which flattens a surface by pressing the wafer grinding on a rotating polishing pad.

The chemical-mechanical planarization is processed by transferring the slurry in between the wafer and the polishing pad stably and evenly to soak the polishing pad surface with slurry which containing chemicals (for instance, acid and oxidant) to erode the film on the wafer surface. The myriad of nano ceramics abrasive (ex. SiO2, Al2O3 and CeO2) in the slurry further pierce and scrape trace film, and simultaneously conduct chemical etching and mechanical grinding to remove the protruding deposit layer on the wafer. The purpose of planarization can be achieved by polishing the wafer surface.

A conditioner, whose function is to polish pad, is a necessary supply for chemical-mechanical planarization. The so-called condition is directed to shaving polishing pad surface and removing the waste accumulated on the polishing pad surface to maintain the roughness of the polishing pad surface. In addition, a conditioner could also produce tiny uplift and compression on the surface, and it is known as the height different of the asperities. Accordingly, the contact area on the polishing pad would be reduced significantly. Once the contact area becomes smaller, the contact pressure becomes greater to make the slurry at the contact point extrusion the protruding part of the wafer and the chemicals (ex. H2O2) in the slurry would be oxidation to soften or erode the wafer.

However, the waste produced in the process of the chemical-mechanical planarization, including the debris of the wafer, ex. copper wires, tungsten filled holes, oxidation film, slurry, abrasive and polishing pad scrap etc, the debris usually accumulates on the polishing pad surface and is pressed to become glaze. Once the glaze is formed, polishing pad would become slippery and difficult to maintain grinding power. Therefore, after polishing each wafer, it is necessary to utilize a conditioner to achieve the grinding efficiency of the process (ex. polishing speed) and flatness (ex. the thickness distributed of the wafer coating) as to stabilize the wafer quality.

However, previous diamond conditioner is usually secured the diamond on the metal table surface by binder. Although it is adapted for conditioning the polishing pad, with regard to the more accurate chemical-mechanical planarization process, scratch, partial dishing,

erosion and thickness non-uniformity of the wafer would cause easily by the rough asperities of the polishing pad. The smaller the required IC critical dimension, the greater the demand to the flatness of the wafer surface, also the greater the demand to the conditioner, and which can lead to previous diamond conditioner not being able to satisfy the pre-requirement of the chemical-mechanical planarization process smaller than 45nm.

In an effort to obtain a dresser with abrasive tips that are as level as possible, it is often prudent to use diamond abrasives of high shape regularity, and the crystal surface coverage of this kind of crystal is quite high, otherwise, using the crystal with low crystal surface coverage would cause an overshooting height difference. Additionally, a diamond having the highest polishing pad penetration ability, the so-called killer diamond, presents no difference when compared to other diamonds with respect to pad height. In order to meet the demand for a smaller critical dimension, continuing improvement of the conditioner is necessary. Using the sharper polishing tips and maintaining the flatness of the tips height (i.e. the difference of the polishing tips top height), making conditioner forming the subtler and more uniform nick on the polishing pad, and improving the removal ratio to the polishing pad simultaneously, by this, it is efficient to remove the hard layer and recover the asperities on the polishing pad to polish the coating on the wafer rapidly.

SUMMARY OF THE INVENTION

The present disclosure provides efficient methods, devices, and systems of conditioning a CMP pad. In one aspect, for example, a composite conditioner can include a base plate and a plurality of polishing units secured to a surface of the base plate by an adhesive layer, where each polishing unit includes a plurality of polishing tips secured in a binding layer. Additionally, a height difference between a first highest polishing tip and a second highest polishing tip is less than or equal to about 10μm, a height difference between the first highest polishing tip and a tenth highest polishing tip is less than or equal to about 20μm, and a height difference between the first highest polishing tip and a 100th highest polishing tip is less than or equal to about 40μm. Furthermore, the first highest polishing tip protrudes from the binding layer to a height of greater than or equal to about 50μm. In some aspects, at least 1% of the plurality of polishing tips actively condition a

CMP pad during a conditioning process. In other aspects, less than 40% of the surface of the base plate is covered with polishing units. The polishing units can be arranged on the plate in a variety of configurations. Non-limiting examples can include a single ring, double rings, multi-rings, radial arrangements, spiral arrangements, and the like.

In another aspect of the present disclosure, a method of making a composite conditioner can include disposing a second mold on a first mold, wherein the second mold includes a plurality of holes, placing a polishing unit in each of the plurality of holes of the second mold, where each polishing unit includes a plurality of polishing tips secured in a binding layer, applying an adhesive layer over the second mold to secure the polishing units, and removing the first mold and the second mold to form a composite conditioner. Regarding the conditioner, a height difference between a first highest polishing tip and a second highest polishing tip is less than or equal to about 10μm, a height difference between the first highest polishing tip and a tenth highest polishing tip is less than or equal to about 20μm, and a height difference between the first highest polishing tip and a 100th highest polishing tip is less than or equal to about 40μm and the first highest polishing tip protrudes from the binding layer to a height of greater than or equal to about 50μm.

In yet another aspect, a process of chemical-mechanical planarization (CMP) can include polishing a wafer against a rotating polishing pad and applying the conditioner as has been described against the polishing pad to remove debris and condition the pad.

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. 1A to 1G show a flow chart of manufacturing a composite conditioner in accordance with one embodiment of the present invention;

FIG. 2A to 2G show a flow chart of manufacturing the composite conditioner in

accordance with one embodiment of the present invention;

FIG. 2H shows a top view of a composite CMP pad dresser in accordance with an embodiment of the present invention;

FIG. 3 shows a comparison chart of the tip height difference and the working granule amount of a conditioner in accordance with one embodiment of the present invention as compared to a prior art conditioner;

FIG. 4 shows a comparison chart of the height difference (μm) and the scratch amount of the first highest point and the second highest point of a conditioner in accordance with one embodiment of the present invention as compared to a prior art conditioner;

FIG.5 shows a comparison chart of the scratch amount and the removal rate of a conditioner in accordance with one embodiment of the present invention as compared to a prior art conditioner;

FIG.6 shows a comparison chart of the in use time and the removing rate of a conditioner in accordance with one embodiment of the present invention as compared to a prior art conditioner;

FIG.7 shows a comparison chart of the polishing wafer amount and the removal rate of a conditioner in accordance with one embodiment of the present invention as compared to a prior art conditioner;

FIG.8 shows a schematic diagram of the chemical-mechanical planarization process in accordance with one embodiment of the present invention; and

FIG.9 shows a schematic diagram of polishing granules in accordance with one 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" and "polishing unit" can be used interchangeably, and refer 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

One objective according to some aspects of the present invention is to provide a composite conditioner, which is utilizing the shape of polishing tips while maintaining the distribution of tip height to avoid the over aggressive cutting of highly exposed abrasives (e.g. killer diamond) so as to improve the ratio of working polishing tips on the polishing pad during conditioning, and to increase the removal ratio from the wafer and the durability of the conditioner.

In one embodiment, for example, a composite conditioner can include a base plate and a plurality of polishing units secured to a surface of the base plate by an adhesive layer, where each polishing unit includes a plurality of polishing tips secured in a binding layer. The height difference between the first highest polishing tip and the second highest polishing tip is less than or equal to about 10μm, the height difference between the first

highest polishing tip and the tenth highest polishing tip is less than or equal to about 20μm, and the height difference between the first highest polishing tip and the 100th highest polishing tip is less than or equal to about 40μm. Furthermore, the first highest polishing tip protrudes from the binding layer to a height of greater than or equal to about 50μm.

Due to the fact the chemical-mechanical planarization (CMP) is directed to planar polishing, the contact distribution between the high point of the wafer and the polishing pad leads to confirm the quality (i.e. yield rate) and the efficiency (i.e. production capacity) of the CMP. If the distribution of the high point of the asperities in the CMP pad is uneven, the abrasion rate of the wafer in different parts of the polishing pad would be different and the wafer surface may become uneven. Furthermore, it is possible that some areas would be over-polished to result in dishing or layer erosion; or in some cases can result in under-polished regions producing residuals. If within wafer non-uniformity (WIWNU) is high, the effect of the chemical-mechanical planarization would be not desired. Accordingly, the present disclosure provides an enhanced conditioner based on extensive data from testing and experiment, and especially adjusts the height of the working polishing tips by a thickness adjustable adhesive layer to compensate for the difference of polishing units thickness and achieve aforesaid height distribution of the highest tip to avoid producing killer diamond and improve the ratio of the effective working polishing tips on the polishing units.

With regard to the present invention aforesaid composite conditioner the material of the thickness adjustable adhesive layer could be any suitable organic adhesive. Each polishing unit further comprises a unit substrate, and a binding layer is positioned between the surface of the unit substrate and the polishing tips. This binding layer can be a solder, a braze, an organic polymer, a plating layer, a sintered layer, a resin layer, or the like. The polishing tips refer to tips, a plurality of which are located on each polishing unit, can achieve the polishing effect, and the tips are exposed at the binding layer, that is, the height of the polishing tips means the height of the polishing tips protrude over the binding layer surface.

With regard to high-precision measurement for the height of the polishing tips,

various techniques are contemplated. In one aspect, for example, measurement can be accomplished by an optical detection system such as the surface detector manufactured by Germany FRT (Fries Research & Technology GmbH). The surface detector detects the height of total polishing tips (ex. more than 10000) of the conditioner, that is, detects the height of the polishing tips protrusion over the binding layer surface. Using the least square method with the height data, a theorized plane may be calculated across the tops of the polishing tips. The first highest tip is the height of the polishing tip that protrudes over the theorized plane to the greatest extent, the second highest tip is the height of the polishing tip that protrudes over the theorized plane to the second most extent, and so on.

The position of the wafer to be polished mostly depends on the distribution of the polishing pad top, and the distribution depends on the height of the polishing tips of the conditioner. The conditioner can be disk-shaped (as adapted for Applied Materials) or ring-shaped (as adapted for Ebara). The diameter of the disk can be about 100mm in general, and the diameter of the ring can be greater than 250mm. Generally, there are tens of thousands polishing tips (e.g. diamond particles) distributed on the conditioner, for example: the polishing tips of 80/90 mesh, 100/120 mesh and 120/140 mesh, but the difference in size might be greater than 30% even in the same mesh. Additionally, there can also be a difference in size in similar polishing tips when oriented different directions; for example, the distance between the tips is 30% greater than that between particle planes, and if the shape of the polishing tips is not regular, the difference would be greater.

Therefore, the present invention regulates the polishing particles such that the height difference between the first highest polishing tip and the second highest polishing tip is less than or equal to about 10μm, the height difference between the first highest polishing tip and the tenth highest polishing tip is less than or equal to about 20μm, and the height difference between the first highest polishing tip and the 100th highest polishing tip is less than or equal to about 40μm, and the first highest polishing tip protrudes from the binding layer to a height of greater than or equal to about 50μm to ensure that there are sufficient working tips in the conditioner to achieve effective polishing of the IC wafer surface rapidly and uniformly. Additionally, it should be noted that the first highest tip and the second highest tip might be located in different polishing units or at the same polishing

unit, similar with the other polishing tips; in another words, the tip distribution is not centralized at single polishing unit.

With respect to the aforesaid composite conditioner, the particles of the polishing tips can be any superabrasive material, including, without limitation, artificial or natural diamond, polycrystalline diamond (PCD), cubic boron nitride (CBN), poly crystal cubic boron nitride (PCBN), or a combination thereof. In some aspects, particle size can be in the range from about 100μm to about 500μm. The aforesaid diamond can be deposition diamond, polycrystalline diamond or the combination thereof. Additionally, there is no restriction to the material of the base plate, for example, resin, metal, alloy, plastic, rubber, ceramics, glass or the combination thereof (examples include the admixture between resin and alloy).

Additionally, in some cases diamond is formed by transforming graphite with iron-nickel alloy at high pressure (ex. 5GPa), thus leaving iron-nickel inclusion inside. The alloy can transform the diamond back to the graphite or carbon at high temperature, and the volume will increase substantially to often split the diamond. Thus, diamond secured to the diamond disk by brazing can be problematic in these cases where high temperature of the braze can cause the diamond to split. In the CMP process, such fractured diamond tips may crumble along the micro-fracture surface, releasing diamond chips that can scratch the wafer. In some aspects, an electromagnetic separator can be used to separate diamond particles having excessive metal inclusions. In some aspects, it can be beneficial to choose a low melting point solder for attaching the diamond particles to the polishing units.

In the present invention, when choosing the solder as the material of the binding layer, the solder layer could comprise greater than about 1 weight percentage of at least one of Cr, B, P, Ti and alloys thereof, and greater than about 50 weight percentage of at least one of: Ni, Cu and alloys thereof. The Cr, B, P, Ti etc. can be used for activating the solder layer. Specifically, the solder can be is Ni-Cr-B, Ni-Cr-P, or Cu-Sn-Ti alloy, where the melting point is between about 800°C and about 1000°C.

Additionally, each polishing unit can further selectively include a metal cover layer which covers on the surface of the binding layer, and the material of the metal cover layer can be at least one of Ni, Cr, Pd, Co, Pt, Au, Ti, Cu, W and alloys thereof to further

adhere the polishing tips on the unit substrate of the polishing unit. Furthermore, each polishing unit further selectively comprises a protective layer which is thin inert material and covers on the surface of the metal cover layer, and the material of the protective layer is metal such as Pd, Pt, or non-metal as silicon carbide, silicon nitride, aluminum nitride, alumina, zirconia, diamond-like carbon (DLC), organic film (as Teflon) or a combination thereof to avoid the solder be eroded by the polishing slurry (ex. pH=3 tungsten process slurry).

Generally, the height of the diamond tips will determine the power pressed into the polishing pad but the sharpness will affect the depth of the diamond penetration into the polishing pad. In general, when the diamond gets sharper, the polishing pad deformation of elasticity and plasticity will be smaller, and the depth of cut is greater. Because the diamond abrasive formed on the conditioner surface is different in shape and direction, the top hundred-height diamond could contact the polishing pad. If the diamond is regular in shape and in symmetry, the angle will be obtuse and may be difficult to pierce into the polishing pad, and the diamond will push onto the polishing pad surface and form protruding plastic deformation from two sides; on the other hand, if the diamond has a sharp surface, which if not regular in shape, it may pierce into the polishing pad, cut the polishing pad surface and remove the waste simultaneously.

Therefore, with regard to the aforesaid composite conditioner of the present invention, the crystal surface coverage of more than 50% of the top 100 height polishing tips are less than 80%, and the preferred is less than 50%.

In some aspects, the highest 100 diamond tips may dress or condition less than 50% of a CMP pad. In other words, the 100 highest diamond tips perform less than 50% of the overall dressing workload. In some aspects, the workload is less than 80% of the overall dressing workload. In additional aspects, the 10 highest tips may dress or condition less than 50% of the CMP pad. Again, in this respect, the 10 highest diamond tips perform less than 50% of the overall dressing workload. In some aspects, the workload is less than 80%. Conversely described, the diamond particles in the dresser outside of the highest 100 or 10 may perform more than 50% of the overall dressing workload. In some aspects, the overall workload may be more than 80%.

As the crystal surface coverage of the polishing tips becomes lower that those polishing tips have more crack surface (sharp surface) and the polishing tips of the polishing unit working surface comprises corners and ridges, have more ridges with the angle less than 45° (means the angle of the two crystal surface which form the ridge), or have more angle less than 90°, therefore have higher removal ratio for the polishing pad.

Referring now to figure 9, the schematic diagram to polish abrasive, wherein A, E and S respectively points to corner, ridge and surface, wherein S_1 is the crystal surface, A_1 is the angle of the crystal surface, S_2 is the crack surface and A_2 is the angle of at least one crack surface. From this, A_1 and A_2 are two different angle, A_1 is greater than 90° (obtuse angle) and A_2 is less than 90° (acute angle), means that when using A_2 as polishing tips, the removal efficiency is greater than A_1.

The polishing tips can be arranged in a pattern on the unit substrate or the polishing tips form a plurality of clusters and the clusters are arranged in a pattern on the unit substrate, where each the cluster is composed of two to six polishing tips.

Additionally, the polishing units can be arranged on the base plate as single ring, double rings, multi-rings, radial or spiral in shape and it could be disk in shape, blade in shape or polygon. If the polishing units are disk in shape, the diameter is in the range of 5mm to 30mm. The diameter in the range of 14mm to 18mm is preferred. The base plate also could be disk in shape and the diameter is in the range of 80mm to 120mm. It also could secure the polishing unit on the ring substrate, which is 250mm to 270mm in diameter, for actual use. Additionally, if the polishing unit with a disk shape is positioned at an inner surface, the polishing unit on the inner surface can be obstructed by the outer polishing units when conditioning the polishing pad, so that the inner polishing unit may not function well. Therefore, the polishing units can be arranged at the outer edge of the disk-shape base plate surface, it could create a fringe effect on a outer edge during the CMP process, such that it will increase the working amount of the polishing units, and the CMP process is adapted to apply for the nanometer process of 18inch wafer or 22nm wire.

In the aforesaid composite conditioner of the present invention, the spacing between the polishing units is not less than 0.1mm, a spacing of greater than 0.5mm is

preferred, so it could be sure that in the CMP process, the slurry could flow favorably along the spacing between the polishing units and would not remain inside due to defect as the scratch to the wafer. Besides, the thickness of the binding layer of the polishing unit's bottom is in the range less than 0.6mm so that it could fully adjust the polishing tips of each polishing unit to the same plane to increase the amount of the diamonds efficiently.

Formerly, the height difference of the tips is too big so that the amount of the tips utilized by traditional conditioner (comprising the tips piercing into polishing pad) is less than 1% of the tips total amount and the diamond coverage area of traditional conditioner is over 40%; on the other hand, in the aforesaid composite conditioner of the present invention, the amount of the polishing tips used for conditioning a polishing pad is more than 1% of the total polishing tips; besides, the area of the polishing units covering the base plate is less than 40% of the total area of the base plate surface to achieve the best remove ratio.

It is another objective of the present invention to provide a manufacture method of a composite conditioner, wherein using the polishing unit with sharp polishing tips, through the inversion method, setting the top several tips of each polishing unit as standard plane, adjusting the thickness of the binding material under each polishing unit to secure on the substrate so that the top several tips of the polishing units would not protrude to form the destructive "killer diamond". Using coplanar to make the highest point of each polishing unit achieve common height could also improve the ratio of working polishing tips, further to improve the removal ratio and durability of the conditioner.

As such, another embodiment of the present invention provides a manufacture method of a composite conditioner, including the following steps: installing a second mold on a first mold, wherein the second mold comprises a plurality of holes; installing a polishing unit in each the hole of the second mold, wherein each the polishing unit comprises a plurality of polishing tips and a binding layer securing the polishing tips; forming a thickness adjustable adhesive layer to secure the polishing units on a base plate; and removing the first mold and the second mold to form a composite conditioner, wherein the height difference between a first and a second highest tip, between the first

and a tenth highest tip, and between the first and a 100th highest tip protruding from a predetermined plane is respectively less than 10μm, 20μm, and 40μm, and the height of the first highest tip protruding the binding layer is greater than 50μm.

Generally, diamond conditioner is made by additive manufacturing method, that is, arranging in flat the diamond abrasive (like matrix distribution) on the plane substrate (as stainless steel No.316). Obtaining a diamond conditioner or tool with diamond particles having a height difference of the diamond top of single conditioner surface of about 50μm or less is difficult using know methods of securing the diamond abrasive such as electroplating with nickel or by brazing with an alloy, which suffers from curdling and gripping due to surface tensions when the braze is liquified. The former diamond surface is usually upward and the latter tips or the ridge usually protrude, and the diamonds with surface upward pierce into the polishing pad with difficultly and the diamond with tips or the ridge cut the polishing pad more easily. In contrast, the present invention can utilize electroplating first to form polishing units, combines organic flexible material and compensates a plurality of polishing units to arrange the polishing units at the base plate, it not only has the advantage of brazing but also could avoid the disadvantage thereof. Therefore, when the diamond conditioner is conditioning the polishing pad, the protrusion diamond top could contact and pierce into the polishing pad without having the polishing tips protruding too much, so piercing into the polishing pad too deep may increase the height difference between the asperities and therefore preventing the outstanding asperities from dipping too much slurry, and ultimately leading to the nano particle of the slurry to penetrate excessively into the wafer surface to produce over-polishing, and even scratching the wafer.

With the aforesaid manufacture method of a composite conditioner of the present invention, the thickness adjustable adhesive layer could be formed after using another plate to leveling the top tips of the polishing units, or after forming the thickness adjustable adhesive layer before the thickness adjustable adhesive layer solidified using the base plate to leveling the top tips of the polishing units.

The thickness adjustable adhesive layer could be composed by the organic adhesive, for example: a plastic material, a rubber material, a hot melt adhesive or the combination

thereof; besides, the holes of the second mold are arranged at the second mold outer. Specifically, the holes of the second mold are arranged at the outer ring of the second mold as 12 holes. Additionally, with the composite conditioner, the polishing units also could be arranged at inner or outer ring, and the smaller the polishing units are, the more the amount of the working granules when polishing, but the cost of manufacturing will be higher, so manufacturing the conditioner with appropriate amount of polishing units could balance the effect and cost.

With the aforesaid manufacture method of a composite conditioner of the present invention, before the second mold set on the first mold, further comprising: forming a temporary adhesive layer on the first mold surface and setting the second mold on the temporary adhesive layer to fold the temporary adhesive layer between the second mold and the first mold, and after removing the second mold, further comprising: removing the temporary adhesive layer. With a preferred embodiment of the present invention, the temporary adhesive layer is a double-sided tape.

In another preferred embodiment of the present invention, a side of the polishing units has the polishing tips, and the thickness adjustable adhesive layer is set on the opposite side, and the polishing units set at the holes contact the first mold through the polishing tips. Besides, after removing the first mold and the second mold, further comprising: forming a mold sealing glue layer to secure the polishing units. From the above, it is possible to make the polishing units arranged orderly on the same plane by adding pressure at the side having the polishing tips of the polishing units.

With the aforesaid manufacture method of a composite conditioner of the present invention, further selectively comprising: after removing the first mold, moving the second mold. That is, the conditioner might comprise the second mold.

It is another object of the present invention to provide a chemical-mechanical planaization (CMP) process, wherein sharp polishing tips may be used while the planarity of the working surface is maintained and the ratio of the effective working polishing tips on the polishing units is improved, and the removing rate and durability are increased.

To achieve aforesaid object, another embodiment of the present invention provides a process of chemical-mechanical planaization, including the following steps: providing a

polishing pad; installing a wafer on the surface of the polishing pad to polish the polishing pad with each other; and using a conditioner, which is installed on the surface of the polishing pad and removing the scrap of the polished wafer, wherein the conditioner is the aforesaid composite conditioner of the present invention.

The Moore's law states that the amount of the transistor in the same size in the same wafer will double every 18 months because of the improvement of the process technology and improved speed of the wafer executing computing. In accordance with the continuing improvement suggested by the Moore's law, the critical dimension of integrated circuit (IC) gradually exceeds the limit of 32nm, however, the bottleneck that is occuring now is that the it is current state of art of the chemical-mechanical process is difficult to surpass, so the process could not enter a stage for finer critical dimension. In other hand, because we utilize the improved conditioner in the chemical-mechanical planaization process of the present invention, we could use the diameter of the wafer in 200mm, 300mm or 450mm in the chemical-mechanical planaization process of the present invention, and the wafer surface having the IC critical dimension is smaller than or equal to 45nm, 28nm, or 22nm. Therefore, if the related technology utilizes the technology of the present invention, for example applying the chemical-mechanical planaization process of the present invention for manufacturing logic device, DRAM, flash memory or hard drive, the speed of the each aforesaid device executing computing will improve as postulated by the Moore's law.

Generally speaking, because diamond sizes differ in great variety, shape and direction, and the random distance of the high point, the plough of the polishing pad and the cut manner are multifarious and which leads to uneven height of the conditioned polishing pad and then leads to difficult controlling the high point position of the wafer contacting, the area and the pressure of each contact point, and result the high variability to the polishing rate and the uniformity. When the wafer varies greatly (8 inch to 12 inch even to 18 inch in the future), the variability to the polishing rate and the uniformity will become higher, and the quality and the efficiency of the chemical-mechanical planaization process will become lower. Not only that, when the critical dimension becomes more and

more narrow (45nm to 32nm even to 22nm in the future), the high point of the polishing pad gets broken more easily. When the electric circuit becomes narrow, the dielectric materials therebetween have to be hollower to avoid leakage, but the hollow dielectric layer is weak, so the contact pressure of the chemical-mechanical planaization process has to weaken, but lowered contact pressure will cause the speed of polishing wafer become lower and the poor productivity.

Currently, the diamond conditioner usually adopts the method of adding diamond on the plane to manufacture, for the reason that the top height is difficult to control. Besides, under the high temperature manufacturing (as brazing), the substrates will distort to make the top height more irregular, and significantly decrease the working granule amount, and even lead to tens of thousands of diamond on the conditioner, but there are only several hundred of working granule. Because the diamond conditioner adopts addition (flattening by the top which has diamond abrasive presses thereon) to manufacture, the size, shape and direction of the diamond are difficult to control, and the diamond amount, position and piercing depth are also difficult to control, cause the predictability of the chemical-mechanical planaization process effect to greatly reduce. Although it strictly controls the flatness, diamond shape, etc. of the conditioner substrate in industry, but the improving effect is not enough.

In view that the top of the diamond is not easy to be regular, the present invention adopts a method of reversed composite, and manufactures the diamond polishing units first, and inverts the diamond polishing units on a plane to make each of the polishing tips regular. At this time, set a thickness adjustable adhesive layer at the contrary side of the polishing tips, and finally irrigate the mold sealing glue layer in (as resin) to secure all polishing units. As the aforesaid manufacturing method could avoid the “killer diamond” that is common on the diamond dish, that is, the stand up protrusion diamond. If the “killer diamond” is too sharp, it may pierce into the polishing pad too deep and pull to fracture, and then form the “killer asperities” on the polishing pad surface to cause the wafer scratch.

The following explains the implemented method of the present invention by specific concrete embodiment, and a skilled person in this art could easily understand other

advantage and effect of the present invention through the content disclosed by the specification. The present invention also could be implemented and applied by other different concrete embodiment, and the detail of the present invention also could base on different view and apply for various decorations and changing without deviating the spirit of the present invention,

The drawings in the embodiment of the present invention are simplified schematic diagram. But the drawings only illustrate the components about the present invention, and the illustrated components are not the example when actually implement. The components amount, shape and ratio are s selective design when actually implement, and the components layout patterns might be more complicated.

References Fig. 1A to 1G, are the flow charts of manufacturing the composite conditioner.

First of all, as shown in Fig. 1A, it provides a first mold10, and first mold10 has an opening101. Then, as Fig. 1B illustrates, it forms a temporary adhesive layer11 on opening101 surface of first mold10, and then sets a second mold12 on temporary adhesive layer11 surface to make second mold12 position in opening101 of first mold10, so temporary adhesive layer11 is between second mold12 and first mold10, wherein second mold12 has a plurality of holes121. In this preparation, temporary adhesive layer11 is a double-sided tape.

As shown in Figs. 1C and 1G, it sets a polishing unit20 in each holes121 of second mold12, wherein each polishing unit20 comprises a plurality of polishing granules22 (each polishing granules22 at least has a polishing tip221), a unit substrate21 and a binding layer213 set between unit substrate21 surface and polishing tips221. Polishing units20 are made by general brazing, which simply explains as follow: spot-weld the solder sheet (BNi_2) at unit substrate21 with 20mm in diameter, 4mm in thickness, and then arrange the polishing granules (as diamond granules) on the solder sheet which is coated with viscose through the template, and then put those into the vacuum furnace exhaust to about 5torr to 10torr, and heating to not more than 1020°C with 12min last (during this the viscose is volatilized), and the solder melts to liquid and welds the diamond granules.

Because the diamond is originally positioned on a fixed solder material, when the solder melting to form as the liquid, the carbide elements (Cr) therein will diffuse to the diamond surface. So the solder will pull sink the diamonds with capillary power and climb the diamond surface to form a gradient. The diamonds will stick the solder sheet surface with plane originally, but now the rotation in the liquid will make the sharp corners up.

Then, as shown in Fig. 1D and 1E, flatten polishing units20 by a base plate24, and fill in a thickness adjustable adhesive layer23 to secure polishing units20. Then, as shown in Fig. 1F, remove first mold10, temporary adhesive layer11 and second mold12 to form a composite conditioner2.

Composite conditioner2 comprises: a base plate24; a plurality of polishing units20 placed on a surface of base plate24, and each polishing unit20 including a plurality of polishing tips221 and a thickness adjustable adhesive layer23 which used to secure polishing units20 on the surface of base plate24, wherein, with polishing tips221, the height difference between a first and a second highest tip, between the first and a tenth highest tip, and between the first and a one hundredth highest tip protruding from a predetermined plane is respectively less than 10μm, 20μm, and 40μm, and the height of the first highest tip protruding the binding layer213 is greater than 50μm.

Referring now to Fig. 1G, it is an enlarged schematic of the polishing units of the present invention. Each polishing unit 20 comprises a plurality of polishing granules22 (each polishing granules22 at least has a polishing tip221), a unit substrate21, a binding layer213 set between unit substrate21 surface and polishing tips221, a metal cover layer212 covering on binding layer213 surface and a protective layer211 covering on metal cover layer212 surface.

Binding layer213 could be a solder layer, a plating layer, a sintering layer or a resin layer. If it is a solder layer, the solder layer comprises greater than 1 weight percentage selected at least one from the group consisting of: Cr, B, P, Ti and the alloy thereof, and greater than 50 weight percentage selected at least one from the group consisting of: Ni, Cu and the alloy thereof.

Metal cover layer212 could be formed by plating, the material of the metal cover

layer is selected at least one from the group consisting of: Ni, Cr, Pd, Co, Pt, Au, Ti, Cu, W and the alloy thereof; the material of the protective layer211 is Pd, Pt, silicon carbide, aluminum nitride, alumina, zirconia, diamond-like carbon (DLC) or the combination thereof.

Reference to Fig. 2A to 2G, these are the flow chart of manufacturing the composite conditioner.

First of all, as shown in Fig. 2A, it provides a second mold12, and second mold12 has 12 holes121, and these holes are arranged at the outer of second mold12. Then, as shown in Fig. 2B, it set second mold12 on a first mold12 surface.

As shown in Fig. 2C, set a polishing unit20 in each holes121 of second mold12, wherein the working surface (the surface having polishing tips) of second mold12 faces to first mold12 surface. In this preparation, polishing unit20 is similar to said in preparation1. Then, as shown in Fig. 2D, it glues on the contrary side of the polishing tips of each polishing units20 to form a thickness adjustable adhesive layer23. Then, as shown in Fig. 2E, with a base plate24 presses on thickness adjustable adhesive layer23, wherein base plate24 has a notch to make base plate24 be an inverted U-shaped, and the inverted U-shaped base plate24 presses thickness adjustable adhesive layer23 by the notch.

As shown in Fig. 2F, it removes first mold10. Then, as shown in Fig. 2G, it inverts the structure of Fig. 2F, that removing second mold12, forming a mold sealing glue layer25, strengthening polishing unit20 and securing base plate24 forms a composite conditioner.

Although the polishing units made by the brazing might yield the killer diamond, but with the aforesaid method of the present invention, it could combine and use different polishing units having similar height killer diamond, and flatten the polishing tips, so the height of the killer diamond is close to that of the diamond tips on other polishing units.

Referring now to Fig. 3, it illustrates the composite conditioner manufactured by aforesaid preparation2 (embodiment1, wherein the crystal cover ratio of the chosen diamond granules is 60% to 80%, that is PDA 878 manufactured by Element Six), the

composite conditioner manufactured by conventional ceramic sintering (comparison1) and the composite conditioner manufactured by the brazing (comparison2), the comparison diagram of the tips height and working granules amount of the three conditioners. As shown in Fig. 3, when polishing, the present invention conditioner in embodiment1 has 60 to 70 working granules at the height different about 10μm; in contrast, comparison1 and comparison2 need more tips height difference to provide more working granules amount, even the conditioner in comparison1, it needs the height difference of the polishing granules more than 40μm to provide more than 100 working granules amount. From this, the actual working polishing granules amount for the same height difference, the present invention conditioner is far more than the prior art conditioner.

Reference to Fig. 4 and Fig. 5, it illustrates the composite conditioner manufactured by aforesaid preparation2 (embodiment1, wherein the crystal cover ratio of the chosen diamond granules is 60% to 80%, that is PDA 878 manufactured by Element Six; embodiment2, wherein the crystal cover ratio of the chosen diamond granules is 40% to 60%, that is PDA 657 manufactured by Element Six), and the composite conditioner manufactured by the brazing (comparison2), the comparison diagram of the height difference of the first high point and the second high point to the scratch amount, and the scratch amount to the removing rate of the three conditioners.

As shown in Fig. 4, with the present invention conditioner in embodiment1 and embodiment2, when the height difference of the first high point and the second high point is about 0mm, the polishing scratch amount is between 100 to 150 scratches, that means the quality of the conditioner in embodiment1 and embodiment2 are fine, and the quality stability is nice when polishing; in contrast, with the conditioner in comparison2, the high point difference of the polishing granules is scattered mixed, and the polishing scratch amount is less than between 50 to 150 scratches, that means the quality of the conditioner in comparison2 is scattered mixed, and the quality stability is bad when polishing. On the other hand, as shown in Fig. 5, the polishing removing rate of the present invention conditioner in embodiment2 is about 250μm to 300μm per hour, and the scratch amount is about 100 to 150 scratches, the polishing removing rate of the present invention conditioner in embodiment1 is about 150μm to 200μm per hour, and the scratch amount is

about 125 to 150 scratches, that means the present invention conditioner in embodiment1 and embodiment2 are high stability and is easy for quality control; in contrast, the polishing removing rate of the conditioner in comparison2 is about 125μm to 225μm per hour, and the scratch amount is about 50 to 75 scratches, that means the conditioner in comparison2 has low scratch amount and is unstable in quality.

Referring next to Fig. 6, it illustrates the composite conditioner manufactured by aforesaid preparation2 (embodiment1 and embodiment2), the composite conditioner manufactured by the brazing (comparison2) and the composite conditioner manufactured by the plating (comparison3), the comparison diagram of the using time to the removing rate of the four conditioners. As shown in Fig. 6, when polishing, the removing rate of the present invention conditioner in embodiment1 and embodiment2 decrease slowly with the longer using time, that means the present invention conditioner has the fastest conditioning rate and the longest durability cause of controlling the nice planeness; in contrast, the removing rate of the conditioner in comparison2 is unstable and then lead the problem in quality controlling, and the removing rate of the conditioner in comparison3 is bad, it cannot saving the polishing process time and cost.

Reference to Fig. 7 illustrates the composite conditioner manufactured by aforesaid preparation2 (embodiment2), the composite conditioner manufactured by the brazing (comparison2), the comparison diagram of the polishing wafer amount to the removing rate of the two conditioners. As shown in Fig. 7, in contrast to the comparison2, the present invention conditioner in embodiment2 could use in the chemical-mechanical planaization process to achieve polishing more wafer amount.

Referring to Fig. 8, it is a schematic diagram of the chemical- mechanical planaization (CMP) process. As shown in Fig. 8, generally, set a wafer4 on a polishing pad6 surface, and provide the polishing slurry by pipeline8 to polish polishing pad6 with each other, and set a conditioner2 at an another margin portion of polishing pad6 surface to remove the scrap after the wafer polished, wherein, the conditioner herein could use the conditioner manufactured by aforesaid preparation of the present invention.

From of all, recently, with convention whole piece brazing diamond conditioner, the

highest polishing tip is usually higher than the second highest one in more than 20μm, and the top 10 highest polishing granules protrusion only under 10mm; in contrast, with the aforesaid method of the present invention, manufactured the composite conditioner by inverting uniform head method to make the difference of the highest polishing tip to the second highest one in less than 10μm, therefore, it could improve the ratio of the effective working polishing tips on the polishing units, removing rate and durability.

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 composite conditioner comprising:

a base plate; and

a plurality of polishing units secured to a surface of the base plate by an adhesive layer, each polishing unit including a plurality of polishing tips secured in a binding layer, wherein a height difference between a first highest polishing tip and a second highest polishing tip is less than or equal to about 10μm, a height difference between the first highest polishing tip and a tenth highest polishing tip is less than or equal to about 20μm, and a height difference between the first highest polishing tip and a 100th highest polishing tip is less than or equal to about 40μm, and the first highest polishing tip protrudes from the binding layer to a height of greater than or equal to about 50μm.

2. The composite conditioner of claim 1, wherein at least 1% of the plurality of polishing tips actively condition a CMP pad during a conditioning process.

3. The composite conditioner of claim 1, wherein less than 40% of the surface of the base plate is covered with polishing units.

4. The composite conditioner of claim 1, wherein the polishing units are arranged on the base plate in an arrangement selected from the group consisting of a single ring, double rings, multi-rings, radial arrangements, and spiral arrangements.

5. The composite conditioner of claim 1, wherein at least one thousand polishing tips of the plurality of polishing tips condition a polishing pad during a conditioning process.

6. The composite conditioner of claim 1, wherein the plurality of polishing tips include diamond, cubic boron nitride, or a combination thereof.

7. The composite conditioner of claim 6, wherein the plurality of polishing tips include diamond, and the diamond is deposition diamond, polycrystalline diamond, or a combination thereof.

8. The composite conditioner of claim 1, wherein the crystal surface coverage of more than 50% of the top 100 height polishing tips are less than 80%.

9. The composite conditioner of claim 8, wherein the crystal surface coverage of more than 50% of the top 100 height polishing tips are less than 50%.

10. The composite conditioner of claim 1, wherein each polishing unit further includes a polishing unit substrate upon which the plurality of polishing tips are secured with the binding layer.

11. The composite conditioner of claim 1, wherein the binding layer is selected from the group consisting of a solder layer, a plating layer, a sintering layer, and a resin layer.

12. The composite conditioner of claim 11, wherein each polishing unit further comprises a metal cover layer that covers the binding layer, wherein the metal cover layer includes at least one material selected from the group consisting of Ni, Cr, Pd, Co, Pt, Au, Ti, Cu, W, and alloys thereof.

13. The composite conditioner of claim 12, wherein each polishing unit further comprises a protective layer covering the metal cover layer, wherein the protective layer includes a material selected from the group consisting of Pd, Pt, silicon carbide, aluminum nitride, alumina, zirconia, diamond-like carbon (DLC), and combinations thereof.

14. The composite conditioner of claim 11, wherein binding layer is a solder layer comprising 1 wt% or more of a first material selected from the group consisting of Cr, B, P, Ti, or an alloy thereof, or 50 tw% or more of a second material selected from the group

consisting of Ni, Cu, or an alloy thereof, or 1 tw% or more of the first material and 50 wt% or more of the second material.

15. The composite conditioner of claim 1, wherein the polishing units are disc-shaped, radial-shaped, or polygon-shaped.

16. The composite conditioner of claim 15, wherein the polishing units are disc-shaped having a diameter of from about 5mm to about 30mm.

17. The composite conditioner of claim 1, wherein the base plate is disc-shaped having a diameter of from about 80mm to about 120mm.

18. The composite conditioner of claim 1, wherein the polishing tips have a particle size of from about 100μm to about 500μm.

19. The composite conditioner of claim 1, wherein the adhesive layer is an organic adhesive.

20. The composite conditioner of claim 1, wherein the polishing tips are arranged in a predetermined pattern on the polishing unit substrate.

21. The composite conditioner of claim 1, wherein the polishing units are spaced no closer than about 0.1mm.

22. The composite conditioner of claim 21, wherein the polishing units are spaced at least 0.5mm apart.

23. The composite conditioner of claim 1, wherein the adhesive layer between the base plate and the polishing units has a thickness of less than or equal to about 0.6 mm.

24. A method of making a composite conditioner, comprising:

disposing a second mold on a first mold, wherein the second mold includes a plurality of holes;

placing a polishing unit in each of the plurality of holes of the second mold, each polishing unit including a plurality of polishing tips secured in a binding layer;

applying an adhesive layer over the second mold to secure the polishing units; and

removing the first mold and the second mold to form a composite conditioner wherein a height difference between a first highest polishing tip and a second highest polishing tip is less than or equal to about 10μm, a height difference between the first highest polishing tip and a tenth highest polishing tip is less than or equal to about 20μm, and a height difference between the first highest polishing tip and a 100th highest polishing tip is less than or equal to about 40μm and the first highest polishing tip protrudes from the binding layer to a height of greater than or equal to about 50μm.

25. The method of claim 24, wherein placing the polishing unit in the plurality of holes of the second mold further comprises orienting the polishing units such that the polishing tips face the first mold.

26. The method of claim 25, wherein the polishing tips contact the first mold such that the polishing unit are leveled across the first mold.

27. The method of claim 26, wherein the holes of the second mold are arranged around an outer periphery of the second mold.

28. A process of chemical-mechanical planarization (CMP), including the following steps:

polishing a wafer against a rotating polishing pad; and

applying the conditioner of claim 1 against the polishing pad to remove debris and condition the pad.

29. The process of claim 28, wherein the diameter of the wafer is 200mm, 300mm, or 450mm.

30. The process of claim 28, wherein a surface of the wafer has an IC critical dimension of less than or equal to 45nm.

31. The process of claim 30, wherein a surface of the wafer has an IC critical dimension of less than or equal to 28nm.

32. The process of claim 28, wherein the surface of the wafer has an IC critical dimension of less than or equal to 22 nm.

ABSTRACT

CMP pad dressers having leveled tips and associated methods are provided. In one aspect, for example, a composite conditioner can include a base plate and a plurality of polishing units secured to a surface of the base plate by an adhesive layer, where each polishing unit includes a plurality of polishing tips secured in a binding layer. Additionally, a height difference between a first highest polishing tip and a second highest polishing tip is less than or equal to about 10μm, a height difference between the first highest polishing tip and a tenth highest polishing tip is less than or equal to about 20μm, and a height difference between the first highest polishing tip and a 100th highest polishing tip is less than or equal to about 40μm. Furthermore, the first highest polishing tip protrudes from the binding layer to a height of greater than or equal to about 50μm.



FIG. 1A

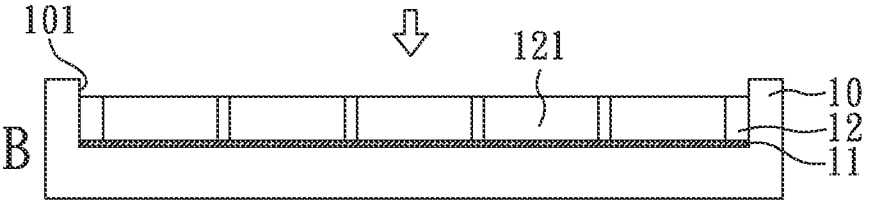


FIG. 1B

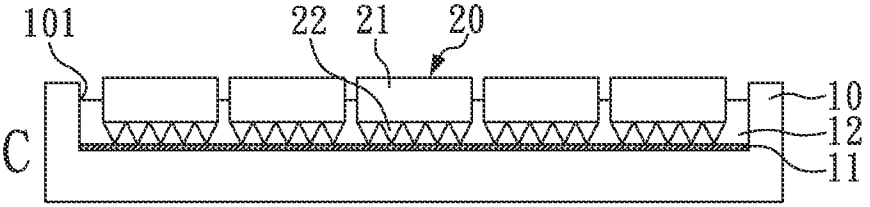


FIG. 1C

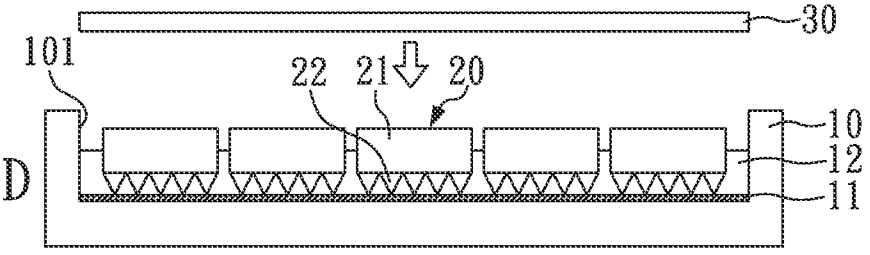


FIG. 1D

FIG. 1E

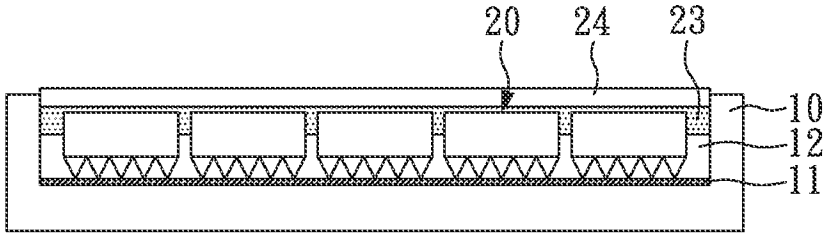


FIG. 1F

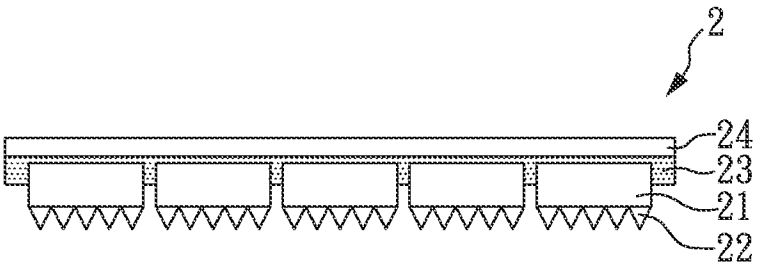


FIG. 1G

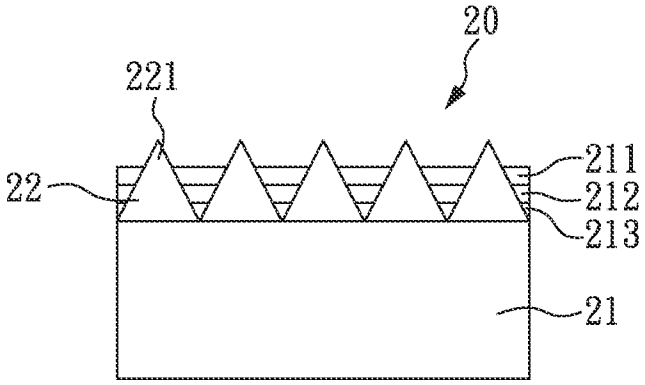




FIG. 2A

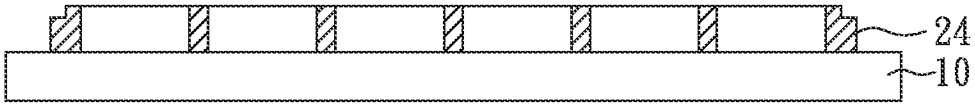


FIG. 2B

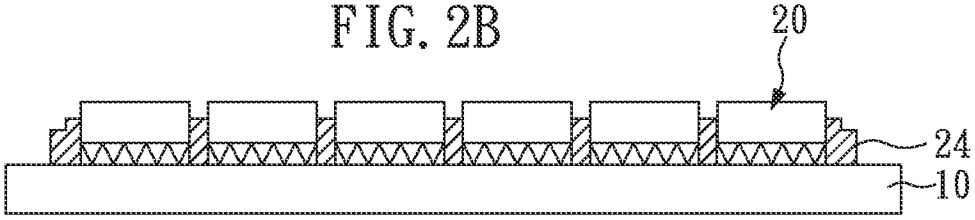


FIG. 2C

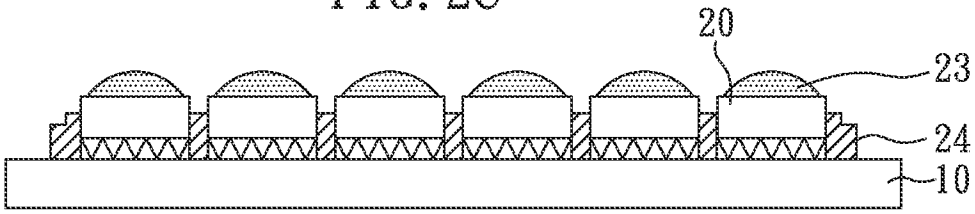


FIG. 2D

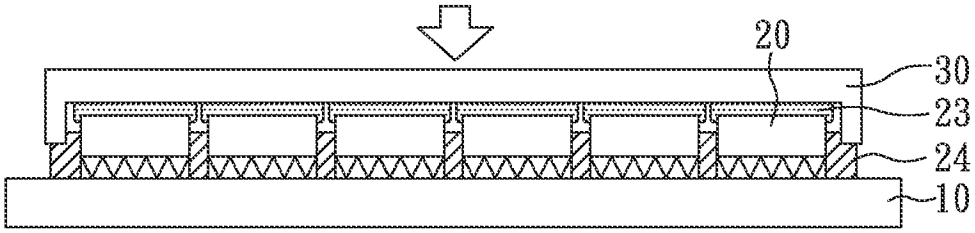


FIG. 2E

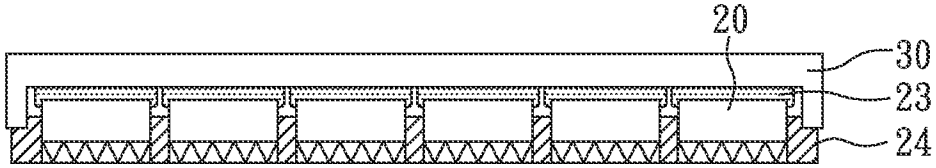


FIG. 2F

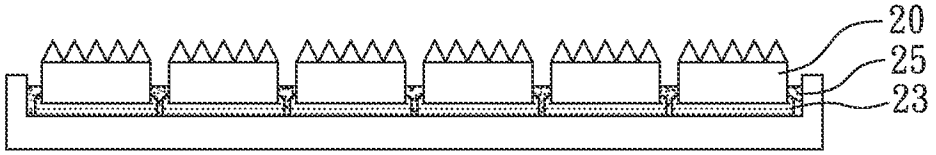


FIG. 2G

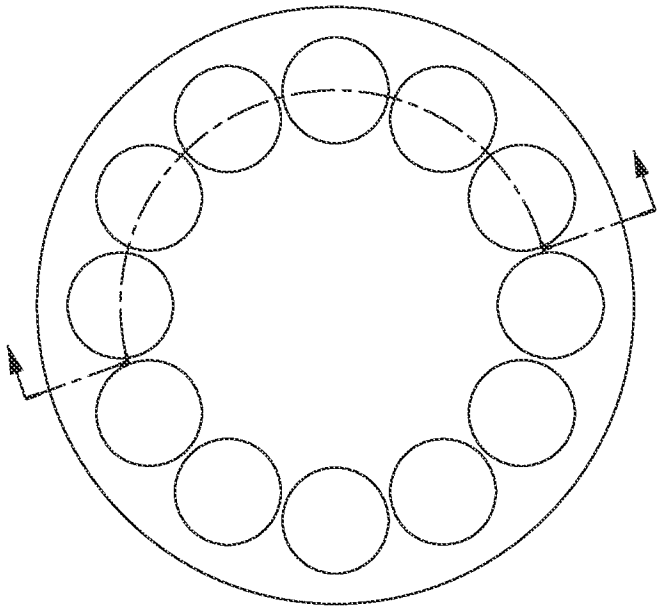


FIG. 2H

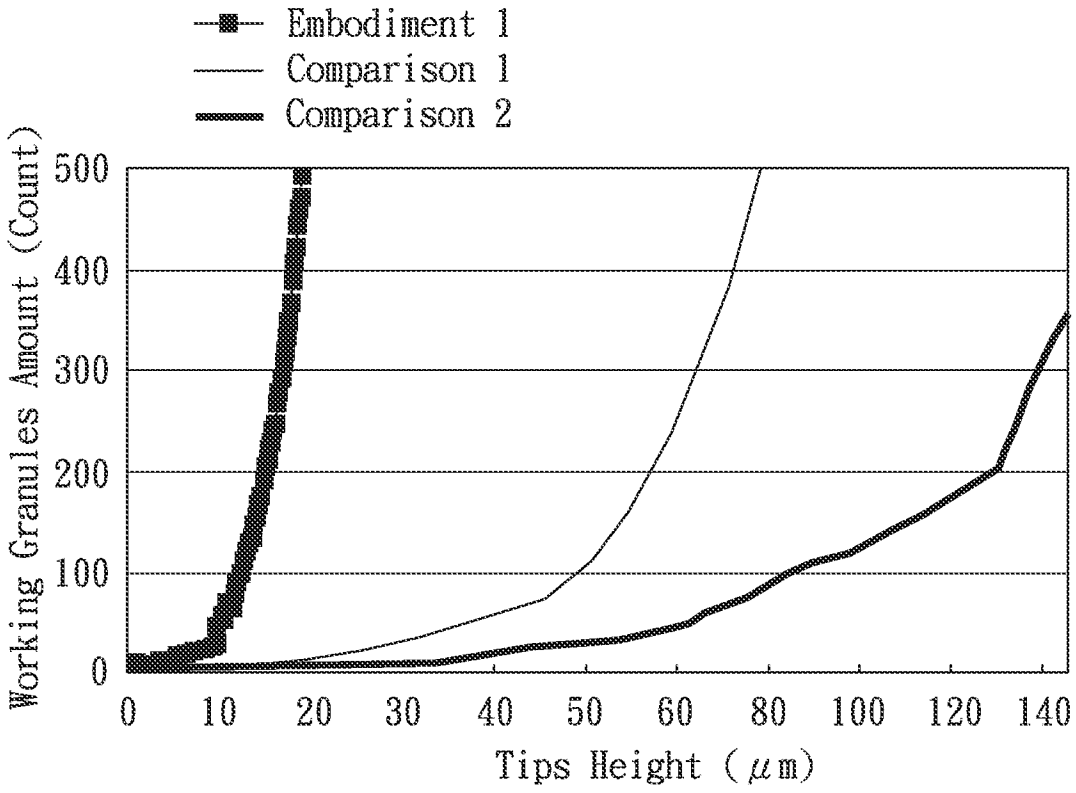

Embodiment 1
Comparison 1
Comparison 2
Working Granules Amount (Count)
500
400
300
200
100
0
0
10
20
30
40
50
60
80
100
120
140
Tips Height (μm)

FIG. 3

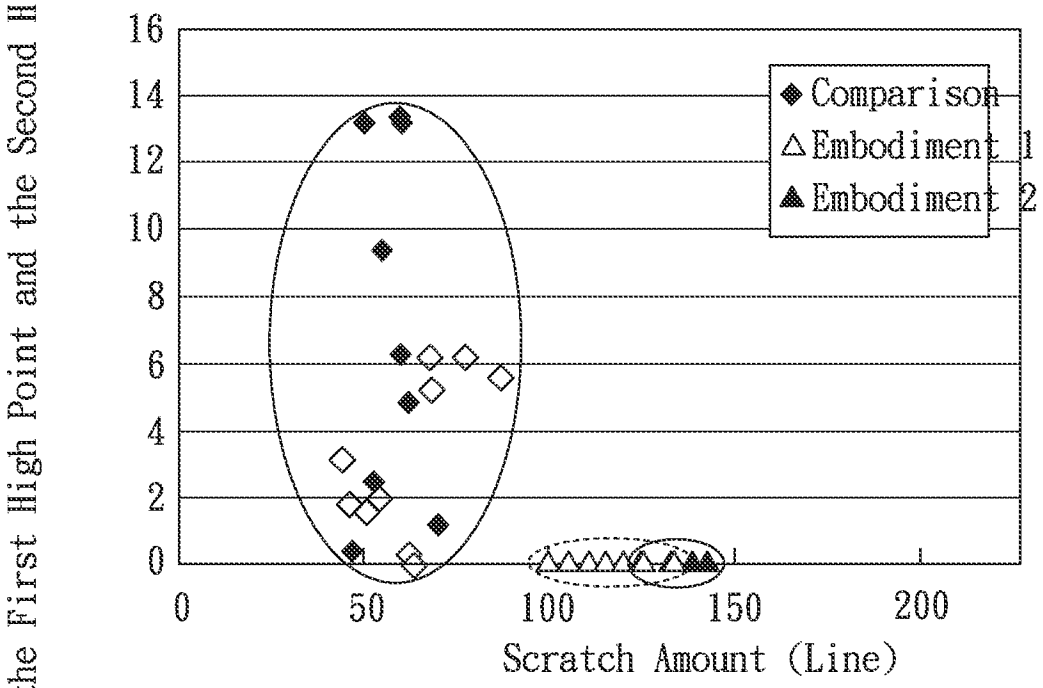

Height Difference of the First High Point and the Second High Point
16
14
12
10
8
6
4
2
0
0
50
100
150
200
Scratch Amount (Line)
Comparison
Embodiment 1
Embodiment 2

FIG. 4

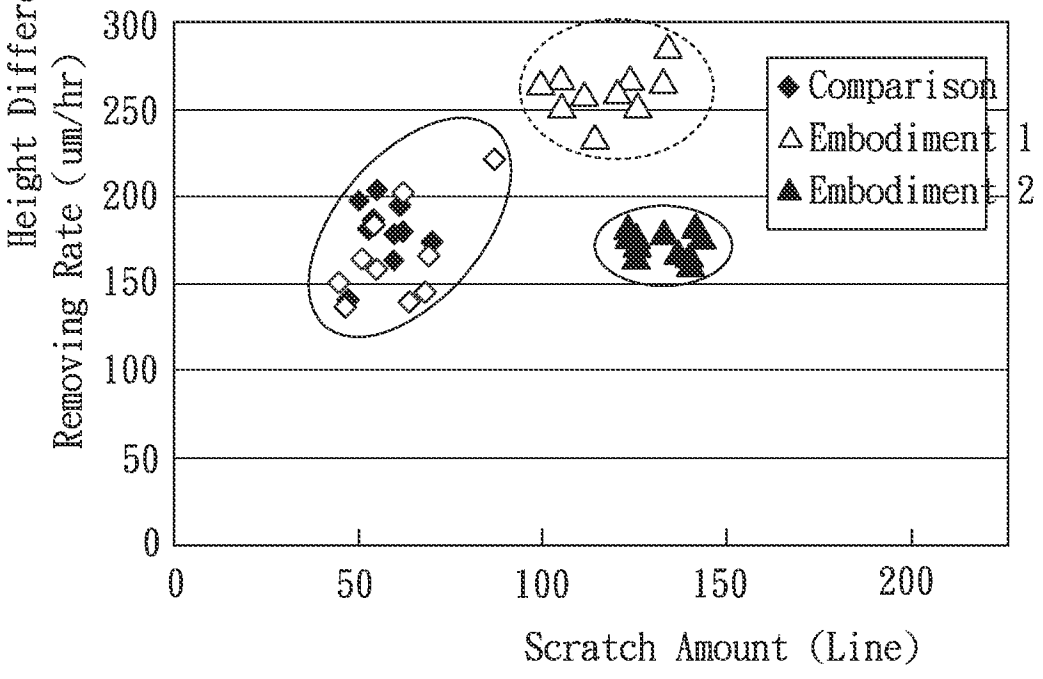

Removing Rate (um/hr)
300
250
200
150
100
50
0
0
50
100
150
200
Scratch Amount (Line)
Comparison
Embodiment 1
Embodiment 2

FIG. 5

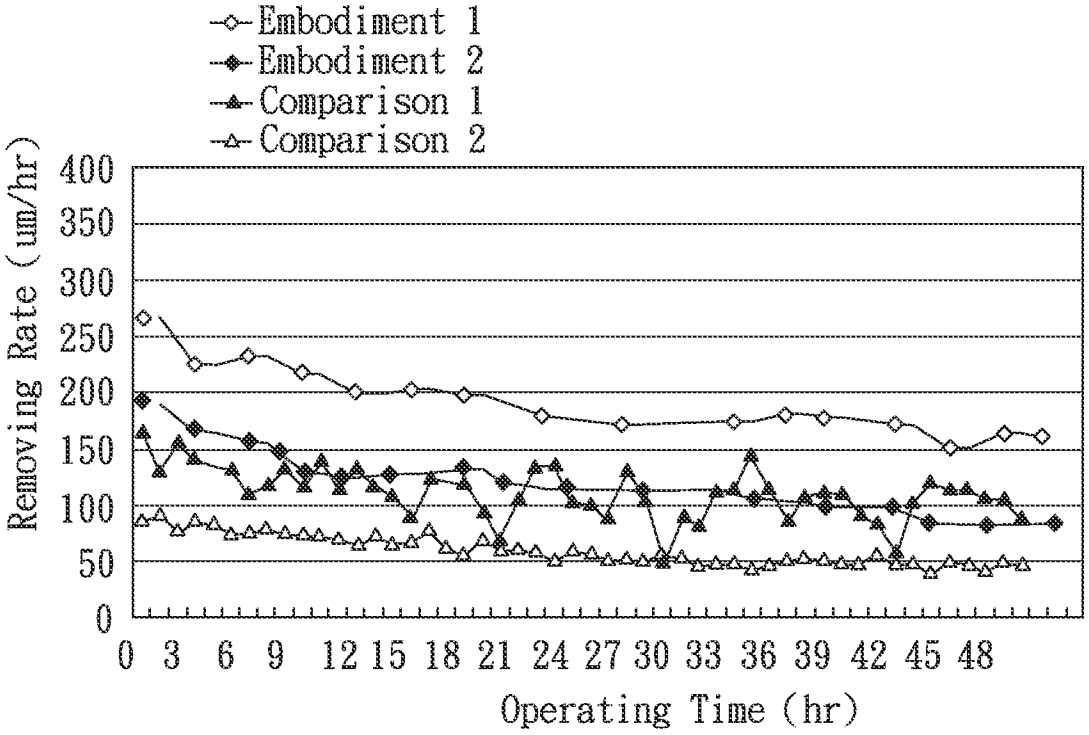

Embodiment 1
Embodiment 2
Comparison 1
Comparison 2
Removing Rate (um/hr)
400
350
300
250
200
150
100
50
0
0 3 6 9 12 15 18 21 24 27 30 33 36 39 42 45 48
Operating Time (hr)

FIG. 6

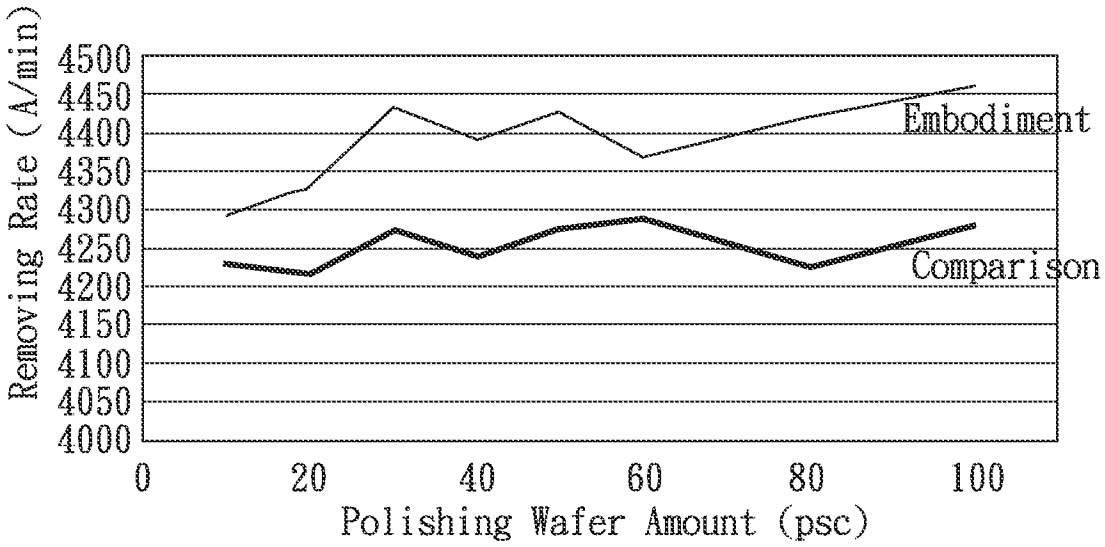

Removing Rate (A/min)
4500
4450
4400
4350
4300
4250
4200
4150
4100
4050
4000
0 20 40 60 80 100
Polishing Wafer Amount (psc)
Embodiment
Comparison

FIG. 7

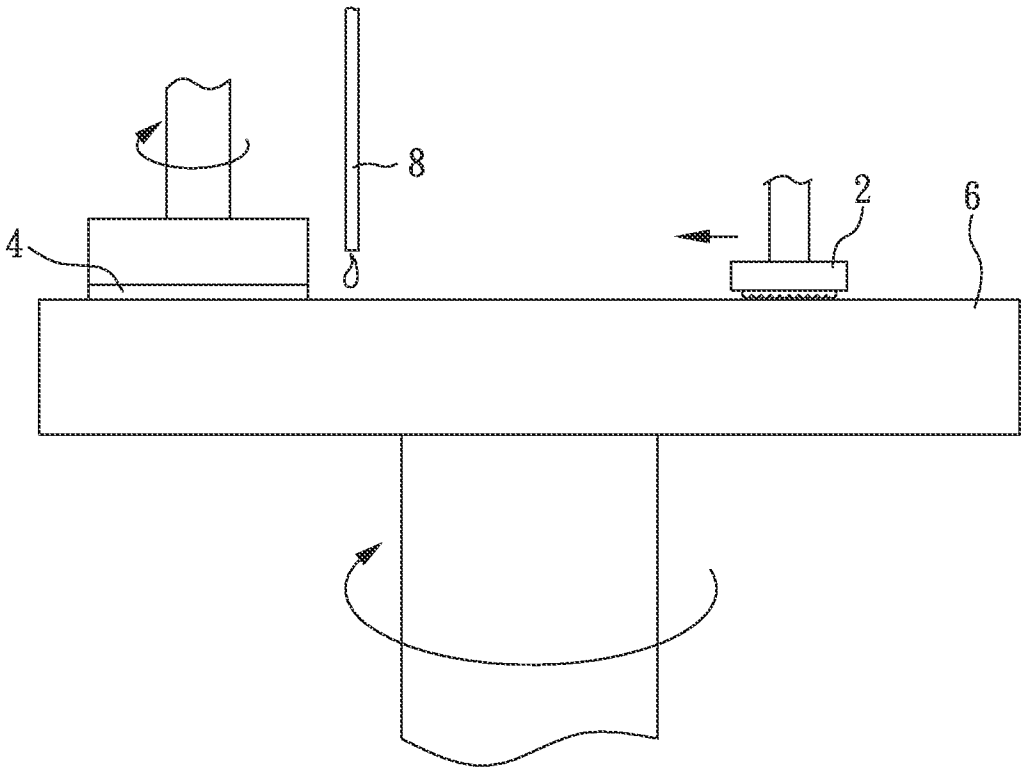
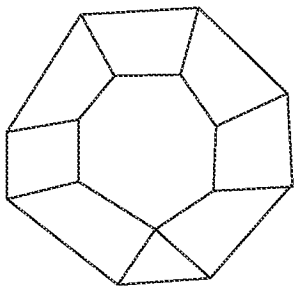


FIG. 8



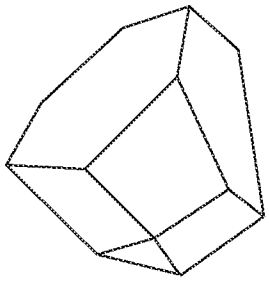


FIG. 9

Electronic Acknowledgement Receipt

EFS ID:	15197487
Application Number:	13797704
International Application Number:	
Confirmation Number:	1089
Title of Invention:	COMPOSITE CONDITIONER 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:	02074-32836.US
Receipt Date:	12-MAR-2013
Filing Date:	
Time Stamp:	20:05:23
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	02074-32836USappdata.pdf	1386204 4e0fdc1ba26a38faac152e98086dab5a5f39abbc	no	7

Warnings:	
Information:	

2		02074-32836USapplication.pdf	169312 2c91c0ac3c652b7df39d914972b5b430f6667ee8	yes	34

Multipart Description/PDF files in .zip description		
Document Description	**Start**	**End**
Specification	1	28
Claims	29	33
Abstract	34	34

Warnings:

Information:

3	Drawings-only black and white line drawings	02074-32836USdrawings.pdf	171510 0d8a3b64f571fae1df6e418b575eae340d520931	no	9

Warnings:

Information:

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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.

PATENT APPLICATION FEE DETERMINATION RECORD

Substitute for Form PTO-875

Application or Docket Number: 13/797,704

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			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))	32 minus 20 =	* 12			OR	x 80 =	960
INDEPENDENT CLAIMS (37 CFR 1.16(h))	3 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	2560

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	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.

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	FIRST NAMED APPLICANT	ATTY. DOCKET NO./TITLE
13/797,704	03/12/2013	Chien-Min Sung	02074-32836.US

CONFIRMATION NO. 1089

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

FORMALITIES LETTER

OC000000060415100

Date Mailed: 04/17/2013

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:

- Additional claim fees of $ **960** as an undiscounted entity, including any required multiple dependent claim fee, are required. Applicant must submit the additional claim fees or cancel the additional claims for which fees are due.
- 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 $ **2700** 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.
- Total additional claim fee(s) for this application is $ **960**
  - $ **960** for **12** total claims over 20.

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

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
13/797,704	03/12/2013	3723	0.00	02074-32836.US	32	3

CONFIRMATION NO. 1089

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

FILING RECEIPT



Date Mailed: 04/17/2013

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 12/726,786 03/18/2010
which is a CIP of 12/255,823 10/22/2008 PAT 8393934
which is a CIP of 12/168,110 07/05/2008 PAT 8398466
which claims benefit of 60/976,198 09/28/2007
and is a CIP of 11/560,817 11/16/2006 PAT 7762872
which is a CIP of 11/357,713 02/17/2006 ABN
which claims benefit of 60/681,798 05/16/2005
This application 13/797,704
is a CIP of 12/628,859 12/01/2009 PAT 7901272
which is a CIP of 11/804,221 05/16/2007 PAT 7651386
which is a CIP of 11/223,786 09/09/2005 ABN
This application 13/797,704
is a CIP of 12/850,747 08/05/2010
which claims benefit of 61/246,816 09/29/2009
This application 13/797,704
is a CIP of 13/034,213 02/24/2011
which claims benefit of 61/333,162 05/10/2010

Foreign Applications (You may be eligible to benefit from the **Patent Prosecution Highway** program at the USPTO. Please see http://www.uspto.gov for more information.)
TAIWAN 101112625 04/10/2012

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: 04/12/2013

The country code and number of your priority application, to be used for filing abroad under the Paris Convention, is **US 13/797,704**

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

Non-Publication Request: No

Early Publication Request: No

Title

COMPOSITE CONDITIONER AND ASSOCIATED METHODS

Preliminary Class

451

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

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 (1-866-999-4158).

LICENSE FOR FOREIGN FILING UNDER

Title 35, United States Code, Section 184

Title 37, Code of Federal Regulations, 5.11 & 5.15

GRANTED

The applicant has been granted a license under 35 U.S.C. 184, if the phrase "IF REQUIRED, FOREIGN FILING LICENSE GRANTED" followed by a date appears on this form. Such licenses are issued in all applications where the conditions for issuance of a license have been met, regardless of whether or not a license may be required as set forth in 37 CFR 5.15. The scope and limitations of this license are set forth in 37 CFR 5.15(a) unless an earlier license has been issued under 37 CFR 5.15(b). The license is subject to revocation upon written notification. The date indicated is the effective date of the license, unless an earlier license of similar scope has been granted under 37 CFR 5.13 or 5.14.

This license is to be retained by the licensee and may be used at any time on or after the effective date thereof unless it is revoked. This license is automatically transferred to any related applications(s) filed under 37 CFR 1.53(d). This license is not retroactive.

The grant of a license does not in any way lessen the responsibility of a licensee for the security of the subject matter as imposed by any Government contract or the provisions of existing laws relating to espionage and the national security or the export of technical data. Licensees should apprise themselves of current regulations especially with respect to certain countries, of other agencies, particularly the Office of Defense Trade Controls, Department of State (with respect to Arms, Munitions and Implements of War (22 CFR 121-128)); the Bureau of Industry and Security, Department of Commerce (15 CFR parts 730-774); the Office of Foreign AssetsControl, Department of Treasury (31 CFR Parts 500+) and the Department of Energy.

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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community; serves as an ombudsman for existing and potential investors; advocates on behalf of U.S. cities, states, and regions competing for global investment; and counsels U.S. economic development organizations on investment attraction best practices. To learn more about why the United States is the best country in the world to develop technology, manufacture products, deliver services, and grow your business, visit http://www.SelectUSA.gov or call +1-202-482-6800.

Electronic Patent Application Fee Transmittal

Application Number:	13797704
Filing Date:	12-Mar-2013
Title of Invention:	COMPOSITE CONDITIONER AND ASSOCIATED METHODS
First Named Inventor/Applicant Name:	Chien-Min Sung
Filer:	David William Osborne/Nicole Solomon
Attorney Docket Number:	02074-32836.US

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:				
Claims in excess of 20	2202	12	40	480
Miscellaneous-Filing:				
Late Filing Fee for Oath or Declaration	2051	1	70	70

Description	Fee Code	Quantity	Amount	Sub-Total in USD($)
Petition:				
Patent-Appeals-and-Interference:				
Post-Allowance-and-Post-Issuance:				
Extension-of-Time:				
Miscellaneous:				
		Total in USD ($)		**1280**

Electronic Acknowledgement Receipt

EFS ID:	16065155
Application Number:	13797704
International Application Number:	
Confirmation Number:	1089
Title of Invention:	COMPOSITE CONDITIONER 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:	02074-32836.US
Receipt Date:	17-JUN-2013
Filing Date:	12-MAR-2013
Time Stamp:	21:28:59
Application Type:	Utility under 35 USC 111(a)

Payment information:

Submitted with Payment	yes
Payment Type	Credit Card
Payment was successfully received in RAM	$1280
RAM confirmation Number	8009
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	38418 901cd1e192e9fc27114f3f9d453ef846cf2d0e0e	no	2

Warnings:	
Information:	
Total Files Size (in bytes):	38418

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

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New International Application Filed with the USPTO as a Receiving Office

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PATENT APPLICATION FEE DETERMINATION RECORD

Substitute for Form PTO-875

Application or Docket Number: 13/797,704

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))	32 minus 20 =	* 12	x 40 =	480	OR		
INDEPENDENT CLAIMS (37 CFR 1.16(h))	3 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	1210		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	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	

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www.uspto.gov

APPLICATION NUMBER	FILING or 371(c) DATE	GRP ART UNIT	FIL FEE REC'D	ATTY.DOCKET.NO	TOT CLAIMS	IND CLAIMS
13/797,704	03/12/2013	3723	1280	02074-32836.US	32	3

CONFIRMATION NO. 1089

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

UPDATED FILING RECEIPT

OC000000066210663

Date Mailed: 01/24/2014

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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 12/726,786 03/18/2010 PAT 8622787
which is a CIP of 12/255,823 10/22/2008 PAT 8393934
which is a CIP of 12/168,110 07/05/2008 PAT 8398466
which claims benefit of 60/976,198 09/28/2007
and is a CIP of 11/560,817 11/16/2006 PAT 7762872
which is a CIP of 11/357,713 02/17/2006 ABN
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is a CIP of 12/850,747 08/05/2010
which claims benefit of 61/246,816 09/29/2009
This application 13/797,704
is a CIP of 13/034,213 02/24/2011 ABN
which claims benefit of 61/333,162 05/10/2010

Foreign Applications (You may be eligible to benefit from the **Patent Prosecution Highway** program at the USPTO. Please see http://www.uspto.gov for more information.)
TAIWAN 101112625 04/10/2012

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: 04/12/2013
The country code and number of your priority application, to be used for filing abroad under the Paris Convention, is **US 13/797,704**
Projected Publication Date: 05/01/2014
Non-Publication Request: No
Early Publication Request: No
**** SMALL ENTITY ****
Title

COMPOSITE CONDITIONER AND ASSOCIATED METHODS

Preliminary Class

451

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

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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).

LICENSE FOR FOREIGN FILING UNDER

Title 35, United States Code, Section 184

Title 37, Code of Federal Regulations, 5.11 & 5.15

GRANTED

The applicant has been granted a license under 35 U.S.C. 184, if the phrase "IF REQUIRED, FOREIGN FILING LICENSE GRANTED" followed by a date appears on this form. Such licenses are issued in all applications where the conditions for issuance of a license have been met, regardless of whether or not a license may be required as set forth in 37 CFR 5.15. The scope and limitations of this license are set forth in 37 CFR 5.15(a) unless an earlier license has been issued under 37 CFR 5.15(b). The license is subject to revocation upon written notification. The date indicated is the effective date of the license, unless an earlier license of similar scope has been granted under 37 CFR 5.13 or 5.14.

This license is to be retained by the licensee and may be used at any time on or after the effective date thereof unless it is revoked. This license is automatically transferred to any related applications(s) filed under 37 CFR 1.53(d). This license is not retroactive.

The grant of a license does not in any way lessen the responsibility of a licensee for the security of the subject matter as imposed by any Government contract or the provisions of existing laws relating to espionage and the national security or the export of technical data. Licensees should apprise themselves of current regulations especially with respect to certain countries, of other agencies, particularly the Office of Defense Trade Controls, Department of State (with respect to Arms, Munitions and Implements of War (22 CFR 121-128)); the Bureau of Industry and Security, Department of Commerce (15 CFR parts 730-774); the Office of Foreign AssetsControl, Department of Treasury (31 CFR Parts 500+) and the Department of Energy.

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).

SelectUSA

The United States represents the largest, most dynamic marketplace in the world and is an unparalleled location for business investment, innovation, and commercialization of new technologies. The U.S. offers tremendous resources and advantages for those who invest and manufacture goods here. Through SelectUSA, our nation works to promote and facilitate business investment. SelectUSA provides information assistance to the international investor

community; serves as an ombudsman for existing and potential investors; advocates on behalf of U.S. cities, states, and regions competing for global investment; and counsels U.S. economic development organizations on investment attraction best practices. To learn more about why the United States is the best country in the world to develop technology, manufacture products, deliver services, and grow your business, visit http://www.SelectUSA.gov or call +1-202-482-6800.

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	FIRST NAMED APPLICANT	ATTY. DOCKET NO./TITLE
13/797,704	03/12/2013	Chien-Min Sung	02074-32836.US

CONFIRMATION NO. 1089

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

NOTICE

OC000000066210664

Date Mailed: 01/24/2014

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):
  Chien-Min Sung

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
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APPLICATION NUMBER	FILING OR 371(C) DATE	FIRST NAMED APPLICANT	ATTY. DOCKET NO./TITLE
13/797,704	03/12/2013	Chien-Min Sung	02074-32836.US

CONFIRMATION NO. 1089

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

PUBLICATION NOTICE

OC00000068171047

Title:COMPOSITE CONDITIONER AND ASSOCIATED METHODS

Publication No.US-2014-0120724-A1
Publication Date:05/01/2014

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

UNITED STATES PATENT AND TRADEMARK OFFICE

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

APPLICATION NO.	FILING DATE	FIRST NAMED INVENTOR	ATTORNEY DOCKET NO.	CONFIRMATION NO.
13/797,704	03/12/2013	Chien-Min Sung	02074-32836.US	1089

20551 7590 07/11/2014

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

EXAMINER	
RACHUBA, MAURINA T	

ART UNIT	PAPER NUMBER
3727	

NOTIFICATION DATE	DELIVERY MODE
07/11/2014	ELECTRONIC

Please find below and/or attached an Office communication concerning this application or proceeding.

The time period for reply, if any, is set in the attached communication.

Notice of the Office communication was sent electronically on above-indicated "Notification Date" to the following e-mail address(es):

patentdocket@tnw.com
rich@tnw.com
laron.lemon@tnw.com

Office Action Summary	Application No. 13/797,704	Applicant(s) SUNG, CHIEN-MIN	
	Examiner MAURINA RACHUBA	Art Unit 3727	AIA (First Inventor to File) Status No

-- The MAILING DATE of this communication appears on the cover sheet with the correspondence address --

Period for Reply

A SHORTENED STATUTORY PERIOD FOR REPLY IS SET TO EXPIRE 2 MONTHS FROM THE MAILING DATE OF THIS COMMUNICATION.

- Extensions of time may be available under the provisions of 37 CFR 1.136(a). In no event, however, may a reply be timely filed after SIX (6) MONTHS from the mailing date of this communication.
- If NO period for reply is specified above, the maximum statutory period will apply and will expire SIX (6) MONTHS from the mailing date of this communication.
- Failure to reply within the set or extended period for reply will, by statute, cause the application to become ABANDONED (35 U.S.C. § 133). Any reply received by the Office later than three months after the mailing date of this communication, even if timely filed, may reduce any earned patent term adjustment. See 37 CFR 1.704(b).

Status

1)☐ Responsive to communication(s) filed on _____.

☐ A declaration(s)/affidavit(s) under **37 CFR 1.130(b)** was/were filed on _____.

2a)☐ This action is **FINAL**. 2b)☐ This action is non-final.

3)☐ An election was made by the applicant in response to a restriction requirement set forth during the interview on _____; the restriction requirement and election have been incorporated into this action.

4)☐ Since this application is in condition for allowance except for formal matters, prosecution as to the merits is closed in accordance with the practice under *Ex parte Quayle*, 1935 C.D. 11, 453 O.G. 213.

Disposition of Claims*

5)☒ Claim(s) *1-32* is/are pending in the application.

5a) Of the above claim(s) _____ is/are withdrawn from consideration.

6)☐ Claim(s) _____ is/are allowed.

7)☐ Claim(s) _____ is/are rejected.

8)☐ Claim(s) _____ is/are objected to.

9)☒ Claim(s) *1-32* are subject to restriction and/or election requirement.

* If any claims have been determined allowable, you may be eligible to benefit from the **Patent Prosecution Highway** program at a participating intellectual property office for the corresponding application. For more information, please see http://www.uspto.gov/patents/init_events/pph/index.jsp or send an inquiry to PPHfeedback@uspto.gov.

Application Papers

10)☐ The specification is objected to by the Examiner.

11)☐ The drawing(s) filed on _____ is/are: a)☐ accepted or b)☐ objected to by the Examiner.

Applicant may not request that any objection to the drawing(s) be held in abeyance. See 37 CFR 1.85(a).

Replacement drawing sheet(s) including the correction is required if the drawing(s) is objected to. See 37 CFR 1.121(d).

Priority under 35 U.S.C. § 119

12)☐ Acknowledgment is made of a claim for foreign priority under 35 U.S.C. § 119(a)-(d) or (f).

Certified copies:

a)☐ All b)☐ Some** c)☐ None of the:

1.☐ Certified copies of the priority documents have been received.

2.☐ Certified copies of the priority documents have been received in Application No. _____.

3.☐ Copies of the certified copies of the priority documents have been received in this National Stage application from the International Bureau (PCT Rule 17.2(a)).

** See the attached detailed Office action for a list of the certified copies not received.

Attachment(s)

1) ☐ Notice of References Cited (PTO-892)

2) ☐ Information Disclosure Statement(s) (PTO/SB/08a and/or PTO/SB/08b) Paper No(s)/Mail Date _____.

3) ☐ Interview Summary (PTO-413) Paper No(s)/Mail Date. _____ .

4) ☐ Other: _____.

1. The present application is being examined under the pre-AIA first to invent provisions.

Election/Restriction

2. Restriction to one of the following inventions is required under 35 U.S.C. 121:

I. Claims 1-23 and 28-32, drawn to a product and method of use of the product, classified in 451/56.

II. Claims 24-27, drawn to a method of making a product, classified in 51/297.

3. The inventions are distinct, each from the other because of the following reasons:

4. Inventions I and II are related as process of making and product made. The inventions are distinct if either or both of the following can be shown: (1) that the process as claimed can be used to make another and materially different product or (2) that the product as claimed can be made by another and materially different process (MPEP § 806.05(f)). In the instant case the product can be made by another materially different process, such as first forming the polishing units, then adhering them to the base plate.

5. Restriction for examination purposes as indicated is proper because all these inventions listed in this action are independent or distinct for the reasons given above and there would be a serious search and/or examination burden if restriction were not required because one or more of the following reasons apply:

The search for the method of making the product is not required for the product, as evinced by the classification above.

Applicant is advised that the reply to this requirement to be complete must include (i) an election of an invention to be examined even though the requirement may be traversed (37 CFR 1.143) **and (ii) identification of the claims encompassing the elected invention**.

The election of an invention may be made with or without traverse. To reserve a right to petition, the election must be made with traverse. If the reply does not distinctly and specifically point out supposed errors in the restriction requirement, the election shall be treated as an election without traverse. Traversal must be presented at the time of election in order to be considered timely. Failure to timely traverse the requirement will result in the loss of right to petition under 37 CFR 1.144. If claims are added after the election, applicant must indicate which of these claims are readable upon the elected invention.

Should applicant traverse on the ground that the inventions are not patentably distinct, applicant should submit evidence or identify such evidence now of record showing the inventions to be obvious variants or clearly admit on the record that this is the case. In either instance, if the examiner finds one of the inventions unpatentable over the prior art, the evidence or admission may be used in a rejection under 35 U.S.C. 103 or pre-AIA 35 U.S.C. 103(a) of the other invention.

6. The examiner has required restriction between product or apparatus claims and process claims. Where applicant elects claims directed to the product/apparatus, and all product/apparatus claims are subsequently found allowable, withdrawn process claims that include all the limitations of the allowable product/apparatus claims should be

considered for rejoinder. All claims directed to a nonelected process invention must include all the limitations of an allowable product/apparatus claim for that process invention to be rejoined.

In the event of rejoinder, the requirement for restriction between the product/apparatus claims and the rejoined process claims will be withdrawn, and the rejoined process claims will be fully examined for patentability in accordance with 37 CFR 1.104. Thus, to be allowable, the rejoined claims must meet all criteria for patentability including the requirements of 35 U.S.C. 101, 102, 103 and 112. Until all claims to the elected product/apparatus are found allowable, an otherwise proper restriction requirement between product/apparatus claims and process claims may be maintained. Withdrawn process claims that are not commensurate in scope with an allowable product/apparatus claim will not be rejoined. See MPEP § 821.04. Additionally, in order for rejoinder to occur, applicant is advised that the process claims should be amended during prosecution to require the limitations of the product/apparatus claims. **Failure to do so may result in no rejoinder.** Further, note that the prohibition against double patenting rejections of 35 U.S.C. 121 does not apply where the restriction requirement is withdrawn by the examiner before the patent issues. See MPEP § 804.01.

Any inquiry concerning this communication or earlier communications from the examiner should be directed to MAURINA RACHUBA whose telephone number is (571)272-4493. The examiner can normally be reached on Monday-Thursday, 5:30AM-4:00PM.

If attempts to reach the examiner by telephone are unsuccessful, the examiner's supervisor, Monica Carter can be reached on 571 272 4475. The fax phone number for the organization where this application or proceeding is assigned is 571-273-8300.

Information regarding the status of an application may be obtained from the Patent Application Information Retrieval (PAIR) system. Status information for published applications may be obtained from either Private PAIR or Public PAIR. Status information for unpublished applications is available through Private PAIR only. For more information about the PAIR system, see http://pair-direct.uspto.gov. Should you have questions on access to the Private PAIR system, contact the Electronic Business Center (EBC) at 866-217-9197 (toll-free). If you would like assistance from a USPTO Customer Service Representative or access to the automated information system, call 800-786-9199 (IN USA OR CANADA) or 571-272-1000.

/MAURINA RACHUBA/
Primary Examiner, Art Unit 3727

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

ART UNIT: 3727 EXAMINER: Maurina Rachuba FIRST NAMED INVENTOR: Chien-Min Sung SERIAL NO.: 13/797,704 FILED: 3/12/2013 CONF. NO.: 1089 FOR: COMPOSITE CONDITIONER AND ASSOCIATED METHODS DOCKET NO.: 02074-32836.US	**CERTIFICATE OF MAILING UNDER 37 C.F.R. § 1.8** DATE OF DEPOSIT: **October 14, 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. /Nicole Solomon/ Nicole Solomon

ELECTION AND RESPONSE UNDER 37 C.F.R. § 1.121

Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313

Dear Sir:

In response to the communication mailed July 11, 2014, please reconsider this application in view of the election and remarks provided below. A petition for a 1 mo. extension of time is enclosed herewith.

ELECTION

Applicants elect without traverse, Group I as outlined in the office action mailed July 11, 2014, containing Claims 1-23 and 28-32 for further examination in the present application.

REMARKS

Applicants thank the Examiner for consideration of the subject patent application. In the office action mailed July 11, 2014, Claims 1-32 were pending, and made subject to restriction requirement under 35 U.S.C. § 121. Particularly, the Examiner indicated the existence of two patentably distinct inventions in the claims as follows: 1) Group I constituting Claims 1-23 and 28-32 drawn to a product and method of use of the product, classified in class 451/56; and 2) Group II constituting Claims 24-27, drawn to methods of making a product classified in class 51/297.

Therefore, pursuant to the present election of Group I, Claims 1-23 and 28-32 remain pending for consideration in the present application, and Claims 24-27 are withdrawn.

CONCLUSION

If any impediment remains to examination after consideration of the above-recited remarks, which could be removed during a telephone interview, the Examiner is invited to telephone the undersigned attorney, at (801) 566-6633 so that such issues may be resolved as expeditiously as possible.

The Commissioner is hereby authorized to charge any additional fees associated with this communication or credit any overpayment to Deposit Account No. 20-0100.

DATED this 14th day of October, 2014.

Respectfully submitted,

THORPE, NORTH & WESTERN, LLP

/David W. Osborne/

David W. Osborne
Reg. No. 44,989
8180 South 700 East, Suite 350
Sandy, UT 84070
Telephone: (801) 566-6633
Facsimile: (801) 566-0750

Electronic Patent Application Fee Transmittal

Application Number:	13797704
Filing Date:	12-Mar-2013
Title of Invention:	COMPOSITE CONDITIONER AND ASSOCIATED METHODS
First Named Inventor/Applicant Name:	Chien-Min Sung
Filer:	David William Osborne/Nicole Solomon
Attorney Docket Number:	02074-32836.US

Filed as Large Entity

Utility under 35 USC 111(a) Filing Fees

Description	Fee Code	Quantity	Amount	Sub-Total in USD($)
Basic Filing:				
Pages:				
Claims:				
Miscellaneous-Filing:				
Petition:				
Patent-Appeals-and-Interference:				
Post-Allowance-and-Post-Issuance:				
Extension-of-Time:				
Extension - 1 month with $0 paid	1251	1	200	200

Description	Fee Code	Quantity	Amount	Sub-Total in USD($)
Miscellaneous:				
			Total in USD ($)	**200**

Electronic Acknowledgement Receipt

EFS ID:	20413030
Application Number:	13797704
International Application Number:	
Confirmation Number:	1089
Title of Invention:	COMPOSITE CONDITIONER 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:	02074-32836.US
Receipt Date:	14-OCT-2014
Filing Date:	12-MAR-2013
Time Stamp:	17:02:52
Application Type:	Utility under 35 USC 111(a)

Payment information:

Submitted with Payment	yes
Payment Type	Credit Card
Payment was successfully received in RAM	$200
RAM confirmation Number	4062
Deposit Account	
Authorized User	

File Listing:

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

1	Extension of Time	02074-32836-US-Extension.pdf	59039 888c537c970c0fa214afb7d616329fd95519c19b	no	1
Warnings:					
Information:					
2		02074-32836-US-Response.pdf	64261 d2541f987259ecc2f0fc19f0ffd7b59d0405bbaa	yes	4

	Multipart Description/PDF files in .zip description		
	Document Description	**Start**	**End**
	Amendment/Argument after Patent Board Decision	1	1
	Response to Election / Restriction Filed	2	2
	Applicant Arguments/Remarks Made in an Amendment	3	4

Warnings:					
Information:					
3	Fee Worksheet (SB06)	fee-info.pdf	30604 346bbb957cb19abdeacc7b32f1ec18acfe472576	no	2
Warnings:					
Information:					
		Total Files Size (in bytes):	153904		

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.

<u>New Applications Under 35 U.S.C. 111</u>
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.

<u>National Stage of an International Application under 35 U.S.C. 371</u>
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.

<u>New International Application Filed with the USPTO as a Receiving Office</u>
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.

PTO/SB/22 (03-13)
Approved for use through 3/31/2013. OMB 0651-0031
U.S. Patent and Trademark Office; U.S. DEPARTMENT OF COMMERCE
Under the Paperwork Reduction Act of 1995, no persons are required to respond to a collection of information unless it displays a valid OMB control number.

PETITION FOR EXTENSION OF TIME UNDER 37 CFR 1.136(a)

Docket Number (Optional): 02074-32836.US

Application Number	Filed
13/797,704	3/12/2013

For: Composite Conditioner and Associated Methods

Art Unit	Examiner
3727	Maurina T. Rachuba

This is a request under the provisions of 37 CFR 1.136(a) to extend the period for filing a reply in the above-identified application.

The requested extension and fee are as follows (check time period desired and enter the appropriate fee below):

		Fee	Small Entity Fee	Micro Entity Fee	
☑	One month (37 CFR 1.17(a)(1))	$200	$100	$50	$ 200
☐	Two months (37 CFR 1.17(a)(2))	$600	$300	$150	$
☐	Three months (37 CFR 1.17(a)(3))	$1,400	$700	$350	$
☐	Four months (37 CFR 1.17(a)(4))	$2,200	$1,100	$550	$
☐	Five months (37 CFR 1.17(a)(5))	$3,000	$1,500	$750	$

☐ Applicant asserts small entity status. See 37 CFR 1.27.

☐ Applicant certifies micro entity status. See 37 CFR 1.29.
Form PTO/SB/15A or B or equivalent must either be enclosed or have been submitted previously.

☐ A check in the amount of the fee is enclosed.

☑ Payment by credit card. Form PTO-2038 is attached.

☐ The Director has already been authorized to charge fees in this application to a Deposit Account.

☑ The Director is hereby authorized to charge any fees which may be required, or credit any overpayment, to Deposit Account Number 20-0100.

☑ Payment made via EFS-Web.

WARNING: Information on this form may become public. Credit card information should not be included on this form. Provide credit card information and authorization on PTO-2038.

I am the

☐ applicant/inventor.

☐ assignee of record of the entire interest. See 37 CFR 3.71. 37 CFR 3.73(b) statement is enclosed (Form PTO/SB/96).

☑ attorney or agent of record. Registration number 44989.

☐ attorney or agent acting under 37 CFR 1.34. Registration number _____.

/David W. Osborne/	October 14, 2014
Signature	Date
David W. Osborne	801-566-6633
Typed or printed name	Telephone Number

NOTE: This form must be signed in accordance with 37 CFR 1.33. See 37 CFR 1.4 for signature requirements and certifications. Submit multiple forms if more than one signature is required, see below*.

☐ * Total of _____ forms are submitted.

This collection of information is required by 37 CFR 1.136(a). 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 1.11 and 1.14. This collection is estimated to take 6 minutes 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, 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.**

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

UNITED STATES PATENT AND TRADEMARK OFFICE

UNITED STATES DEPARTMENT OF COMMERCE
United States Patent and Trademark Office
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Alexandria, Virginia 22313-1450
www.uspto.gov

APPLICATION NO.	FILING DATE	FIRST NAMED INVENTOR	ATTORNEY DOCKET NO.	CONFIRMATION NO.
13/797,704	03/12/2013	Chien-Min Sung	02074-32836.US	1089

20551 7590 01/12/2015

THORPE NORTH & WESTERN, LLP.
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EXAMINER
RACHUBA, MAURINA T

ART UNIT	PAPER NUMBER
3727	

NOTIFICATION DATE	DELIVERY MODE
01/12/2015	ELECTRONIC

Please find below and/or attached an Office communication concerning this application or proceeding.

The time period for reply, if any, is set in the attached communication.

Notice of the Office communication was sent electronically on above-indicated "Notification Date" to the following e-mail address(es):

patentdocket@tnw.com
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laron.lemon@tnw.com

Office Action Summary	Application No. 13/797,704	Applicant(s) SUNG, CHIEN-MIN	
	Examiner MAURINA RACHUBA	Art Unit 3727	AIA (First Inventor to File) Status No

-- The MAILING DATE of this communication appears on the cover sheet with the correspondence address --

Period for Reply

A SHORTENED STATUTORY PERIOD FOR REPLY IS SET TO EXPIRE *3* MONTHS FROM THE MAILING DATE OF THIS COMMUNICATION.

- Extensions of time may be available under the provisions of 37 CFR 1.136(a). In no event, however, may a reply be timely filed after SIX (6) MONTHS from the mailing date of this communication.
- If NO period for reply is specified above, the maximum statutory period will apply and will expire SIX (6) MONTHS from the mailing date of this communication.
- Failure to reply within the set or extended period for reply will, by statute, cause the application to become ABANDONED (35 U.S.C. § 133). Any reply received by the Office later than three months after the mailing date of this communication, even if timely filed, may reduce any earned patent term adjustment. See 37 CFR 1.704(b).

Status

1)☒ Responsive to communication(s) filed on *14 October 2014*.

☐ A declaration(s)/affidavit(s) under **37 CFR 1.130(b)** was/were filed on _____.

2a)☐ This action is **FINAL**. 2b)☒ This action is non-final.

3)☐ An election was made by the applicant in response to a restriction requirement set forth during the interview on _____; the restriction requirement and election have been incorporated into this action.

4)☐ Since this application is in condition for allowance except for formal matters, prosecution as to the merits is closed in accordance with the practice under *Ex parte Quayle*, 1935 C.D. 11, 453 O.G. 213.

Disposition of Claims*

5)☒ Claim(s) *1-32* is/are pending in the application.

5a) Of the above claim(s) *24-27* is/are withdrawn from consideration.

6)☐ Claim(s) _____ is/are allowed.

7)☒ Claim(s) *1-23 and 28-32* is/are rejected.

8)☐ Claim(s) _____ is/are objected to.

9)☐ Claim(s) _____ are subject to restriction and/or election requirement.

* If any claims have been determined allowable, you may be eligible to benefit from the **Patent Prosecution Highway** program at a participating intellectual property office for the corresponding application. For more information, please see http://www.uspto.gov/patents/init_events/pph/index.jsp or send an inquiry to PPHfeedback@uspto.gov.

Application Papers

10)☐ The specification is objected to by the Examiner.

11)☒ The drawing(s) filed on *12 March 2013* is/are: a)☒ accepted or b)☐ objected to by the Examiner.

Applicant may not request that any objection to the drawing(s) be held in abeyance. See 37 CFR 1.85(a).

Replacement drawing sheet(s) including the correction is required if the drawing(s) is objected to. See 37 CFR 1.121(d).

Priority under 35 U.S.C. § 119

12)☒ Acknowledgment is made of a claim for foreign priority under 35 U.S.C. § 119(a)-(d) or (f).

Certified copies:

a)☐ All b)☐ Some** c)☒ None of the:

1.☒ Certified copies of the priority documents have been received.

2.☐ Certified copies of the priority documents have been received in Application No. _____.

3.☐ Copies of the certified copies of the priority documents have been received in this National Stage application from the International Bureau (PCT Rule 17.2(a)).

** See the attached detailed Office action for a list of the certified copies not received.

Attachment(s)

1) ☒ Notice of References Cited (PTO-892)

2) ☐ Information Disclosure Statement(s) (PTO/SB/08a and/or PTO/SB/08b) Paper No(s)/Mail Date _____.

3) ☐ Interview Summary (PTO-413) Paper No(s)/Mail Date. _____ .

4) ☐ Other: _____.

1. The present application is being examined under the pre-AIA first to invent provisions.

DETAILED ACTION

Election/Restrictions

2. Applicant's election without traverse of group I, claims 1-23 and 28-32 in the reply filed on 12 March 2014 is acknowledged.

3. Claims 24-27 are withdrawn from further consideration pursuant to 37 CFR 1.142(b) as being drawn to a nonelected invention, there being no allowable generic or linking claim.

Claim Rejections - 35 USC § 112

4. The following is a quotation of the first paragraph of 35 U.S.C. 112(a):

> (a) IN GENERAL.—The specification shall contain a written description of the invention, and of the manner and process of making and using it, in such full, clear, concise, and exact terms as to enable any person skilled in the art to which it pertains, or with which it is most nearly connected, to make and use the same, and shall set forth the best mode contemplated by the inventor or joint inventor of carrying out the invention.

The following is a quotation of the first paragraph of pre-AIA 35 U.S.C. 112:

> The specification shall contain a written description of the invention, and of the manner and process of making and using it, in such full, clear, concise, and exact terms as to enable any person skilled in the art to which it pertains, or with which it is most nearly connected, to make and use the same, and shall set forth the best mode contemplated by the inventor of carrying out his invention.

5. Claims 28-32 are rejected under 35 U.S.C. 112(a) or 35 U.S.C. 112 (pre-AIA), first paragraph, as failing to comply with the written description requirement. The claim(s) contains subject matter which was not described in the specification in such a way as to reasonably convey to one skilled in the relevant art that the inventor or a joint

inventor, or for pre-AIA the inventor(s), at the time the application was filed, had possession of the claimed invention. Applicant, in for example pages 20-22 of the specification, discloses that the sizes of wafers are in the future growing in diameter, and shrinking in thickness, and that the conditioning tool disclosed is capable of conditioning a polishing pad to work on these future (but nonexistent) wafers. It is not clear if the conditioning pad is capable of conditioning the polishing pad to process these wafers, as they do not yet exist. As such, the examiner believes that the method as disclosed is not capable of being practiced by one of ordinary skill.

6. The following is a quotation of 35 U.S.C. 112(b):

> (b) CONCLUSION.—The specification shall conclude with one or more claims particularly pointing out and distinctly claiming the subject matter which the inventor or a joint inventor regards as the invention.

The following is a quotation of 35 U.S.C. 112 (pre-AIA), second paragraph:

> The specification shall conclude with one or more claims particularly pointing out and distinctly claiming the subject matter which the applicant regards as his invention.

7. Claims 29-32 are rejected under 35 U.S.C. 112(b) or 35 U.S.C. 112 (pre-AIA), second paragraph, as being indefinite for failing to particularly point out and distinctly claim the subject matter which the inventor or a joint inventor, or for pre-AIA the applicant regards as the invention. As the dimensions of the wafer as claimed are considered by applicant to be the dimensions of future wafers, the scope of the claims cannot be readily determined. Applicant is advised to amend these claims to reflect the current sizes of the workpiece, as supported by the specification.

Claim Rejections - 35 USC § 103

8. In the event the determination of the status of the application as subject to AIA 35 U.S.C. 102 and 103 (or as subject to pre-AIA 35 U.S.C. 102 and 103) is incorrect, any

correction of the statutory basis for the rejection will not be considered a new ground of rejection if the prior art relied upon, and the rationale supporting the rejection, would be the same under either status.

9. The following is a quotation of pre-AIA 35 U.S.C. 103(a) which forms the basis for all obviousness rejections set forth in this Office action:

> (a) A patent may not be obtained though the invention is not identically disclosed or described as set forth in section 102 of this title, if the differences between the subject matter sought to be patented and the prior art are such that the subject matter as a whole would have been obvious at the time the invention was made to a person having ordinary skill in the art to which said subject matter pertains. Patentability shall not be negatived by the manner in which the invention was made.

10. Claims 1-23 and 28-32 are rejected under pre-AIA 35 U.S.C. 103(a) as being unpatentable over Sung et al, 2002/0173234 in view of Romero, 6,371,842. '234, discloses a composite conditioner comprising: a base plate **40**; a plurality of polishing tips **50** secured in a binding layer **90** on the base plate, wherein a height difference between a first highest polishing tip and a second highest polishing tip is **less than** or equal to about 10 μm, a height difference between the first highest polishing tip and a tenth highest polishing tip is **less than** or equal to about 20 μm, and a height difference between the first highest polishing tip and a 100th highest polishing tip is **less than** or equal to about 40 μm, [0071]and the first highest polishing tip protrudes from the binding layer to a height of greater than or equal to about 50 μm [0071], (as the polishing tips are all approximately the same distance from the binding layer, then the relative heights of the tips, relative to the other tips, will be less than the claimed ranges); at least 1% of the plurality of polishing tips actively condition a CMP pad during a conditioning process (as the tips are all the same distance from the binding layer, the percentage of tips

conditioning the pad falls within the claimed range); the plurality of polishing tips include diamond, and the diamond is deposition diamond, polycrystalline diamond, or a combination thereof [0061]. '234 further teaches placing individual abrasive particles such that the plurality of polishing tips are secured with the binding layer; the binding layer is a **solder (brazing) layer** [0062]; a metal cover layer [0063] that covers the binding layer, the metal cover layer includes at least one material selected from the group consisting of **Ni,** Cr, Pd, Co, Pt, Au, Ti, Cu, W, and alloys thereof [0063]; a protective layer covering the metal cover layer, wherein the protective layer includes a material selected from the group consisting of Pd, Pt, silicon carbide, aluminum nitride, alumina, zirconia, diamond-like carbon (DLC) (which includes diamond, as diamond is carbon, and is "diamond-like", and combinations thereof [0064]; binding layer is a solder layer comprising 1 wt% or more of a first material selected from the group consisting of , B, 30 P, Ti, or an alloy thereof [0074]; the polishing tips have a particle size of from about 100 μm to about 500 μm [0068]; wherein the polishing tips are arranged in a predetermined pattern on the polishing unit substrate [0078], '234 further discloses a process of chemical-mechanical planarization (CMP), including the following steps: polishing a wafer against a rotating polishing pad; and applying the disclosed against the polishing pad to remove debris and condition the pad; the wafer has a diameter; a surface of the wafer has an IC critical dimension.

11. '234 does not disclose that the polishing tips are arranged as a plurality of polishing units secured to the base plate by an adhesive layer; the percent area of surface coverage of the substrate by the units; the number of tips conditioning the pad;

the crystal surface coverage of the tips; the polishing units are disc-shaped, radial-shaped, or polygon-shaped; the polishing units are disc-shaped having a diameter range; diameter of the substrate **40**; wherein the adhesive layer is an organic adhesive; the spacing of the polishing units; the thickness of the adhesive layer between the substrate and the units. '842, figures 1-4, teaches a conditioning device having a substrate and a plurality of polishing units each having a plurality of polishing tips adhered to the substrate, the units having various forms including disc- or radially shaped (column 3, lines 18-33) having a diameter of 10-50 mm; the units adhered to a substrate by organic adhesive, (column 5, lines 1-60) having a thickness. Regarding the range of surface coverage by the units, the number of tips conditioning the pad, the crystal surface coverage, and the thickness of the adhesive, it would have been obvious to one having ordinary skill in the art at the time the invention was made to have made the various components with the claimed size and amount ranges, for the predictable result of forming a conditioning tool, since it has been held that where the general conditions of a claim are disclosed in the prior art, discovering the optimum or workable ranges involves only routine skill in the art. Regarding the diameter and IC dimension of the workpiece, applicant appears to be claiming dimensions not of currently available workpieces, but of possible future workpieces, see for example pages 20-22 of applicant's specification. As such, it is the examiner's position that one of ordinary skill in the art would have considered it obvious to fashion a conditioning tool that conditions a polishing pad such that it is capable of working not just on present workpieces, but on possible future workpieces of the claimed sizes as well.

Conclusion

12. The prior art made of record and not relied upon is considered pertinent to applicant's disclosure. Other similar conditioners and abrasive tools are cited of interest.

Any inquiry concerning this communication or earlier communications from the examiner should be directed to MAURINA RACHUBA whose telephone number is (571)272-4493. The examiner can normally be reached on Monday-Friday, between the hours of 5:30AM-4:00PM.

If attempts to reach the examiner by telephone are unsuccessful, the examiner's supervisor, Monica Carter can be reached on 571 272 4475. The fax phone number for the organization where this application or proceeding is assigned is 571-273-8300.

Information regarding the status of an application may be obtained from the Patent Application Information Retrieval (PAIR) system. Status information for published applications may be obtained from either Private PAIR or Public PAIR. Status information for unpublished applications is available through Private PAIR only. For more information about the PAIR system, see http://pair-direct.uspto.gov. Should you have questions on access to the Private PAIR system, contact the Electronic Business Center (EBC) at 866-217-9197 (toll-free). If you would like assistance from a USPTO Customer Service Representative or access to the automated information system, call 800-786-9199 (IN USA OR CANADA) or 571-272-1000.

/MAURINA RACHUBA/
Primary Examiner, Art Unit 3727

Notice of References Cited	Application/Control No. 13/797,704	Applicant(s)/Patent Under Reexamination SUNG, CHIEN-MIN	
	Examiner MAURINA RACHUBA	Art Unit 3727	Page 1 of 1

U.S. PATENT DOCUMENTS

*		Document Number Country Code-Number-Kind Code	Date MM-YYYY	Name	Classification
*	A	US-5,197,249 A	03-1993	Wiand, Ronald C.	451/529
*	B	US-6,190,240 B1	02-2001	Kinoshita et al.	451/56
*	C	US-6,213,856 B1	04-2001	Cho et al.	451/443
*	D	US-6,371,842 B1	04-2002	Romero, Vincent D.	451/540
*	E	US-2002/0173234 A1	11-2002	Sung et al.	451/21
*	F	US-2006/0079160 A1	04-2006	Balagani et al.	451/285
*	G	US-7,201,645 B2	04-2007	Sung, Chien-Min	451/443
*	H	US-2007/0155298 A1	07-2007	Sung, Chien-Min	451/527
*	I	US-7,658,666 B2	02-2010	Sung, Chien-Min	451/56
*	J	US-2012/0302146 A1	11-2012	Sung, Chien-Min	451/443
*	K	US-8,398,466 B2	03-2013	Sung et al.	451/443
*	L	US-2014/0099868 A1	04-2014	Sung, Chien-Min	451/56
	M	US-			

FOREIGN PATENT DOCUMENTS

*		Document Number Country Code-Number-Kind Code	Date MM-YYYY	Country	Name	Classification
	N					
	O					
	P					
	Q					
	R					
	S					
	T					

NON-PATENT DOCUMENTS

*		Include as applicable: Author, Title Date, Publisher, Edition or Volume, Pertinent Pages)
	U	
	V	
	W	
	X	

*A copy of this reference is not being furnished with this Office action. (See MPEP § 707.05(a).)
Dates in MM-YYYY format are publication dates. Classifications may be US or foreign.

EAST Search History

EAST Search History (Prior Art)

Ref #	Hits	Search Query	DBs	Default Operator	Plurals	Time Stamp
L1	81	("0970618" | "1854508" | "2410506" | "2712987" | "2907146" | "3048482" | "3991527" | "4314827" | "4525177" | "4554765" | "4652275" | "4653236" | "4744802" | "4770671" | "4881951" | "4903440" | "4906523" | "5015266" | "5142829" | "5174795" | "5197249" | "5201916" | "5213591" | "5287662" | "5316812").PN. OR ("6371842").URPN.	US-PGPUB; USPAT; USOCR	OR	ON	2015/01/07 08:49
L2	4694	(b24b37/34 b24b53/00 b24b53/017 b24b53/02).cpc.	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2015/01/07 09:41
L3	342	((Chien-Min) near2 (Sung)).INV.	US-PGPUB; USPAT; USOCR	OR	ON	2015/01/07 09:42
L4	67	l2 and L3	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2015/01/07 09:42
L5	11049	((B24B53/017 OR B24B53/12 OR B24B1/00 OR B24B7/228 OR B24B53/02 OR B24B53/00).CPC.)	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2015/01/07 09:44
L6	361365	condition$4 with (wafer substrate semiconductor)	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2015/01/07 09:45
L7	1666	l5 and l6	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2015/01/07 09:45
L8	754	l7 and (unit segment)	US-PGPUB; USPAT; USOCR;	OR	ON	2015/01/07 09:45

			FPRS; EPO; JPO; DERWENT; IBM_TDB			
L9	33	l8 and l3	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2015/01/07 09:45
L10	207	highest adj tip	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2015/01/07 09:46
L11	5	l8 and L10	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT; IBM_TDB	OR	ON	2015/01/07 09:46

EAST Search History (Interference)

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1/7/2015 9:46:47 AM
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Search Notes	Application/Control No.	Applicant(s)/Patent Under Reexamination
	13797704	SUNG, CHIEN-MIN
	Examiner	Art Unit
	MAURINA RACHUBA	3727

CPC- SEARCHED		
Symbol	Date	Examiner
B24B 37/34, 53/00, 53/017, 53/02	1/7/2015	MTR

CPC COMBINATION SETS - SEARCHED		
Symbol	Date	Examiner

US CLASSIFICATION SEARCHED			
Class	Subclass	Date	Examiner
SEE eas			

SEARCH NOTES		
Search Notes	Date	Examiner
See EAST, inventor and citation searches	1/7/2015	MTR

INTERFERENCE SEARCH			
US Class/ CPC Symbol	US Subclass / CPC Group	Date	Examiner

	/MAURINA RACHUBA/ Primary Examiner.Art Unit 3727

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

ART UNIT: 3727 EXAMINER: Maurina Rachuba FIRST NAMED INVENTOR: Chien-Min Sung SERIAL NO.: 13/797,704 FILED: 3/12/2013 CONF. NO.: 1089 FOR: COMPOSITE CONDITIONER AND ASSOCIATED METHODS DOCKET NO.: 02074-32836.US	**CERTIFICATE OF MAILING UNDER 37 C.F.R. § 1.8** DATE OF DEPOSIT: **July 13, 2015** 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. /Nicole Solomon/ Nicole Solomon

RESPONSE UNDER 37 C.F.R. § 1.111

Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313

Dear Sir:

In response to the communication mailed on January 12, 2015, please reconsider this application in view of the remarks provided herein. A petition for a 3-month extension of time and associated fees are submitted herewith.

CLAIM LISTING

1. (original) A composite conditioner comprising:

a base plate; and

a plurality of polishing units secured to a surface of the base plate by an adhesive layer, each polishing unit including a plurality of polishing tips secured in a binding layer, wherein a height difference between a first highest polishing tip and a second highest polishing tip is less than or equal to about 10μm, a height difference between the first highest polishing tip and a tenth highest polishing tip is less than or equal to about 20μm, and a height difference between the first highest polishing tip and a 100th highest polishing tip is less than or equal to about 40μm, and the first highest polishing tip protrudes from the binding layer to a height of greater than or equal to about 50μm.

2. (original) The composite conditioner of claim 1, wherein at least 1% of the plurality of polishing tips actively condition a CMP pad during a conditioning process.

3. (original) The composite conditioner of claim 1, wherein less than 40% of the surface of the base plate is covered with polishing units.

4. (original) The composite conditioner of claim 1, wherein the polishing units are arranged on the base plate in an arrangement selected from the group consisting of a single ring, double rings, multi-rings, radial arrangements, and spiral arrangements.

5. (original) The composite conditioner of claim 1, wherein at least one thousand polishing tips of the plurality of polishing tips condition a polishing pad during a conditioning process.

6. (original) The composite conditioner of claim 1, wherein the plurality of polishing tips include diamond, cubic boron nitride, or a combination thereof.

7. (original) The composite conditioner of claim 6, wherein the plurality of polishing tips include diamond, and the diamond is deposition diamond, polycrystalline diamond, or a combination thereof.

8. (original) The composite conditioner of claim 1, wherein the crystal surface coverage of more than 50% of the top 100 height polishing tips are less than 80%.

9. (original) The composite conditioner of claim 8, wherein the crystal surface coverage of more than 50% of the top 100 height polishing tips are less than 50%.

10. (original) The composite conditioner of claim 1, wherein each polishing unit further includes a polishing unit substrate upon which the plurality of polishing tips are secured with the binding layer.

11. (original) The composite conditioner of claim 1, wherein the binding layer is selected from the group consisting of a solder layer, a plating layer, a sintering layer, and a resin layer.

12. (original) The composite conditioner of claim 11, wherein each polishing unit further comprises a metal cover layer that covers the binding layer, wherein the metal cover layer includes at least one material selected from the group consisting of Ni, Cr, Pd, Co, Pt, Au, Ti, Cu, W, and alloys thereof.

13. (original) The composite conditioner of claim 12, wherein each polishing unit further comprises a protective layer covering the metal cover layer, wherein the protective layer includes a material selected from the group consisting of Pd, Pt, silicon carbide, aluminum nitride, alumina, zirconia, diamond-like carbon (DLC), and combinations thereof.

14. (original) The composite conditioner of claim 11, wherein binding layer is a solder layer comprising 1 wt% or more of a first material selected from the group consisting of Cr, B, P, Ti, or

an alloy thereof, or 50 tw% or more of a second material selected from the group consisting of Ni, Cu, or an alloy thereof, or 1 tw% or more of the first material and 50 wt% or more of the second material.

15. (original) The composite conditioner of claim 1, wherein the polishing units are disc-shaped, radial-shaped, or polygon-shaped.

16. (original) The composite conditioner of claim 15, wherein the polishing units are disc-shaped having a diameter of from about 5mm to about 30mm.

17. (original) The composite conditioner of claim 1, wherein the base plate is disc-shaped having a diameter of from about 80mm to about 120mm.

18. (original) The composite conditioner of claim 1, wherein the polishing tips have a particle size of from about 100μm to about 500μm.

19. (original) The composite conditioner of claim 1, wherein the adhesive layer is an organic adhesive.

20. (original) The composite conditioner of claim 1, wherein the polishing tips are arranged in a predetermined pattern on the polishing unit substrate.

21. (original) The composite conditioner of claim 1, wherein the polishing units are spaced no closer than about 0.1mm.

22. (original) The composite conditioner of claim 21, wherein the polishing units are spaced at least 0.5mm apart.

23. (original) The composite conditioner of claim 1, wherein the adhesive layer between the base plate and the polishing units has a thickness of less than or equal to about 0.6 mm.

24. (withdrawn) A method of making a composite conditioner, comprising:

disposing a second mold on a first mold, wherein the second mold includes a plurality of holes;

placing a polishing unit in each of the plurality of holes of the second mold, each polishing unit including a plurality of polishing tips secured in a binding layer;

applying an adhesive layer over the second mold to secure the polishing units; and

removing the first mold and the second mold to form a composite conditioner wherein a height difference between a first highest polishing tip and a second highest polishing tip is less than or equal to about 10μm, a height difference between the first highest polishing tip and a tenth highest polishing tip is less than or equal to about 20μm, and a height difference between the first highest polishing tip and a 100th highest polishing tip is less than or equal to about 40μm and the first highest polishing tip protrudes from the binding layer to a height of greater than or equal to about 50μm.

25. (withdrawn) The method of claim 24, wherein placing the polishing unit in the plurality of holes of the second mold further comprises orienting the polishing units such that the polishing tips face the first mold.

26. (withdrawn) The method of claim 25, wherein the polishing tips contact the first mold such that the polishing unit are leveled across the first mold.

27. (withdrawn) The method of claim 26, wherein the holes of the second mold are arranged around an outer periphery of the second mold.

28. (original) A process of chemical-mechanical planarization (CMP), including the following steps:

polishing a wafer against a rotating polishing pad; and

applying the conditioner of claim 1 against the polishing pad to remove debris and condition the pad.

29. (original) The process of claim 28, wherein the diameter of the wafer is 200mm, 300mm, or 450mm.

30. (original) The process of claim 28, wherein a surface of the wafer has an IC critical dimension of less than or equal to 45nm.

31. (original) The process of claim 30, wherein a surface of the wafer has an IC critical dimension of less than or equal to 28nm.

32. (original) The process of claim 28, wherein the surface of the wafer has an IC critical dimension of less than or equal to 22 nm.

REMARKS

The Applicant expresses appreciation to the Examiner for consideration of the subject patent application. This communication is in response to the Office Action mailed on January 12, 2015. In the Office Action the Examiner rejected claims 28-32 under pre-AIA 35 U.S.C. § 112 first paragraph as allegedly failing to comply with the written description requirement, claims 29-32 under pre-AIA 35 U.S.C. § 112 second paragraph as allegedly indefinite, and claims 1-23 and 28-32 under 35 U.S.C. § 103 as allegedly obvious.

Claim Rejections – 35 U.S.C. § 112, first paragraph

In the Office Action claims 28-32 were rejected under pre-AIA 35 U.S.C. § 112, first paragraph, for allegedly failing to comply with the written description requirement. The Examiner alleged that the claims contain subject matter which was not described in the Specification in a way as to reasonably convey to one skilled in the relevant art that the inventors had possession of claimed invention at the time application was filed. The Examiner's allegations are based on the Specification stating, "[w]hen the wafer varies greatly (8 inch to 12 inch even to 18 inch in the future), the variability to the polishing rate and the uniformity will become higher, and the quality and the efficiency of the chemical-mechanical planarization process will become lower. Not only that, when the critical dimension becomes more and more narrow (45nm to 32nm even to 22nm in the future), the high point of the polishing pad gets broken more easily." In essence, the Examiner takes this language because of its future tense and alleges that the present claims lack written description because the wafer products produced by the claimed process are not yet commercially available. The Applicant respectfully disagrees with the Examiner's assertion.

The purposes behind the written description requirement are (i) "to clearly convey the information that an applicant has invented the subject matter which is claimed," (ii) to put in the hands of the public what the Applicant claims as the invention, and (iii) to satisfy the inventor's obligation to disclose the technologic knowledge upon which the patent was based. *see In re Barker*, 559 F.2d 588, 592 (C.C.P.A. 1977); *Regents of the Univ. of Cal. v. Eli Lily*, 119 F.3d 1559, 1566 (Fed. Cir. 1997); and *Capon v. Eshhar*, 418 F.3d 1349, 1357 (Fed. Cir. 2005).

All that is required in the Specification to satisfy the written description requirement is a description of "the claimed invention in sufficient detail that one skilled in the art can reasonably conclude that the inventor had possession of the claimed invention." *Moba, B.V. v. Diamond Automation, Inc.* 325 F.3d 1306, 1319 (Fed. Cir. 2003). "There is a strong presumption that an adequate written description of the claimed invention is present when the application is filed." *In re Wertheim,* 541 F.2d 257, 263 (C.C.P.A. 1976). The patent act does not require a physical embodiment of the invention; thus there is no express "reduction to practice" requirement. *Pfaff v. Wells Electronics, Inc.* 525 U.S. 55, 66 (1998). *see also* MPEP § 2163.II.3.a.

In regards to claim 28, the Applicant respectfully submits that the Examiner's basis for this rejection is improper. Claim 28 is directed to a process of CMP comprising polishing a wafer against rotating polishing pad and applying the conditioner of claim 1. Claim 28 does not include claim elements that disclose the wafer size or IC critical dimension of the wafers. Therefore, the Examiner's reason for this rejection is irrelevant to claim 28. The Applicant respectfully requests that the Examiner's rejection to claim 28 be withdrawn.

In regards to claim 29, the Applicant respectfully submits that the Examiner's rejection to this claim is improper because (i) the claims are drawn to a process not the wafers themselves, and (ii) the Specification does not expressly state the claimed wafer sizes are not currently available. In contrast, the Specification states that the process "could use the diameter of the wafer in 200mm, 300mm or 450mm in the chemical-mechanical planaization process of the present invention." [pg. 21, lins. 20-23].

In regards to claims 30-32 the Applicant submits that these claims are drawn to a process allowing for a level of precision that is otherwise not available and therefore, the Applicant has satisfied the written description requirement. The claims are directed to a level of precision that can be obtained *by a process* that incorporates the CMP pad conditioner of claim 1. CMP processing techniques and pad dressers limit the critical dimensions of integrated circuits on the wafer. The critical dimension of the wafer varies based on the level of polishing precision and/or the size of the wafer. In order to achieve lower critical dimension without increasing the surface area, the wafer has to be polished more precisely to achieve a more even surface. As the surface becomes more level, more nodes can be placed on the surface, and the integrated circuits critical

dimension can be lowered. Claims 30-32 are directed *to the process* and the critical dimension that can be achieved using the claimed process. The claims are not directed to a wafer having that critical dimension; therefore, the Applicant respectfully submits that these claims comply with the written description requirement and are supported by the originally filed Specification. The Applicant respectfully requests that the Examiner's rejection to these claims be withdrawn.

For all of the reasons stated above the Applicant respectfully submits that the originally filed Specification meets the written description requirement for claims 28-32. The originally filed Specification clearly conveys the necessary information to support the process which is claimed. It is irrelevant as to whether or not a wafer having an IC critical dimension as claimed in claims 30-32 is commercially available because the patent act does not include an actual "reduction to practice" requirement. Therefore, the Applicant respectfully submits that the originally filed Specification meets the written description requirements of 35 U.S.C. § 112, first paragraph and the Applicant respectfully requests that the Examiner's rejections be withdrawn.

Claim Rejections - 35 U.S.C. § 112, second paragraph

In the Office Action the Examiner rejected claims 29-32 as allegedly indefinite under pre-AIA 35 U.S.C. § 112, second paragraph. To determine whether a patent claim is indefinite, a court must consider whether one skilled in the art would understand the bounds of the claim when read in light of the Specification. *Personalized Mass Media Communications, LLC v. International Trade Commission*, 161 F.3d 696, 48 U.S.P.Q.2d 1880 (Fed. Cir. 1998). If the claims, when read in the light of the Specification, reasonably apprise those skilled in the art both of the utilization and scope of the invention and, if the language is as precise as the subject matter permits the courts can demand no more. *North Am. Vaccine, Inc. v. American Cyanamid Co.*, 7 F.3d 1571, 28 USPQ 2d 1333 (Fed. Cir. 1993); *Personalized Mass Media Communications, LLC v. International Trade Commission*, *supra*. The meaning of a claim should be what would be reasonable from the perspective of one of ordinary skill in the art. *In re Buszard,* 504 F.3d 1364, 84 U.S.P.Q.2d 1749 (Fed. Cir. 2007).

The Applicant respectfully asserts that the bounds of claims 29-32 as previously presented would be understood by one skilled in the art when read in light of the originally filed Specification. Claims 29-32 are directed *to a process* of chemical mechanical planarization. These claims merely

specify either the diameter size of the wafer used in the process, claim 29, or the integrated circuit critical dimension of the resultant wafer, claims 30-32. One skilled in the art would be aware of both the utilization and scope of the invention that is covered by these claims. Therefore, the Applicant respectfully submits that these claims are sufficiently definite and respectfully requests that the Examiner's rejection be withdrawn.

Claim Rejections - 35 U.S.C. § 103

In the Office Action the Examiner rejected claims 1-23 and 28-32 under pre-AIA U.S.C. § 103(a) as allegedly unpatentable over U.S. Pub. No. 2002/0173234 (hereinafter "Sung") in view of U.S. Pat. No. 6, 371,842 (hereinafter "Romero").

Before discussing the obviousness rejection herein, it is thought proper to briefly state what is required to sustain such a rejection. The issue under 35 U.S.C. § 103 is whether the USPTO has stated a case of *prima facie* obviousness. The Applicant does not deem it necessary to recite the entire case law standard required in order to establish a *prima facie* case of obviousness. However, the Applicant would like to briefly remind the Examiner that a *prima facie* case of obviousness generally includes establishing 1) that the asserted references as modified or combined teach or suggest each and every element of the claimed invention, 2) that the asserted references as modified or combined provide a sufficient likelihood of successfully making the modification or combination, and 3) a reason for the modification or combination asserted. Under the guidelines in the MPEP, an Examiner must establish that the reference teaches or suggests each and every claim element or explain "why the difference(s) between the prior art and the claimed invention would have been obvious." See MPEP § 2143.03 and MPEP § 2141.III.

Additionally, under *KSR*, and as outlined under the MPEP § 2143, additional rationales include (a) combining prior art elements according to known methods to yield predictable results; (b) simple substitution of one known element for another to obtain predictable results; (c) use of known technique to improve similar devices (methods, or products) in the same way; (d) applying a known technique to a known device (method, or product) ready for improvement to yield predictable results; (e) "obvious to try" - choosing from a finite number of identified, predictable solutions, with a reasonable expectation of success; (f) known work in one field of endeavor may

prompt variations of it for use in either the same field or a different one based on design incentives or other market forces if the variations are predictable to one of ordinary skill in the art; and (g) some teaching, suggestion, or motivation in the prior art that would have led one of ordinary skill to modify the prior art reference or to combine prior art reference teachings to arrive at the claimed invention. Additionally, the obviousness inquiry asks "whether the claim inventions are rendered obvious by the teachings of the prior art as a whole." *In re Chevalier*, 500 Fed. Appx. 932, 934 (Fed. Cir. 2013).

The Applicant respectfully asserts the Examiner has not satisfied the requirements for establishing a *prima facie* case of obviousness. The Applicant respectfully submits that the Examiner has not established a *prima facie* case of obviousness because Romero is not analogous art and cannot be used to establish a *prima facie* case of obviousness and Sung does not teach each and every element of the claims. Therefore, the cited references do not render the claims obvious.

First, Romero is not analogous art and cannot be used to establish a *prima facie* case of obviousness. In order to sustain an obviousness rejection the cited references must be analogous prior art. MPEP § 2141.01(a)I. A reference is analogous art if: (1) the reference is from the same field of endeavor as the claimed invention (even if it addresses a different problem); or (2) the reference is reasonably pertinent to the problem faced by the inventor (even if it is not in the same field of endeavor as the claimed invention). MPEP § 2141.01(a)I *see Bigio*, 381 F.3d at 1325, 72 USPQ2d at 1212. In order for a reference to be "reasonably pertinent" to the problem, it must "logically have commended itself to an inventor's attention in considering his problem." *In re Icon Health and Fitness, Inc.*, 496 F.3d 1374, 1379-80 (Fed. Cir. 2007).

Romero is not in the same field of endeavor as the claimed invention. The claims are directed to a chemical mechanical planarization (CMP) conditioner and a CMP process. Romero does not teach a CMP conditioner or process. Romero teaches an abrasive disc for grinding, sanding, and polishing work pieces. [col. 2, lins. 41-44]. The work piece exemplified in Romero is a mild steel work piece, not a CMP pad. [Examples, col. 9, lin. 67]. Therefore, Romero is not in the same field of endeavor as the claimed invention.

Romero is also not reasonably pertinent to the problem faced by the Applicant. The Applicant sought to provide a composite conditioner that avoided over aggressive cutting, improved the ratio of working polishing tips during conditioning, and increased the removal ratio from the wafer and the durability of the conditioner. The abrasive disc taught in Romero is not reasonably pertinent to these problems because of the differences in scale. The scale of the abrasive disc in Romero is not suitable for functioning as a CMP pad conditioner. The scale of the abrasive disc in Romero is substantially larger than the scale of the claimed CMP pad conditioner. The substrate of Romero has a diameter from about 5 cm to about 30 cm and the central aperture diameter ranges from 1.27 cm to about 2.54 cm. [col. 2, lins. 59-62]. The thickness of the substrate, not including the raised portions, ranges from about 100 μm to about 2500 μm. [col. 2, lins. 62-65]. The length and width of the raised portions are between 0.1 cm and 5 cm. [col. 3, lins 21-23]. By contrast, the diameter of the CMP pad conditioner disk can be about 100 mm in general and the diameter of the ring can be greater than 250 mm. [pg. 14, lins. 13-14]. The particle size can range from 100 μm to 500 μm. [pg. 15, lins. 6-7]. The claimed composite conditioner includes polishing units having the highest polishing tip protruding from the binding layer to a height of greater than or equal to about 50 μm. Therefore, the claimed CMP pad conditioner is substantially smaller than the abrasive disc taught in Romero and the abrasive particles in the claims are substantially smaller than the substrate in Romero. This scale difference is crucial in chemical mechanical planarization. CMP pad dressers have small leveling tolerances. As more precise leveling is achieved, more nodes can be placed in a given area of the wafer. This is important because smaller critical wafer dimensions are in demand. In addition, one purpose of the claimed composite conditioner is to avoid over aggressive cutting. Therefore, this scale of the abrasive disc taught in Romero is not compatible with the scale required in the claimed invention. The abrasive disc taught in Romero is not reasonably pertinent to the problem faced by the Applicant.

Therefore, Romero is not analogous art and cannot be used to create a *prima facie* case of obviousness. Sung as acknowledged by the Examiner does not teach each and every element of the claims. Therefore, the Examiner has not presented a *prima facie* case of obviousness. The Applicant respectfully requests that that Examiner's rejection be withdrawn.

CONCLUSION

In view of the foregoing, the Applicant asserts that the pending claims present allowable subject matter and the allowance thereof is respectfully requested. If any impediment remains to examination after consideration of the above-recited remarks, which could be removed during a telephone interview, the Examiner is invited to telephone the undersigned attorney, at (801) 566-6633 so that such issues may be resolved as expeditiously as possible.

The Commissioner is hereby authorized to charge any additional fees associated with this communication or credit any overpayment to Deposit Account No. 20-0100.

DATED this 13th day of July, 2015.

Respectfully submitted,

THORPE, NORTH & WESTERN, LLP

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Electronic Patent Application Fee Transmittal

Application Number:	13797704
Filing Date:	12-Mar-2013
Title of Invention:	COMPOSITE CONDITIONER AND ASSOCIATED METHODS
First Named Inventor/Applicant Name:	Chien-Min Sung
Filer:	David William Osborne/Nicole Solomon
Attorney Docket Number:	02074-32836.US

Filed as Small Entity

Filing Fees for Utility under 35 USC 111(a)

Description	Fee Code	Quantity	Amount	Sub-Total in USD($)
Basic Filing:				
Pages:				
Claims:				
Miscellaneous-Filing:				
Petition:				
Patent-Appeals-and-Interference:				
Post-Allowance-and-Post-Issuance:				
Extension-of-Time:				

Description	Fee Code	Quantity	Amount	Sub-Total in USD($)
Extension - 3 months with $0 paid	2253	1	700	700
Miscellaneous:				
			Total in USD ($)	**700**

Electronic Acknowledgement Receipt

EFS ID:	22902467
Application Number:	13797704
International Application Number:	
Confirmation Number:	1089
Title of Invention:	COMPOSITE CONDITIONER 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:	02074-32836.US
Receipt Date:	13-JUL-2015
Filing Date:	12-MAR-2013
Time Stamp:	17:12:24
Application Type:	Utility under 35 USC 111(a)

Payment information:

Submitted with Payment	yes
Payment Type	Credit Card
Payment was successfully received in RAM	$700
RAM confirmation Number	4544
Deposit Account	
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The Director of the USPTO is hereby authorized to charge indicated fees and credit any overpayment as follows:	

File Listing:

Document Number	Document Description	File Name	File Size(Bytes)/ Message Digest	Multi Part /.zip	Pages (if appl.)
1	Extension of Time	02074-32836_US_Extension.pdf	127861 ebb9e7cf41981ec4d0ea6daf72eedc5fe7567cb8	no	1
Warnings:					
Information:					
2		02074-32836_US_Response.pdf	122506 7704d7ac6b10c177aacee4f75ca3c46a2929567f	yes	13

	Multipart Description/PDF files in .zip description		
	Document Description	**Start**	**End**
	Amendment/Req. Reconsideration-After Non-Final Reject	1	1
	Claims	2	6
	Applicant Arguments/Remarks Made in an Amendment	7	13

Warnings:					
Information:					
3	Fee Worksheet (SB06)	fee-info.pdf	30868 65017c6cd938b31498817aa1dad839db9b29e5cd	no	2
Warnings:					
Information:					
			Total Files Size (in bytes):	281235	

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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

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PTO/AIA/22 (03-13)
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PETITION FOR EXTENSION OF TIME UNDER 37 CFR 1.136(a)

Docket Number (Optional): 02074-32836.US

Application Number: 13/797,704

Filed: 03/12/2013

For: Composite Conditioner and Associated Methods

Art Unit: 3727

Examiner: Maurina T. Rachuba

This is a request under the provisions of 37 CFR 1.136(a) to extend the period for filing a reply in the above-identified application.

The requested extension and fee are as follows (check time period desired and enter the appropriate fee below):

	Fee	Small Entity Fee	Micro Entity Fee	
☐ One month (37 CFR 1.17(a)(1))	$200	$100	$50	$
☐ Two months (37 CFR 1.17(a)(2))	$600	$300	$150	$
☑ Three months (37 CFR 1.17(a)(3))	$1,400	$700	$350	$ 700
☐ Four months (37 CFR 1.17(a)(4))	$2,200	$1,100	$550	$
☐ Five months (37 CFR 1.17(a)(5))	$3,000	$1,500	$750	$

☑ Applicant asserts small entity status. See 37 CFR 1.27.

☐ Applicant certifies micro entity status. See 37 CFR 1.29.
Form PTO/SB/15A or B or equivalent must either be enclosed or have been submitted previously.

☐ A check in the amount of the fee is enclosed.

☑ Payment by credit card. Form PTO-2038 is attached.

☐ The Director has already been authorized to charge fees in this application to a Deposit Account.

☑ The Director is hereby authorized to charge any fees which may be required, or credit any overpayment, to Deposit Account Number 20-0100.

☑ Payment made via EFS-Web.

WARNING: Information on this form may become public. Credit card information should not be included on this form. Provide credit card information and authorization on PTO-2038.

I am the

☐ applicant.

☑ attorney or agent of record. Registration number 44989.

☐ attorney or agent acting under 37 CFR 1.34. Registration number _____.

/David W. Osborne/ — Signature

July 13, 2015 — Date

David W. Osborne — Typed or printed name

801-566-6633 — Telephone Number

NOTE: This form must be signed in accordance with 37 CFR 1.33. See 37 CFR 1.4 for signature requirements and certifications. Submit multiple forms if more than one signature is required, see below*.

☐ * Total of _____ forms are submitted.

This collection of information is required by 37 CFR 1.136(a). 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 1.11 and 1.14. This collection is estimated to take 6 minutes 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, 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: Mail Stop PCT, Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450.

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PATENT APPLICATION FEE DETERMINATION RECORD Substitute for Form PTO-875	Application or Docket Number 13/797,704	Filing Date 03/12/2013	☐ To be Mailed

ENTITY: ☐ LARGE ☒ SMALL ☐ MICRO

APPLICATION AS FILED – PART I

FOR	(Column 1) NUMBER FILED	(Column 2) NUMBER EXTRA	RATE ($)	FEE ($)
☐ BASIC FEE (37 CFR 1.16(a), (b), or (c))	N/A	N/A	N/A	
☐ SEARCH FEE (37 CFR 1.16(k), (i), or (m))	N/A	N/A	N/A	
☐ EXAMINATION FEE (37 CFR 1.16(o), (p), or (q))	N/A	N/A	N/A	
TOTAL CLAIMS (37 CFR 1.16(i))	minus 20 =	*	X $ =	
INDEPENDENT CLAIMS (37 CFR 1.16(h))	minus 3 =	*	X $ =	
☐ 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).			
☐ MULTIPLE DEPENDENT CLAIM PRESENT (37 CFR 1.16(j))				
* If the difference in column 1 is less than zero, enter "0" in column 2.			TOTAL	

APPLICATION AS AMENDED – PART II

AMENDMENT

07/13/2015	(Column 1) CLAIMS REMAINING AFTER AMENDMENT		(Column 2) HIGHEST NUMBER PREVIOUSLY PAID FOR	(Column 3) PRESENT EXTRA	RATE ($)	ADDITIONAL FEE ($)
Total (37 CFR 1.16(i))	* 32	Minus	** 32	= 0	X $40 =	0
Independent (37 CFR 1.16(h))	* 3	Minus	***3	= 0	X $210 =	0
☐ Application Size Fee (37 CFR 1.16(s))						
☐ FIRST PRESENTATION OF MULTIPLE DEPENDENT CLAIM (37 CFR 1.16(j))						
					TOTAL ADD'L FEE	0

AMENDMENT

	(Column 1) CLAIMS REMAINING AFTER AMENDMENT		(Column 2) HIGHEST NUMBER PREVIOUSLY PAID FOR	(Column 3) PRESENT EXTRA	RATE ($)	ADDITIONAL FEE ($)
Total (37 CFR 1.16(i))	*	Minus	**	=	X $ =	
Independent (37 CFR 1.16(h))	*	Minus	***	=	X $ =	
☐ Application Size Fee (37 CFR 1.16(s))						
☐ FIRST PRESENTATION OF MULTIPLE DEPENDENT CLAIM (37 CFR 1.16(j))						
					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 number found in the appropriate box in column 1.

LIE
/LAWANDA MILTON/

This collection of information is required by 37 CFR 1.16. 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 12 minutes 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, 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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INFORMATION DISCLOSURE STATEMENT BY APPLICANT

(Use as many sheets as necessary)

Sheet | | of |

Complete if Known	
Application Number	13/797,704
Filing Date	3/12/2013
First Named Inventor	Chien-Min Sung
Art Unit	3727
Examiner Name	Maurina Rachuba
Attorney Docket Number	02074-32836.US

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
		0187593	02/20/1877	Brown et al.	
		1854071	04/12/1932	Schacht	
		1988065	01/1935	Wooddell	
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		2005/0260939	11/24/2005	Andrews et al.	
		2006/0073774	04/06/2006	Sung	
		2006/0079160	04/2006	Balagani et al.	

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

(Use as many sheets as necessary)

Sheet | | of | |

Complete if Known	
Application Number	13/797,704
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Art Unit	3727
Examiner Name	Maurina Rachuba
Attorney Docket Number	02074-32836.US

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INFORMATION DISCLOSURE STATEMENT BY APPLICANT

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Sheet | | of |

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Filing Date	3/12/2013
First Named Inventor	Chien-Min Sung
Art Unit	3727
Examiner Name	Maurina Rachuba
Attorney Docket Number	02074-32836.US

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INFORMATION DISCLOSURE STATEMENT BY APPLICANT

(Use as many sheets as necessary)

Sheet | | of | |

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Application Number	13/797,704
Filing Date	3/12/2013
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Art Unit	3727
Examiner Name	Maurina Rachuba
Attorney Docket Number	02074-32836.US

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Substitute for form 1449/PTO

INFORMATION DISCLOSURE STATEMENT BY APPLICANT

(Use as many sheets as necessary)

Sheet | | of | |

Complete if Known	
Application Number	13/797,704
Filing Date	3/12/2013
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Art Unit	3727
Examiner Name	Maurina Rachuba
Attorney Docket Number	02074-32836.US

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Substitute for form 1449/PTO

INFORMATION DISCLOSURE STATEMENT BY APPLICANT

(Use as many sheets as necessary)

Sheet | | of |

Complete if Known	
Application Number	13/797,704
Filing Date	3/12/2013
First Named Inventor	Chien-Min Sung
Art Unit	3727
Examiner Name	Maurina Rachuba
Attorney Docket Number	02074-32836.US

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Substitute for form 1449/PTO

INFORMATION DISCLOSURE STATEMENT BY APPLICANT

(Use as many sheets as necessary)

Sheet | | of | |

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Application Number	13/797,704
Filing Date	3/12/2013
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Art Unit	3727
Examiner Name	Maurina Rachuba
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Substitute for form 1449/PTO

INFORMATION DISCLOSURE STATEMENT BY APPLICANT

(Use as many sheets as necessary)

Sheet | | of |

Complete if Known	
Application Number	13/797,704
Filing Date	3/12/2013
First Named Inventor	Chien-Min Sung
Art Unit	3727
Examiner Name	Maurina Rachuba
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INFORMATION DISCLOSURE STATEMENT BY APPLICANT

(Use as many sheets as necessary)

Sheet | | of |

Complete if Known	
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Filing Date	3/12/2013
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Substitute for form 1449/PTO

INFORMATION DISCLOSURE STATEMENT BY APPLICANT

(Use as many sheets as necessary)

Sheet | | of | |

Complete if Known	
Application Number	13/797,704
Filing Date	3/12/2013
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Art Unit	3727
Examiner Name	Maurina Rachuba
Attorney Docket Number	02074-32836.US

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Sheet | | of | |

Complete if Known	
Application Number	13/797,704
Filing Date	3/12/2013
First Named Inventor	Chien-Min Sung
Art Unit	3727
Examiner Name	Maurina Rachuba
Attorney Docket Number	02074-32836.US

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Attorney Docket Number	02074-32836.U.S.

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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]
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Art Unit	3727
Examiner Name	Maurina Rachuba
Attorney Docket Number	02074-32836.U.S.

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Attorney Docket Number	02074-32836.U.S.

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Sheet | | of |

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Examiner Signature		Date Considered	

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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] +
Applicant(s):	CHINA EMERG WHEEL ENTPR CO LTD [CN] +
Classification:	- **International:** ***B24B53/12;*** (IPC1-7): B24B53/12 - **European:**
Application number:	CN20001028301 20001107
Priority number(s):	CN20001028301 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.

Last updated: 14.02.2012 Worldwide Database 5.7.39.32p

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地址 中国台湾

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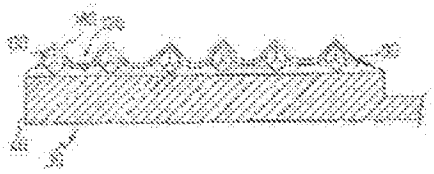
[74]专利代理机构 上海专利商标事务所
代理人 张民荣

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-机械抛光软垫的修整器及其制造方法

[57]摘要

一种化学—机械抛光软垫的修整器及一种制造这种软垫修整器的方法，该修整器表面有大量均匀间隔的磨料颗粒，颗粒是电为金刚石或立方氮化硼等。磨料颗粒纤焊于基底，再覆以一层含有金刚石或仿金刚石石墨的防腐层，以防纤焊料合金受化学研磨膏的腐蚀。这种结构使修整器得以在软垫进行抛光工作的同时修整软垫。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修理或修整软垫。



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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

料颗粒靠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。

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。研磨膏持续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膜的堆积。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

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，

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，请附图为：

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锐顶峰，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

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，当加请于基底 40 时，融熔状金属合金的表面张

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域。

磨料颗粒的簇状所产生的另一效应，是会于纤焊材料 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，防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 以纤焊材料 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 可保护纤焊材料 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 置于纤焊料合金片 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

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，以满足该软垫修整器的特定使用要求。

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，而延长软垫修整器 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 60 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聚集。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

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理置于钎焊料合金片 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。该软垫浸调 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。

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，以特别情形加以详细描述，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

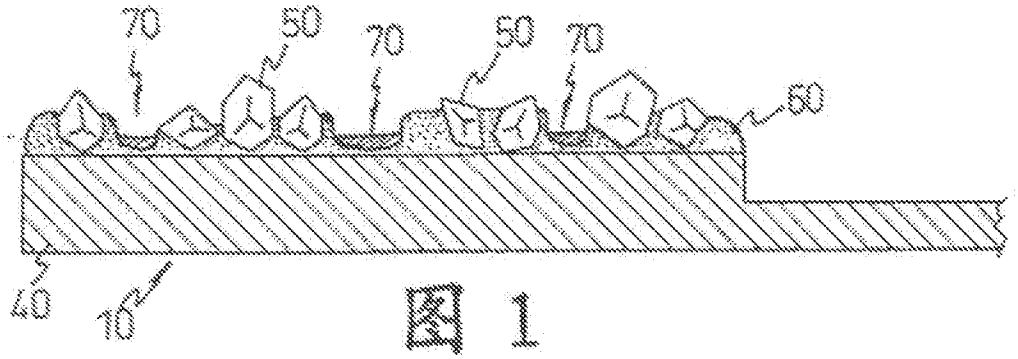


图 1

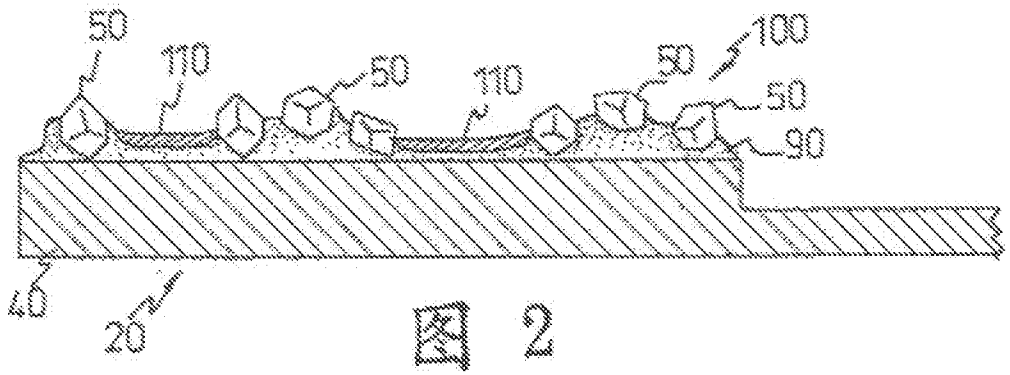


图 2

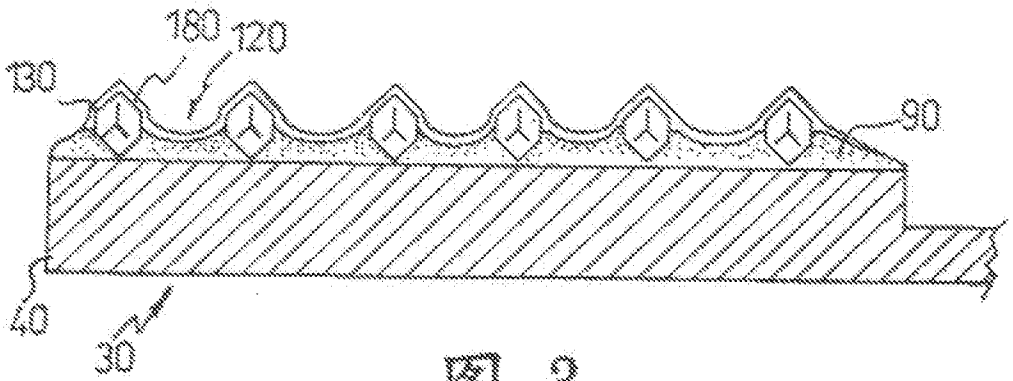


图 3

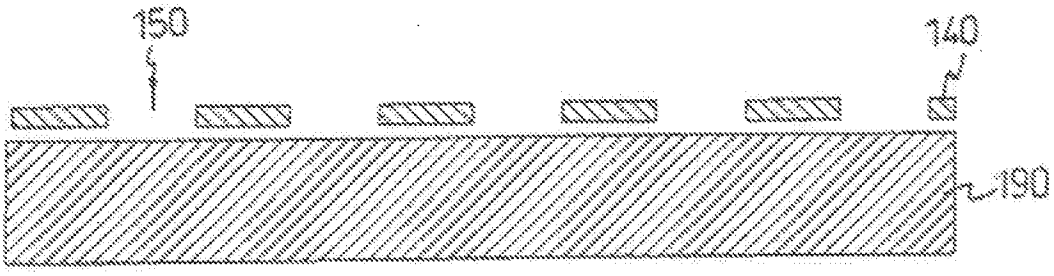


图 4

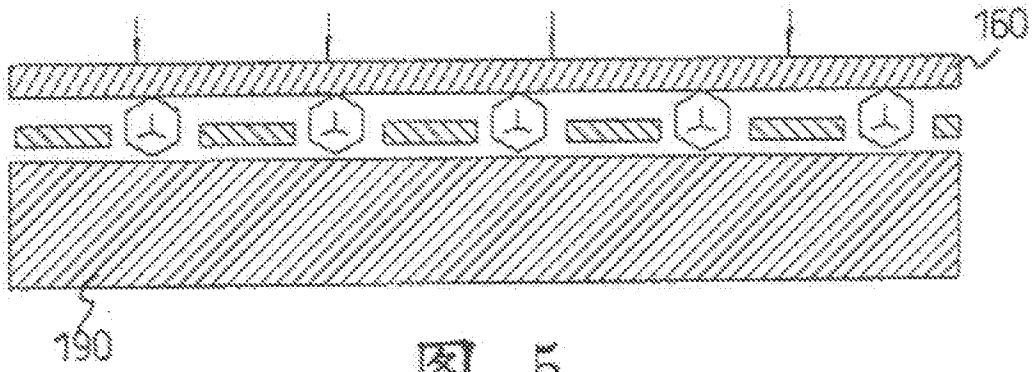


图 5

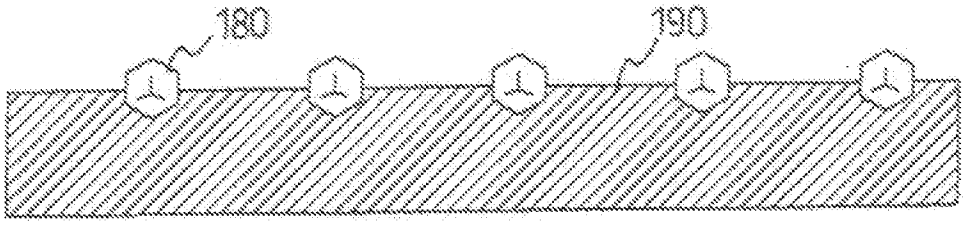


图 6

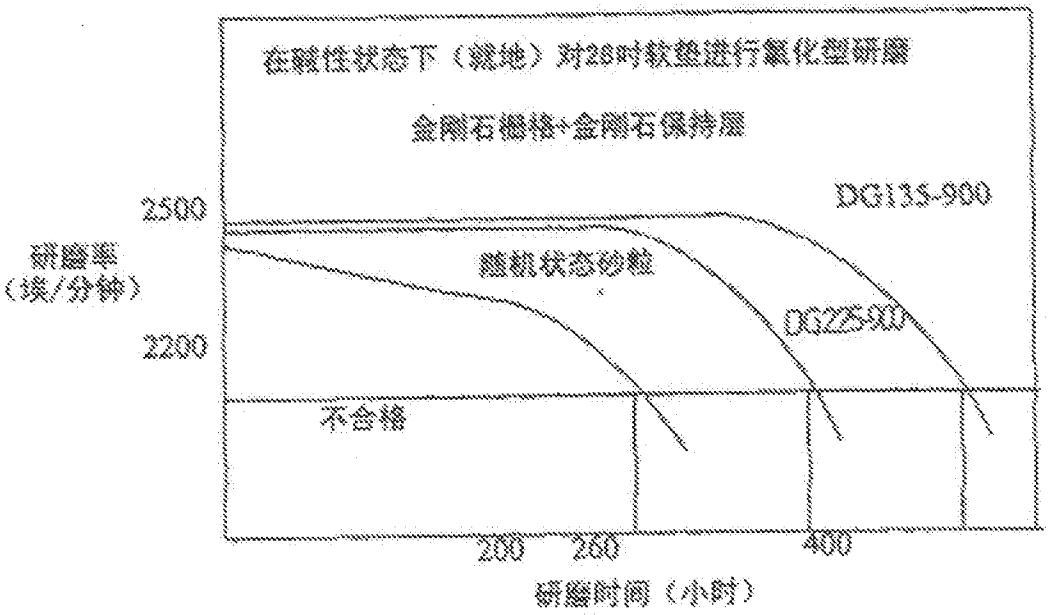

在碱性状态下（就地）对28吋软垫进行氧化型研磨
金刚石栅格+金刚石保持器
DG135-900
2500
研磨率
（埃/分钟）
随机状态砂粒
DG225-900
2200
不合格
200
260
400
研磨时间（小时）

图 7

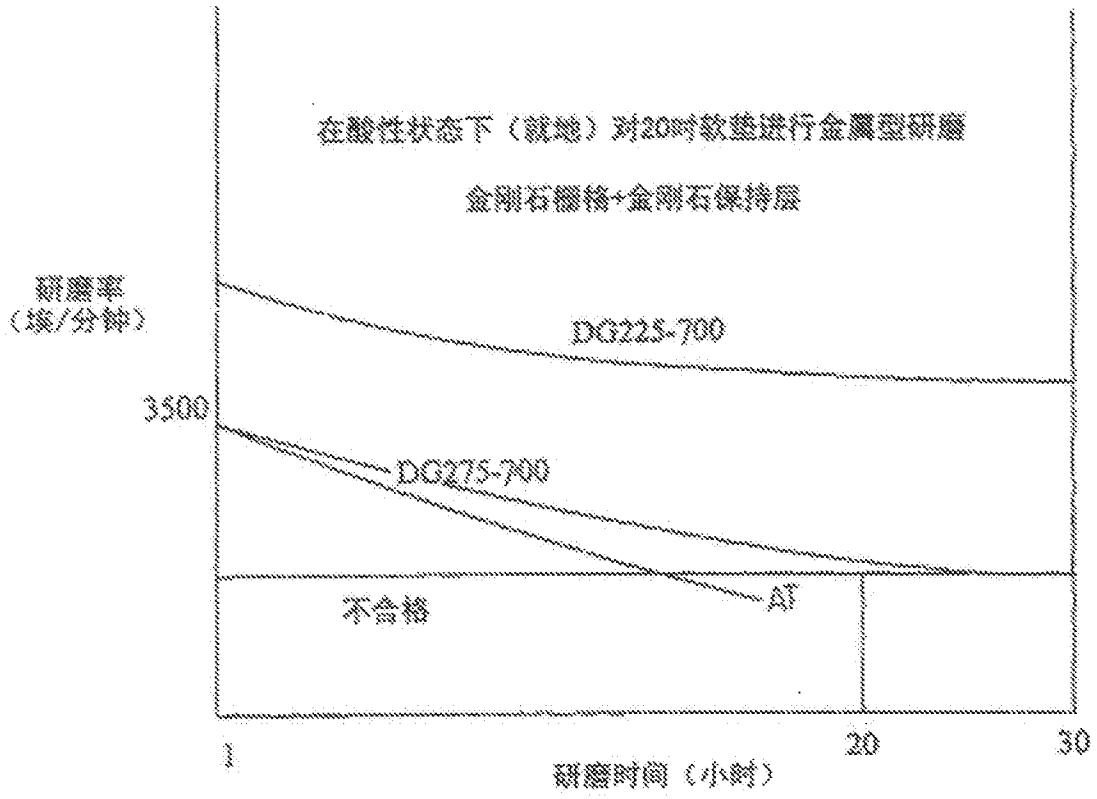

在酸性状态下（就地）对20吋软垫进行金属型研磨
金刚石栅格+金刚石保持座
研磨率
（埃/分钟）
DG225-700
3500
DG275-700
不合格
AT
1
20
30
研磨时间（小时）

图 8

Espacenet

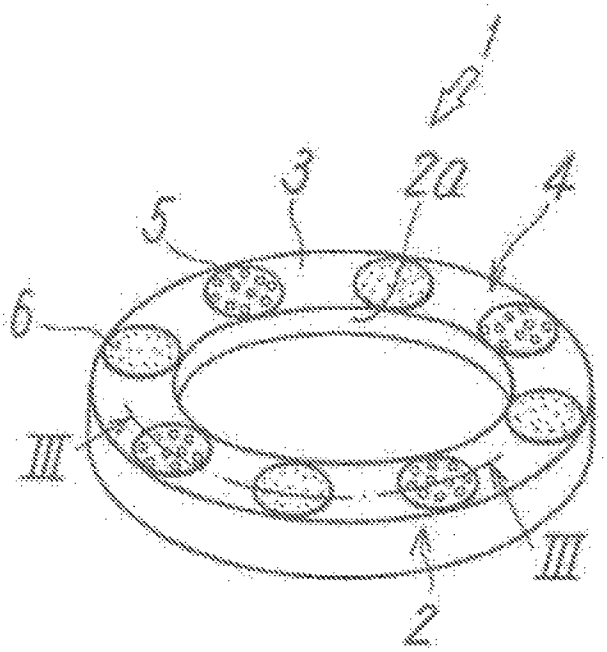
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Sander for polishing cloth and polishing cloth sanding method using said sander

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An abrasive cloth dresser is provided which is capable of adjusting the state of its dressing face so as to give an abrasive cloth a uniform polishing surface, and capable of giving the surfaces of an abrasive cloth a suitable polishing capability according to polishing objects, even if the dressing face state exhibits individual differences in, for example, the end shapes of abrasive grains. The abrasive cloth dresser 1 includes a base metal 2 having a ring-shaped dressing face 4 in the outer region of the base metal 2. First abrasive grain units 5 and second abrasive grain units 6 formed of abrasive grains with different grain sizes from each other are alternately arranged on the dressing face 4. The base metal 2 includes adjusters 7 for arbitrarily adjusting the height difference delta between reference planes S1 and S2 of the respective abrasive grain units 5 and 6. The reference planes S1 and S2 each include the ends of the abrasive grains with the largest grain size.



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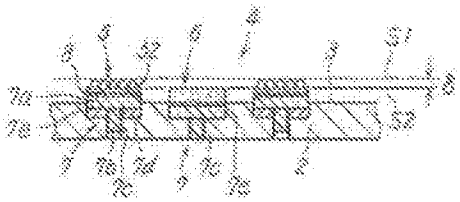
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权利要求书2页 说明书13页 附图4页

[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，即使在颗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)的圆盘面上备有打磨面(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被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)。



CN 1494984A 2004.05.12

1. 一种抛光布用打磨器，是在能够转动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

EX1057, Page 156

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三三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。

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，用使氧化铈、碳酸钡、氧化钛、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纤料把金刚

EX1057, Page 158

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材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，由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

EX1057, Page 159

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

EX1057, Page 160

具有规律地排列，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，上述课题可以通过拿上述调节机构，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

EX1057, Page 161

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，以使在各磨粒群 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

EX1057, Page 162

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 D 变得较大的场合，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连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～

EX1057, Page 163

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养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，磨粒靠保持材 52 保持而形成，通过借助粘接剂 8 等把保持该磨粒的保持材 52 固定于上述调节机构 7 中的基台 7a 的表面，磨粒群 5、6 固定于上述基台 7a。

此外，作为上述保持材 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材没有特别限制，靠一般的电镀镍或借助纤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

EX1057, Page 164

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，也可以达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

EX1057, Page 165

高低差 δ 而把打磨面 4 的状态调整成想要的状态，这不仅即使在打磨面 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 的前端的平面的纵差为 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 φ 100 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

EX1057, Page 166

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
		σ_{n-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
		σ_{n-1}	0.12	0.15	0.14
晶片抛光速度（nm／min）		Ave.	126	129	121
		σ_{n-1}	6.7	7.2	5.6
划痕发生率（%）			0.00	0.00	0.00

EX1057, Page 167

如此一来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权小，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磨粒彼此连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，使用由这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

EX1057, Page 168

40～60 μm，并且邻接的磨粒彼此连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	ave.	63.6	189	138
（μm／Hour）	σn－1	2.84	14.6	2.43
抛光布的表面粗糙度	ave.	3.01	3.32	3.45
Ra（μm）	σn－1	0.36	0.36	0.13
晶片研光速度	ave.	105	98.3	120
（nm／min）	σn－1	33.2	23.3	9.6
刮痕发生率（%）		0.52	0.00	0.00

EX1057, Page 169

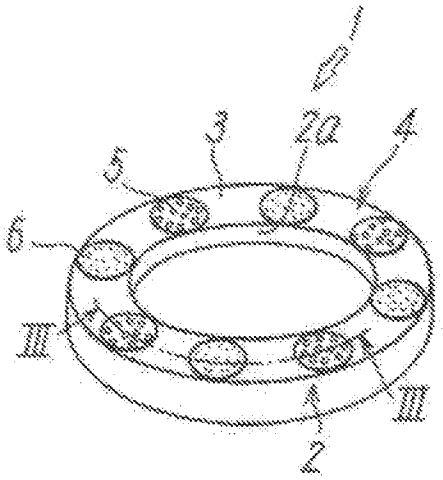


图 1A

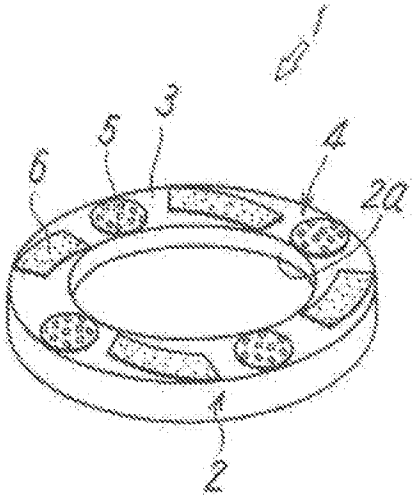


图 1B

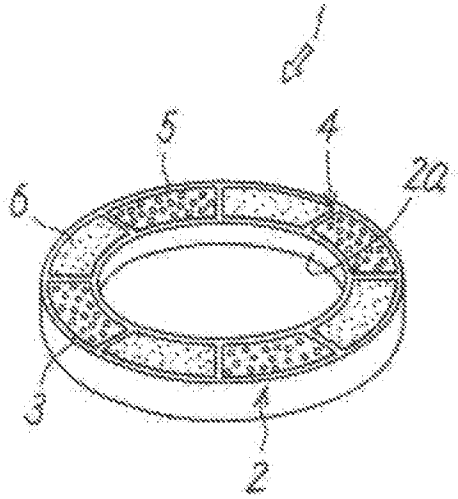


图 1C

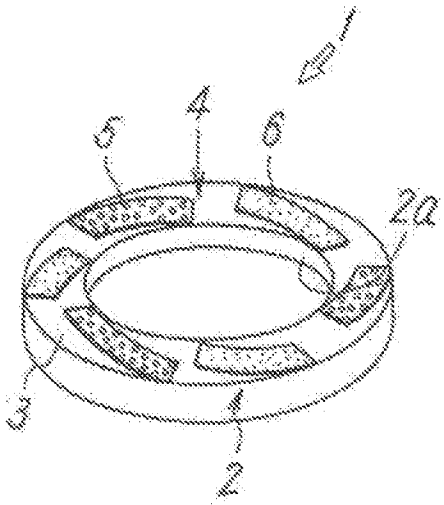


图 1D

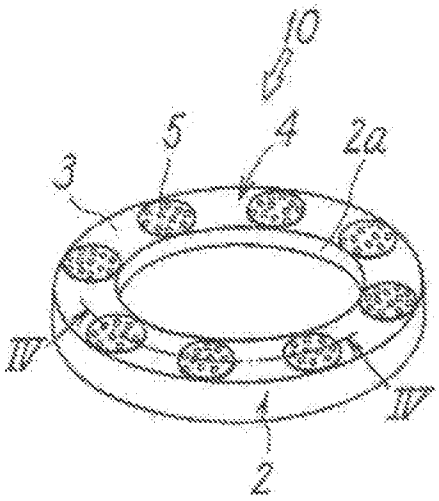


图 2A

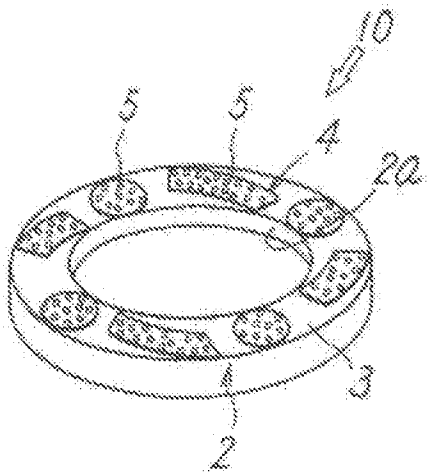


图 2B

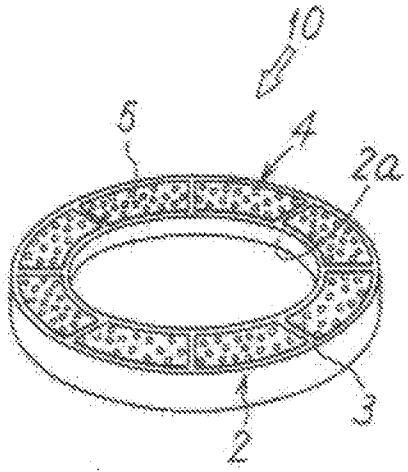


图 2C

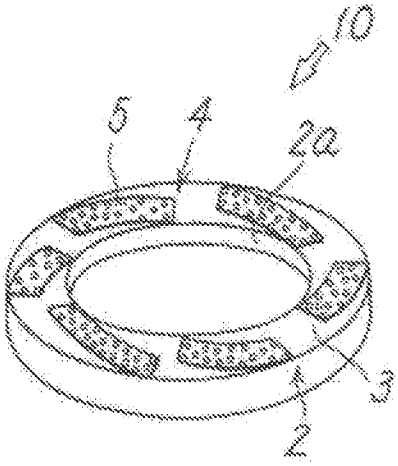


图 2D

EX1057, Page 171

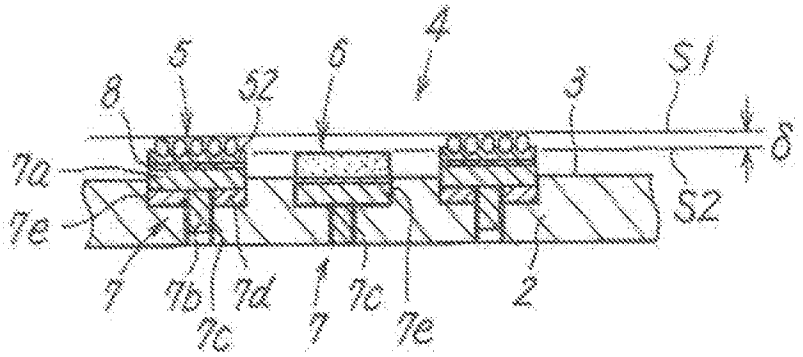


图 3

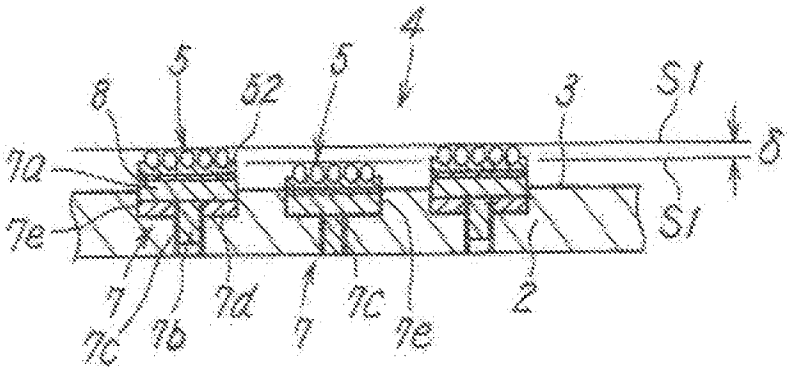


图 4

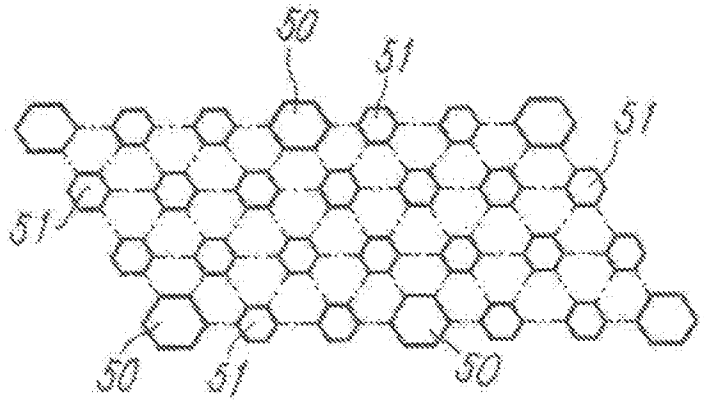


图 5

EX1057, Page 172

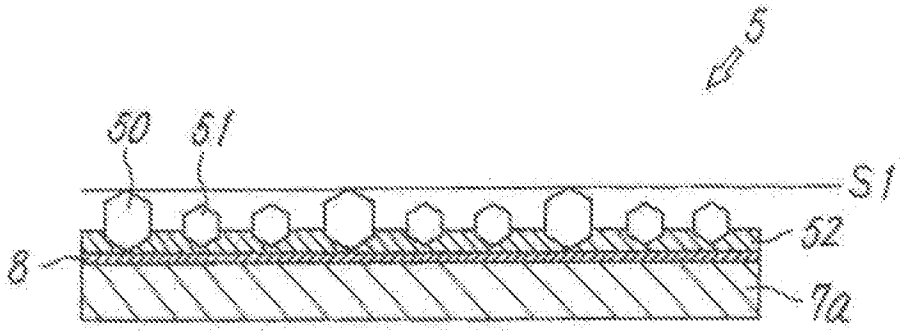


图 6

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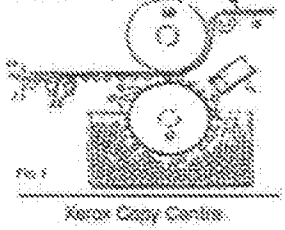
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(54) Abrasive sheeting having individually positioned abrasive granules.

(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.



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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 (Gonton), it was discovered that unbroken "abrading surfaces soon become clogged and gummed by the fine grit and minute particles worn from the abrading 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. 1,867,784 (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. 4,113,831 (Howard); and No. 4,541,842 (Rostoker). U.S. Pat. No. Re. 28,808 (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 disky 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

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. 4,539,195 (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 um 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) cindering 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

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 80% of the mean dimension of the abrasive granules. When using spherical abrasive granules 80 um in diameter, good results have been attained with dots 62 um 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

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, 80 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" #606 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 8.25/8.25 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 670 granules/cm². 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.

TABLE I

Ex.	Rows per inch	Rows per cm	mm of lens removed in 2 mins	
			2.12 d. lens	6.25/6.25 d. lens
1	20	7.9	0.001	0.000
2	40	15.7	0.031	0.056
3	65	25.6	0.025	0.076
4	85	33.5	0.028	0.124
5	150	59.0	0.012	0.032
6	200	78.7	0.009	0.025
Control			0.028	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 these 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 70 N was applied to the nip roll per cm of width. The composite was then placed in an oven at 113° 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 63 µm in diameter from a fluidized bed to create 8-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".

TABLE II

Ex.	Inner Circle Rows/cm	Outer Circle Rows/cm	mm of lens removed	
			212 lens	625/625 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	0.108
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 $X = k\theta$ and $X = k\theta + 0.317$ mm, starting from a circle 2.1 cm in diameter and extending to a circle 18.2 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 83 µm in diameter equally spaced in rows extending in the X and Y directions. There were 156 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 66 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. 807 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 4.46 cm I.D. and 8.4 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).

TABLE III

Example	Total Cut	R_a (µm)	R_t (µm)
13	2.84 g	0.30	2.87
Control	1.44 g	0.45	3.46
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.

FIG. 1

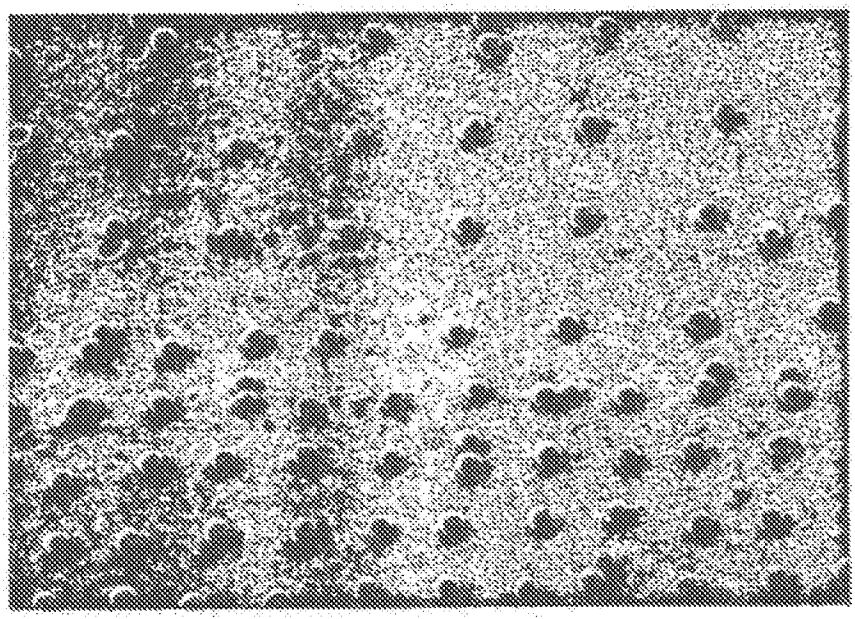


FIG. 2

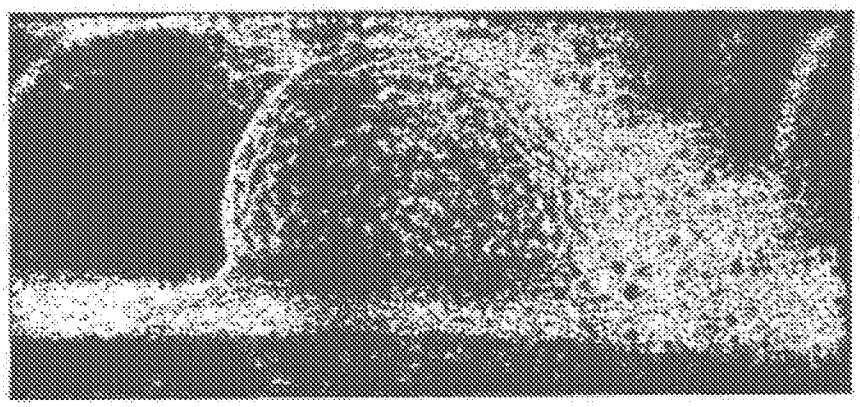


FIG. 3

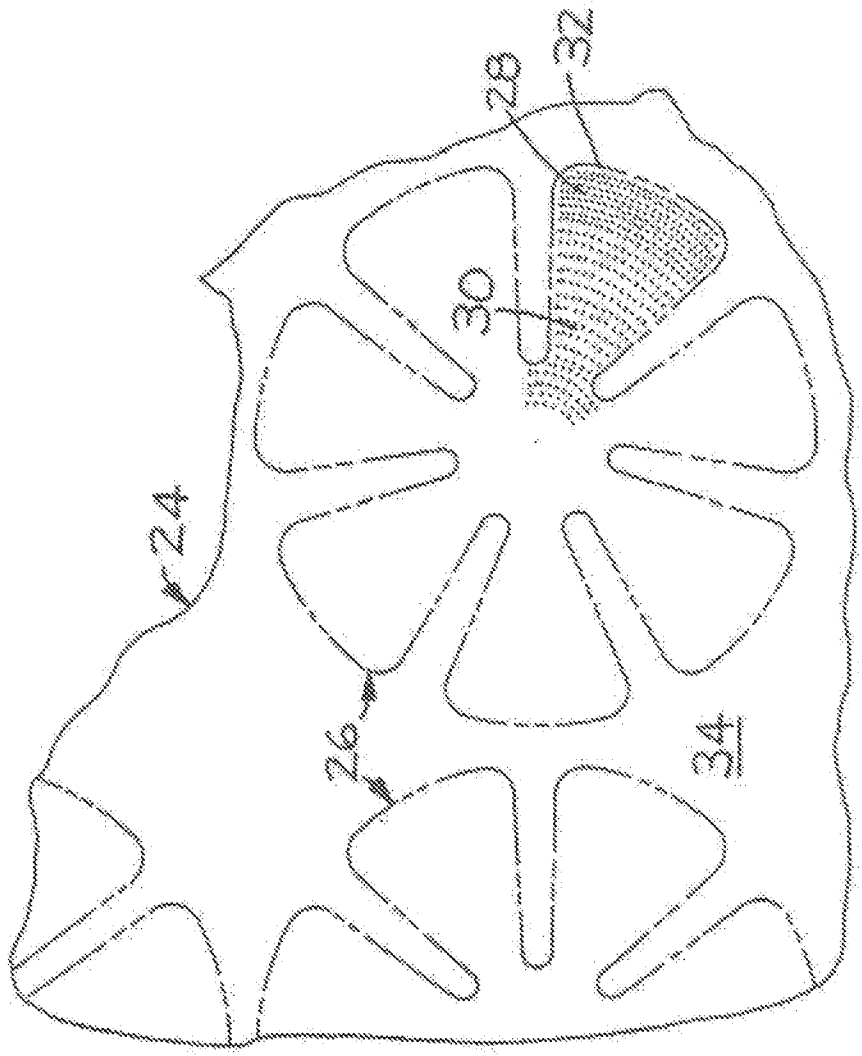


Fig. 4

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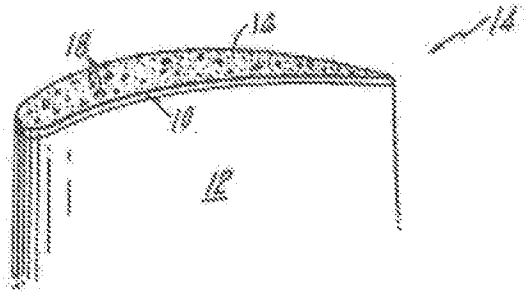
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(54) Method for depositing a layer of abrasive material on a substrate.

(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 (16) which are evenly spaced apart, in a high density arrangement, within a metallic matrix (18). In the deposition of the particles (16) 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 (16) on the article surface (11). The vacuum holds one particle (16) 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 (16) fall onto the article (12). The matrix (18) 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 case. 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 abseals; 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. 4,366,112. 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

EX1057, Page 187

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 1.3 to 1, so that less than about 10% 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 50-60% of the particulate surface area (excluding that surface area

EX1057, Page 188

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 perforations 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

EX1057, Page 189

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, relevent 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 1.8 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

EX1057, Page 190

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.

FIG. 1

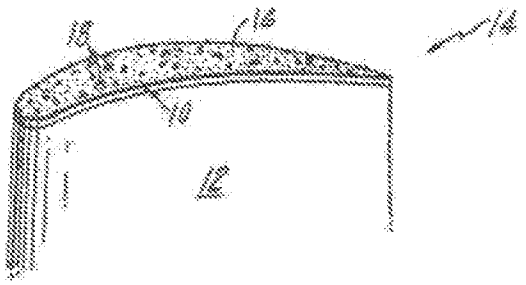
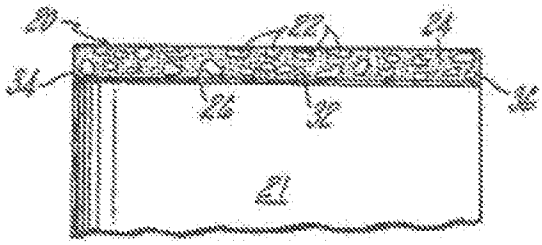


FIG. 2 PRIOR ART



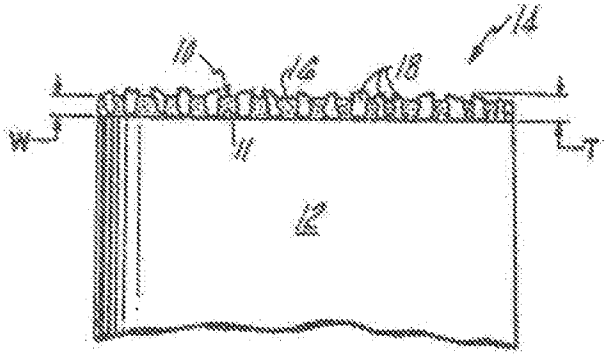


FIG. 3

FIG. 4

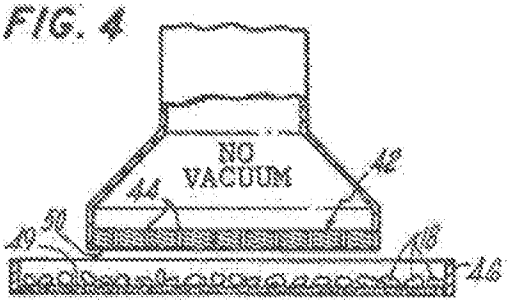


FIG. 5

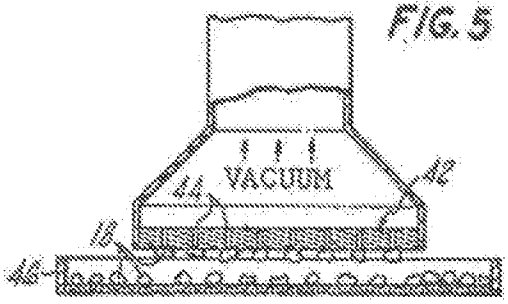


FIG. 6

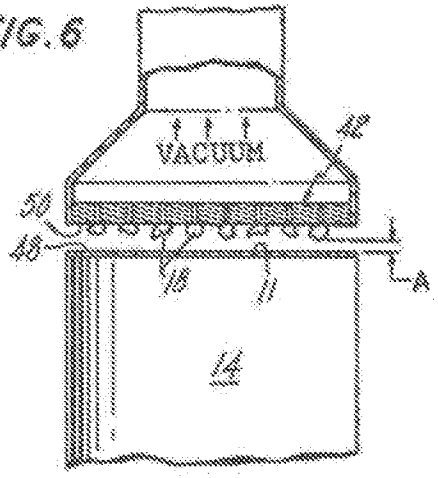
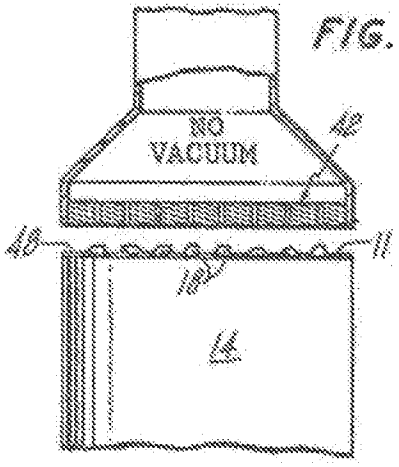


FIG. 7



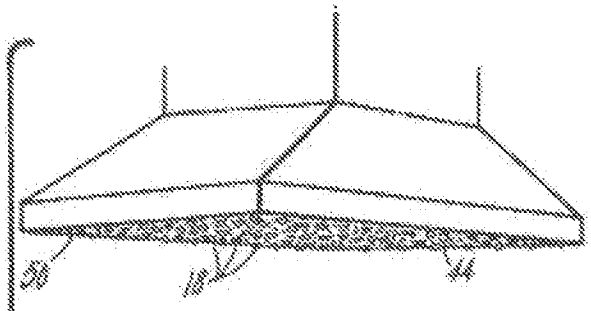
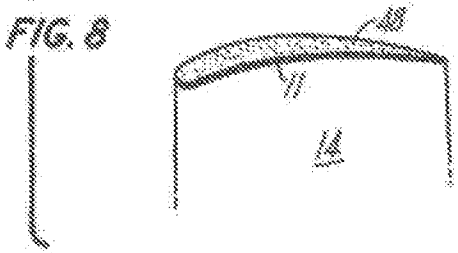


FIG. 8



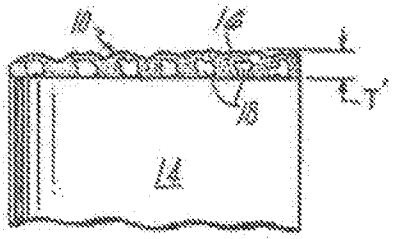


FIG. 9

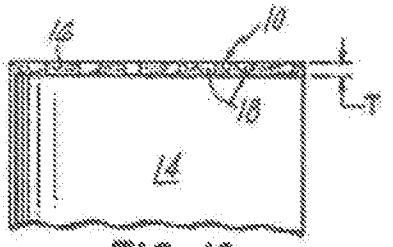


FIG. 10

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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.

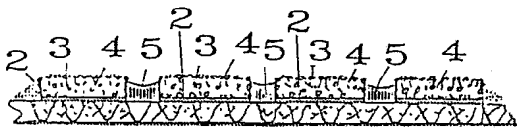


Fig. 1

EP 0 280 657 A2

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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

EX1057, Page 195

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

EX1057, Page 196

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:

EX1057, Page 197

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

EX1057, Page 199

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.

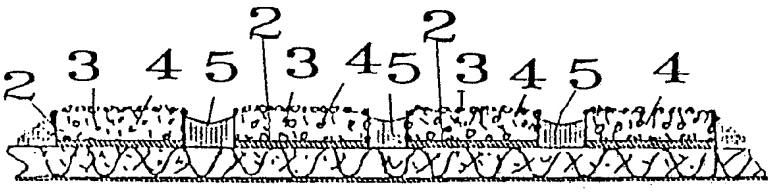


Fig. 1

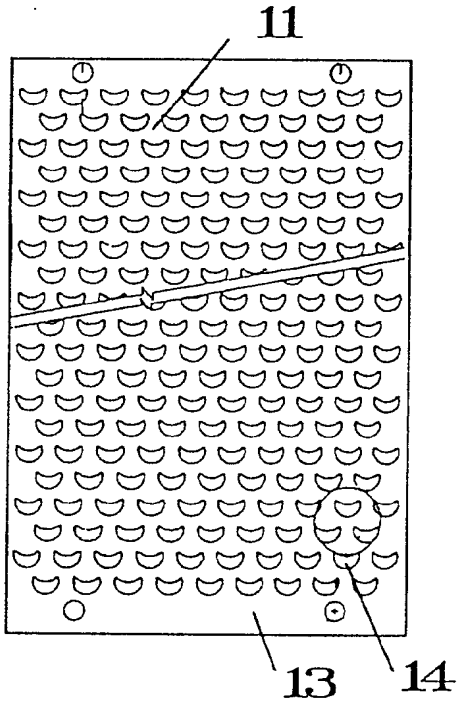


Fig. 2

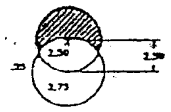


Fig. 3a

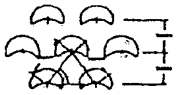


Fig. 3b

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(54) 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 diamond, 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 various 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 backing 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 80 millimeters in diameter, see for example United States Patent Application Serial Number 909,168, 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-

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 obtains 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 797,098; 794,569 and 797,568, 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

EX1057, Page 204

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 binds 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 deposition 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 650 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 8,000 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

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

EX1057, Page 206

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 infiltration 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 850 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 for the binder system tested. Coatings of less the 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

or hot isostatic pressing of the cold pressed part. If processed by solid state sintering, the materials used for casting 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 aggregate.

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 perspective, 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

EX1057, Page 208

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 80% 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 example, 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 starting 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 configuration and content of the final part. Since one of the advantages of the present invention is the ability to form large TSPCD structures, 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 both 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 dissolved 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

EX1057, Page 209

micron and up to 80 microns. Thicker coatings may be used but do not seem to increase the bond stregth over that aclieved 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 absolutely 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 ,esulting 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 infiltrant includes copper or nickel or alloys thereof, as is normally the case in abrasive 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 subsequent processing problems, even if coated with an oxidation protective layer. Thicker 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 carbids 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,

EX1057, Page 210

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-lithium-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 80 degrees 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 600 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 compaction 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 temperature 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 completely 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 tilled 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

EX1057, Page 211

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 60 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 60 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 69 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 preferred 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.

EX1057, Page 212

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 illustrated 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 of 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.

EX1057, Page 213

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 plurality 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 81b-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, 81ea 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 fabrication of products that are other than abrading or cutting tools, 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 600 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.

EX1057, Page 214

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 28,000 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 combinations thereof, thinner coatings of one mi-

EX1057, Page 215

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 18 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 18 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 letter 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 176.

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 178, 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

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 179 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 178a, 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 208.

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 bodies 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.

EX1057, Page 217

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 simultaneously bonded to a supporting 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 3. 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 hexagonal 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

800 degrees 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 though 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.

EX1057, Page 219

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 80% 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 80% 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 cutter 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.

Claim 31. An earth boring bit as set forth in claim 28 wherein said cutter element is of a diameter greater than 14mm.

Claim 32. An earth boring bit as set forth in claim 28 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 thickness 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 powder 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 80% 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 800 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.

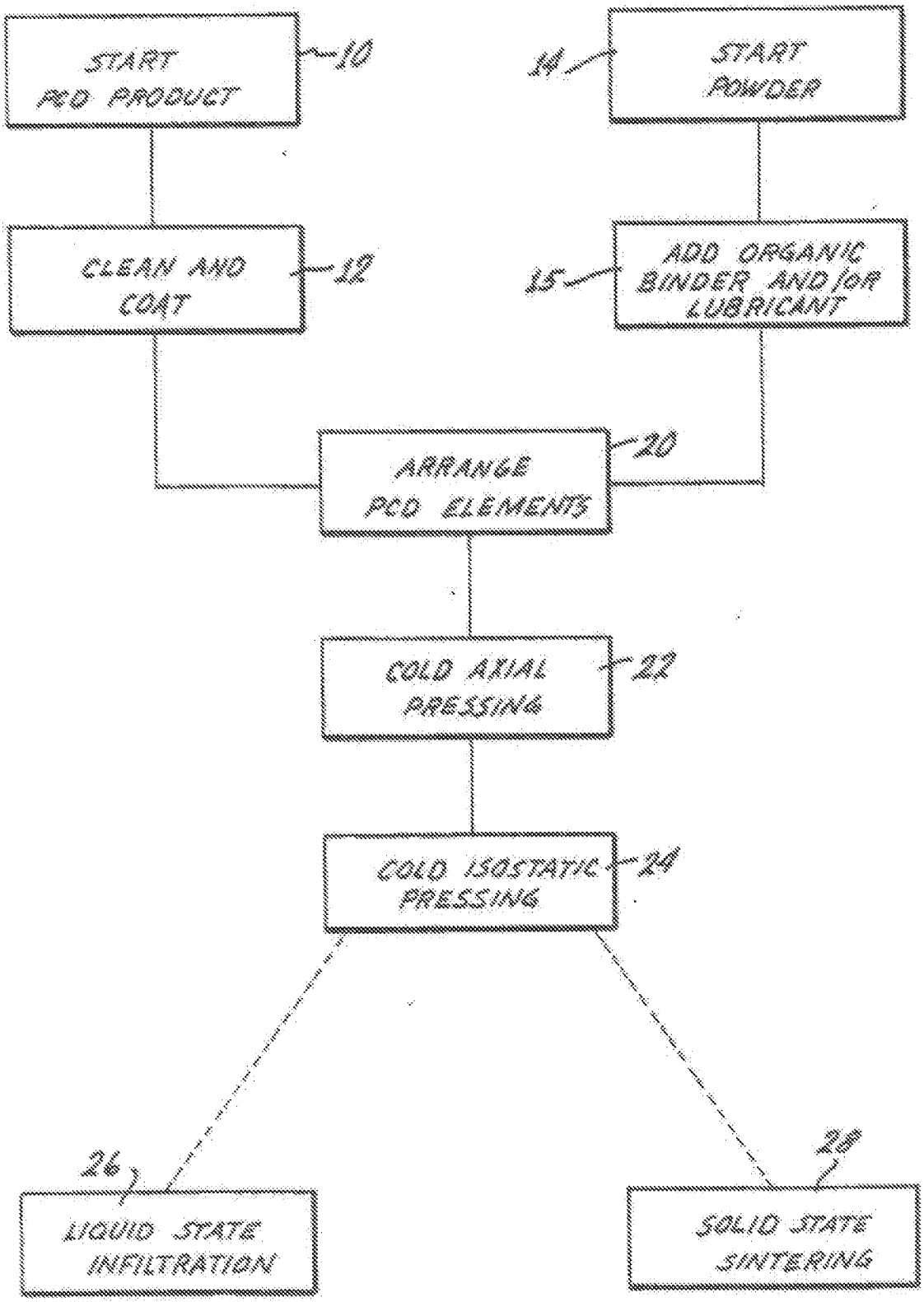
Claim 38. A method as set forth in Claim 37 wherein said elements are assembled such that there is a gap between adjacent elements.

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.

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

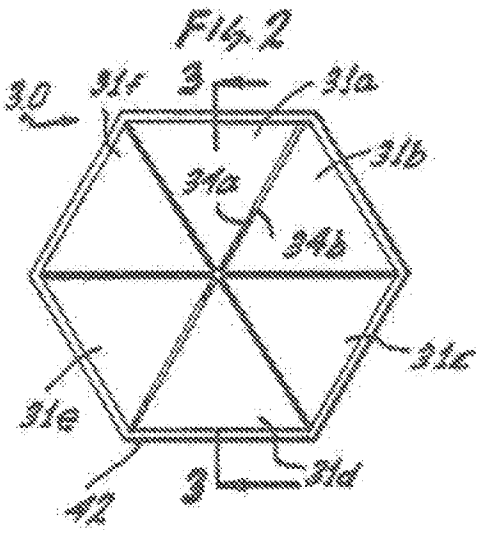


Fig. 2

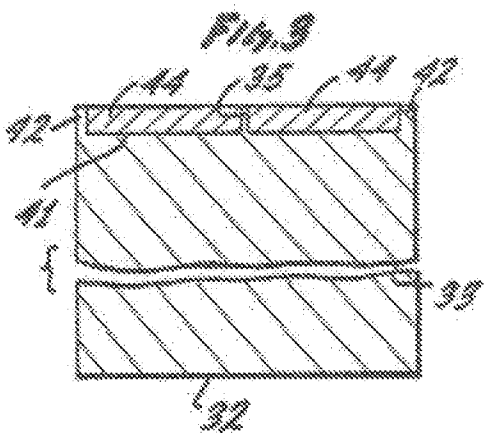


Fig. 3

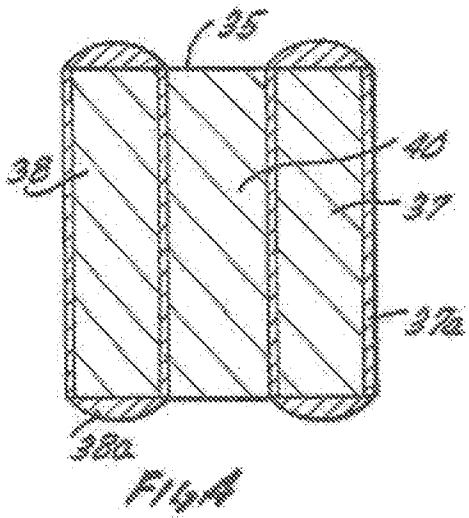


Fig. 4

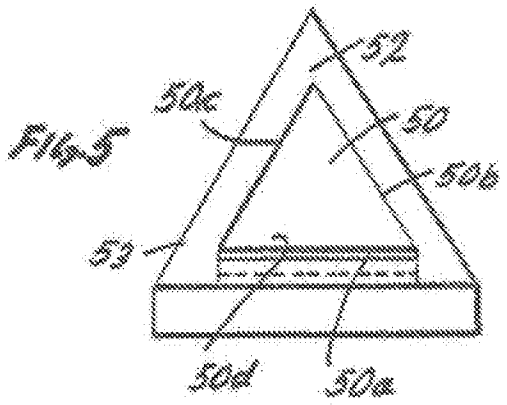


Fig. 5

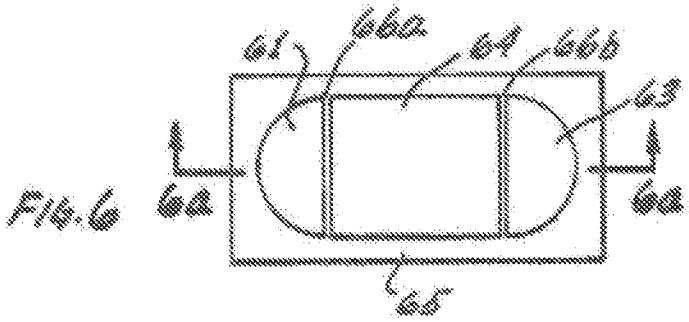


Fig. 6

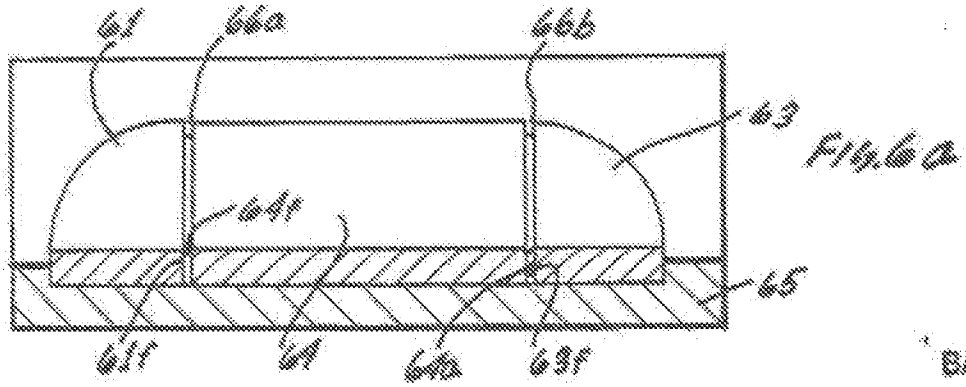


Fig. 6a

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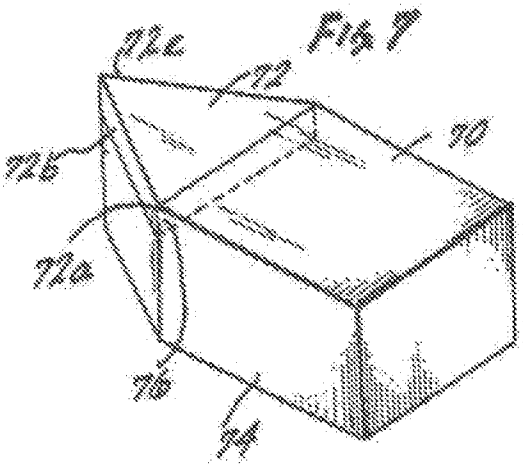


Fig. 7

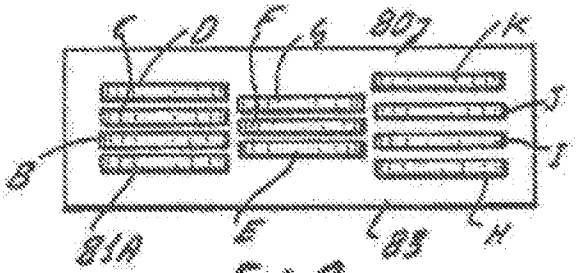


Fig. 8

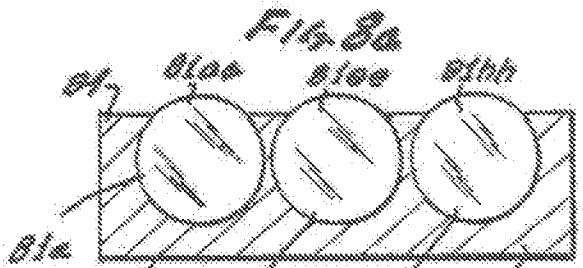


Fig. 8a

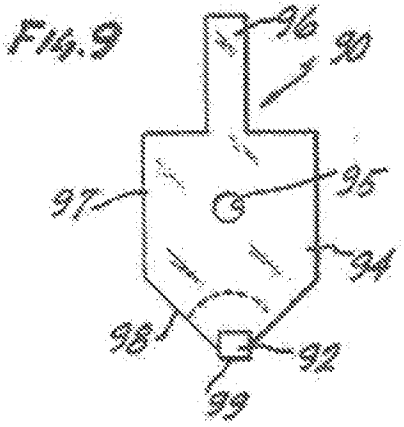


Fig. 9

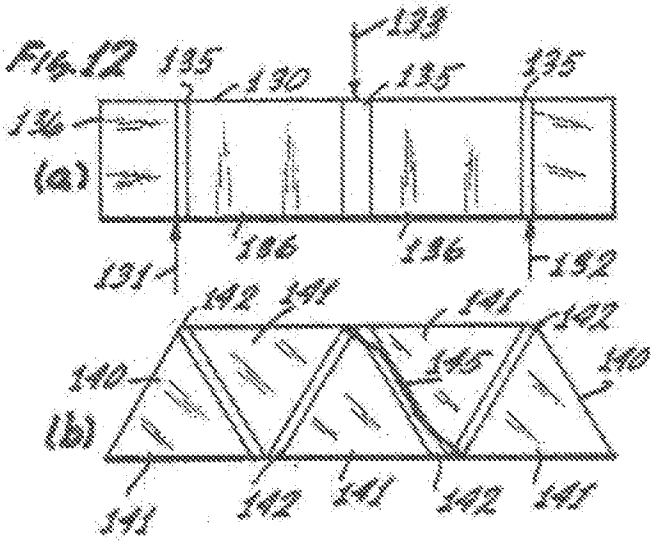


Fig. 12

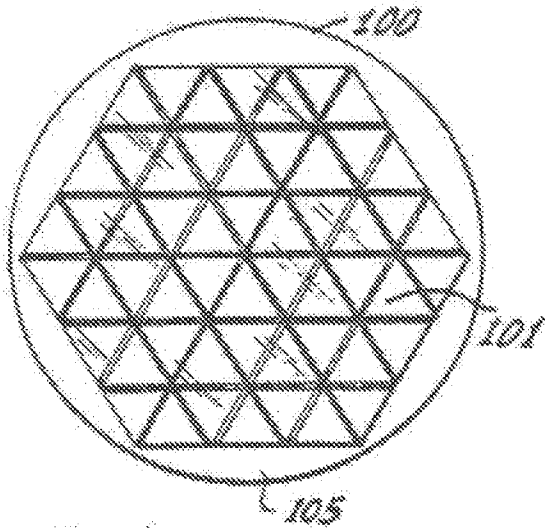
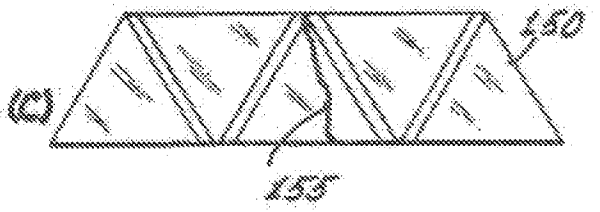


Fig. 10





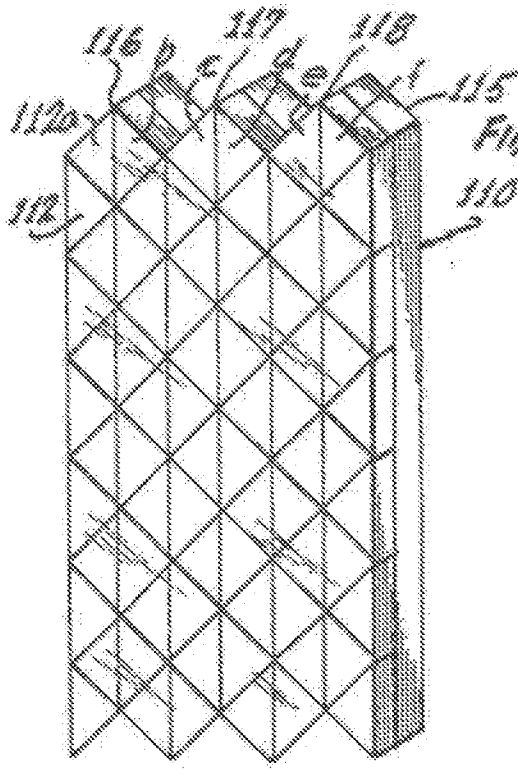


Fig.11

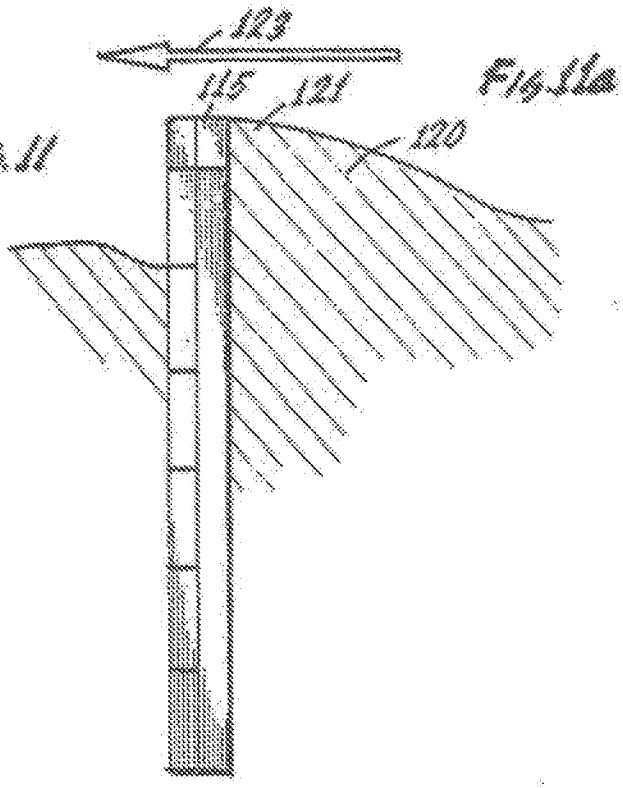


Fig.11a

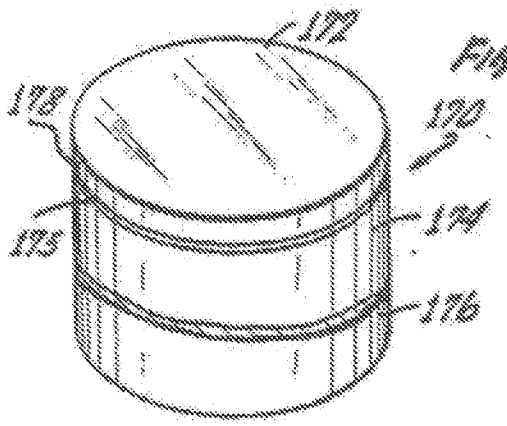


Fig.13

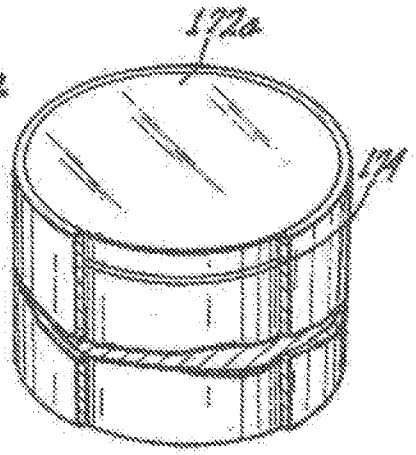


Fig.12a

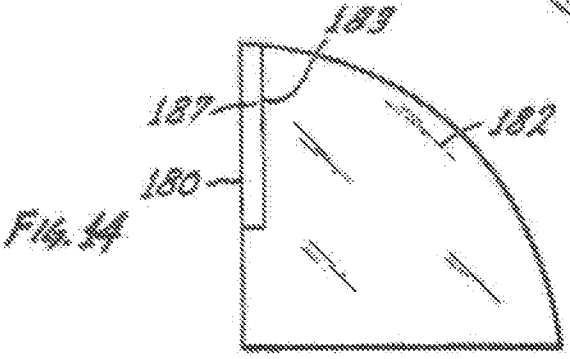


Fig.14

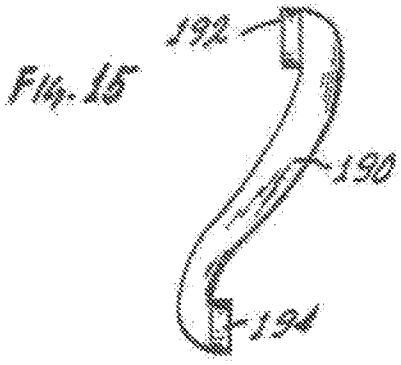


Fig. 15

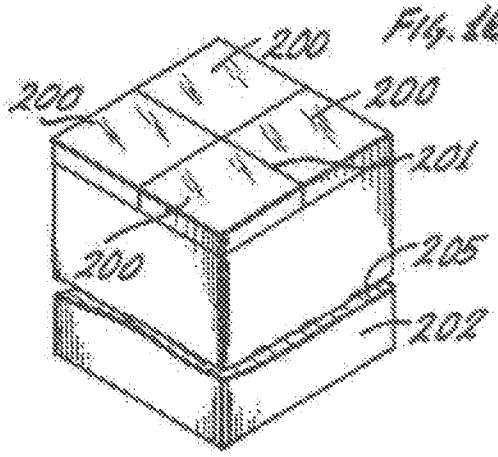


Fig. 16

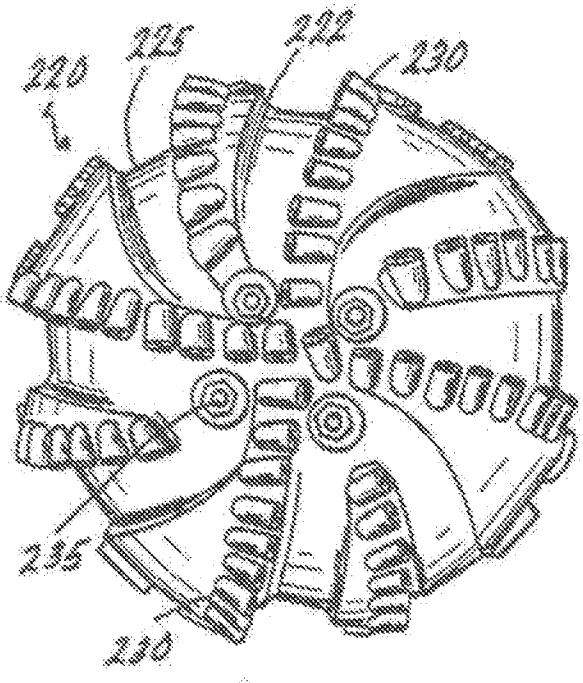


Fig. 17

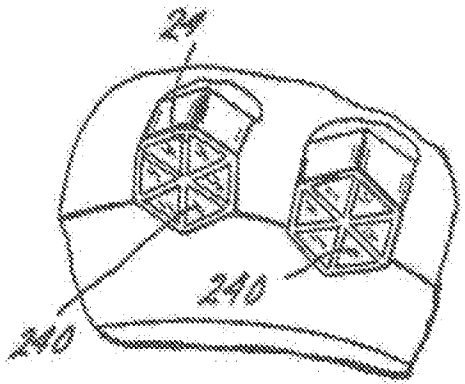


Fig. 18

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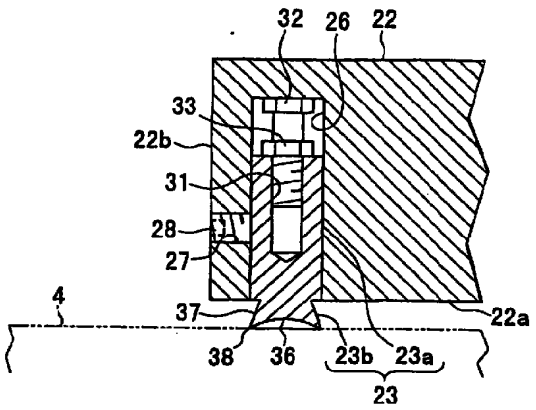
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(54) Dresser and dressing apparatus

(57) A dresser for dressing a polishing pad used for polishing a material to be polished is disclosed, the dresser comprising: a dresser principal and a dressing-working part which is provided as a body other than the dresser principal; and a holding means for holding the dressing-working part in such a manner that the dressing-working part can be attached to and detached from the dresser principal.

FIG. 2



EP 1 075 898 A2

Description

BACKGROUND OF THE INVENTION

Field of the invention:

[0001] The present invention relates to a dresser and a dressing apparatus for dressing the surface of a polishing pad which is used for polishing materials to be polished, for example, semiconductor wafer or the like.

[0002] The present specification is based on Japanese patent applications (N0. HEI 11-229495, N0. HEI 11-271176, N0. 2000-131784 and N0. 2000-131785) and the contents of these applications are incorporated herein as a part of the present specification.

Description of the Related Art:

[0003] A chemical and mechanical polishing method (CMP method) which provides a high flatness is used for polishing the surface of materials to be polished for which excellent polishing accuracy is required, for example, for polishing semiconductor wafers or the like.

[0004] The CMP method is a method where the surface of wafer is polished chemically and mechanically to obtain a flatness of the surface, using an alkali slurry containing SiO_2, a neutral slurry containing CeO_2, an acid slurry containing Al_2O_3 or a slurry containing abrasive grains. For example, the following polishing apparatus is known as an apparatus for polishing the surfaces of wafers.

[0005] As schematically shown in Fig 17 of an enlarged perspective view of an important part, a polishing pad 4 comprising, for example, hard urethane, is provided on a disc-shaped platen 3 attached to a center shaft 2 in a polishing apparatus 1 and a wafer-polishing head 5 which holds a wafer 6 is disposed facing the polishing pad 4 and in a position eccentric from the center shaft 2 of the platen 3 to the polishing pad such that the head can be rotated on its own axis.

[0006] In the polishing apparatus 1, the polishing pad 4 and the wafer 6 are moved relatively each other while the wafer 6 is held in the state of being pressed against the polishing pad 4 by the wafer-polishing head 5 and the above-mentioned slurry S is interposed between the wafer 6 and the polishing pad 4, thereby to polish one surface of the wafer 6.

[0007] A lot of fine pores, channels or the like are provided on the polishing pad 4 for holding the slurry S, whereby the wafer 6 is polished by the slurry S hold on the polishing pad 4.

[0008] However, repeating the polishing causes the decrease of the surface flatness of the polishing pad 4 and causes clogging and dulling thereof with the result of bringing about a problem to decrease the polishing accuracy and efficiency of the wafer 6.

[0009] Accordingly, a dressing apparatus 7 has conventionally been arranged in a CPM apparatus as shown in Fig 17, thereby to dress the surface of the polishing pad 4.

[0010] In the above-mentioned dressing apparatus, a dresser 11 is, through an arm 9, attached to a rotary shaft 8 which is disposed outside a platen 3, while the dresser 11 is moved to perform a reciprocating and oscillatory motion on the rotating polishing pad 4 by rotating the arm 9 with the rotary shaft 8 to shave a slight amount of the surface of the polishing pad 4, whereby the surface flatness and the like of the polishing pad are recovered and maintained, and the clogging, dulling and the like are dissolved. The surface of the polishing pad is shaved off little by little in such a manner that while pressed against the surface of the polishing pad 4, the dresser 11 is rotated by a friction force between the dresser and the polishing pad or is rotated by being driven by a drive unit which is not shown in the drawing.

[0011] The conventional dresser 11 has a nearly circular shape having an opening in the center, for example, as shown in Fig.18 (a) of a side cross-sectional view and Fig.19 of a plane view and is provided with a dresser principal 11a wherein a projection 11b is formed in the outer peripheral portion of one end face. As shown in Fig.18 (b) of a partially enlarged side sectional view, the conventional dresser having a dressing-working part is used which comprises a diamond electrodeposited part 11d formed by electrodepositing on the projection 11b abrasive grains 11c (super abrasive grain) such as diamond or cBN. The dressing-working part shaves little by little the polishing pad 4 (such a dresser is called electrodeposited grindstone). Fig.19 is a plane view illustrating the under surface of the dresser 11. A dressing apparatus using this type of dresser is disclosed in, for example, JP-A 9-168961.

[0012] In the conventional dresser 11, there has been encountered the dropping off of the abrasive grains 11 such as diamond grain during dressing. In this case, a trouble is brought about due to scratching the surface of the wafer 6, when the wafer is polished while the dropped grains are filled up in the surface of the polishing pad.

[0013] In order to dissolve the above-mentioned problem, there was a proposal in JP-A 10-217103 as in Fig.20. A dresser 12 shown in Fig.20 has the following construction as a dressing-working part: a lot of projections 13 having a shape of pyramid are formed in the surface of a metal base 12a by machining and a surface 13a of the metal base 12a having the formed projections 13 was coated by a hard film. Dressing is carried out by pressing the projections 13 against a polishing cloth (polishing pad). There is no fear in the dressing apparatus having the dresser 12 for dropping of abrasive grains such as diamond grain with the result that there is little anxiety for suffering from a damage in the surface of materials to be polished.

[0014] However, in the dressers 11 and 12 as men-

EX1057, Page 228

tioned above, a cutting quality degreases due to reasons such as wear of the dressing-working part in proportion to repeated dressings. In this case, changing a dresser unit is required, which takes a high cost.

[0015] In addition, as shown in Fig.19, the dressing of the polishing pad 4 is carried out by the whole surface of the dressing-working part which is projected in a shape of ring in the under surface of the outer peripheral portion of a dresser principal 11a. Chatter is apt to be caused due to a force acting all over the whole surface of the dressing-working part. Further, if once the chatter is caused, the vibration of the dressing 11 continues due to reasons such as the resonance of the vibration, which causes cases leading to a failure to polish uniformly the surface of the polishing pad.

[0016] Further, chips generate from the polishing pad 4 shaved little by little during dressing with dresser 12. In the above-mentioned construction having a lot of projections 13, the chips clog the spaces between each projection, thereby to bring about a problem of degreasing the dressing performance.

[0017] Furthermore, it is difficult to maintain always a stable dressing performance, as it is difficult to estimate how much the wear of the hard film 14 proceeds with the passage of time and a criterion for use becomes unclear.

[0018] The Constitution of the dresser 11 is shown in a side sectional view illustrated in Fig.21 (a schematically illustrated view). A dresser 11 is attached to an arm 9 by the aid of a setting shaft 10 and on a dresser principal 11a connected to the setting shaft 10, there is formed a diamond-electrodepositing part 11d (an abrasive grain layer) including abrasive grains 11c, thereby to form a dressing-working face 15. The diamond-electro-depositing part 11d has the following construction: electroplating is carried out on a ground face positioned on the dresser principal 11a, whereby the abrasive grains 11c such as diamond, cBN or the like are fastened dispersedly on the ground face.

[0019] In the dresser 11, the dressing-working face 15 comprises the under surface of the diamond-electrodepositing part 11d (an abrasive grain layer). In Fig.21, the thickness of the diamond-electro-depositing part 11d and the size of abrasive grains are illustrated larger than actual ones so as to understand easily the shape of the dressing-working face 15.

[0020] If the dresser 11 contacts partially the polishing pad 4 during dressing the pad 4 with the dresser 11, the contact area of the dressing-working face 15 to the polishing pad 4 becomes small with the result that the dressing efficiency becomes lower and it takes a longer time to dress the pad than in unpartial contact cases, though the pad 4 can be dressed at any rate. Every time after polishing a wafer 6 has finished, the polishing pad 4 is dressed and it is carried out at every two and half to three minutes. Therefore, the working efficiency of polishing the wafer 6 becomes low when it takes a long time to dress the polishing pad 4.

[0021] Still further, when the dressing condition is unstable as mentioned above, the surface condition of the polishing pad varies widely and the polishing condition of the wafer 6 with the polishing pad 4 scatters. As the result, it becomes difficult to keep the rate of operation of CMP apparatus and the flatness of the wafer 6.

[0022] Because of the above-mentioned reasons, in order to stabilize the dressing condition, an arrangement is carried out such that the dressing-working face 15 is flat and the dressing-working face cross orthogonally the rotary axis of the dresser 11.

[0023] In a dresser having a prescribed shape formed by fixing abrasive grains by a binder, the above-mentioned arrangement is available by shaving the dressing-working face in the state that the dresser is attached to the setting shaft of a dressing apparatus 7 (this treatment is called "tooling"). However, the tooling can not be carried out in the case of an electrodeposited grindstone due to its own property.

[0024] Accordingly, in the case of using an electrodeposited grindstone for the dresser 11, it is necessary to increase the shape accuracy of the dresser principal 11a where the abrasive grain layer is formed and to increase the accuracy of attaching the dresser principal 11a to the setting shaft.

[0025] However, there are cases in the electrodeposited grindstone that the shape of the dresser principal 11a changes during the process of making thereof and the flatness of the dressing-working face 15 becomes inferior. The flatness of the dresser 11 is measured while contacing directly a dial gauge to the dressing-working face 15, and in the case of the electrodeposited grindstone, as the surface roughness of the dresser-working face is coarse which is a degree of #100, the measurement error is large and it is difficult to measure the flatness thereof accurately.

[0026] Further, there are cases during attaching the dresser 11 to the setting shaft that the dressing-working face 15 of the dresser 11 inclines against the rotary axis due to troubles such as an attaching error caused by a worker, dust interposed between the dresser 11 and the setting shaft, and so on. In order to detect the inclination, there is no way except measuring the deviation of the dressing-working face 15 while contacting directly the dial gauge to the dressing-working face, which has a problem in having a large measurement error. Therefore, it is difficult to obtain the attaching accuracy within a prescribed criterion.

[0027] In addition, as the polishing pad has a softness, it is difficult to detect the partial contact of the dresser 11 to the polishing pad 4 by observing the dressed state of the polishing pad 4, which is different from polishing materials having rigidity such as metal.

[0028] As mentioned above, it has been difficult to stabilize the dressing condition of the polishing pad 4 with the dresser 11.

EX1057, Page 229

SUMMARY OF THE INVENTION

[0029] An object of the present invention is to provide a dresser which can be used economically and a dressing apparatus having the same.

[0030] In order to attain the above-mentioned object, the dresser of the present invention is one used for dressing a polishing pad for polishing materials to be polished and comprises a dresser principal, a dressing-working part which is provided as a body other than the dresser principal and a holding means for holding the dressing-working part in such a manner that the dressing-working part can be attached to and detached from the dresser principal.

[0031] According to the dresser of the present invention, the dressing-working part can be attached and detached, and when a dressing performance thereof degreases due to reasons such as the wear of the dressing-working part, the dressing-working part is exchanged for a new one to recover the dressing performance. In this way, the dressing performance can be recovered only by exchanging a dressing-working part, thereby to degrease a cost for a dresser.

[0032] Another object of the present invention is to provide a dresser which enables a vibration hardly to take place, and which enables to suppress the vibration within a short time if the vibration takes place, and is to provide a dressing apparatus having the same.

[0033] In order to attain the above-mentioned object, the dresser of the present invention is one used for dressing a polishing pad for polishing materials to be polished and comprises a plurality of dressing-working parts which are disposed at unequal spaces in the circular direction of a dresser principal.

[0034] According to the dresser of the present invention, the dressing-working parts are disposed at unequal spaces in the circular direction of the dresser principal, whereby forces which cause the vibration of the dresser are not brought about periodically and are brought about aperiodically to suppress the resonance of the dresser.

[0035] As the result, a vibration hardly takes place and the vibration can be suppressed within a short time if the vibration takes place.

[0036] The interval between each dressing-working part can be decided by any way and for example, can be decided by using random numbers.

[0037] As another way, the interval can be unequal by arranging positions in such a manner that positions are designated as a standard position in the case of disposing dressing-working parts at an equal space each other, and each position is deviated from each standard position to the circular direction. In this case, the deviation of each dressing-working part from the corresponding standard position can be decided by using the random numbers or, for example, can be decided by designating as a starting point any position among each standard position, designating as θ an angle between the starting point and each standard position when looked from the center of axis, and specifying $x=\sin\theta$ as the deviation x of each dressing-working part from each standard position (or $x=\cos\theta$).

[0038] In the above-mentioned ways, when the deviation is too large, dressing-working parts having the too large deviation have a life shorter than the other dressing-working parts as a result of dressing more amount of a polishing pad. In contrast, when the deviation is too small, an effect on suppressing a resonance is lowered. Therefore, the deviation value is preferably by the absolute value within 10 to 30% of an interval between the corresponding standard positions.

[0039] The dresser is rotated in a position having an eccentric relation with the polishing pad, and the speed of the dresser relative to the polishing pad is lower in half a range of the circular direction (an inward peripheral side or an outward peripheral side of the polishing pad when looked from the center of axis of the dresser) than in the other half range and a load imposed on the dressing-working part is small. Therefore, it is estimated that the vibration of the dresser is hardly caused by a force imposed on a side which has a lower speed relative to the polishing pad. That is, dressing-working parts can be provided which are disposed in a same pattern every half a round.

[0040] A further object of the present invention is to provide a dresser which enables to always maintain an excellent dressing performance and can be used economically.

[0041] In order to attain the above-mentioned object, the dresser of the present invention is applied to an equipment which is used for polishing materials to be polished having a flat surface such as semiconductor wafer with a dressing pad, and is a dresser used for dressing the surface of the polishing pad, and is characterized by the following construction: the cutters are arranged in such a manner that the cutters are projected from the under surface of a dresser principal; the polishing pad surface is shaved by moving the edge of cutter which is a movement relative to and parallel to the polishing pad surface while the edge of the cutter is pressed against the polishing pad surface.

[0042] As the dressing pad is shaved with a cutter in the dresser of the present invention, the dresser is protected from the lowering of the dressing performance which is caused by clogging of the polishing pad with the shaved chips of the dressing pad in between abrasive grains, as compared with the conventional one, whereby a stable dressing performance is available. In addition, as an amount of wear can be decided from the flank of the cutter, a criterion for use is clear as compared with the conventional one, thereby to enable to maintain easily the dressing performance. Further, as a cutter is used, reusing the cutter is possible, which is different from the conventional one, with result of providing excellence in a cost for the dresser.

[0043] Still further object of the present invention is

EX1057, Page 230

to provide a dresser wherein the polishing condition of the polishing pad can be stabilized.

[0044] In order to attain the above-mentioned object, the dresser of the present invention comprises: a dressing-working face formed by providing an abrasive grain layer including abrasive grains on the surface of a dresser principal, wherein the dresser principal comprises a ground face on which the abrasive grain layer is provided and a basic face which is disposed in a portion of the dresser principal more inward than the ground face and is formed in parallel to the ground face.

[0045] In the dresser of the present invention, as the basic face of the dresser principal is a plane parallel to the ground face, it is possible to predict the flatness of the ground face having the abrasive-grain layer formed thereon, that is, the flatness of the dressing-working face, by measuring the flatness of the basic face, whereby dressers can be selected which have a good flatness with regard to a dressing-working face.

[0046] In addition, when the dresser is attached to the dressing apparatus, the attaching situation can be confirmed according to the basic face as a basis, as the basic face is a plane parallel to the ground face.

BRIEF DESCRIPTION OF THE DRAWINGS

[0047]

Fig.1 is a plan view illustrating the under surface of the dresser of Example 1 of the present invention.

Fig.2 is a sectional view looking in the direction of the arrows A-A in the Fig.1.

Fig.3 (a) is a side sectional view showing another embodiment of the dresser-working part used in the dresser of Example 1.

Fig.3 (b) is an end surface view showing another embodiment of the dresser-working part used in the dresser of Example 1.

Fig.4 (a) is a side sectional view showing a further embodiment of the dresser-working part used in the dresser of Example 1.

Fig.4 (b) is a side sectional view showing still another embodiment of the dresser-working part used in the dresser of Example 1.

Fig.5 is a side sectional view illustrating another construction of the dresser in Example 1 of the present invention.

Fig.6 is an under surface view illustrating the shape of the dresser in Example 2 of the present invention.

Fig.7 (a) is a side sectional view illustrating the shape of the dresser in Example 2 of the present invention.

Fig.7 (b) is a side sectional view illustrating another shape of the dresser in Example 2 of the present invention.

Fig.8 is an under surface view illustrating the shape of the dresser in Example 3 of the present invention.

Fig.9 is a schematic view illustrating the dresser of Example 4 of the present invention, which is an enlarged side view of the important part of the dresser principal.

Fig.10 is a schematic view illustrating the dresser of Example 4 of the present invention, which is an under surface view of the dresser principal.

Fig.11 is a schematic view illustrating the dresser of Example 4 of the present invention, which is a side sectional view of the dresser principal.

Fig. 12 is an enlarged side view of a cutter used in the dresser shown in Figs.9 to 11.

Fig.13 is an enlarged under surface view of the cutter used in the dresser shown in Figs.9 to 11.

Fig.14 is a plan view showing a positional relationship between the dresser principal shown in Fig.9 to 13 and a platen to which a polishing pad is attached, when dressing is carried out.

Fig.15 is an enlarged side sectional view showing another embodiment of the cutter used in the dresser of Example 4.

Fig. 16 is a side sectional view illustrating the constitution and construction of the dresser in Example 5 of the present invention.

Fig.17 is an enlarged perspective view of the important part of the conventional polishing equipment and dressing apparatus, which is illustrating the schematic constitutions thereof.

Fig.18 (a) is a side sectional view illustrating the shape of the conventional dresser.

Fig. 18 (b) is a partially enlarged side sectional view of Fig. 18 (a).

Fig.19 is a plan view showing the under surface of the conventional dresser.

Fig.20 is an enlarged side sectional view of an important part illustrating the shape of the conventional dresser.

Fig.21 is a side sectional view illustrating the constitution and construction of the conventional dresser.

DESCRIPTION OF PREFERRED EMBODIMENTS

[0048] The dresser of the present invention and the dressing apparatus having the same are described in detail hereunder.

[0049] Fig.1 is a plan view illustrating the under surface of the dresser of Example 1 of the present invention. Fig.2 is a sectional view looking in the direction of the arrows A-A in the Fig.1.

[0050] The dressing apparatus of the present invention comprises a constitution almost similar to that of, for example, a dressing apparatus 7 shown Fig.17, and a dresser 21 of the present invention is used as a dresser in place of a dresser 11.

[0051] The dresser 21 of the present invention comprises a dresser principal 22 having an approximately circular shape and a plurality of dressing-working parts

EX1057, Page 231

23 disposed in the circular direction in the under surface of the outer peripheral portion of the dresser principal 22. The dresser-working parts 23 which are provided as bodies other than the dresser principal 22 are arranged and are held to the dresser 22 by a means mentioned after in such a manner that the dresser-working parts 23 can be attached to and detached from the dresser principal 22. The number and disposition of the dressing-working parts 23 shown in Fig.1 is only an example. The dressing-working parts 23 can be disposed at equal spaces in the circular direction of the dresser principal or at irregular spaces in the circular direction. The dressing-working parts 23 can be arranged in a plurality of lines in the circular direction of the dresser principal 22, which is similar to that of the conventional dresser.

[0052] In the present example, the dresser principal 22 is composed of an approximately circular shape having an opening in the center thereof.

[0053] As shown in Fig.2, the dressing-working part 23 is composed of a member having an approximately pin-like shape. A receiving hole 26 for receiving a part of the dressing-working part 23 is formed from the under surface 22a to a predetermined depth in the dresser principal 22. In the dresser principal 22, a tapped hole 27 is formed which leads to the intermediate position of the receiving hole 26 from the outer periphery 22b thereof. In the tapped hole 27, a clamp screw 28 is screwed such that the end of the clamp screw can be advanced and retreated in the receiving hole 26.

[0054] The dressing-working part 23 is fixed in the receiving hole 26 by pressing the side of dressing-working part 23 received in the receiving hole 26 to the inner wall of the receiving hole with the end of the clamp screw 28 and is released from being fixed by releasing the pressing of dressing-working part 23 with the end of the clamp screw.

[0055] The present example comprises a holding means which hold dressing-working part 23 to the dresser principal 22 in such a manner that the dressing-working part 23 can be attached to and detached from the dresser principal 22 by the receiving hole 26, the tapped hole 27 and the clamp screw 28.

[0056] Here, in the present example, the receiving hole 26 is approximately parallel to the axis of the dresser principal 22 and the bottom surface thereof is a plane which approximately crosses orthogonally the axis of the dresser principal 22.

[0057] As shown in Fig.2, the dressing-working part 23 is a cutter having approximately a pin-like shape, and comprises a base 23a having an approximately columnar shape and a cutting edge 23b formed in the end of the base 23a.

[0058] The base 23a is received in the receiving hole 26 of the dresser 22 and the side of the base is subjected to the pressure given by the clamp screw 28. A position-adjusting screw hole 31 is formed in the base 23a, which has a prescribed depth along the axis from the end face of the base.

[0059] A position-adjusting screw 32 is screw-clamped to the position-adjusting screw hole 31 and an amount of projection of the position-adjusting screw 32 from the base 23a can be adjusted by controlling a screwed length to the position-adjusting screw hole 31.

[0060] The upper surface of the head of the position-adjusting screw 32 is pressed against the bottom surface of the receiving hole 26 when the dressing-working part 23 is received in the receiving hole 26. An amount of projection of the dressing-working part 23 from the receiving hole 26, that is, an amount of projection of the dressing-working part 23 from the dresser principal 22 can be adjusted by controlling an amount of projection of the position-adjusting screw 32 from the base 23a. In the present example, the upper surface of the head of the position-adjusting screw 32 is a plane crossing approximately orthogonally to the axis thereof.

[0061] Further, a fixing nut 33 is screw-clamped to the position-adjusting screw 32 which is positioned on the position-adjusting screw hole 31. The position-adjusting screw 32 is fixed in a state projected at any distance from the base 23a by screwing the fixing nut toward the edge of the position-adjusting screw 32 while pressed against the base 23a.

[0062] The present example comprises a projection adjusting means having the position-adjusting screw hole 31, the position-adjusting screw 32 and the fixing nut 33 for controlling an amount of projection of the dresser-working part 23 from the dresser principal 22.

[0063] The cutting edge portion 23b of the dressing-working part 23 comprises: a flank 36 which is formed by concaving one end face of the cutter toward the base end of the cutter; a rake face 37 comprising a cicumferential surface in the end portion of the cutter; and a cutting edge 38 which is a ridgeline formed by crossing of the end face and the circumferential surface in the end portion of the cutter. Namely, the cutting edge portion 23b has a nearly Japanese soup cup-like shape.

[0064] Here, ceramics, diamond, cBN and the like can be used for the cutting edge 38, and the surface of the cutting edge portion 23b can be covered by a hard film comprising hard materials such as diamond or the like.

[0065] Almost in the same manner as in the dressing apparatus shown in Fig. 17, the surface of a polishing pad 4 is dressed by the dressing apparatus of the present invention, which comprises the steps of: oscillating a dresser 21 while the dresser 21 is pressed against the surface of the polishing pad 4; rotating of the dresser owing to a friction force caused by friction with the polishing pad 4 or rotating the dresser by driving by a drive unit which is not shown in Figs while the dresser 21 is pressed against the surface of the polishing pad.

[0066] In the dresser 21, the surface of the polishing pad 4 is shaved little by little by the dresser 21 by providing to the dresser a relative movement to a direction parallel to the polishing pad 4, while the cutting edge 38 of the dressing-working part 23 is pressed

EX1057, Page 232

against the surface of the polishing pad 4.

[0067] As an amount of projection of the dressing-working part 23 from the dresser principal 22 can be adjusted, the dressing performance of the dresser 21 is controlled by adjusting the depth of cut of the dressing-working part 23 into the dressing pad 4 according to the material and hardness of the dressing pad.

[0068] The dressing apparatus of the present invention can be composed of a constitution other than the above-mentioned one, in which the dresser 21 is connected to an arm 9 through a ball bearing, and the dresser can freely be inclined according to the surface of the polishing pad 4. In this case, in the dressing apparatus, the dresser is pressed against the dressing pad 4 by the dead weight thereof or by a force obtained by pressing the arm 9, and a depth of cut of the dressing-working part 23 into the polishing pad 4 is adjusted by controlling an amount of force of the pressing.

[0069] Adjusting an amount of projection of the dressing-working part 23 is carried out as follows: first, an amount of the position-adjusting screw 32 from the base 23a is adjusted by turning round the position-adjusting screw 32 which is screw-clamped to the base 23a to any of two directions in the state of detaching the dressing-working part 23 from the dresser principal 22 and the position-adjusting screw 32 is fixed to the base 23a by the fixing nut 33; then, the dressing-working part 23 is inserted into the receiving hole 26 of the dresser principal 22 and the clamp screw 28 is driven and tightened which is screw-clamped to the tapped hole 27, whereby the dressing-working part 23 is fixed in such a state that the cutting edge 38 of the dressing-working part 23 is projected from the under surface 22a of the dresser principal 22 by a prescribed amount.

[0070] In addition, as the dressing-working part 23 has the cutting edge 38 in the whole round thereof, the shaving of the polishing pad 4 can be carried out for any direction thereto, thereby to enable to increase a dressing performance.

[0071] And, as the dressing-working part 23 is a cutter, the dressing performance can be recovered by repolishing the cutter edge 38 when the dressing performance degreases, thereby to enable to lower a cost for the dresser.

[0072] Furthermore, though shaved chips generate due to shaving a little the polishing pad 4, the polishing pad 4 is hardly clogged with the shaved chips, which is different from a dresser having a dressing-working part comprising an abrasive grain layer and projections, thereby to obtain a stable dressing performance.

[0073] Still further, as the dressing performance can be judged from an amount of wear of the cutting edge 38 to clear a criterion for use, the stable dressing performance can always be obtained.

[0074] Yet further, there is no fear to scratch the surfaces of materials to be polished due the dropping of abrasive grains, as the abrasive grains are not used in the dressing-working part 23.

[0075] Still furthermore, if a dressing performance lowers due to reasons such as the wear of the dressing-working part 23, the dressing performance can be recovered by exchanging only the dressing-working part 23, thereby to enable to decrease a cost for a dresser.

[0076] The example is exemplified by a case wherein the dressing-working part 23 is used which comprises a cutting edge portion 23b having a Japanese soup cup-like shape and the example is not limited to the case. For another example, a dressing-working part 39 can be used which has a plurality of projections in the end thereof as shown in Fig.3 (a) of a side sectional view and Fig.3 (b) of an end surface view. The projection 40 of a dressing-working part 39 has a cross section approximately equal to an end face 40a from one end of the projection to near another end of the projection from which the projection is started and a cutting edge 40b is provided by a ridgeline formed by crossing of the end face 40a and the side surface of the projection. The projections are disposed in any way, regularly or irregularly. In addition, any shape can be provided to the end face 40a of the projection 40, for examples, the end face 40a can be nearly circular (the projection 40 is approximately columnar) or can be a polygon (the projection 40 is a square pillar having a polygonal section).

[0077] Further, ceramics, diamond, cBN and the like can be used for the cutting edge 40b, and the surface of the cutting edge 40b can be covered by a hard film comprising hard materials such as diamond.

[0078] In the dressing-working part 39 formed as mentioned above, the surface of the polishing pad 4 is dressed by the cutting edge 40b of each projection 40. Here, as the cutting edge 40b is formed in the wholly around of each projection 40, the polishing pad 4 can be dressed from any direction. The projection 40 has a cross section approximately equal to an end face 40a from one end of the projection to near another end of the projection from which the projection is started and the change of shape and area of the end face 40a is small when the projection 40 wears with the result that the dressing performance hardly changes and the stable dressing performance can be obtained.

[0079] Further, for example, as shown in Fig.4 (a) of a side sectional view, a dressing-working part 41 can be used which has a cutting edge 42 having a cutting edge portion 41b directed to one direction of the circular direction of dressing-working part. Here, ceramics, diamond, cBN and the like can be used for the cutting edge 42 and the surface of the cutting edge portion 41b can be covered by a hard film comprising hard materials such as diamond.

[0080] In addition, the polishing pad can be shaved from any direction, as a whole dresser, by providing to the dressing-working part 41 the cutting edges 42 which are directed in various directions each other.

[0081] Further, a dressing-working part 43 can be used as a dressing-working part, which comprises a grindstone 44 in the end of the base 43a which is

EX1057, Page 233

approximately columnar as shown in Fig.4 (b). In this case, the grindstone 44 can be a grindstone which is conventionally used for dressing the polishing pad 4, for examples, can be a grindstone formed by an abrasive grain layer prepared by electrodepositing abrasive grains such as diamond or can be a metal bonded grindstone.

[0082] Further, a means for controlling an amount of projection can be composed of a constitution other than the above-mentioned one, in which a bolt inserting hole 29 leading to the upper surface of the dresser principal 22 is formed in the bottom face of a receiving hole 26 as shown in Fig.5 of a side sectional view and a position-adjusting screw 32 is inserted into the bolt inserting hole 29 from the upper surface of the dresser principal 22 and screw-clamped to a position-adjusting screw hole 31 (a fixing nut becomes unnecessary). Here, the position adjusting screw 32 is attached to the dresser principal 22 in such a manner that the screw is possible to turn around an axis while the head of the screw is received in the bolt inserting hole 29. When a means for controlling an amount of projection is provided as mentioned above, as the dressing-working part 23 can be moved along the position adjusting screw 32 by handling the position adjusting screw 32 in the state of attaching the dresser-working part 23 to the dresser principal 22, an amount of projection of the dressing-working part 23 can be controlled without detaching the dressing-working part 23 from the dresser principal 22.

Example 2:

[0083] The dresser of Example 2 of the present invention and the dressing apparatus having the same are described in detail hereunder, referring to Figs.

[0084] Fig.6 is an under surface view illustrating the shape of the dresser of Example 2. Fig.7 is a side sectional view illustrating the dresser.

[0085] The dressing apparatus of the present invention comprises a constitution almost similar to that of a dressing apparatus 7 shown Fig. 17, and the dresser 51 of the present invention is used as a dresser in place of a dresser 11.

[0086] A dresser 51 of the present invention comprises a dresser principal 52 having an approximately circular shape and a plurality of dressing-working parts B disposed in the circular direction in the under surface of the outer peripheral portion of the dresser principal 52. The dresser principal 22 of the present invention is composed of a shape having an opening in the center thereof in the same manner as in the conventional dresser. The dresser-working parts B are provided as bodies other than the dresser principal 52 and are fixed to the dresser principal 52 by, for examples, brazing, a clamp method, a blade method and so on.

[0087] In the present example, the dressing-working parts are members having a approximately pin-like shape.

[0088] The dressing-working parts B are disposed at unequal spaces in the circular direction of the dresser principal 52. A space between each dressing-working part B is decided by any way. In the present example, the positions in the case of disposing dressing-working parts B at equal spaces are designated as a standard position P and any one among the standard positions is designated as a starting point P_0 and an angle between each standard position P and the starting point P_0 when looked from the center of axis O of the dresser 52 is designated as θ , and $x=\sin\theta$ is specified as a deviation x of each dressing-working part B from each standard position P. Here, in Fig.6, accompanied characters are provided to standard positions P (and a dressing-working part B, angle θ and an amount of deviation x corresponding to each standard position P), in a counterclockwise order starting from the starting point P_0.

[0089] In the above-mentioned way, when the deviation x of a dressing-working part B from the corresponding standard position P is too large, a distance becomes large which is between one dressing-working part B and another dressing-working part B next to the former dressing-working part. In this case, the life of the dressing-working parts B become shorter as an amount of dressing of the polishing pad 4 carried out by the dressing-parts B becomes always too much as compared with the other dressing-working parts B. In contrast, when the deviation is too small, an effect on suppressing a resonance is lowered. Therefore, a deviation value of each dressing working part B from the corresponding standard position P is preferable by the absolute value within 10 to 30% of a space H between standard positions P.

[0090] The dressing-working part is approximately pin-like and can comprise any construction such as a dressing-working part having a diamond-electro-deposited part formed by electrodepositing abrasive grains such as diamond in the end which is directed to the under surface side of the dresser principal 52 or a dressing-working part having a cutter having a cutting edge formed in the end which is directed to the under surface side of the dresser principal 52 if dressing the polishing pad 4 is possible.

[0091] In the present example, as shown in Fig.7 (a), there is provided to the dresser principal 52 a receiving hole 56 having a prescribed depth from an under surface 52a and there is provided thereto a tapped hole 57 which leads to the intermediate position of the receiving hole 56 from the outer periphery 52b of the dresser principal 52 and to which a clamp screw 58 is clamped. The dressing-working part B is fixed to the receiving hole 56 by the following mechanism: the side thereof is pressed by the clamp screw 58 which is screw-clamped to the tapped hole 57, whereby the dressing-working part B is fixed to the receiving hole 56. And, as shown in Fig.7 (a), in the dressing-working part B, a position-adjusting screw hole 59 having a prescribed depth is formed along an axis from the end face

EX1057, Page 234

of the base end thereof and a position-adjusting screw 60 is screw-clamped to the position-adjusting screw hole 59. An amount of projection of the dressing-working part B from the base end can be controlled by controlling a screw-clamping length of the position-adjusting screw 60 to the position-adjusting screw hole 59, and the upper face of head of the position adjusting screw 60 can be contacted to the bottom face of the receiving hole 56 when the dressing-working B is received in the receiving hole 56. Here, an amount of projection of the dressing-working part B from the receiving hole 56, that is, an amount of projection of the dressing working part B from the under surface 52a of the dresser principal 52a can be controlled by adjusting an amount of projection of the position-adjusting screw 60 from of the base end of the dressing-working part B.

[0092] In the present example, the dressing-working part B is a cutter having approximately a pin-like shape. A cutting edge portion 54 formed in one end of the cutter comprises: a flank 54a which is formed by concaving the end face of the cutting edge portion toward the base end of the cutter; a rake face 54b comprising a circumferential surface in the end portion of the cutting edge portion; and a cutting edge 54c which is a ridgeline formed by crossing of the end face and the circumferential surface in the end portion. Namely, the cutting edge 54 has a nearly Japanese soup cup-like shape and whole the round of the edge makes it possible to shave the polishing pad 4.

[0093] For example, as shown in Fig.7 (b) of a side sectional view, the dresser 51 can be composed of a constitution other than the above-mentioned one, in which a bolt inserting hole 53 is formed in the dresser principal 52 from the bottom face of a receiving hole 56 to the upper surface of the dresser principal 52 and a position-adjusting screw 60 is inserted into the bolt inserting hole 53 from the upper side of the dresser principal 52, thereby to screw-clamped the screw to a position adjusting screw hole 59. In this case, the position-adjusting screw 60 is attached to the dresser principal 52 in such a manner that the screw is possible to turn around an axis while the head of the screw is received in the bolt inserting hole 53. When the dresser 51 is constituted as mentioned above, as the dressing-working part B can be moved along the position adjusting screw 60 by handling the position-adjusting screw 60 in the state of attaching the dresser-working part B to the dresser principal 52, an amount of projection of the dressing-working part B can be controlled without detaching the dressing-working part B from the dresser principal 52.

[0094] Dressing the polishing pad 4 by using a dressing apparatus having the dresser 51 constructed as mentioned above is carried out in the same manner as in the conventional dressing apparatus.

[0095] In the present example, as the dressing-working parts B are disposed at unequal spaces in the circular direction of the dresser principal 52, forces which cause the vibration of the dresser 51 are not brought about periodically and are brought about aperiodically to suppress the resonance of the dresser 51.

[0096] Thus, according to the dresser 51 of the present invention and the dressing apparatus having the same, the vibration of the dresser 51 hardly takes place and the vibration can be suppressed within a short time if the vibration takes place.

[0097] The number and disposition of arranged dressing-parts B are not limited to the example shown in Fig.6 and any embodiment is available as long as the dressing-working parts B are disposed at unequal spaces in the circular direction of a dresser principal 52.

Example 3:

[0098] The dresser of Example 3 of the present invention and the dressing apparatus having the same are described in detail hereunder, referring to Fig.8 showing an under surface view.

[0099] A dresser 61 of the present example comprises a dresser principal 62 having an approximately circular shape and a dressing-working part C comprising a grindstone disposed along the circular direction in the under surface of the outer peripheral portion of the dresser principal 62.

[0100] Nicks N are provided to the dressing-working part C by forming a plurality of notches at unequal spaces in the circular direction of the dresser principal 62.

[0101] The arrangement of the nicks N can be decided in the same manner as in the dressing-working parts of Example 2. In the present example, the positions in the case of disposing dressing-working parts C at equal spaces are designated as a standard position P, and any one among the standard positions P is designated as a starting point P_0, and an angle between each standard position and the starting point when looked from the center of axis of the dresser principal 62 is designated as θ, and $x=\sin\theta$ is specified as a deviation x of each dressing-working part C from each standard position P. Here, in Fig.8, accompanied characters are provided to standard positions P (and a dressing-working part C, nick N, angle θ and an amount of deviation x corresponding to each standard position P), in a counterclockwise order starting from the starting point P_0.

[0102] Dressing a polishing pad 4 is carried out in the thus-constituted dresser 61 and a dressing apparatus having the same, in the same manner as in the dresser 51 of Example 2 and the dressing apparatus having the same.

[0103] In this example, as the nicks N are disposed at unequal spaces in the circular direction of the dresser principal 62, forces which cause the vibration of the dresser 61 are not brought about periodically and are brought about aperiodically to suppress the resonance of the dresser 61.

EX1057, Page 235

[0104] Thus, according to the dresser 61 of the present invention and the dressing apparatus having the same, the vibration of the dresser 61 hardly takes place and the vibration can be suppressed within a short time if the vibration takes place.

[0105] The number and disposition of arranged nicks N are not limited to the example shown in Fig.8 and any embodiment is available as long as the nicks are disposed at unequal spaces in the circular direction of a dresser principal 62.

Example 4:

[0106] Example 4 of the present invention is described in detail hereunder, referring to Figs.

[0107] Fig.10 and 11 are schematically illustrating the present example. In the Figs., symbol 71 shows a dresser.

[0108] As shown in the Figs., the dresser 71 comprises a plurality f cutters 75 disposed at a prescribed spaces in the circular direction of a dresser principal 72, in a peripheral edge portion 74 of an under surface 73 of the dresser principal 72 having a circular shape.

[0109] The cutters 75 are arranged in the state that the cutter is projected from the under surface73 of the dresser principal 72 as shown in Fig.9 of an enlarged view (a view illustrating the vicinity of the cutter 75 when looked from a p direction in Fig.11). As further enlarged in Fig.12, the cutter 75 comprises an edge of cutter 76 having a flat surface 77 parallel to a polishing pad surface to be dressed (not show in Figs.). The width of the flat surface (a land width:1) is 0.001 to 0.5mm.

[0110] The rake angle D of the cutter 75 is 10° and the clearance angle E thereof is also 10°. The edge of cutter 76 of the cutter 75 is arranged in such a manner having a -15° of an inclined angle α, wherein, as shown in Fig.13, the inclined angle is defined as an angle of the edge of the cutter 76 inclined from a straight line L1 passing through an edge of end 76a in the edge of cutter 76 at the center side of the dresser principal 72 (not shown in the Fig.) and the center of the dresser principal 72 (not shown in the Fig.), and a movement direction of the cutter 75 relative to the polishing pad, that is, a rotational direction of the dresser principal 72 (mentioned after) is defined as positive (a q direction in Fig.13). In addition, nicks 78 are provide to the edge of cutter 76 of the cutter 75 as shown in Fig.13.

[0111] Hard materials such as diamond having a single crystal, cBN, sintered compact produced under a high pressure such as polycrystalline diamond, and materials coated by hard materials as mentioned above are used for the edge of cutter 76.

[0112] When a polishing pad is dressed by using the dresser 71, the polishing pad is shaved by the cutter 75 by rotating a platen 79 having the polishing pad attached thereto an h direction in the Fig.14 and rotating the dresser principal 72 to an i direction in the Fig.14 while pressing the under surface of the dresser principal 72 in such a manner that the flat surface 77 of the cutter 75 is pressed against the under surface of the dresser principal 72, thereby to dress the upper surface of the polishing pad. Here, the i direction is a movement direction of the cutter 75 mentioned above relative to the polishing pad (a q direction in Fig.13).

[0113] In the above-mentioned dresser 71, as the shaving of the polishing pad surface is carried out by providing to the edge of cutter 76 of the cutter 75 a movement relative to the polishing pad in a direction parallel to the polishing pad surface while the edge of cutter 76 is pressed against the polishing pad surface, the dresser is protected from the lowering of the dressing performance thereof which is caused by clogging of the polishing pad surface with the shaved chips in between abrasive grains, as compared with the conventional one, whereby a stable dressing performance is available. Further, as the polishing pad is shaved by using the cutter 75 as mentioned above, an amount of wear can be decided from the flank of the cutter 75 with result that the criterion for use is clear as compared with the conventional one, thereby to enable always to maintain easily a stable dressing performance. Furthermore, as the cutter 75 is used, reusing a cutter 75 which has worn is possible, which is different from the conventional one, with the result of providing excellence in a cost for the dresser.

[0114] In the dresser 71, as the flat surface 77 is formed in the edge of cutter 76 of the cutter 75, the edge of cutter 76 of the cutter 75 is disposed always at a settled position to the polishing pad without causing the vibration of the edge of cutter 76 of the cutter 75 when the dresser principal 72 is pressed against the polishing pad surface, whereby the flatness of the polishing pad can be obtained and in its turn, the polished performance of materials to polished such as semiconductor wafer can be increased.

[0115] Still further, in the dresser 71, the shaved chips of the polishing pad and the residues of slurries left in the polishing pad which have generated in polishing materials to be polished by methods such as a CMP method can be scooped up by the cutter 75 owing to providing a positive angle to a rake angle D of the cutter 75, whereby the dressing performance can be increased and the polishing pad can be easily cleaned.

[0116] In addition, in the dresser 71, the edge of cutter 76 of the cutter 75 is arranged in such a manner having a negative angle wherein a rotational direction of the dresser principal 72 (a q direction in Fig.13) is defined as positive to a straight line L1 passing through an edge of end 76a at the center side of the dresser principal 72 (not shown in the Fig.) and the center of the dresser principal 72. In such a construction, as a normal line L2 (refer to Fig.13) of the rank face 80 of the cutter 75 (refer to Figs.12 and 13) is always directed to the outward of the dresser principal, the shaved chips of the polishing pad scraped away and the residues left in the polishing pad of slurries which have generated in polish-

EX1057, Page 236

ing materials to be polished by the CMP are scraped out by the edge of cutter 76 from the center side of the dresser 72 to the outward thereof. As mentioned above, as the shaved chips of the polishing pad and the residues of slurries left in the polishing pad which have generated in polishing materials to be polished by the CMP are scraped out to the outside by the cutter 75, the polishing pad can easily be cleaned.

[0117] It is to be understood that, within the spirit of the present invention, designs may be practiced otherwise than as specifically described in the present example.

[0118] For example, the rake angle D can be from -20 to 30°. Specifically, in case where channels having a embossed pattern or a latticed pattern are provide to the polishing pad, the rake angle D is preferably a negative angle such that the side wall of the channel can not be shaved off. In case where the polishing pad is too soft, a negative rake angle is also effective as the rake angle D in order to protect the polishing pad from an excessive shaving.

[0119] Further, in case where the rake angle D is from -5° to -15° , the mechanical strength of the cutter 75 can be ensured and it is useful for cases which require the strength of an edge of cutter.

[0120] Further, a width of land 1 can be within the range of 0.005 to 0.5mm and is preferably from 0.05 to 0.1mm. Further, the clearance angle can be from 3° to 15°. An amount of biting depth of the cutter to the polishing pad can be controlled by adjusting an amount of clearance angle E and a width of flank, as mentioned above. Thereby, an optimum shape can be adopted according to the materials and hardness of the polishing pads. Further, the angle α of inclination of the edge of cutter 76 of the cutter 75 can be within the rage of -10 to -30°. In addition, a construction is possible which has no nick 78.

[0121] Further, the edge of cutter 76 can be selected according to the materials of polishing pads to be dressed and slurry materials used in polishing materials to polished by a CMP method. With regard to a method for attaching the edge of cutter 75, brazing, a clamp method, a blade method and so on can be used.

[0122] Further, the clearance angle of the cutter 75 can comprise two steps as in shown in Fig.15 (for example, α 1=5° , β=15° ,in Fig.15). In this case, an amount of biting depth of the cutter 75 to the polishing pad is controlled by adjusting the clearance angle and the width of the first flank W, thereby to enable to obtain a pertinent dressing rate.

Example 5:

[0123] The dresser 81 of Example 5 of the present invention is described in detail hereunder, referring to Fig.16. The same symbols are provided to the same parts as those of the conventional dresser 11 described in the related art and the description thereon is omitted.

[0124] A dresser 81 is attached to an arm 9 of a dressing apparatus 7 by the aid of a setting shaft 82 in a CPM apparatus 1 shown in Fig.17, in the same manner as in the dresser 11.

[0125] Here, with regard to the setting shaft 82, one end of the setting shaft directed to a rotary shaft is connected to a drive unit (not shown in Figs.) by the aid of the arm and the setting shaft 82 is rotated around the rotary shaft. In the other end thereof, a flange 84 is formed for fixing the dresser 81 (refer to Fig.16 illustrating a side sectional view). In the flange 84, a plurality of bolt inserting holes 86 for bolts 85 for screw-clamping the dresser 81 to the flange 84 are disposed at appropriate spaces in the circular direction thereof (only one portion is illustrated in Fig. 16). Hereunder, in the present specification, the side of the dresser 81 contacting to the setting shaft 82 is defined as the upper side thereof and another side of the dresser 81 where a dressing-working part 15 is formed is defined as the lower side thereof.

[0126] The dresser 81 is an electrodeposited grindstone having a dressing-working face 15 formed by providing an abrasive grain layer 93 including abrasive grains 93 on the surface of a dresser principal 83 having a approximately annular shape, as shown in Fig.16, and the dresser is fixed to the setting shaft 82 while, the center of the dresser is matched with the center of the setting shaft 82.

[0127] The material of the dresser principal 83 comprises aluminum, stainless steel, steel or a combination of aluminum and steel and the section thereof is approximately a rectangle. A convex 87 of the outer peripheral side which is projected at the outer peripheral side of the under surface of the dresser principal 83 toward the lower side is formed all around the periphery, and the under surface of the convex 87 comprises a ground face 88 having an abrasive grain layer 93 formed thereon. The ground face is formed in such a manner that the face thereof is orthogonally crossed to a rotary axis AX of the dresser principal 83. In Fig.16, the thickness of the abrasive grain layer 93 is illustrated larger than the actual one so as to easily understand the shape of the dressing-working face 15.

[0128] A convex 89 of the inward peripheral side having an amount of projection to the lower side smaller than that of the convex 87 of the outer peripheral side is formed in a position closer to the center of rotation than the convex 87 of the outer peripheral side in such a manner that the convex 89 is concentric with the convex 87 and the under surface of the convex 89 comprises a basic face 90 which is parallel to the ground face 88. A recess 92 is formed between the convex 87 and the convex 89 so as to obtain the working accuracy thereof. A plurality of holes for bolt 91 for screw-clamping the bolts 85 are formed at appropriate spaces in the upper face of the dresser principal 83 all around the periphery (only one portion is illustrated in Fig.16).

[0129] Here, the dresser principal 83 is screw-

EX1057, Page 237

clamped to the under surface of the flange 84 of the setting shaft 82 by screw-clamping the bolts 85 to the holes for bolt 91.

[0130] When the dressing of a polishing pad 4 is carried out with the dresser 81 constructed as mentioned above, the following operations are carried out so as to stabilize conditions for dressing the polishing pad 4.

[0131] First, dressers 81 are checked on whether or not a dresser 81 has a flatness of the dressing-working face 15 within a range which is proper to a use for the dressing apparatus 7, and dressers 81 are selected which have the proper flatness.

[0132] The flatness of a dressing-working face 15 can be predicted by inspecting the flatness of the ground face 88 for forming the dressing-working face 15, and the flatness of the ground face 88 can be predicted by inspecting the flatness of the basic face 90 parallel to the ground face 88.

[0133] Namely, if the flatness of a basic face 90 is within the range proper for a use in the dressing apparatus 7, the flatness of the corresponding ground face 88 and dressing-working face 15 can be predicted to be also within the range proper for the use, thereby to select dressers 81 which have the flatness of a basic face which is within the range proper for the use and to use the selected dressers.

[0134] Next, a dresser 81 which has been confirmed to have a flatness within the proper range is attached to the under surface of the flange 84 of the setting shaft 82 by the bolts 85.

[0135] Then, the deflection of the basic face 90 of the dresser 81 is measured with dial gauge, while rotating the setting shaft 82 together with the dresser 81 by a drive unit which is not shown in Figs., and the setting condition of the dresser is adjusted so as to attain a deflection having a proper range.

[0136] When the deflection is not within the proper range, it is considered as a reason that dust is contained in between the setting shaft 82 and the dresser 81, together with another reason due to an error in attaching the setting shaft 82. If the dust problem is considered, the dresser 81 is detached from the setting shaft 82 to confirm whether or not the dust is interposed in between. When the dust is interposed, the dust is removed, followed by attaching again the dresser 81 to the setting shaft 82.

[0137] According to the thus constructed dresser 81, as a ground face 88 having a dressing-working face 15 formed thereon can be predicted whether to be flat or not, dressers having a good flatness of the dressing-working face can be selected and used.

[0138] Further, the condition of attaching a dresser 81 to the setting shaft 82 of the dressing apparatus 7 is confirmed whether to be proper or not, the accuracy of attaching to the dressing apparatus 7 can be increased.

[0139] Thereby, the condition of dressing a polishing pad 4 with the dresser 81 can be stabilized.

[0140] Furthermore, as the basic face 90 is disposed in a position closer to the center of rotation as mentioned above than the ground face 88, that is, at the inward peripheral side of the dresser principal 83, a possibility of damaging the basic face 90 due to hitting the basic face 90 against something is decreased during delivering the dresser 81 or attaching the dresser 81 to the dressing apparatus 7, thereby to ensure an accuracy for predicting the flatness of the dressing-working face 15.

[0141] Still further, as the basic face 90 is disposed in a position close to the center of rotation, the outside diameter of the dresser 81 can be minimized and the dresser 81 can be easily handled.

[0142] In the above-mentioned example, a metal plating layer comprising the abrasive grain layer 93 can be formed by a metal which is not limited to Ni, and other metals can used. A method for forming the metal layer is not limited to electroplating, and the metal layer can be formed on the dresser principal 83 by PVD or CVD.

[0143] The present invention is not limited to the examples as mentioned above and various modified examples are included which contain the combinations of the examples (for examples, a combination of Example 1 and Example 2, and the combinations of any of Examples 1 to 4 and Example 5).

Claims

1. A dresser for dressing a polishing pad used for polishing a material to be polished, the dresser comprising:

   a dresser principal and a dressing-working part which is provided as a body other than the dresser principal; and
   a holding means for holding the dressing-working part in such a manner that the dressing-working part can be attached to and detached from the dresser principal.

2. The dresser as claimed in claim 1, wherein a projection adjusting means is provided for controlling an amount of projection of said dressing-working part from said dresser principal.

3. The dresser as claimed in claim 2, wherein said dressing-working part is a cutter.

4. The dresser as claimed in claim 3, wherein said cutter has an approximately columnar shape and comprises: a flank which is formed by concaving one end face of the cutter toward the base end of the cutter; a rake face comprising a circumferential surface in the end portion of the cutter; and a cutting edge which is a ridgeline formed by crossing of the end face and the circumferential surface in the end

EX1057, Page 238

portion.

5. The dresser as claimed in claim 3, wherein said cutter comprises:

a plurality of projections formed in one end of the cutter;
said projection having a cross section approximately equal to an end face of the projection from one end of the projection to near another end of the projection from which the projection is started; and
a cutting edge which is a ridgeline formed by crossing of the end face of the projection and the side surface of the projection.

6. The dresser as claimed in claim 1, wherein said dressing-working part is a cutter.

7. The dresser as claimed in claim 6, wherein said cutter has an approximately columnar shape and comprises: a flank which is formed by concaving one end face of the cutter toward the base end of the cutter; a rake face comprising a circumferential surface in the end portion of the cutter; and a cutting edge which is a ridgeline formed by crossing of the end face and the circumferential surface in the end portion.

8. The dresser as claimed in claim 6, wherein said cutter comprises:

a plurality of projections formed in one end of the cutter;
said projection having a cross section approximately equal to an end face of the projection from one end of the projection to near another end of the projection from which the projection is started; and
a cutting edge which is a ridgeline formed by crossing of the end face of the projection and the side surface of the projection.

9. A dressing apparatus for dressing with a dresser the surface of a polishing pad used for polishing a material to be polished, said dresser comprising a dresser according to claim 1.

10. A dresser for dressing a polishing pad used for polishing a material to be polished, said dresser comprising a plurality of dressing-working parts for dressing the polishing pad which are disposed at unequal spaces in the circular direction of a dresser principal.

11. A dressing apparatus for dressing with a dresser the surface of a polishing pad used for polishing a material to be polished, said dresser comprising a dresser according to claim 10.

12. A dresser for dressing a polishing pad used for polishing a material to be polished, said dresser comprising: a dressing-working part for dressing the polishing pad along the circular direction of a dresser principal; and a plurality of nicks formed in the dressing-working part at unequal spaces in the circular direction of the dresser principal.

13. A dressing apparatus for dressing with a dresser the surface of a polishing pad used for polishing a material to be polished, said dresser comprising a dresser according to claim 12.

14. A dresser for dressing a polishing pad surface which is used for an apparatus for polishing, with the polishing pad, materials to be polished such as a semiconductor wafer having a flat surface, said dresser comprising at least one cutter projected from the under surface of a dresser principal,
wherein shaving the polishing pad surface is carried out by providing to the edge of cutter of the cutter a movement relative to the polishing pad surface in a direction parallel to the polishing pad surface, while the edge of cutter is pressed against the polishing pad surface.

15. The dresser as claimed in claim 14, wherein said edge of cutter comprises a flat surface which is parallel to the polishing pad surface and is pressed against the polishing pad surface.

16. The dresser as claimed in claim 14, wherein said cutter comprises a positive rake angle.

17. The dresser as claimed in claim 14, wherein said cutter comprises a rake angle having from -5° to -15°.

18. The dresser as claimed in claim 14, wherein said dresser principal has a circular shape and is constructed in such a manner that the dresser principal is rotated around a rotary shaft formed in the center thereof; and the edge of cutter of the cutter is arranged in such a manner that the edge of cutter has a negative angle wherein a rotational direction of the dresser principal is defined as positive to a straight line passing through the edge of end of the edge of cutter at the center side of the dresser principal and the center of the dresser principal.

19. A dresser which comprises a dresser principal and a dresser-working part having an abrasive grains layer including abrasive grains formed on the dresser principal,
wherein the dresser principal comprises a ground face on which the abrasive-grain layer is provided

EX1057, Page 239

and a basic face which is disposed in a portion of the dresser principal more inward than the ground face and is formed in parallel to the ground face.

20. The dresser as claimed in claim 19, wherein said ground face comprises an approximately annular shape having a center which is the same as the center of rotation of said dresser principal; and the basic face is disposed in a position closer to the center of rotation than the ground face and is concentric with the ground face.

EX1057, Page 240

FIG. 1

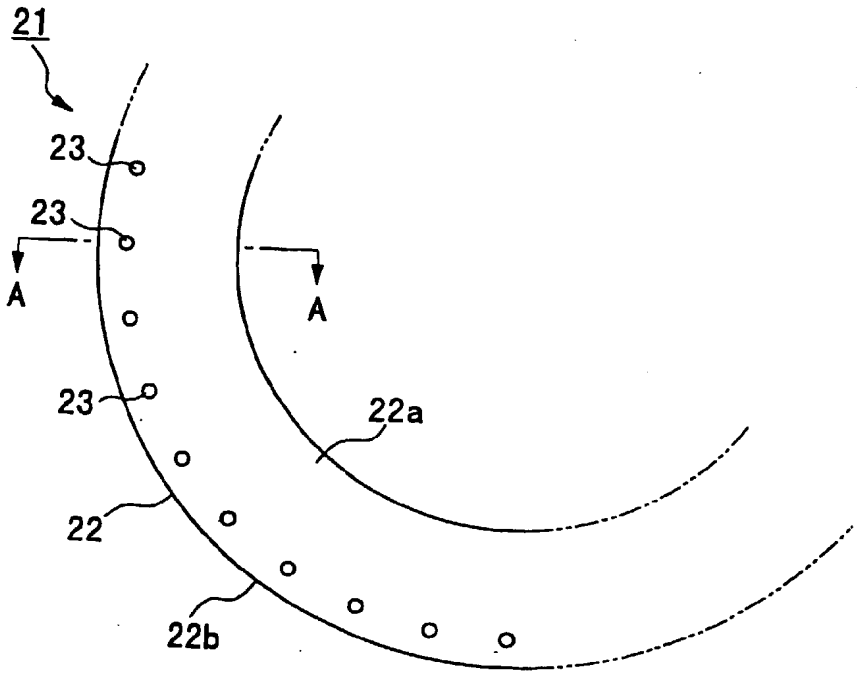


FIG. 2

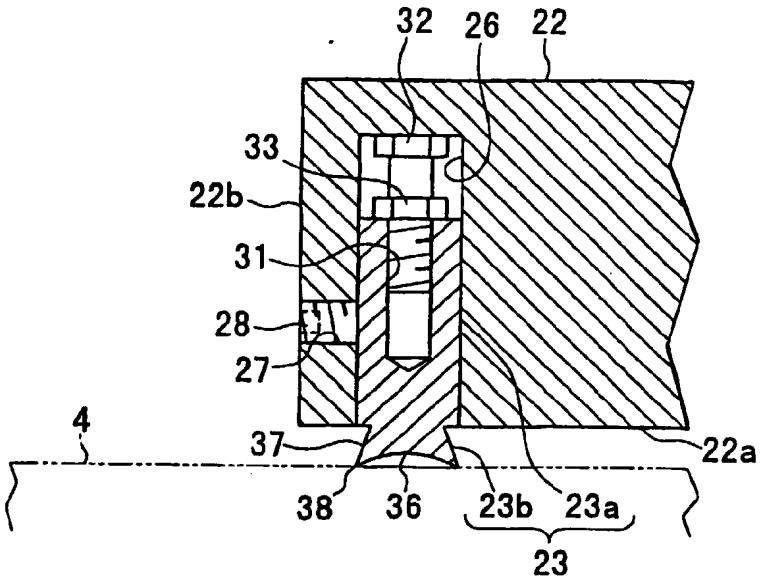

32
26
22
33
22b
31
28
27
4
37
22a
38
36
23b
23a
23

FIG. 3

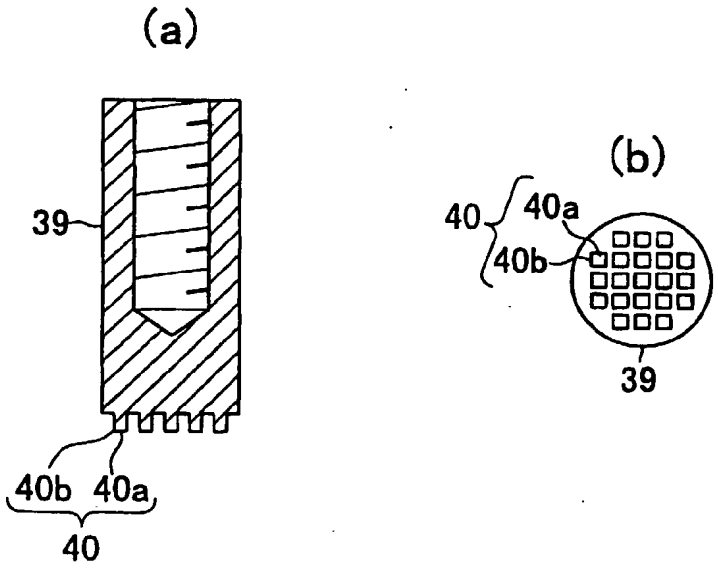

(a)
39
40b
40a
40
(b)
40
40a
40b
39

FIG. 4

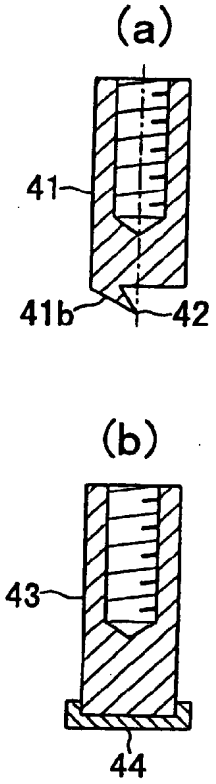


FIG. 5

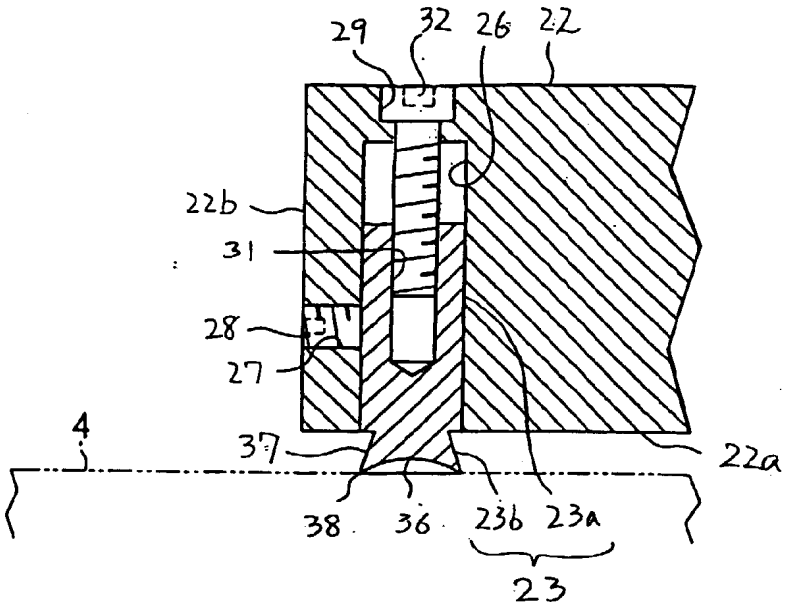


FIG. 6

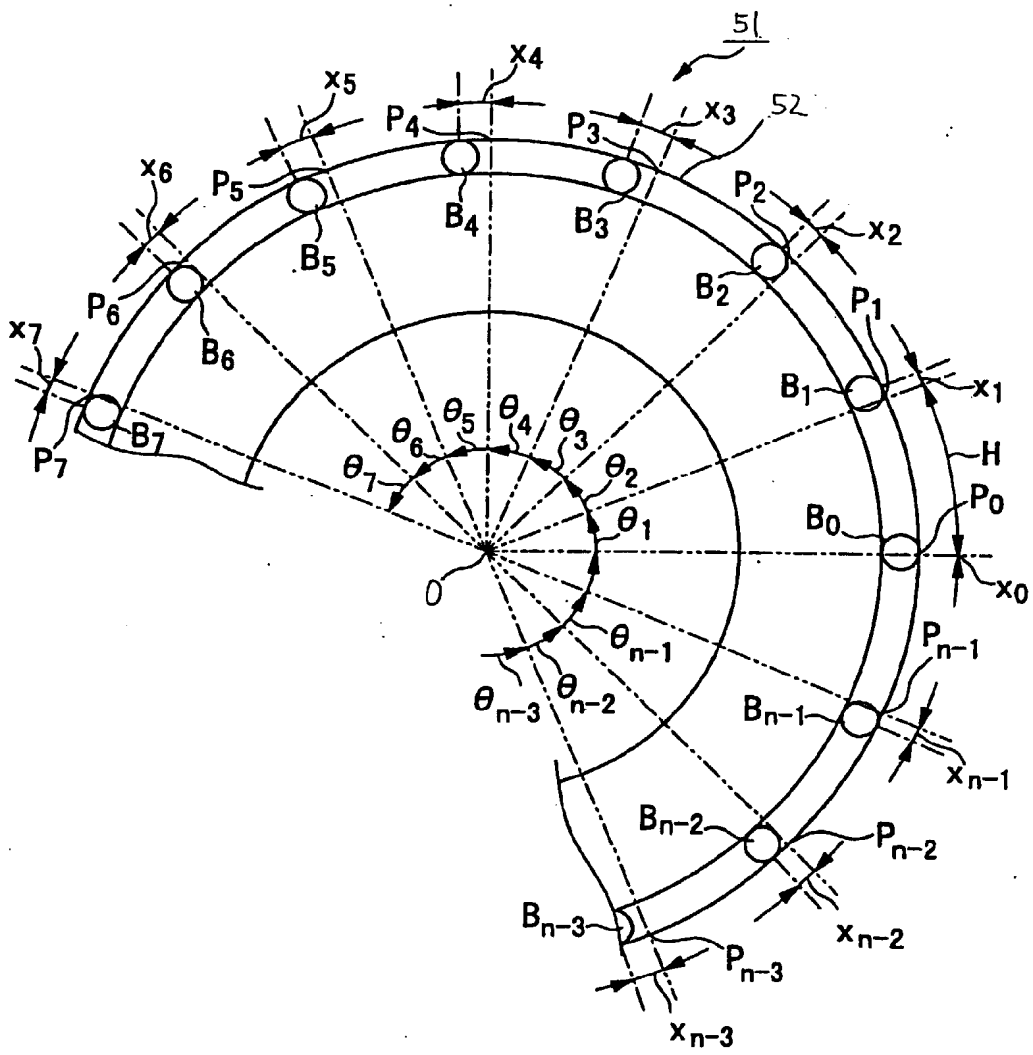

51
52
x_4
x_5
P_4
P_3
x_3
x_6
P_5
P_2
x_2
B_4
B_3
B_5
B_2
P_6
P_1
x_7
B_6
B_1
x_1
B_7
θ_6
θ_5
θ_4
θ_3
H
P_7
θ_7
θ_2
P_0
θ_1
B_0
0
x_0
θ_{n-1}
P_{n-1}
θ_{n-3}
θ_{n-2}
B_{n-1}
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P_{n-2}
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P_{n-3}
x_{n-3}

FIG. 7

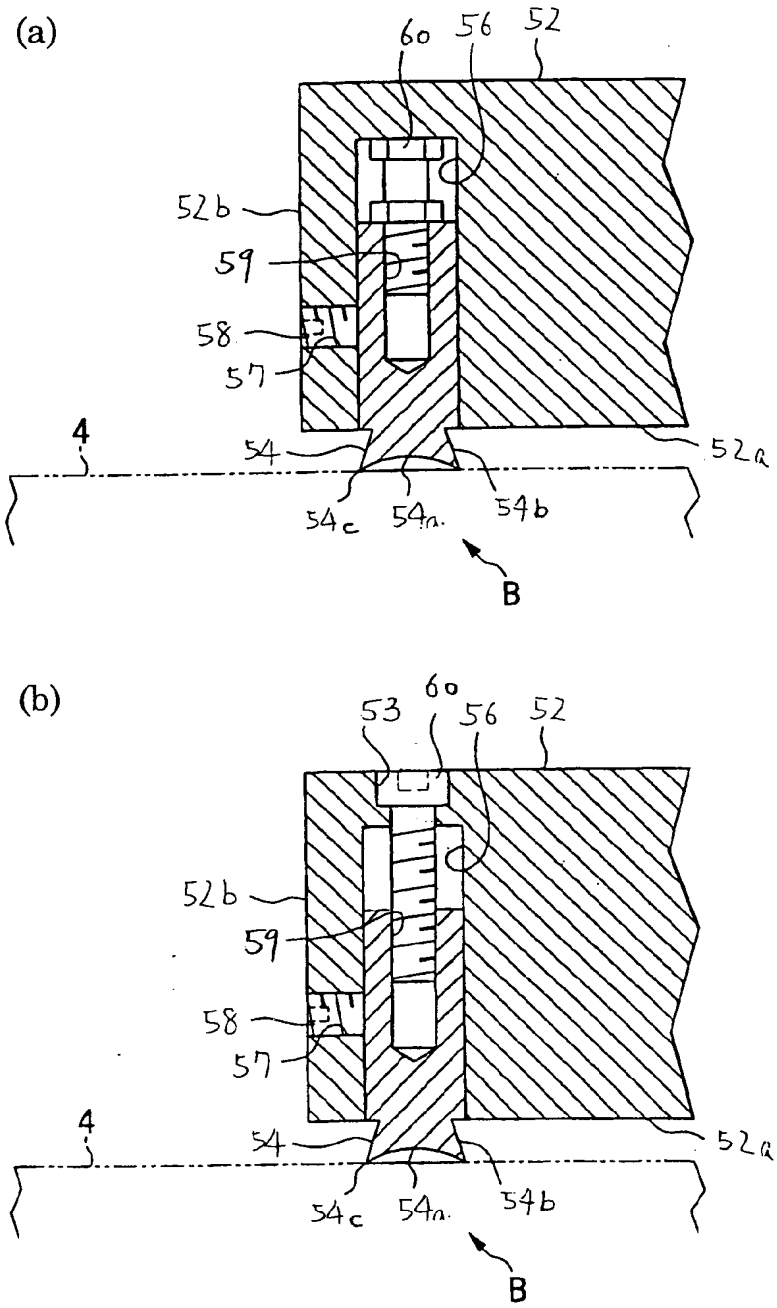

(a)
60
56
52
52b
59
58
57
4
54
52a
54c
54a
54b
B
(b)
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52
52b
59
58
57
4
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52a
54c
54a
54b
B

FIG. 8

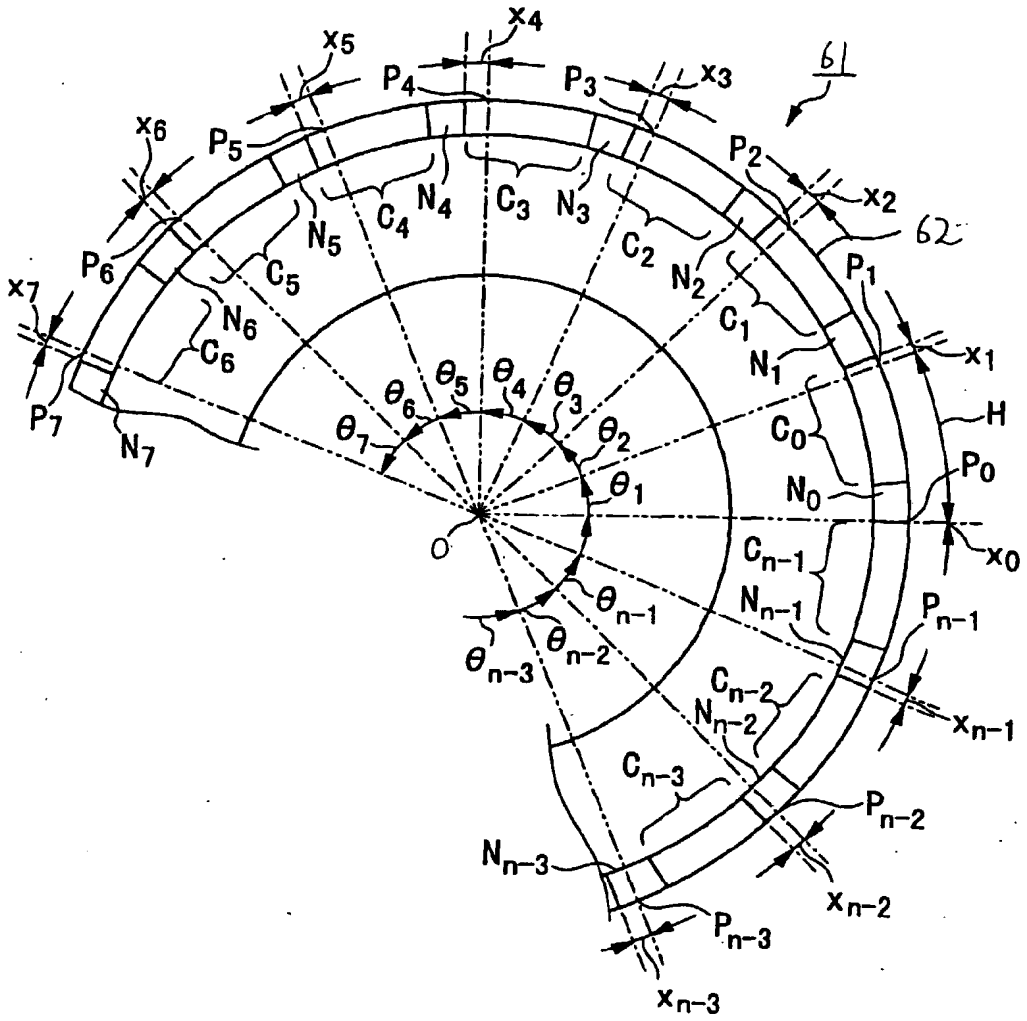

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62
H
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x_1
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x_3
x_4
x_5
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P_0
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FIG. 9

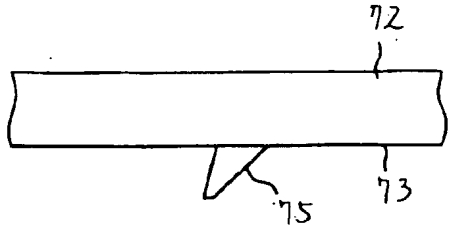


FIG. 10

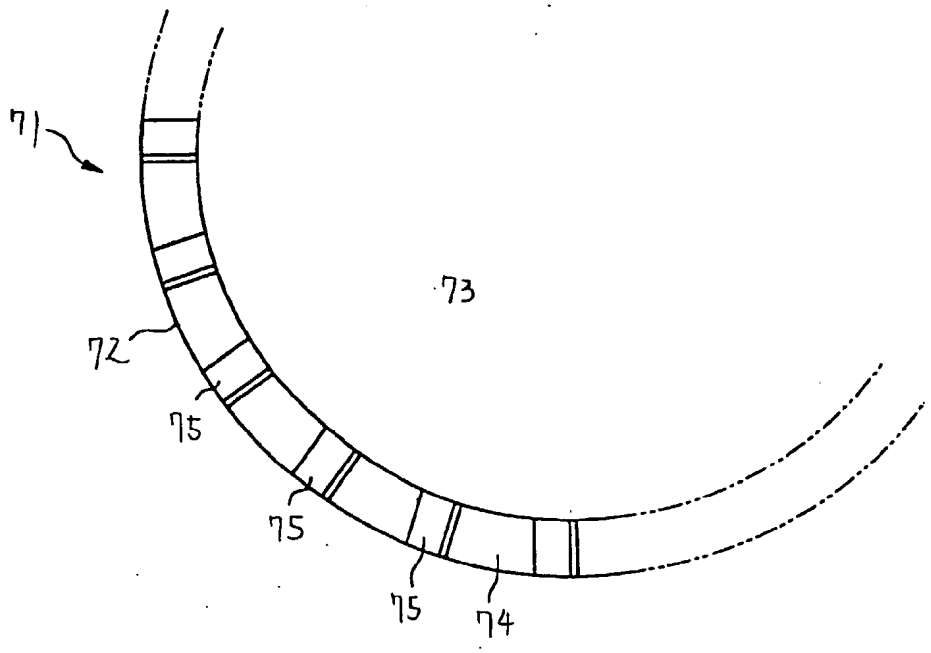


FIG. 11

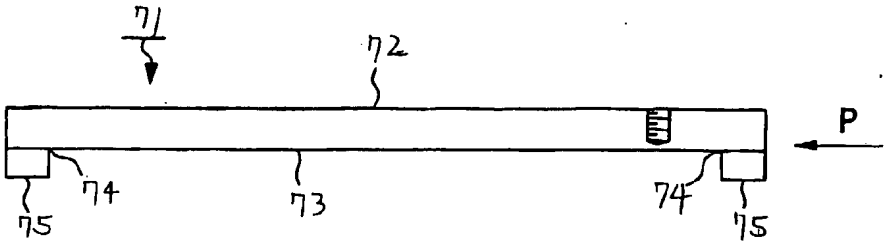


FIG. 12

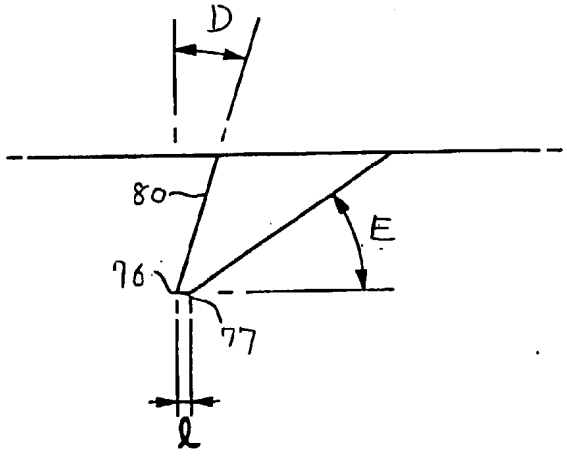


FIG. 13

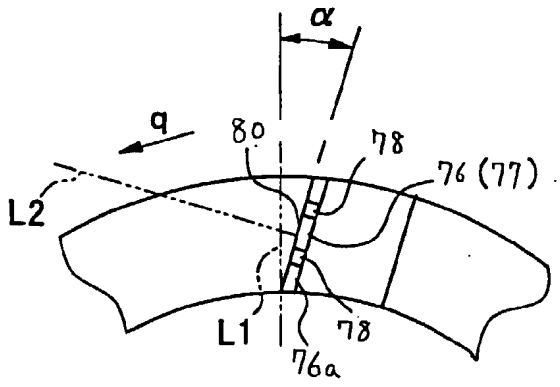


FIG. 14

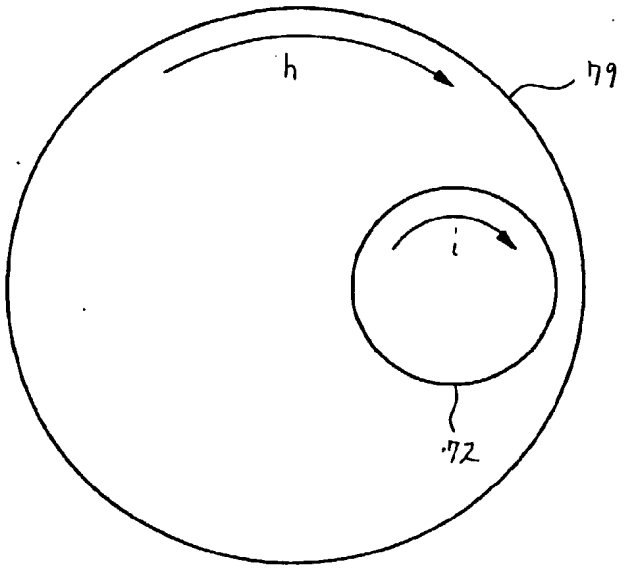


FIG. 15

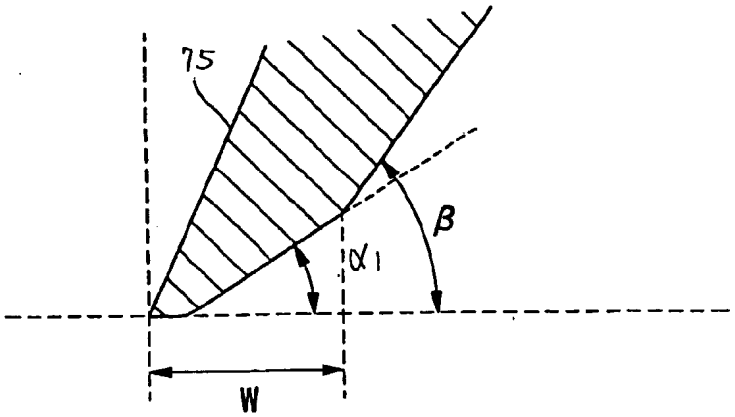


FIG. 16

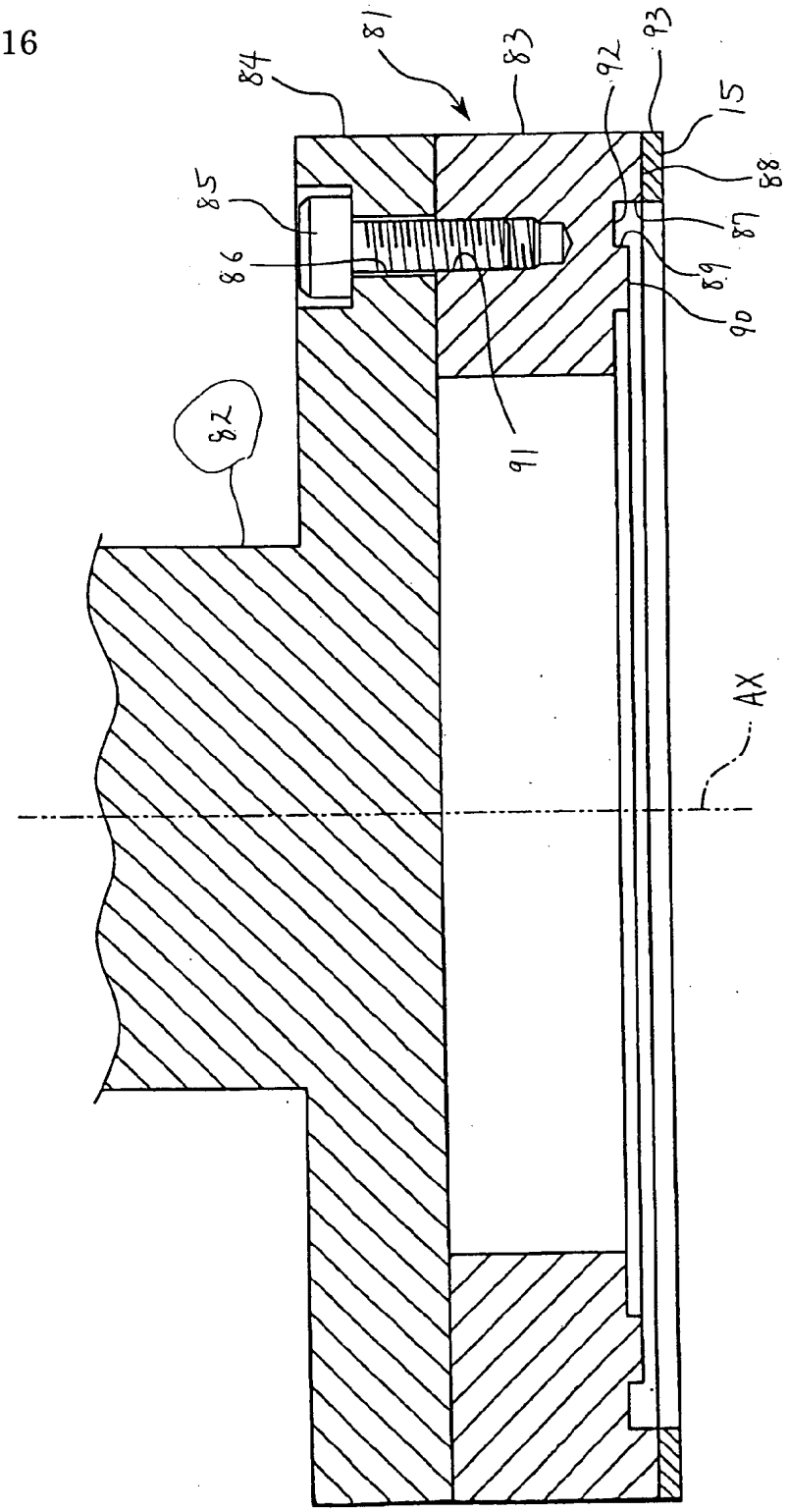


FIG. 17

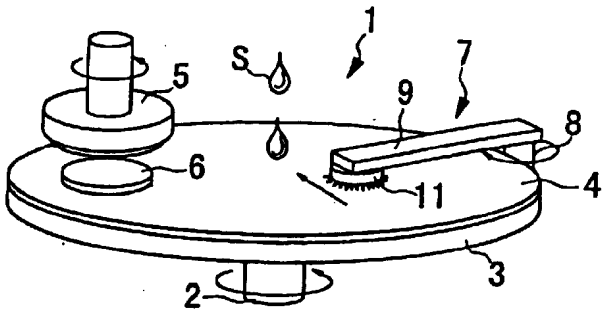


FIG. 18

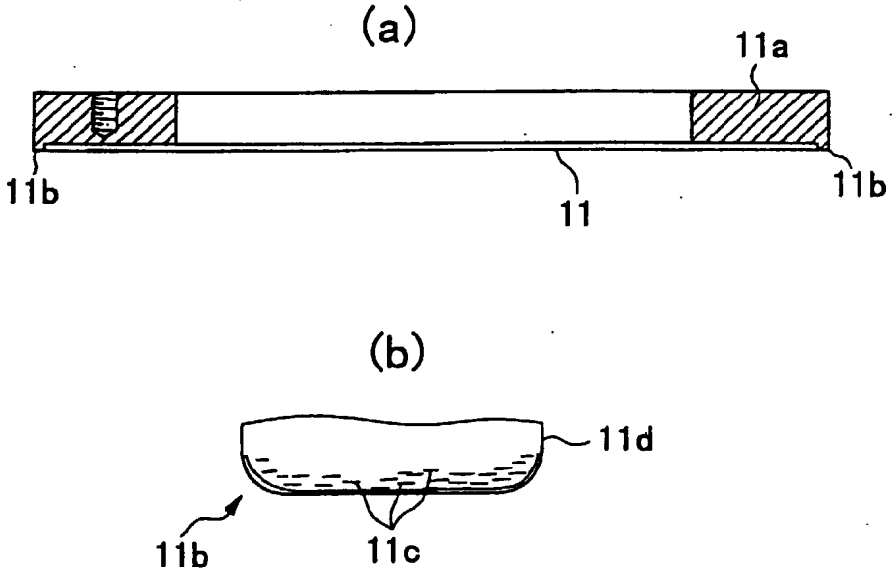


FIG. 19

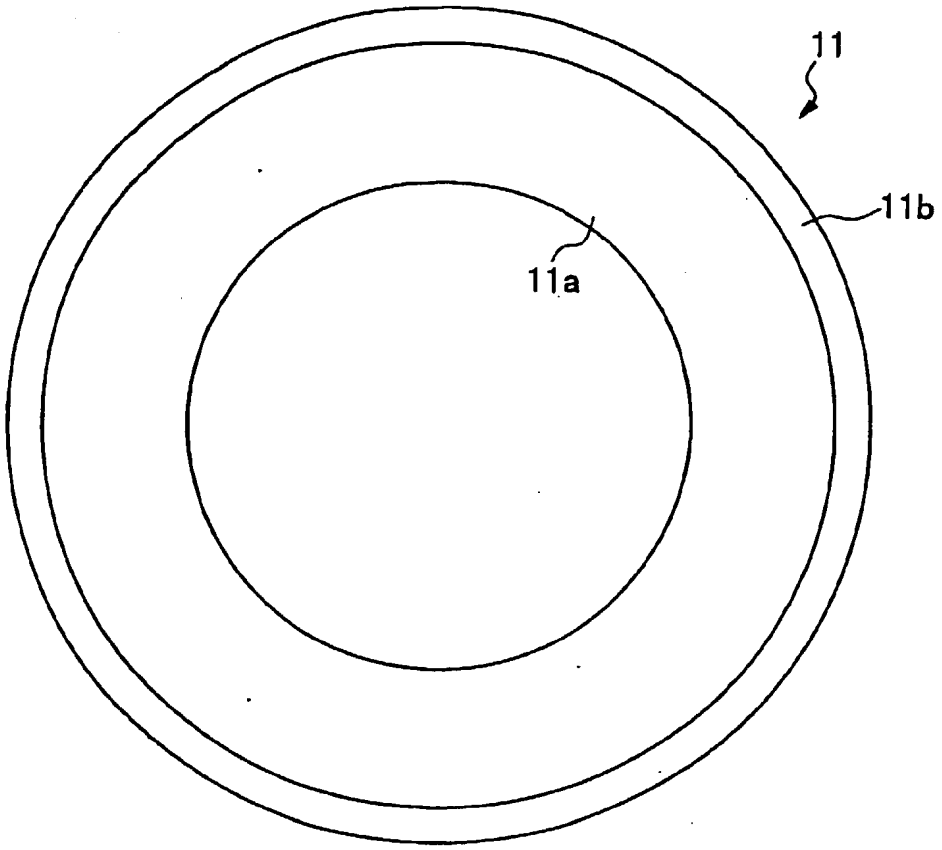


FIG. 20

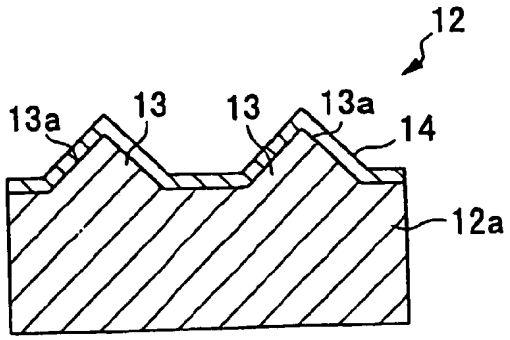
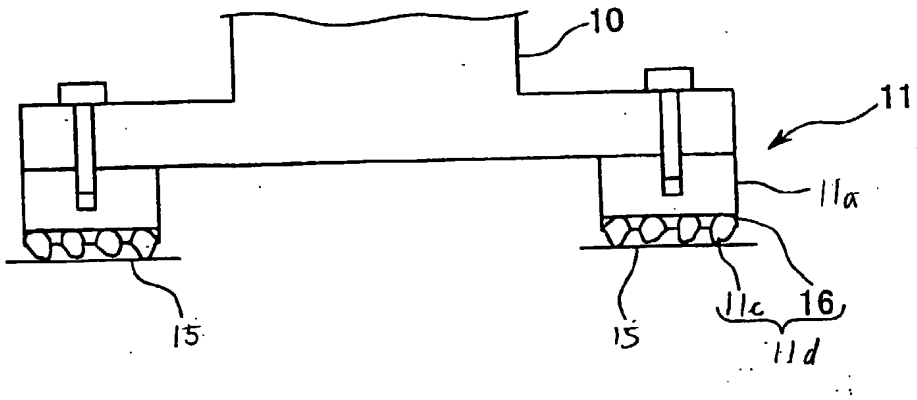


FIG. 21



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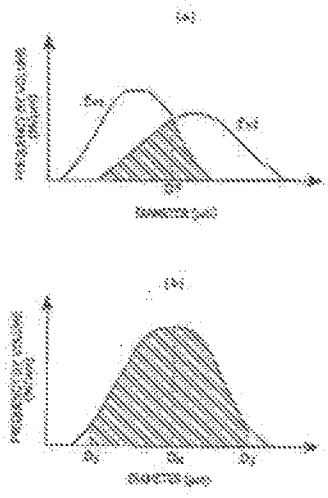
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(54) Diamond sinter, high-pressure phase boron nitride sinter, and processes for producing those sinters

(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 gasified 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 3000 MPa, the invention offers the advantage of eliminating the constraint of the need to use equipment capable of creating ultrahigh pressures of at least 3000 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



EP 0 712 941 A1

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 these sinters.

Diamond, with strong covalent bonding, has many excellent properties. On the other hand, because of the 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 9500 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

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. Mater. Sci., 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 60-35456. 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 (1973) 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 (HIP) 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 migrates 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,

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 these 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

EX1057, Page 258

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 58-31075 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 61-30683 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 3-150364 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 3-43577 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 64-60437 teaches a method in which the agglom-

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 64-159789 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 64-60437 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 cylindrical sputtering chamber equipped with a vertical target, whereby the particles in powder are provided with a coating. Unexamined Published Japanese Patent Application (kokai) Hei 2-153883 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.

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 diamond 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 20000 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

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 comprises 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

EX1057, Page 262

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 sinterable to a density of at least 85% at pressures less than 2000 MPa and at temperatures not exceeding 1800°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 1800°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 87% 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 98% 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 1800°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 sinterable to a density of at least 85% at

pressures less than 2000 MPa and at temperatures not exceeding 1800°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 1800°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 600 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 600 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 cooling 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 cooling 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 cooling 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

EX1057, Page 264

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

EX1057, Page 265

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 30 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 30 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 ($D_{90}/5$, $5D_{90}$, ≥ 90%) in terms of frequency distribution by volume, where D_{90} 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 1550°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

EX1057, Page 266

pressure of less than 2000 MPa and at a temperature not exceeding 1800°C under such conditions that diamond or high-pressure phase boron nitride is thermodynamically metastable, is sintered at a pressure of less than 3000 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 first treating means in a group of means for high dispersion treatment of particles are allowed to combine with each other in the cooling start region of a cooling 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 3000 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 3000 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;

EX1057, Page 267

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_{50} satisfies the relation ($[D_{50}/5, 5D_{50}]$, ≥ 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 ($[D_{50}/3, 3D_{50}]$, ≥ 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 ($[D_{50}/2, 2D_{50}]$, ≥ 80%) in terms of frequency distribution by volume.

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 burnt 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 uncombined.

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", "feed 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 ($(D_M/5, 5D_M)$, ≥ 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.

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 0.0005 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, BiC, 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, VB_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 $Al_6Si_2O_{13}$ (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 filling 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

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 23th 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 $f_{m,2}$ and the frequency distribution by mass of completely dispersed powder particles $f_{m,1}$ and is expressed by β according to the following equation:

$$\beta = \int_0^{D_x} f_{m2}(D)dD + \int_{D_x}^{\infty} f_{m1}(D)dD$$

where

D: particle diameter (μm);
D_x: 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.

$$\beta = \int_0^{D_x} f_{v2}(D)dD + \int_{D_x}^{\infty} f_{v1}(D)dD$$

where

D: particle diameter (μm);
D_x: 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], x 90%)

A ([D1,D2], x 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.