Definition of Frequency Distribution by Volume ($(D_M/5, 50_{(f)}) \geq 90\%$)

A particle size distribution that is expressed by ($(D_M/5, 50_{(f)}) \geq 90\%$) in terms of frequency distribution by volume represents one in which particles ranging in diameter from one fifth of D_M to five times D_M (D_M is the average particle diameter by volume) account for at least 90% of the total volume of the particles present. Consider, for example, particles having an average diameter D_M of 10 μm on a volume basis; a particle size distribution that is expressed by ($(D_M/5, 50_{(f)}) \geq 90\%$) in terms of frequency distribution by volume represents one in which particles ranging in diameter from 2 μm to 50 μm account for at least 90% of the total volume of the particles present. The average particle diameter D_M on a volume basis is expressed by:

$$D_M = \frac{\int_0^\infty Df(D)dD}{\int_0^\infty f(D)dD}$$

where f(D) is the frequency distribution by volume. Or D_M is technically expressed by:

$$DM = \Sigma(V_i D_i)/\Sigma V_i$$

where Vi is the volume of a group of particles within a given interval of diameters $D_i \pm \Delta D_i/2$ (ΔD_i is the width of interval).

Coating Start Region

The region where coating is first started after the final treatment by the group of means for high dispersion treatment of particles is called the "coating start region". Therefore, prior to the final treatment by the group of means for high dispersion treatment of particles, no region is called the "coating start region" according to this definition even if coating is first started there.

Dispersity β in the Coating Start Region

In the present invention, the coating chamber is provided in such a way that the coating start region of the coating space is located in a region that satisfies one of the following conditions:

(1) a powder of core particles that have an average diameter of no more than 10 μm in the frequency distribution by volume should be dispersed in a gaseous atmosphere by the final treatment by the group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and the dispersity β of the particles in said powder of core particles should be adjusted to at least 70%;

(2) a powder of core particles that have an average diameter in excess of 10 μm but no more than 20 μm in the frequency distribution by volume should be dispersed in a gaseous atmosphere by the final treatment by the group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and the dispersity β of the particles in said powder of core particles should be adjusted to at least 80%;

(3) a powder of core particles that have an average diameter in excess of 20 μm but no more than 50 μm in the frequency distribution by volume should be dispersed in a gaseous atmosphere by the final treatment by the group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and the dispersity β of the particles in said powder of core particles should be adjusted to at least 90%;

(4) a powder of core particles that have an average diameter in excess of 50 μm but no more than 300 μm in the frequency distribution by volume should be dispersed in a gaseous atmosphere by the final treatment by the group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and the dispersity β of the particles in said powder of core particles should be adjusted to at least 95%;

(5) a powder of core particles that have an average diameter in excess of 300 μm but no more than 800 μm in the frequency distribution by volume should be dispersed in a gaseous atmosphere by the final treatment by the group

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of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and the dispersity β of the particles in said powder of core particles should be adjusted to at least 97%; and

(6) a powder of core particles that have an average diameter in excess of 500 μm in the frequency distribution by volume should be dispersed in a gaseous atmosphere by the final treatment by the group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and the dispersity β of the particles in said powder of core particles should be adjusted to at least 99%.

Given one of those dispersities in the coating start region of the coating space, one can disperse the particles in the powder of core particles to such a state that they are substantially single particles in a gaseous atmosphere, so that the precursor of the coat forming substance will uniformly contact and/or impinge against the surfaces of all particles in the powder of core particles that pass through the coating start region of the coating space, whereby the coat forming substance can be deposited on single particles in equal amounts.

In the present invention, dispersity β varies continuously with the average diameter of the particles in the powder of core particles but because of the difficulty involved in expressing it, the convenient stepwise expression is adopted.

Advantageously, the coating chamber is provided in such a way that the coating start region of the coating space is located in a space region that satisfies one of the following conditions.

(1) a powder of core particles that have an average diameter of no more than 10 μm in the frequency distribution by volume should be dispersed in a gaseous atmosphere by the final treatment by the group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and the dispersity β of the particles in said powder of core particles should be adjusted to at least 80%;

(2) a powder of core particles that have an average diameter in excess of 10 μm but no more than 20 μm in the frequency distribution by volume should be dispersed in a gaseous atmosphere by the final treatment by the group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and the dispersity β of the particles in said powder of core particles should be adjusted to at least 90%;

(3) a powder of core particles that have an average diameter in excess of 20 μm but no more than 50 μm in the frequency distribution by volume should be dispersed in a gaseous atmosphere by the final treatment by the group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and the dispersity of the particles in said powder of core particles should be adjusted to 95%;

(4) a powder of core particles that have an average diameter in excess of 50 μm but no more than 200 μm in the frequency distribution by volume should be dispersed in a gaseous atmosphere by the final treatment by the group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and the dispersity β of the particles in said powder of core particles should be adjusted to 97%; and

(5) a powder of core particles that have an average diameter in excess of 200 μm in the frequency distribution by volume should be dispersed in a gaseous atmosphere by the final treatment by the group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and the dispersity β of the particles in said powder of core particles should be adjusted to at least 99%.

Given the coating chamber in which the coating start region of the coating space is located in such a way as to satisfy those conditions, the particles in the powder of core particles are at least free from areas that are substantially shaded by two adjacent core particles and it is possible to assure that the precursor of the coat forming substance is allowed to contact and/or impinge against the surfaces of individual particles from every direction, whereby coating with the coat forming substance can be accomplished more uniformly on a single particle basis and, accordingly, coating can be done to provide more uniform coating weights for the individual core particles.

The particles in a powder of core particles that have an average diameter exceeding 10 μm in the frequency distribution by volume are subjected to a cohesive action in a gaseous atmosphere, although not as intense as in the case of the particles in a powder of core particles that have an average diameter no more than 10 μm, and they contact and/or impinge against one another, causing unevenness in the distribution of the particles in the powder of highly dispersed core particles which form a mixture with a gas. However, if the coating of the particles in the powder of core particles is started as they are dispersed to provide one of the above-specified dispersities in accordance with their diameter, the particles in the powder of core particles or the particles in a powder that consists chiefly of the core particles can be coated with the coat forming substance more uniformly on a single particle basis and, accordingly, the individual particles can be coated with the coat forming substance in more uniform weights.

More advantageously, in the coating start region of the coating space,

(1) a powder of core particles that have an average diameter of no more than 10 μm in the frequency distribution by volume should be dispersed in a gaseous atmosphere by the final dispersing treatment by the group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles; and the dispersity β of the particles in said powder of core particles should be adjusted to at least 90%, or

(2) a powder of core particles that have an average diameter in excess of 10 μm but no more than 20 μm in the frequency distribution by volume should be dispersed in a gaseous atmosphere by the final dispersing treatment by the group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and the dispersity β of the particles in said powder of core particles should be adjusted to at least 95%, or

(3) a powder of core particles that have an average diameter in excess of 20 μm but no more than 50 μm in the frequency distribution by volume should be dispersed in a gaseous atmosphere by the final dispersing treatment by the group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and the dispersity β of the particles in said powder of core particles should be adjusted to at least 97%, or

(4) a powder of core particles that have an average diameter in excess of 50 μm in the frequency distribution by volume should be dispersed in a gaseous atmosphere by the final dispersing treatment by the group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and the dispersity β of the particles in said powder of core particles should be adjusted to at least 99%.

If one of these dispersities is attained in the coating start region of the coating space, the core particles are practically free from agglomeration and practically uniform coating can be performed on all surfaces of the individual particles.

If, in a particular case, high-quality coating is desired at the sacrifice of the efficiency of treatment, the dispersity is preferably at least 95%. In this case, the throughput of the treatment of the particles in the powder of core particles may be reduced to a very small level, namely, the concentration by number of the particles in the powder of core particles in the gaseous atmosphere may be reduced so that the particles in the powder of core particles are dispersed completely, whereby uniform coating can thoroughly be accomplished on the entire surfaces of the individual particles.

Group of Means for High Dispersion Treatment of Particles

The group of means for high dispersion treatment of particles which is to be used in the present invention is such that: (A) it has at least one dispersing means; and (B) it has, as the final treating means,

(a) dispersing means for dispersing the particles in a powder of core particles in a gaseous atmosphere or
(b) means for selecting a mixture of a gas and the particles in a powder of highly dispersed particles which comprises:

(b-1) means for selecting a mixture of a gas and the particles in a powder of highly dispersed particles that separates a portion of the powder that is composed of less highly dispersed particles from the mixture of a gas and the particles in the powder of core particles in which the particles in the powder of core particles are dispersed in a gaseous atmosphere, thereby selecting a mixture of a gas and the particles in a powder of highly dispersed core particles in which the particles in the powder of core particles are present in the gaseous atmosphere mainly in a single particle state and
(b-2) feedback means by which the portion of the powder that is composed of less highly dispersed particles and which has been separated by the means for selecting a mixture of a gas and the particles in a powder of highly dispersed particles is transported to the final dispersing means among the dispersing means in the group of means for high dispersion treatment of particles and/or treating means upstream of the final dispersing means.

Advantageously, the group of means for high dispersion treatment of particles has such dispersing performance that one of the following conditions is satisfied:

(1) a powder of core particles that have an average diameter of no more than 10 μm in the frequency distribution by volume should be dispersed in a gaseous atmosphere by the final treatment by said group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and the dispersity β of the particles in said powder of core particles should be adjusted to at least 70%,
(2) a powder of core particles that have an average diameter in excess of 10 μm but no more than 20 μm in the frequency distribution by volume should be dispersed in a gaseous atmosphere by the final treatment by said group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder

of highly dispersed core particles and the dispersity β of the particles in said powder of core particles should be adjusted to at least 80%;

(3) a powder of core particles that have an average diameter in excess of 20 μm but no more than 50 μm in the frequency distribution by volume should be dispersed in a gaseous atmosphere by the final treatment by the group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and the dispersity β of the particles in said powder of core particles should be adjusted to at least 90%;

(4) a powder of core particles that have an average diameter in excess of 50 μm but no more than 300 μm in the frequency distribution by volume should be dispersed in a gaseous atmosphere by the final treatment by the group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and the dispersity β of the particles in said powder of core particles should be adjusted to at least 95%;

(5) a powder of core particles that have an average diameter in excess of 300 μm but no more than 800 μm in the frequency distribution by volume should be dispersed in a gaseous atmosphere by the final treatment by the group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and the dispersity β of the particles in said powder of core particles should be adjusted to at least 97%; and

(6) a powder of core particles that have an average diameter in excess of 800 μm in the frequency distribution by volume should be dispersed in a gaseous atmosphere by the final treatment by the group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and the dispersity β of the particles in said powder of core particles should be adjusted to at least 99%.

By insuring that the group of means for high dispersion treatment of particles have dispersing capabilities comparable to or better than the various dispersities to be attained in the above-described coating start region and by providing them in correspondence to those dispersities, high-quality coating can be applied in the coating start region to comply with the respective dispersities.

Final Treating Means

If the final treating means in the group of means for high dispersion treatment of particles is a dispersing means, said dispersing means is referred to as the final treatment means in the group of means for high dispersion treatment of particles. If the final treating means in the group of means for high dispersion treatment of particles is a means for selecting a mixture of a gas and the particles in a powder of highly dispersed particles which is equipped with a feedback means by which the portion that has been separated selectively on account of less highly dispersed state during the step of treatment for selecting the mixture of a gas and the particles in the powder of highly dispersed core particles is to be transported to the final dispersing means at the last stage of the means for high dispersion treatment of particles, or if said final treating means is a means for selecting a mixture of a gas and the particles in a powder of highly dispersed particles which is equipped with a feedback means by which the portion that has been separated selectively on account of less highly dispersed state during the step of treatment for selecting the mixture of a gas and the particles in the powder of highly dispersed core particles is to be transported to a treating means upstream of the final dispersing means, said means for selecting a mixture of a gas and the particles in a powder of highly dispersed core particles is referred to as the final treating means in the group of means for high dispersion treatment of particles.

It should be noted here that any means for selecting a mixture of a gas and the particles in a powder of highly dispersed particles that is provided upstream of the means for selecting a mixture of a gas and the particles in a powder of highly dispersed particles that is equipped with a feedback means and which is the final treating means in the group of means for high dispersion treatment of particles ("upstream" may typically mean between the means for selecting a mixture of a gas and the particles in a powder of highly dispersed particles that is equipped with a feedback means and the final dispersing means, or upstream of the final dispersing means) is a constituent element of the group of means for high dispersion treatment of particles and this is true irrespective of whether the feedback means is used or not.

Dispersing Means

The means used to disperse particles is named a dispersing means. Any mechanism that has even a small or slight dispersing effect is usable as a dispersing means and it is used as a dispersing means in the invention. Consider, for example, a rotary feeder and an injection feeder for pneumatic conveying which are commonly used as supply means ["Funtai Kogaku Binran (A Handbook of Particle Technology)", Ed. by Funtai Kogakukai (Society of Particle Technology), published by Nikkan Kogyo Shimbunsha in 1966, pp. 568 and 571]. These apparatus also have a dispersing effect and, hence, they are dispersing means if used as means to achieve dispersion. The dispersion maintaining and promoting means which are to be discussed later are also dispersing means if they are used to achieve dispersion (for increasing

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5). Such dispersing means may be a single apparatus or machine or it may be a complex of apparatus and machines; to cover all of these cases, the dispersing means is collectively referred to as the "group of means for high dispersion treatment of particles".

The group of means for high dispersion treatment of particles is such that it operates on at least one selected mechanism of dispersion, such as dispersing the particles in a powder of core particles by a gas stream which accelerates and/or places them in a velocity gradient, dispersing the particles in a powder of core particles by impingement against a static obstacle and/or an obstacle composed of a rotating body, and dispersing the particles in a powder of core particles by mechanical deagglomeration which consists of a fluidized bed and/or pulsation and/or a rotary drum and/or vibrations and/or scraping.

Specifically, the group of means for high dispersion treatment of particles is equipped with at least one selected dispersing means, such as an ejector-type disperser, a Venturi-type disperser, a disperser using a capillary, an agitator, and an obstacle in a gas stream, a disperser using a jet blow, a disperser using a spiral tube and rotating blades, a disperser using rotating pins (Kacy mill), a fluidized bed-type disperser, a disperser using pulsation, a disperser using a rotary drum, a disperser using vibrations, a disperser using a vibrating screen and the scraping action of a scraper, an SAEI Gonnet-type disperser, a Chujo-type disperser, a Roller-type disperser, an office-type disperser, a B.M. type disperser, a Timbrell-type disperser, and a Wright-type disperser ["Funtai Kogaku Binran (A Handbook of Particle Technology)", Ed. by Funtai Kogakukai (Society of Particle Technology), published by Nikkan Kogyo Shimbunsha in 1966, p. 430].

Also usable are the dispersers described in official gazettes and exemplified blow: the disperser using agitating blades which is described in Unexamined Published Japanese Patent Application (kokai) Sho 56-1038; the disperser using a high-speed gas stream and dispersing nozzles which is described in Unexamined Published Japanese Patent Application (kokai) Sho 58-163454; the disperser using the dispersing action of rotating blades and the dispersing action of plasma jets which is described in Unexamined Published Japanese Patent Application (kokai) Sho 59-199027; the disperser using the dispersing action of plasma jets which is described in Unexamined Published Japanese Patent Application (kokai) Sho 59-207318; the disperser using an ejector and the dispersing action of plasma jets which is described in Unexamined Published Japanese Patent Application (kokai) Sho 59-216616; the disperser using an ejector and the dispersing action of ion streams which is described in Unexamined Published Japanese Patent Application (kokai) Sho 59-225739; the disperser using the dispersing action of plasma jets which is described in Unexamined Published Japanese Patent Application (kokai) Sho 59-156845; the disperser using the dispersing action of dispersing blades and a pressure gas which is described in Unexamined Published Japanese Patent Application (kokai) Sho 62-168421; the disperser using linear or annular slit jet ports which is described in Unexamined Published Japanese Patent Application (kokai) Sho 62-176527; the disperser using mesh blades which is described in Unexamined Published Japanese Patent Application (kokai) Sho 63-221828; the disperser using the dispersing action of high-speed gas streams issuing from jet nozzles which is described in Unexamined Published Japanese Patent Application (kokai) Sho 63-1629; the disperser using many small holes which is described in Unexamined Published Utility Model Application (kokai) Sho 62-8215; the ejector-type disperser which is described in Unexamined Published Japanese Utility Model Application (kokai) Sho 62-156954; and the disperser using small holes and orifices which is described in Unexamined Published Japanese Utility Model Application (kokai) Sho 62-6034.

Dispersing means advantageous for use in the group of means for high dispersion treatment of particles include the apparatus that are described in Japanese Patent Application Sho 63-311258, Japanese Patent Application Hei 1-71071, Japanese Patent Application Hei 2-218587, etc.

Means for Selecting a Mixture of a Gas and the Particles in a Powder of Highly Dispersed Core Particles

The means for selecting a mixture of a gas and the particles in a powder of highly dispersed core particles is a means that separates a mixture of a gas and the particles in a powder of less highly dispersed core particles from the mixture of a gas and the particles in the powder of core particles so as to select a mixture of a gas and the particles in a powder of highly dispersed core particles that is chiefly composed of those particles which are in a single-particle state. Agglomerating particles which are association of primary particles have a greater apparent diameter than the primary particles and, hence, they can be separated by a suitable means such as dry classifying means. An example of the means for selecting a mixture of a gas and the particles in a powder of highly dispersed core particles is at least one dry classifying means selected from among the following: classifying means using gravity, classifying means using inertia force, classifying means using centrifugal force, classifying means using static electricity, classifying means using a fluidized bed, etc.

Specific examples of the means for selecting the mixture of a gas and the particles in a powder of highly dispersed core particles include: a gravity classifier, an inertia classifier, a centrifugal classifier, a cyclone, an air separator, a micron separator, a microplex, a multiplex, a zigzag classifier, an acu-cut, a conical separator, a turbo classifier, a super separator, a dispersion separator, an elbow jet, a fluidized bed classifier, a virtual impactor, an O-Sepa, a sieve, a vibrating

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screen, a sifter ["Funtai Kogaku Binran (A Handbook of Particle Technology)", Ed. by Funtai Kogakukai (Society of Particle Technology), published by Nikkan Kogyo Shimbunsha in 1966, p. 514].

Mixture of a Gas and the Particles in a Powder of Core Particles

The mixture of a gas and the particles in a powder of core particles refers to (a) a homogeneous flow characterized in that the particles in a powder of core particles are suspended uniformly in a gaseous atmosphere (which may be called a "uniform suspension flow"), (b) a heterogeneous flow characterized in that the particles in a powder of core particles show a nonuniform distribution in certain regions of a gaseous atmosphere (which may be called an "inhomogeneous suspension flow"), (c) a flow involving a sliding layer of the particles in a powder of core particles (which may be called a "sliding flow"), or (d) a flow involving a stationary layer of the particles in a powder of core particles.

Mixture of a Gas and the Particles in a Powder of Less Highly Dispersed Core Particles

The mixture of a gas and the particles in a powder of less highly dispersed core particles refers to that portion of a mixture of a gas and the particles in a powder of core particles in which the powder particles are present in a gaseous atmosphere mainly in states other than the single particle state.

Mixture of a Gas and the Particles in a Powder of Highly Dispersed Core Particles

The mixture of a gas and the particles in a powder of highly dispersed particles refers to a mixture of a gas and the particles in a powder of core particles in which the powder particles are present in a gaseous atmosphere mainly in a single particle state. No matter how much high the degree of dispersion is, the mixture of a gas and the particles in a powder of highly dispersed core particles contains agglomerating particles in practice. The mixture of a gas and the particles in a powder of less highly dispersed particles contains non-agglomerating single particles in practice and can be selectively separated into a mixture of a gas and the particles in a powder of less highly dispersed particles and a mixture of a gas and the particles in a powder of highly dispersed particles. The mixture of a gas and the particles in a powder of less highly dispersed particles is converted to a mixture of a gas and the particles in a powder of highly dispersed particles by selective separation and/or redispersion of the agglomerating particles.

Recovery Means

The recovery means is a means for picking up the coated particles that have been prepared in the coating space. The part of the recovery means in which the recovery treatment is performed is called the "recovery section". The coated particles the coating on which has been provided by passage through the coating start region of the coating space are recovered by direct pickup from the gaseous atmosphere or after temporary storage following pickup from the gaseous atmosphere or together with a gas.

Usable as the recovery section of the recovery means is one of the following members: the recovery section of the recovery means that uses barriers (obstacles), the recovery section of the recovery means that uses gravity, the recovery section of the recovery means that uses inertia forces, the recovery section of the recovery means that uses centrifugal forces, the recovery section of the recovery means that uses coulombic attractive forces, the recovery section of the recovery means that uses thermophoretic forces, the recovery section of the recovery means that uses Brownian diffusion, the recovery section of the recovery means that uses the pulling force of the back pressure, evacuation, etc.

Advantageous examples of the recovery section of the recovery means include a gravity dust collector, an inertia dust collector, a centrifugal dust collector, a filtration dust collector, an electrostatic precipitator, a scrubbing dust collector, a particle packed layer, a cyclone, a bag filter, a ceramic filter, a scrubber, etc.

Bonding Material

The bonding material to be used to manufacture diamond or high-pressure phase boron nitride sinters in accordance with the invention is selected from among those bonding materials which can be sintered densely to a density of at least 80% by sintering at pressures less than 3000 MPa and at temperatures not exceeding 1850°C. Advantageously, those bonding materials are selected which will not promote the phase transition of diamond or high-pressure phase boron nitride to the lower pressure phase (graphite or hBN). Alternatively, those bonding materials are selected which, when sintered at pressures less than 3000 MPa and at temperatures not exceeding 1850°C, will react with diamond or boron nitride to yield dense reaction products that have densities of at least 95%. More preferably, those substances are selected which, when sintered at pressures less than 2000 MPa and at temperatures not exceeding 1850°C, will produce bonding materials that are dense (density ≥ 80%) and/or of high hardness (Vickers hardness ≥ 800). Feed powders which, when sintered at pressures less than 3000 MPa and at temperatures not exceeding 1850°C, will yield dense

bonding materials having densities of at least 80% are selected from powders or particles that contain at least one of the metals, semiconductors, metalloids and rare earth metals of groups 1a, 2a, 3a, 4a, 5a, 6a, 7a, 1b, 2b, 3b, 4b, 5b, 6b, 7b and 8 of the periodic table and/or at least one compound containing at least one of those metals, semiconductors, metalloids and rare earth metals. More specifically, such powders or particles can be those of at least one member which is selected from among B, Ti, Zr, Hf, Ta, Nb, V, SiC, TiC, ZrC, B_4C, WC, HfC, TaC, NbC, Si_3N_4, TiN, ZrN, AlN, HfN, TaN, TiB, TiB_2, ZrB_2, HfB, HfB_2, LaB_6 $MoSi_2$, BP, Al_2O_3, Al_2SiO_5 (mullite), ZrO_2 (PSZ: partially stabilized zirconia with Y_2O_3, MgO or CaO, or TZP: tetragonal zirconia polycrystalline compound), and $MgAl_2O_4$ (spinel). To take alumina as an example, a suitable example is a high purity, easily sintrable fine feed, such as the alumina described in Unexamined Published Japanese Patent Application (kokai) Sho 62-151618 which is produced by pyrolysis of an ammonium aluminum carbocate and that is prefered since it can be densified at a temperature of about 1400°C even by normal sintering at atmospheric pressure. Another suitable example is a fine, high-purity alumina powder that contains up to 10% by volume of magnesia (MgO) and/or titania (TiO_x, x = 1 - 2) which are effective in promoting the sintering of alumina and if this condition is met, high-purity aluminas other than the alumina described in Unexamined Published Japanese Patent Application (kokai) Sho 62-151618 supra may be used, as exemplified by high-purity (≥ 99%) and easily sintrable aluminas that are composed of fine (≤ 1 µm) particles and which are typically produced by the Bayer process, hydrolysis of organic aluminum, pyrolysis of aluminum alum, the ethylene chlorohydrin process, submerged arc discharge, etc.

Besides the aluminas mentioned above, zirconium oxides may also be selected and advantageous examples include the easily sintrable yttria partially stabilized zirconia (2-4 mol% Y_2O_3-ZrO_2) powder which is produced by coprecipitation, and an alumina-zirconia base powder [FC Report, 1(5) (1983) 13-17]. Also selectable is a titanium oxide or titania powder (TiO_2: Summaries of Lectures Read at the 19th Symposium on High-Pressure Technology (1978) p. 174].

Another example that can be selected is titanium nitride [TiN; Yamada et al., Yogyo Kyokaishi (Journal of The Ceramic Society of Japan, 89, (1981) 621-625].

In the next place, the group of means for high dispersion treatment of particles which is adopted to prepare the coated diamond or high-pressure phase boron nitride particles is described below with reference to accompanying drawings.

Description of the Drawings of the Group of Means for High Dispersion Treatment of Particles

Fig. 2a is a block diagram showing an example of the basic composition of the group of means for high dispersion treatment of particles as it is used to prepare coated diamond or high-pressure phase boron nitride particles. The group is composed of a final dispersing means A for dispersing the particles in a powder of core particles and a constituent element g of the group of means for dispersion treatment which is upstream of the final dispersing means. Shown by x is a mixture of a gas and the particles in a powder of highly dispersed core particles which are part of the particles in the powder of core particles and which are present in a gaseous atmosphere mainly in a single-particle state. Constituent element g may be any treating means such as dispersing means, supply means, and means for selecting a mixture of a gas and the particles in a powder of highly dispersed core particles, which may be used either individually or in combination. Constituent element g is optional and may be omitted. The group of means for high dispersion treatment of particles is advantageously of such a design that after treatment by the final treating means A, one of the following values of dispersity β can be realized: (1) at least 70% for a powder of core particles that have an average diameter of no more than 10 µm in the frequency distribution by volume; (2) at least 80% for a powder of core particles that have an average diameter in excess of 10 µm but no more than 20 µm in the frequency distribution by volume; (3) at least 90% for a powder of core particles that have an average diameter in excess of 20 µm but no more than 50 µm in the frequency distribution by volume; (4) at least 95% for a powder of core particles that have an average diameter in excess of 50 µm but no more than 300 µm in the frequency distribution by volume; (5) at least 97% for a powder of core particles that have an average diameter in excess of 300 µm but no more than 800 µm in the frequency distribution by volume; and (6) at least 99% for a powder of core particles that have an average diameter in excess of 800 µm in the frequency distribution by volume.

Fig. 2b is a block diagram showing a second example of the basic composition of the group of means for high dispersion treatment of particles as it is used to prepare coated diamond or high-pressure phase boron nitride particles. The group is composed of a final dispersing means A for dispersing the particles in a powder of core particles, final means B for selecting a mixture of a gas and the particles in a powder of highly dispersed core particles at the last stage which is equipped with feedback means C by which a mixture of a gas and the particles in a powder of less highly dispersed core particles η as separated from a mixture of a gas and the particles in a powder of highly dispersed core particles in which the powder particles are present in a gaseous atmosphere mainly in a single-particle state is fed back to the final dispersing means A, a constituent element g of the group of means for dispersion treatment which is upstream of the final dispersing means, and a constituent element g of the group of means for high dispersion treatment of particles which is provided between the final dispersing means and the final selecting means. As for the mixture x and the constituent element g, the explanation made in connection with Fig. 2a will apply and, hence, no detailed description is made. Constituent element g may be any treating means other than the dispersing means, such as supply means and

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selecting means, which may be used either individually or in combination. Constituent elements g and g are optional and may be omitted. The group of means for high dispersion treatment of particles is advantageously of such a design that after treatment by selecting means B which is the final treating means, the above-mentioned values of dispersity can be realized for powders of core particles having the above-mentioned average diameters.

Fig. 3c is a block diagram showing a third example of the basic composition of the group for high dispersion treatment of particles as it is used to prepare coated diamond or high-pressure phase boron nitride particles. The group is composed of a final dispersing means A for dispersing the particles in a powder of core particles, final means B for selecting a mixture of a gas and the particles in a powder of highly dispersed core particles at the last stage which is equipped with feedback means C by which a mixture of a gas and the particles in a powder of less highly dispersed core particles η as separated from a mixture of a gas and the particles in a powder of highly dispersed core particles in which the powder particles are present in a gaseous atmosphere mainly in a single-particle state is fed back to a treating means upstream of the final dispersing means A, a constituent element d of the group of means for high dispersion treatment of particles which is upstream of the final dispersing means, and a constituent element e of the group of means for high dispersion treatment of particles which is provided between the final dispersing means and the final selecting means. As for the mixture c, constituent elements d and e, and the group of means for high dispersion treatment of particles, the foregoing explanation will apply and no detailed description is made.

Having the construction described above, the group of means for high dispersion treatment of particles under discussion may include powder supply sources such as a supply vessel and core particle generating means. To take the case of Fig. 2c as an example, the group of means for high dispersion treatment of particles may of course adopt such a composition that the mixture η is fed back to a supply vessel by the feedback means C. It also goes without saying that the step of dispersion by the group of means for high dispersion treatment of particles may be preceded by a disintegrating step in which the particles in the powder of core particles are disintegrated and/or otherwise reduced in size.

Typical specific examples of these basic compositions of the group of means for high dispersion treatment of particles are described below in greater detail with reference to more detailed block diagrams.

Design 1

Fig. 3a is a block diagram illustrating a first specific design of the group of means for high dispersion treatment of particles as it is used to prepare coated diamond or high-pressure phase boron nitride particles and the drawing corresponds to Fig. 2a. The group shown in Fig. 3a is composed of a supply vessel 100 for supplying the powder of core particles to be coated, and the final dispersing means A for dispersing the powder of core particles to be coated.

Design 2

Fig. 3b is a block diagram illustrating a second specific design of the group of means for high dispersion treatment of particles as it is used to prepare coated diamond or high-pressure phase boron nitride particles and the drawing corresponds to Fig. 2a. The group shown in Fig. 3b is composed of a supply vessel 100 for supplying the powder of core particles to be coated, dispersing means a for dispersing the powder of core particles to be coated, and the final dispersing means A for dispersing the powder of core particles to be coated.

Design 3

Fig. 3c is a block diagram illustrating a third specific design of the group of means for high dispersion treatment of particles as it is used to prepare coated diamond or high-pressure phase boron nitride particles and the drawing corresponds to Fig. 2a. The group shown in Fig. 3c is composed of a supply vessel 100 for supplying the powder of core particles to be coated, dispersing means a for dispersing the powder of core particles to be coated, feedback means C by which a mixture of a gas and the particles in a powder of less highly dispersed core particles η as separated from a mixture of a gas and the particles in a powder of highly dispersed core particles in which the powder particles are present in a gaseous atmosphere mainly in a single-particle state is fed back from the mixture of a gas and the particles in the powder of core particles (which have been dispersed by the dispersing means a) to the dispersing means a, means b for selecting the mixture of a gas and the particles in a powder of highly dispersed particles, by which the mixture of a gas and the particles in a powder chiefly composed of highly dispersed core particles is introduced into the final dispersing means A, and the final dispersing means A for dispersing the powder of core particles to be coated.

Design 4

Fig. 3d is a block diagram illustrating a fourth specific design of the group of means for high dispersion treatment of particles as it is used to prepare coated diamond or high-pressure phase boron nitride particles and the drawing in Fig. 3d corresponds to Fig. 2b. The group shown in Fig. 3d is composed of a supply vessel 100 for supplying the powder

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of core particles to be coated, a final dispersing means A for dispersing the powder of core particles to be coated, feedback means C by which a mixture of a gas and the particles in a powder of less highly dispersed core particles is as separated from a mixture of a gas and the particles in a powder of highly dispersed particles in which the powder particles are present in a gaseous atmosphere mainly in a single-particle state is fed back from the mixture of a gas and the particles in the powder of core particles (which have been dispersed by the final dispersing means A) to the dispersing means A, and final means B for selecting the mixture of a gas and the particles in the powder of highly dispersed core particles at the final stage and which discharges the mixture of a gas and the particles in the powder of highly dispersed core particles.

Design 5

Fig. 3e is a block diagram illustrating a fifth specific design of the group of means for high dispersion treatment of particles as it is used to prepare coated diamond or high-pressure phase boron nitride particles and the drawing corresponds to Fig. 2b. The group shown in Fig. 3e is composed of a supply vessel 100 for supplying the powder of core particles to be coated, dispersing means a for dispersing the powder of core particles to be coated, a final dispersing means A for dispersing the powder of core particles to be coated, feedback means C by which a mixture of a gas and the particles in a powder of less highly dispersed core particles is as separated from a mixture of a gas and the particles in a powder of highly dispersed particles in which the powder particles are present in a gaseous atmosphere mainly in a single-particle state is fed back from the mixture of a gas and the particles in the powder of core particles (which have been dispersed by the final dispersing means A) to the dispersing means A, and final means B for selecting the mixture of a gas and the particles in the powder of highly dispersed core particles at the final stage and which discharges the mixture of a gas and the particles in the powder of highly dispersed core particles.

Design 6

Fig. 3f is a block diagram illustrating a sixth specific design of the group of means for high dispersion treatment of particles as it is used to prepare coated diamond or high-pressure phase boron nitride particles and the drawing corresponds to Fig. 2b. The group shown in Fig. 3f is composed of a supply vessel 100 for supplying the powder of core particles to be coated, means b for selecting a mixture of a gas and the particles in a powder of highly dispersed core particles, which rejects a mixture of a gas and the particles in a powder chiefly composed of less highly dispersed core particles from the mixture of a gas and the particles in the powder of core particles and which introduces into dispersing means A the mixture of a gas and the particles in a powder chiefly composed of highly dispersed core particles, final dispersing means A for dispersing the particles in the selectively separated powder of core particles, feedback means C by which a mixture of a gas and the particles in a powder of less highly dispersed core particles as separated from a mixture of a gas and the particles in a powder of highly dispersed particles in which the powder particles are present in a gaseous atmosphere mainly in a single-particle state is fed back from the mixture of a gas and the particles in the powder of core particles (which have been dispersed by the final dispersing means A) to the dispersing means A, and final means B for selecting the mixture of a gas and the particles in the powder of highly dispersed core particles at the final stage and which discharges the mixture of a gas and the particles in the powder of highly dispersed core particles.

Design 7

Fig. 3g is a block diagram illustrating a seventh specific design of the group of means for high dispersion treatment of particles as it is used to prepare coated diamond or high-pressure phase boron nitride particles and the drawing corresponds to Fig. 2b. The group shown in Fig. 3g is composed of a supply vessel 100 for supplying the powder of core particles to be coated, dispersing means a for dispersing the powder of core particles to be coated, a final dispersing means A for dispersing the powder of core particles to be coated, feedback means C by which a mixture of a gas and the particles in a powder of less highly dispersed core particles as separated from a mixture of a gas and the particles in a powder of highly dispersed particles in which the powder particles are present in a gaseous atmosphere mainly in a single-particle state is fed back from the mixture of a gas and the particles in the powder of core particles (which have been dispersed by the final dispersing means A) to the dispersing means A, and final means B for selecting the mixture of a gas and the particles in the powder of highly dispersed core particles at the final stage and which discharges the mixture of a gas and the particles in the powder of highly dispersed core particles.

To maintain the thus achieved high dispersive state of the particles, means for maintaining dispersion in a gaseous atmosphere may be added to the group of means for high dispersion treatment of particles. The term "means for maintaining dispersion in a gaseous atmosphere" as used herein refers to means that maintains a desired value of dispersibility by preventing reagglomeration of the particles in the powder of core particles that have been carried dispersively in a gaseous atmosphere. To promote the thus achieved high dispersive state of the core particles, means for promoting dispersion in a gaseous atmosphere may be added between the group of means for high dispersion treatment of particles

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and the coating chamber. The term "means for promoting dispersion in a gaseous atmosphere" as used herein refers to means which is primarily intended to promote the redispersion of that part of the particles in the powder of core particles carried dispersively in a gaseous atmosphere which have agglomerated again, thereby retarding the drop in the dispersive state of the powder; the term also refers to means that promotes redispersion in such a way that the state of dispersion that has dropped once can be restored to the initial high dispersive state.

Advantageous examples of said means for maintaining dispersion in a gaseous atmosphere or promoting dispersion in a gaseous atmosphere include a pipe vibrator, a pipe heater, a plasma generator, an electric charger, etc.

The pipe vibrator is means in which a pipe equipped with an oscillator is vibrated to provide the particles dispersed in a gaseous atmosphere with vibrations uncomparable to those imparted by dispersers, whereby reagglomeration is suppressed to maintain the high dispersive state or the dispersion of reagglomerating particles is promoted.

The pipe heater is means in which a heated pipe imparts heat to a carrier gas from outside, thereby expanding the carrier gas so that its flow velocity is accelerated to a level that is too fast to be comparable to that achieved by dispersers, whereby reagglomeration of the particles is suppressed while promoting the dispersion of reagglomerating particles.

The plasma generator is means in which a plasma is generated in the gaseous atmosphere which dispersively carries the powder of core particles, with the resulting plasma ions being allowed to impinge against the core particles, whereby reagglomeration is suppressed to maintain the high dispersive state or the dispersion of reagglomerating particles is promoted.

The electric charger is means in which corona discharge, electron beams, radiations or some other method is used to generate monopolar ions in the gaseous atmosphere which dispersively carries the powder of core particles, which are subsequently passed through the monopolar ion atmosphere to be charged to a single polarity, whereupon electrostatic repulsive forces either suppress reagglomeration to maintain the high dispersive state or promote the dispersion of reagglomerating particles.

The powder of core particles which have been rendered to assume a highly dispersive state in the manner described on the foregoing pages is sent to the coating chamber so that the surfaces of the particles are coated with the coat forming substance. The coating compartment is provided with the coating space including the coating start region.

The group of means for high dispersion treatment of particles is desirably coupled directly to the coating chamber. If necessary, they may be connected together by means of a hollow member and/or a pipe that are indispensable to transport. In this case, too, it is essential that the dispersity *β* in the coating start region be adjusted to one of the values within the ranges set forth hereinabove.

If the group of means for high dispersion treatment of particles is coupled to the coating chamber located separately, the powder of core particles suffices to be introduced into the coating chamber as it maintains the state of dispersion that has been achieved by said group of means for high dispersion treatment of particles. To this end, means for maintaining dispersion in a gaseous atmosphere (which is a device for maintaining the state of dispersion of the powder of core particles) and/or means for promoting dispersion in a gaseous atmosphere (which is a device for enhancing the state of dispersion) and/or means for selecting a mixture of a gas and the particles in a powder of highly dispersed particles (which separates the powder portion of less highly dispersed core particles from the mixture of a gas and the particles in the powder of core particles, thereby selecting a mixture of a gas and the particles in a powder of highly dispersed core particles, the major part of which comprises particles in a single-particle state) may be provided between the group of means for high dispersion treatment of particles and the coating chamber.

The invention also contemplates that when used to prepare coated diamond or high-pressure phase boron nitride particles, the group of means for high dispersion treatment of particles may share one or more portions of the space with (1) the coating chamber, (2) the coating space or (3) the coating start region.

For example, the group of means for high dispersion treatment of particles may share its dispersing space with the coating chamber, or with the coating space having the coating start region, or with the coating start region.

The term "coating start region" as used herein refers to the region where the precursor of the coat forming substance that has been generated via the vapor phase and/or the precursor of the coat forming substance in a vapor phase state contacts and/or impinges against the powder of highly dispersed core particles which has been transported in the state of dispersion that has one of the following values of dispersity *β*, whereupon coating of the individual particles is started: (1) at least 70% for a powder of core particles that have an average diameter of no more than 10 μm in the frequency distribution by volume; (2) at least 80% for a powder of core particles that have an average diameter in excess of 10 μm but no more than 30 μm in the frequency distribution by volume; (3) at least 90% for a powder of core particles that have an average diameter in excess of 30 μm but no more than 80 μm in the frequency distribution by volume; (4) at least 95% for a powder of core particles that have an average diameter in excess of 80 μm but no more than 300 μm in the frequency distribution by volume; (5) at least 97% for a powder of core particles that have an average diameter in excess of 300 μm but no more than 800 μm in the frequency distribution by volume; and (6) at least 99% for a powder of core particles that have an average diameter in excess of 800 μm in the frequency distribution by volume. Five typical embodiments of the coating start region are shown schematically in Figs. 4a - 4e.

In Figs. 4a - 4e, the coating start region is indicated by numeral 2.

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In the case shown in Fig. 4a, the coating start region 2 of the coating space in which the coating of core particles is started as they are dispersed to one of the above-listed values of dispersity β in accordance with the average diameter of those core particles is provided in such a way that it surrounds the group of means for high dispersion treatment of particles or its discharge section 1.

In the case shown in Fig. 4b, the coating start region 2 of said coating space is provided in such a way that it is passed by all the particles 4 in the powder of core particles as they are discharged from the group of means for high dispersion treatment or its discharge section 1.

By adopting these arrangements, the particles in all powders of core particles can start to be coated as they are dispersed to the above-listed values of dispersity β.

In the case shown in Fig. 4c, the coating start region 2 of said coating space is provided in such a way that it is passed in all cases by those particles 4 in the powder of core particles which are discharged from the group of means for high dispersion treatment of particles or its discharge section 1 to enter the recovery section 5.

In the case shown in Fig. 4d, the coating start region 2 of said coating space is provided in such a way that it surrounds the recovery section 5.

In the case shown in Fig. 4e, the recovery section 5 is provided in a position that can be reached solely by the particles in the mixture of a gas and the particles in the powder of highly dispersed core particles. Hence, the region indicated by 6 in Fig. 4e is selection means using gravity. The coating start region 2 of said coating space is provided in the hatched area of Fig. 4e so that it is passed in all cases by those particles in the mixture of a gas and the particles in the powder of highly dispersed core particles which enter the recovery section. This arrangement insures that only the core particles that started to be coated as they were dispersed to the above-listed values of dispersity β can be recovered and there is no chance that the core particles that did not pass through the coating start region will intermingle with the coated particles which emerge from the coating start region.

As one can understand from the foregoing description, the apparatus for implementing the present invention are composed of the group of means for high dispersion treatment of particles and the coating chamber or, alternatively, the group of means for high dispersion treatment of particles, the coating chamber and the recovery means. The constituent elements of the apparatus can be combined in various manners and several designs of these apparatus are described below with reference to drawings.

Apparatus Design 1

Fig. 5a is a block diagram showing the construction of a first apparatus for producing coated diamond or high-pressure phase boron nitride particles. As shown, the apparatus is composed of the main body of the production equipment 2-A (coating apparatus), coating chamber 2-B1, coating space 2-B2, coating start region 2-B3, the group of means for high dispersion treatment of particles 2-C1, and recovery means 2-D. The group of means for high dispersion treatment of particles 2-C1 is coupled directly to the coating chamber 2-B1.

Apparatus Design 2

Fig. 5b is a block diagram showing the construction of a second apparatus for producing coated diamond or high-pressure phase boron nitride particles. As shown, the apparatus is composed of the main body of the production equipment 2-A (coating apparatus), coating chamber 2-B1, coating space 2-B2, coating start region 2-B3, the group of means for high dispersion treatment of particles 2-C1, indispensable hollow member 2-C2, and recovery means 2-D. The group of means for high dispersion treatment of particles 2-C1 is connected to the coating chamber 2-B1 via the indispensable hollow member 2-C2.

Apparatus Design 3

Fig. 5c is a block diagram showing the construction of a third apparatus for producing coated diamond or high-pressure phase boron nitride particles. As shown, the apparatus is composed of the main body of the production equipment 2-A (coating apparatus), coating chamber 2-B1, coating space 2-B2, coating start region 2-B3, the group of means for high dispersion treatment of particles 2-C1, means for maintaining dispersion in gaseous atmosphere 2-C3, and recovery means 2-D. The group of means for high dispersion treatment of particles 2-C1 is connected to the coating chamber 2-B1 via the means for maintaining dispersion in gaseous atmosphere 2-C3.

Apparatus Design 4

Fig. 5d is a block diagram showing the construction of a fourth apparatus for producing coated diamond or high-pressure phase boron nitride particles. As shown, the apparatus is composed of the main body of the production equipment 2-A (coating apparatus), coating chamber 2-B1, coating space 2-B2, coating start region 2-B3, the group of means

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for high dispersion treatment of particles 2-C1, and recovery means 2-D. The group of means for high dispersion treatment of particles 3-C1 shares space with the coating chamber 2-B1.

Apparatus Design 5

Fig. 5e is a block diagram showing the construction of a fifth apparatus for producing coated diamond or high-pressure phase boron nitride particles. As shown, the apparatus is composed of the main body of the production equipment 2-A (coating apparatus), coating chamber 2-B1, coating space 2-B2, coating start region 2-B3, the group of means for high dispersion treatment of particles 2-C1, and recovery means 2-D. The group of means for high dispersion treatment of particles 2-C1 is provided within the coating chamber 2-B1.

Apparatus Design 6

Fig. 5f is a block diagram showing the construction of a sixth apparatus for producing coated diamond or high-pressure phase boron nitride particles. As shown, the apparatus is composed of the main body of the production equipment 2-A (coating apparatus), coating chamber 2-B1, coating space 2-B2, coating start region 2-B3, the group of means for high dispersion treatment of particles 2-C1, and recovery means 2-D. The coating chamber 2-B1 is provided within the dispersing space of the group of means for high dispersion treatment of particles 2-C1.

Apparatus Design 7

Fig. 5g is a block diagram showing the construction of a seventh apparatus for producing coated diamond or high-pressure phase boron nitride particles. As shown, the apparatus is composed of the main body of the production equipment 2-A (coating apparatus), coating chamber 2-B1, coating space 2-B2, coating start region 2-B3, the group of means for high dispersion treatment of particles 2-C1, recovery means 2-D, and recoating supply means 2-E. The coated particles issuing from the recovery means 2-D can be transported by the recoating supply means 2-E to the group of means for high dispersion treatment of particles 2-C1 for performing repeated coating treatments.

Either one of the apparatuses of these designs can produce coated diamond or high-pressure phase boron nitride particles.

The coated particles in which the powder of core particles (diamond or high-pressure phase boron nitride particles) has its surface coated with the coat forming substance may be coated again with the coat forming substance or subjected to repeated recoating operations. In these cases, the coated particles are sent to the recoating supply means. The term "recoating supply means" as used herein refers to means for transporting the coated particles to the group of means for high dispersion treatment of particles to perform recoating. Specifically, the recoating supply means is furnished with (a) means for recovering the coated particles and (b) means for transporting said coated particles from the recovery means (a) to the group of means for high dispersion treatment of particles. Alternatively, the recoating supply means is furnished with (a) means for recovering the coated particles, (b) means for transporting said coated particles from the recovery means (a) to the group of means for high dispersion treatment of particles, and (c) means for classifying the coated particles. Given large coating weights, the particle size distribution of the coated particles will vary from that of the particles in the powder of uncoated core particles. Under these conditions, it is effective to perform the recoating operation after the particle size distribution of the coated particles is adjusted by the classifying means (a).

The recoating operation may be repeated depending on the need and the coating weight of the coat forming substance can be set to a desired level. If necessary, the coating operation may be repeated with the type of the coat forming substance varied and, thus, a substance of more than one component can be coated in multiple layers as the coat forming substance.

The equipment for producing coated particles that is to be used in the present invention is in no way limited as long as it is capable of applying the coat forming substance over the surfaces of the particles in the powder of core particles by vapor phase processes involving transition through the vapor phase. For example, chemical vapor deposition (CVD) equipment can be used, as exemplified by a thermal CVD apparatus, a plasma-assisted CVD apparatus, apparatus for CVD utilizing electromagnetic waves (i.e., visible light CVD, laser CVD, ultraviolet CVD, infrared CVD and far-infrared CVD) and a MOCVD apparatus. Alternatively, physical vapor deposition (PVD) equipment can be used, as exemplified by a vacuum evaporation apparatus, an ion sputtering apparatus and an ion plating apparatus. Stated more specifically, a suitable apparatus for producing coated particles is described in the official gazette of Unexamined Published Japanese Patent Application (Kokai) Hei 3-75502, entitled "Particles Coated with Superfine Grains on the Surfaces and A Process for Producing Such Coated Particles".

As described on the foregoing pages, the process of the present invention for producing coated diamond or high-pressure phase boron nitride comprises the steps of charging into the coating space the particles in the powder of core particles which are diamond or high-pressure phase boron nitride particles and then permitting the precursor of the coat forming substance which is generated via the vapor phase and/or the precursor of the coat forming substance in a vapor-

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phase state to contact and/or impinge against the particles in said powder of core particles so that their surfaces are coated with the coat forming substance. Five basic processes for coating the core particles with the coat forming substance are summarized below.

I. A coating method comprising:

(A) a dispersing step in which the particles in the powder of core particles are dispersed in a gaseous atmosphere by the group of means for high dispersion treatment of particles so as to make a mixture of a gas and the particles in a powder of highly dispersed core particles; and

(B) a coating step in which the core particles in said gas-particle mixture, as they have been dispersed with the dispersity β adjusted to the values within the ranges set forth above, are allowed to contact and/or impinge against the coat forming substance in the coating start region of the coating space, thereby starting the coating operation.

II. A coating method comprising:

(A) a dispersing step in which the particles in the powder of core particles are dispersed in a gaseous atmosphere by the group of means for high dispersion treatment of particles which realize the adjustment of dispersity β to the values within the ranges set forth above, thereby making a mixture of a gas and the particles in a powder of highly dispersed core particles; and

(B) a coating step in which the core particles in said gas-particle mixture, as they have been dispersed with the dispersity β adjusted to the values within the ranges set forth above, are allowed to contact and/or impinge against the coat forming substance in the coating start region of the coating space, thereby starting the coating operation.

III. A coating method comprising:

(A) a dispersing step in which the particles in the powder of core particles are dispersed in a gaseous atmosphere by the group of means for high dispersion treatment of particles which realize the adjustment of dispersity β to the values within the ranges set forth above, thereby making a mixture of a gas and the particles in a powder of highly dispersed core particles;

(B) a transport step in which the highly dispersed core particles in said gas-particle mixture are transported directly to a subsequent coating step; and

(C) the coating step in which the transported core particles in said gas-particle mixture, as they have been dispersed with the dispersity β adjusted to the values within the ranges set forth above, are allowed to contact and/or impinge against the coat forming substance in the coating start region of the coating space, thereby starting the coating operation.

IV. A coating method comprising:

(A) a dispersing step in which the particles in the powder of core particles are dispersed in a gaseous atmosphere by the group of means for high dispersion treatment of particles which realize the adjustment of dispersity β to the values within the ranges set forth above, thereby making a mixture of a gas and the particles in a powder of highly dispersed core particles;

(B) a transport step in which the highly dispersed core particles in said gas-particle mixture are transported via at least one member indispensable to transport that is selected from the group consisting of a hollow member, an intermediate member composed of a member that forms a hollow portion, and a pipe; and

(C) a coating step in which the transported core particles in said gas-particle mixture, as they have been dispersed with the dispersity β adjusted to the values within the ranges set forth above, are allowed to contact and/or impinge against the coat forming substance in the coating start region of the coating space, thereby starting the coating operation.

V. A coating method comprising:

(A) a dispersing step in which the particles in the powder of core particles are dispersed in a gaseous atmosphere by the group of means for high dispersion treatment of particles which realize the adjustment of dispersity β to the values within the ranges set forth above, thereby making a mixture of a gas and the particles in a powder of highly dispersed core particles;

(B) a transport step in which the highly dispersed core particles in said gas-particle mixture are transported to a subsequent coating step via at least one means selected from the group consisting of means for maintaining the dispersion in a gaseous atmosphere of the highly dispersed core particles in said gas-particle mixture, means for

enhancing the dispersion in a gaseous atmosphere of the highly dispersed core particles in said gas-particle mixture, and means for separating a mixture of a gas and the particles in a powder of less highly dispersed core particles from said gas-particle mixture so as to select a mixture of a gas and the particles in a powder of highly dispersed core particles in which the core particles are present in the gaseous atmosphere mainly in a single-particle state; and

(C) the coating step in which the transported particles in said mixture of a gas and the powder of highly dispersed core particles, as they have been dispersed with the dispersity β adjusted to the values within the ranges set forth above, are allowed to contact and/or impinge against the coat forming substance in the coating start region of the coating space, thereby starting the coating operation.

In each of the coating methods I - V, it is preferred that the coating start region of the coating space is located in either one of the following portions of the space region in which the dispersity β of the particles in the powder of highly dispersed core particles as mixed with a gas and which have been dispersed by the group of means for high dispersion treatment of particles is adjusted to the values within the ranges set forth above.

the space region that includes planes through which all particles in the powder of highly dispersed core particles as mixed with a gas will pass; or

the space region that includes planes through which all particles to be recovered into the recovery section of the recovery means will pass.

Alternatively, in each of the coating methods I and II, it is preferred that at least one part of the dispersing step, in which the particles in the powder of core particles are dispersed in a gaseous atmosphere by the group of means for high dispersion treatment of particles which realize the adjustment of dispersity β to the values within the ranges set forth above, thereby making a mixture of a gas and the particles in a powder of highly dispersed core particles, is performed sharing one or more parts of the space with at least one part of the coating step.

It should be noted here that the coated diamond or high-pressure phase boron nitride particles occasionally form lumps as a result of association of several particles that contact one another via the coat forming substance on the coated particles. Thus, the powder of the coated diamond or high-pressure phase boron nitride particles consists of coated particles in a single-particle state, lumps formed by the association of from several to several tens of such discrete coated particles as a result of contacting one another, and larger lumps formed by the association of more of such discrete coated particles as a result of contacting one another, and the shapes and sizes of the particles and lumps are nonuniform and irregular. The lumps which are the associations of the discrete coated particles are preferably deagglomerated and/or otherwise reduced in size before they are subjected to the shaping (molding) or sintering process. To deagglomerate and/or otherwise reduce in size the associating lumps of the coated diamond or high-pressure phase boron nitride particles, various deagglomerating means may be utilized, as exemplified by ball mills, vibrating ball mills, mortars and jet mills, etc. Alternatively, the discrete coated particles may be selectively separated from the associating lumps of such discrete coated particles, so that only the discrete coated particles can be subjected to the shaping or sintering process.

In accordance with the present invention, the thus coated diamond or high-pressure phase boron nitride particles or mixtures containing such particles are then sintered at pressures no less than 2000 MPa and at elevated temperatures, or at pressures of less than 2000 MPa and at temperatures not exceeding 1850°C under such sintering pressure and temperature conditions that diamond or high-pressure phase boron nitride is thermodynamically metastable if not stable.

Alternatively, the coated diamond or high-pressure phase boron nitride particles are mixed with a bonding material in volume ratios of (1 - 80):(99 - 10) that is sinterable to a density of at least 85% at pressures less than 2000 MPa and at temperatures not exceeding 1850°C under such pressure and temperature conditions that diamond or high-pressure phase boron nitride is thermodynamically metastable, and the mixture is then sintered at pressures less than 2000 MPa and at temperatures not exceeding 1850°C under such sintering pressure and temperature conditions that diamond or high-pressure phase boron nitride is thermodynamically metastable if not stable.

If an auxiliary agent, or a substance that develops another function, is to be added, the substance may be in a powder, plate or particulate form. More specifically, the substance may be selected from among powders and/or particles, etc. that are composed of at least one member selected from the group consisting of metals, semiconductors, metalloids, and rare earth metals of groups 1a, 2a, 3a, 4a, 5a, 6a, 7a, 1b, 2b, 3b, 4b, 5b, 6b, 7b and 8 of the periodic table, as well as oxides, nitrides, carbides, oxynitrides, oxycarbides, carbonitrides, oxycarbonitrides, borides and silicides of these elements, as exemplified below:

Al, B, Si, Fe, Ni, Co, Ti, Nb, V, Zr, Hf, Ta, W, Re, Cr, Cu, Mo, TiAl, Ti_3Al, $TiAl_3$, TiB, NiAl, Nb_3Al, SiC, B_4C, Cr_3C_2, TiC, ZrC, WC, W_2C, HfC, TaC, Ta_2C, NbC, VC, Mo_2C, Si_3N_4, TiN, ZrN, Si_2N_2O, AlN, HfN, VxN (x = 1 ~ 3), NbN, TaN, Ta_2N, TiB, TiB_2, ZrB_2, VB, V_3B_2, VB_2, NbB, NbB_2, TaB, TaB_2, MoB, Mo_2B_4, MoB_4, Mo_2B, WB, W_2B, W_2B5, LaB_6, BP, $B_{13}P_2$, $MoSi_2$, Al_2O_3, ZrO_2 (PSZ: partially stabilized zirconia with the addition of Y_2O_3, MgO or CaO; or TZP: tetragonal zirconia polycrystalline compound), $MgAl_2O_4$ (spinel) and Al_6SiO_2 (mullite).

The auxiliary agent may be a fibrous substance. The fibrous substance to be mixed with the coated diamond or high-pressure phase boron nitride particles is composed of at least one of elemental metals and compounds and has such a morphology that the width is no more than 500 μm, with the length to width ratio being at least 2. The fibrous

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substance comprises bar- or rod-like substances having widths of no more than 500 μm, with the length to width ratio being at least 2, and/or filaments which are continuous fibers formed by melt spinning and/or short fibers produced by crystals themselves to give shape and/or whiskers produced by unidirectional crystal growth into a fibrous form. The whiskers comprise intrinsic whiskers which, according to the definition, are formed without experiencing any phase transition or any phenomenon such as chemical reactions that affect the overall volume and/or whiskers in the broad sense of the term which refer to single crystals that are formed as long needles by the growth of only one crystal face of the crystal that is created by a phase transition or a chemical change that affects the overall volume and/or other whiskers that are single crystals having cross sectional areas of no more than 8×10^{-6} in^2, with the length being at least 10 times the average diameter.

The fibrous substance to be used may be those which contain at least one compound containing at least one member of the group consisting of metals, semiconductors, metalloids, rare earth metals and nonmetals of groups 1a, 2a, 3a, 4a, 5a, 6a, 7a, 1b, 2b, 3b, 4b, 5b, 6b, 7b and 8 of the periodic table and which have widths of no more than 500 μm, with the length to width ratio being at least 2. More specifically, the fibrous substances to be used are those which are composed of at least one member selected from the group consisting of metals, semiconductors, metalloids, and rare earth metals of groups 1a, 2a, 3a, 4a, 5a, 6a, 7a, 1b, 2b, 3b, 4b and 8 of the periodic table, as well as oxides, nitrides, carbides, oxynitrides, oxycarbides, carbonitrides, oxycarbonitrides, borides and silicides and which have widths of no more than 500 μm, with the length to width ratio being at least 2. Preferably, the following fibrous substances may be selected: those which are composed of at least member one selected from the group consisting of Al, B, Si, Fe, Ni, Co, Ti, Nb, V, Zr, Hf, Ta, W, Re, Cr, Cu, Mo, TiAl, Ti_3Al, $TiAl_3$, TiNi, NiAl, Ni_3Al, SiC, B_4C, Cr_3C_2, TiC, ZrC, WC, W_2C, HfC, TaC, Ta_2C, NbC, VC, Mo_2C, Si_3N_4, TiN, ZrN, Si_2N_2O, AlN, HfN, VxN (x = 1 ~ 8), NbN, TaN, Ta_2N, TiB, TiB_2, ZrB_2, VB, V_3B_2, VB_2, MoB, NbB_2, TaB, TaB_2, MoB, MoB_2, MoB_4, Mo_2B, WB, W_2B, W_2B5, LaB_6, SP, $B_{13}P_2$, $MoSi_2$, Al_2O_3, ZrO_2 (PSZ: partially stabilized zirconia with the addition of Y_2O_3, MgO or CaO; or TZP: tetragonal zirconia polycrystalline compound), $MgAl_2O_4$ (spinel), Al_2SiO_5 (mullite), etc., and which have widths of no more than 500 μm, with the length to width ratio being at least 2.

As described above, the coated diamond or high-pressure phase boron nitride particles to be used in the present invention have their surfaces coated with the coat forming substance by vapor-phase processes and, hence, the coat forming substance that can be used is basically unlimited. While materials design of diamond or high-pressure phase boron nitride sinters can be made in any suitable manners depending on their use, the coating of diamond or high-pressure phase boron nitride particles with the coat forming substance may be preceded by preliminary coating of the surfaces of those surfaces with a similar and/or dissimilar coat forming substance by a similar and/or dissimilar coating technique.

Take, for example, the case where the surfaces of diamond or high-pressure phase boron nitride particles are to be eventually provided with coats formed of a dense metal carbide. In this case, those particles may be preliminarily coated with carbon. While the preliminary coating technique is not limited in any particular way, suitable examples include not only the molten salt dip method which is described in Unexamined Published Japanese Patent Application (kokai) Hei 2-252060 and the molten salt disproportionation method which is described in Unexamined Published Japanese Patent Application (kokai) Hei 1-207680 but also electroplating, electroless plating, cladding, physical vapor deposition processes (e.g., sputtering and ion plating) and chemical vapor deposition processes. The metals in the metal components to be eventually coated are not limited to any particular types as long as they are within the class of substances that can be applied as bonding materials and/or sintering aids for the purpose of the invention.

The coating films formed by the molten salt dip method are composed of dense substances having high hardness and melting point and, having typically the capability of isolating diamond or high-pressure phase boron nitride particles from other substances, these films offer the advantage of markedly increasing the latitude in the choice of binders. The diamond or high-pressure phase boron nitride particles provided with coating films by the molten salt dip method are difficult to sinter densely by the conventional process for producing diamond or high-pressure phase boron nitride sinters at pressures less than 2000 MPa under such sintering pressure and temperature conditions that diamond or high-pressure phase boron nitride is thermodynamically metastable. However, if diamond or high-pressure phase boron nitride particles that are coated with the films of high-hardness and high-melting substances which have been formed by the molten salt dip method are coated by the vapor-phase coating techniques of the invention with one or more layers in suitable amounts of substances that can be applied as bonding materials that are sinterable to assure satisfactory density and high hardness, favorable results are obtained that are advantageous for the purpose of promoting sintering.

In accordance with the present invention, coated diamond or high-pressure phase boron nitride particles which have the coat forming substance applied to the surfaces of bare diamond or high-pressure phase boron nitride particles by vapor phase processes may be mixed with a bonding material or, alternatively, the mixture of the coated diamond or high-pressure phase boron nitride particles and the bonding material may be mixed with the aforementioned auxiliary substance in a powder, plate or particulate form and/or the fibrous substance also mentioned above which has a width of no more than 500 μm, with the length to width ratio being at least 2, and either mixture may be sintered in a powder form or after being shaped into a compact.

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Diamond or high-pressure phase boron nitride is thermodynamically stable only at ultrahigh pressures and if the pressure is inadequate under elevated temperatures, they will experience a phase transition to the lower-pressure phase (graphite or hexagonal boron nitride (hBN)).

Therefore, in order to prevent the phase transition of diamond or high-pressure phase boron nitride to the lower-pressure phase (graphite or hBN) and to insure sintering to high density, the sinters containing diamond or high-pressure phase boron nitride are produced at ultrahigh pressures exceeding 2000 MPa and at elevated temperatures under such conditions that diamond or high-pressure phase boron nitride is thermodynamically stable.

Therefore, if one selects those sintering pressure and temperature conditions where the pressure is at least 2000 MPa and where diamond or high-pressure phase boron nitride is thermodynamically stable, the temperature is limited by pressure in accordance with the thermodynamic equilibrium line in the phase diagram of diamond or high-pressure phase boron nitride. However, if one selects those sintering pressure and temperature conditions where the pressure is at least 2000 MPa and where diamond or high-pressure phase boron nitride is thermodynamically metastable if not stable, temperatures will suffice if they deviate slightly from the region that is limited by pressure in accordance with the thermodynamic equilibrium line in the phase diagram of diamond or high-pressure phase boron nitride.

On the other hand, if one selects those sintering pressure and temperature conditions where the pressure is less than 2000 MPa and where diamond or high-pressure phase boron nitride is thermodynamically metastable if not stable, one should note that there is a pressure-dependent temperature in the region where diamond or high-pressure phase boron nitride is thermodynamically metastable.

Stated more specifically, in the case of selecting those sintering pressure and temperature conditions where diamond or high-pressure phase boron nitride is thermodynamically metastable if not stable, the upper limit of the sintering temperature will vary with the quality of the diamond or high-pressure phase boron nitride particles to be sintered.

Take, for example, the case of diamonds; Hall conducted an experiment under ultrahigh pressures and reported that when a diamond was thermodynamically metastable if not stable, it took such a long time for a phase transition to occur that it remained stable for practical purposes; he then indicated the temperature 1100°C as the upper limit beyond which the diamond would no longer exist in a practically stable manner (see H.T. Hall, Science, 169 (1970) 868-869, as well as Fig. 15a). Speaking of high-pressure phase boron nitrides, Wakatsuki et al. conducted an experiment on a cubic boron nitride under ultrahigh pressures and reported that when the cubic boron nitride was thermodynamically metastable if not stable, it took such a long time for a phase transition to occur that it remained stable for practical purposes; they then indicated the temperature 1200°C as the upper limit beyond which the cubic boron nitride would no longer exist in a practically stable manner (see Wakatsuki, Ichise, Aoki and Maeda, Abstracts of the Lectures Read at the 14th Symposium on High-Pressure Technology (1972) p. 78, as well as Fig. 83b).

Therefore, if high-purity diamonds (e.g. natural products or high quality synthetic diamonds produced via the vapor phase by means of PVD or CVD processes, or diamonds synthesized by application of ultrahigh pressures over a long time) are especially selected and if they are sintered either by HIP, optionally under ultrahigh pressures, as they are confined within a degassed, pressure-transmitting capsule or by PC or HP under vacuum or in an inert gas, diamonds will actually exist up to 1950°C which is much higher than 1100°C, or the temperature reported by Hall, even if they are not in a thermodynamically stable state. However, if the temperature exceeds 1850°C, the diamonds will turn to the graphite phase in a short time. Generally speaking, synthesis catalysts or the like will sometimes remain as impurities in synthetic diamonds. However, if synthetic diamonds are such that impurities have been removed positively to achieve high purity, the diamond will exist up to 1700°C by application of similar sintering techniques and this is the same as in the case of natural diamonds. Alternatively, with known diamonds that do not contain fine grains smaller than 1 μm, preferably those which are at least 3 μm and whose size is within the class contemplated by the present invention, the diamond will exist up to 1900°C irrespective of whether they are natural or synthetic products. It should also be noted that in the case of known diamonds of comparatively high purity, both natural and synthetic products will exist up to 1800°C. However, with commonly known types of synthetic diamonds, the suitable temperature is up to 1400°C. Therefore, the upper limit of sintering temperatures is 1850°C.

Speaking of high-pressure phase boron nitrides, if those of high purity (e.g. cubic boron nitrides of ultrahigh purity that are produced via the vapor phase by means of PVD or CVD processes, or cubic boron nitrides of ultrahigh purity that are synthesized by application of ultrahigh pressures over a long time) are especially selected and if they are sintered either by HIP (hot isostatic pressing), optionally under ultrahigh pressures, as they are confined within a degassed, pressure-transmitting capsule or by PC (piston-cylinder) or HP (hot pressing) under vacuum or in an inert gas, cubic boron nitrides will actually exist up to 1950°C which is much higher than 1200°C, or the temperature reported by Wakatsuki et al., even if they are not in a thermodynamically stable state. However, if the temperature exceeds 1850°C, the cubic boron nitrides will turn to a graphitic phase in a short time. If conventionally known cubic boron nitrides are used after impurities have been removed positively to achieve high purity, the cubic boron nitride will exist up to 1700°C by application of similar sintering techniques. Alternatively, with known cubic boron nitrides that do not contain fine grains, preferably those which are at least 3 μm in size, the cubic boron nitride will exist up to 1900°C by application of similar sintering techniques. It should also be noted that in the case of known cubic boron nitrides of comparatively high purity, the cubic boron nitride will exist up to 1800°C by application of similar sintering techniques. However, in the case where

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commonly known types of cubic boron nitrides or wurtzitic boron nitrides are contained, the suitable temperature is up to 1400°C.

Consequently, if one selects those sintering pressure and temperature conditions where the pressure is less than 2000 MPa and where diamond or high-pressure phase boron nitride is thermodynamically metastable if not stable, the upper limit of sintering temperatures is 1800°C. It should be remembered that the temperature at which the diamond or high-pressure phase boron nitride is not in a thermodynamically stable state but exists stably for practical purposes varies with the quality of the diamond or high-pressure phase boron nitride to be used and that, therefore, the sintering temperature must be set in accordance with the quality of the feed of diamond or high-pressure phase boron nitride. If it is necessary that the sintering temperature be set to a value that is close to the right temperature which matches the quality of the feed of diamond or high-pressure phase boron nitride, said sintering temperature must be controlled with close tolerance.

The diamond or high-pressure phase boron nitride sinters of the present invention are produced by sintering processes that adopt two kinds of conditions with different pressures. To produce diamond or high-pressure phase boron nitride sinters by performing sintering for a suitable period of time at ultrahigh pressures of 2000 MPa or above and at elevated temperatures, any conventional equipment that produces ultrahigh pressures can be applied without particular limitations, as exemplified by the cubic, tetragonal, girdle, and belt type ultrahigh pressure apparatus.

Known apparatus and methods may be applied without any particular limitations to produce dynamic ultrahigh pressures (≥ 2000 MPa) and elevated temperatures and they include: an explosive charge lens (planar explosion generator), a cylindrical explosion generator, a method involving collision with a flying object, a magnetic implosion method, a Mach explosion method, an excessive explosion method, a conical implosion method, a single-action shock gun, a double-action light gas gun, etc. According to these apparatus and methods which generate dynamic ultrahigh pressures and elevated temperatures, the pressure that can be produced is at least 10 GPa (10,000 MPa), preferably at least 40 GPa and, therefore, they can be selected as preferred examples in cases where particularly high pressures need be exerted for short periods of time.

Depending on the object, for example, in the case where samples need be prepared while giving results with good reproducibility, the above-mentioned cubic apparatus and various other types of equipment that are capable of generating ultrahigh temperatures in a static manner may be used to produce pressures of 2000 MPa and above. The sintering temperature condition may deviate slightly from the region where diamond or high-pressure phase boron nitride is thermodynamically stable.

But, more preferably, one may select those sintering pressure and temperature conditions where the pressure is at least 2000 MPa and where diamond or high-pressure phase boron nitride is thermodynamically stable.

To produce diamond or high-pressure phase boron nitride sinters by performing sintering for a suitable period of time at pressures less than 2000 MPa and at temperatures not exceeding 1800°C under such sintering pressure and temperature conditions that diamond or high-pressure phase boron nitride is thermodynamically metastable if not stable, any conventional equipment can be applied without particular limitations, as exemplified by PC (piston-cylinder) type ultrahigh pressure apparatus, ultrahigh pressure HIP (hot isostatic pressing) apparatus, HIP apparatus and HP (hot pressing) apparatus.

If the PC type ultrahigh pressure apparatus is to be used, pressures less than 2000 MPa may be applied but, considering the durability of this apparatus, the pressure does not preferably exceed 1600 MPa. Conventionally known technologies concerning the production of pressures include ultrahigh pressure HIP apparatus which is capable of operating at HIP pressures up to 1000 MPa, as well as HIP apparatus (excluding the ultrahigh pressure HIP apparatus) and HP apparatus which are each capable of operating at pressures up to 200 MPa.

The diamond or high-pressure phase boron nitride sinters produced by the methods described above have high performance with highly controlled microstructures. While their most common use is as machine parts, it is possible to manufacture diamond or high-pressure phase boron nitride sinters that are superhard, with the Vickers hardness (Hv) being preferably at least 800, and which are dense, with the density being at least 85%. Preferably, it is possible to manufacture diamond or high-pressure phase boron nitride sinters that are superhard, with the Vickers hardness (Hv) being preferably at least 800, and/or which are dense, with the density being at least 90%. More preferably, considering the application to highly wear-resistant machine parts, superhard (Hv ≥ 1000) diamond or high-pressure phase boron nitride sinters can be manufactured by both increasing the relative content of diamond or high-pressure phase boron nitride and sintering to high density. For use in tools and other applications that require even higher levels of wear resistance, diamond or high-pressure phase boron nitride sinters having Vickers hardness values of at least 2000 can be manufactured by further increasing the content of diamond or high-pressure phase boron nitride while sintering to high density.

In a typical case, a diamond sinter may be produced from a diamond powder feed in the following manner using a cubic apparatus to produce ultrahigh pressures. First, the particles in the diamond powder are coated with a suitable coat forming substance on the surfaces. The coated diamond particles are then shaped into a pellet by stamping. The pellet is surrounded by a shaped hexagonal boron nitride (h-BN) part which, in turn, is enclosed with a graphite tubular heater. Provided around the heater as a solid pressure transmitting medium is pyrophyllite from which the walls of

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crystallization has been removed by heat treatment at 700°C. The sample thus prepared is placed in the cubic apparatus; the pressure is increased to a predetermined level and, then, the temperature is also elevated to a predetermined level and sintering is effected for a suitable time. Thereafter, the temperature is lowered and the pressure is also reduced. The pressure-transmitting medium is taken out of the cubic ultrahigh pressure apparatus and the sample is then recovered from the medium. In this way, one can produce a diamond sinter of high performance that is composed of diamond and which is characteristic in that it has a controlled distribution of the bonding material and/or sintering aid and/or surface modifier, that is uniform and dense, and that it has a highly controlled microstructure.

As described in detail on the foregoing pages, a powder of fine diamond or high-pressure phase boron nitride core particles having an average diameter of no more than 10 μm in terms of frequency distribution by volume was dispersed in a gaseous atmosphere and the core particles thus dispersed to a dispersity β of at least 70% were allowed to contact or impinge against a precursor of a coat forming substance, whereby the core particles were covered on their surfaces with the coat forming substance in a single-particle state. In accordance with the invention, the thus coated core particles or mixtures containing them were sintered at ultrahigh atmospheres of at least 2000 MPa and at elevated temperatures, whereby coated diamond or high-pressure phase boron nitride sinters of high performance were produced that had uniform, dense, firmly sintered and highly controlled microstructures.

According to another aspect of the invention, said core particles or mixtures containing them or mixtures of said core particles with bonding materials are sintered at pressures less than 2000 MPa and at temperatures not exceeding 1650°C under such sintering pressure and temperature conditions that diamond or high-pressure phase boron nitride is thermodynamically metastable if not stable, whereby one can produce coated diamond or high-pressure phase boron nitride sinters of high performance that have uniform, dense, firmly sintered and highly controlled microstructures. This offers the advantage of eliminating the constraint of the need to use equipment capable of creating ultrahigh pressures of at least 2000 MPa. It should be particularly noted that the use of ultrahigh pressure HIP apparatus or other types of HIP apparatus offers the added advantage of enabling the production of complexly shaped sinters, thereby presenting significant benefits to manufacture on an industrial scale.

According to yet another aspect of the invention, even pseudo-fine diamond or high-pressure phase boron nitride particles having an average diameter greater than 10 μm in terms of frequency distribution by volume may be covered with coat forming substances after being dispersed to a dispersity as determined by their average diameter. By sintering the thus coated pseudo-fine particles, one can produce sinters that are substantially free from graphite or a graphitic phase and which have the same features as fine particulate sinters having an average particle diameter of no more than 10 μm.

EXAMPLES

The diamond or high-pressure phase boron nitride sinters of the present invention, as well as the processes for their production are described below with reference to examples.

Example 1

Diamond particles having an average diameter (D_{50}) of 1 μm and a frequency distribution by volume of ($D_{50}/2 \leq 3D_{50}/2$), $\alpha \geq 80\%$) were coated with elemental silicon.

The construction of the apparatus used is shown in Fig. 6 and Fig. 7 which is a partial enlarged view of Fig. 6; the apparatus is a specific example of the design shown in Fig. 3a.

As shown, the apparatus comprises: a plasma torch 3-A including a plasma chamber 3-a, a vessel 3-B for coating a coat forming substance precursor generating chamber 3-b, a coating chamber coating vessel 3-C in the narrow sense of the term, a coating chamber 3-c in the narrow sense of the term, a vessel 3-D for coating a coated particle coating chamber 3-d, a supply unit 3-E1 on the side where the feed of the coat forming substance is supplied, an agitating disperser 3-F1 and an ejector-type disperser 3-H1 which are on the side where a powder of core particles is supplied, a capillary disperser 107, and a coated particle recovery section 3-G. The supply unit 3-E1 is coupled to a feeder 112 equipped with a vessel for supplying the feed powder of the coat forming substance, and the agitating disperser 3-F1 is coupled to a feeder 111 equipped with a vessel for supplying the powder of core particles. According to the definition, the coating chamber used in Example 1 is composed of the plasma chamber 3-a, the coat forming substance precursor generating chamber 3-b, the coating chamber 3-c in the narrow sense of the term, and the coated particle coating chamber 3-d. These make up the coating chamber in the broad sense of the term and the chamber 3-c where most part of the coating operation is performed is referred to as the "coating chamber in the narrow sense of the term".

The group of means for high dispersion treatment of particles c used in Example 1 is composed of the feeder 111 equipped with a supply vessel, agitating disperser 3-F1, ejector-type disperser 3-H1, and the capillary disperser 107 which is made of a stainless steel pipe having an inside diameter of 4 mm. The basic concept of the group is shown in Fig. 2a and it is a specific example of the group of means for high dispersion treatment of particles that is of the design shown in Fig. 3b. The group is designed in such a way that it can realize an output value β of at least 70% (β ≥ 70%).

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given the particles in a powder of diamond core particles that are characterized by (D_{90}/5, SD_{90}) ≥ 90%) where D_{90} = 1 μm. The capillary dispenser 107 which is the final means of treatment in the group of means for high dispersion treatment of particles is coupled directly to the coating chamber 3-c and designed in such a way as to realize β ≥ 70% in the coating start region 3-L1 of the coating space 3-L3.

A gas jet port 101 is provided on top of the plasma torch 3-A and an argon gas is supplied from a supply source 102 through this port at a rate of 30 L/min. The supplied argon gas is turned into a plasma by applied high-frequency waves, thereby forming a plasma flame in the plasma chamber 3-a within the plasma torch 3-A.

A silicon powder comprising particles with an average diameter of 10 μm serves as the feed of the coat forming substance and is supplied from the feeder 112 equipped with a vessel for supplying the feed powder of the coat forming substance. The supplied silicon powder is carried by a carrier gas 103 flowing at a rate of 5 L/min so that it is introduced into the plasma flame at a rate of 0.2 g/min via an inlet 104 provided at the bottom of the plasma torch 3-A. Being evaporated by the heat of the plasma flame, the silicon powder goes through the vapor phase to become the precursor of the coat forming substance in the precursor generating chamber 3-b.

The diamond core particles with an average diameter of 1 μm are supplied at a rate of 1.2 g/min from the feeder 111 equipped with a supply vessel of the core particles and they are dispersed with the agitating disperser 3-F1 while being carried by a carrier gas 105 that is supplied at a rate of 5 L/min. The particles are then dispersed to a dispersity (β) of 82% by means of the ejector-type disperser 3-H1 and the capillary disperser 107 with the aid of a dispersing gas 106 that is supplied at a flow rate of 10 L/min. The thusly dispersed core particles are introduced into the coating chamber.

The highly dispersed diamond particles entering the coating start region 3-L1 of the coating space 3-L2 start to contact and/or impinge against the precursor of the coat forming substance as they maintain the dispersity β at 82%.

Thus, the diamond particles are given the coat of the coat forming substance on their surfaces and they descend down the coated particle cooling chamber 3-d together with the gas, eventually reaching the recovery section 3-G. The product comprising the coated particles is separated from the gas 109 by means of a filter 110 and collected for recovery.

The recovered fine diamond particles having silicon coats on the surfaces were examined with a scanning electron microscope. As Fig. 8 shows, all of these particles were uniformly coated with superfine silicon particles of about 0.005 μm in size. The silicon coating weight was 20% by volume.

The resulting powder of diamond particles coated with 20 vol% silicon was stamped into a pellet having an outside diameter of 6 mm and a height of 2 mm. The pellet was buried in a pressure-transmitting medium surrounded by a shaped hexagonal boron nitride (h-BN) and it was vacuum-dried overnight at 800°C and at 10^{-3} torr to remove low-boiling impurities. The pellet was then set in a cubic ultrahigh pressure apparatus, the pressure was raised to 5.5 GPa at room temperature, then the temperature was elevated to 1450°C, which was held for 30 min, thereafter, the temperature was lowered and the pressure reduced.

The resulting sinter was examined by X-ray diffractometry, the elemental silicon reacted with the diamond to change to both diamond and SiC while the elemental silicon disappeared. Quantification by X-ray analysis showed that the volume proportions of the diamond sinter and SiC were about 78% and 22%, respectively.

The surface of the sinter was polished with a diamond paste and its Vickers microhardness (Hv) was measured. The diamond sinter synthesized in Example 1 was superhard with the Hv (0.5/10) being about 12,000 and this value was comparable to or higher than that of a commercial cutting tool made of sintered diamond. Furthermore, the hardness of the sinter of Example 1 was uniform over the entire surface subjected to the hardness measurement and it was substantially free from variations.

The polished surface of the sinter resulting of the example 1 was coated with a gold film by a customary evaporation technique and examined with an electron microscope. The result is shown in the Fig. 9 electron micrograph (X5000), from which one can see that the sinter had a relative density of at least 99%. In addition, the sinter had no portion left unsintered. In areas where diamond particles contacted each other via a thin layer of the coat forming substance, the latter was bonded by the contiguous diamond particles, which hence were coupled directly as a result of sintering. In all other areas, SiC was distributed to surround the diamond particles, rendering the sinter to be characteristic in that it was dense, uniform and had the bonding material distributed provide a highly controlled microstructure. It should also be noted that the diamond particles in the sinter did not grow at all compared to the particles in the feed powder and that the coating on the diamond was formed by chemical reaction between the diamond and the elemental silicon which served as the coat forming substance; these facts combined together to offer the characteristic feature that the diamond particles in the sinter were finer than those in the feed although they were coated with silicon. This is very favorable and, hence, ideal for the mechanical characteristics of the sinter. The differences can be clearly seen by comparing Fig. 9 with Fig. 10 which is an electron micrograph (X5000) of a polished surface of the above-mentioned commercial cutting tool that was made of sintered diamond and which was coated with a gold film by a customary evaporation technique for examination. Obviously, the bonding material and the sintering aid were distributed irregularly in the diamond sinter of the commercial cutting tool to form lumps in certain areas. On the other hand, the number of areas that were devoid of the necessary sintering aid and bonding material was by no means small and unsintered portions were found there. Another major difference from the sinter of Example 1 was that every diamond particle in the cutting tool was coarse.

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Diamond is the most difficult to sinter of all substances that exist on the earth. In spite of this fact, the coated particles in a diamond powder that were synthesized in Example 1 in accordance with the invention behaved as if they were comparatively easy-to-sinter particles under industrially feasible ultrahigh pressures and elevated temperatures, thereby forming a dense, rigid and superhard structure.

Example 2

Cubic boron nitride particles having an average diameter (D_{50}) of 1 μm and a frequency distribution by volume of ($[D_{50}/2, 3D_{50}/2]$, $\alpha \geq 90\%$) were coated with alumina.

The construction of the apparatus used is shown in Fig. 11 and Fig. 12 which is a partial enlarged view of Fig. 11; the apparatus is a specific example of the design shown in Fig. 5d.

As shown, the apparatus comprises: a plasma torch 5-A including a plasma chamber 5-a, a vessel 5-B for coating a coat forming substance precursor generating chamber 5-b, a coating chamber coating vessel 5-C in the narrow sense of the term, a coating chamber 5-c in the narrow sense of the term, a vessel 5-D for cooling a coated particle cooling chamber 5-d, a supply unit 5-E1 on the side where the feed of the coat forming substance is supplied, an agitating disperser 5-F1 which is on the side where a powder of core particles is supplied, a capillary dispenser 211 and a dispenser 5-H2 that utilizes an impingement plate, and a coated particle recovery section 5-G. That part of this apparatus which was to generate the precursor of the coat forming substance was of the same design as in Example 1. The group of means for high dispersion treatment of particles α was composed of a feeder 214 equipped with a supply vessel, the agitating disperser 5-F1, the capillary dispenser 211, and the dispenser 5-H2 that utilizes an impingement plate. The basic concept of the group α is shown in Fig. 3a and it is a specific example of the group of means for high dispersion treatment of particles that is of the design shown in Fig. 3b. The capillary dispenser 211 is made of a stainless steel pipe having an inside diameter of 4 mm. The dispenser 5-H2 is the final means of treatment in the group of means for high dispersion treatment of particles α and it consists of a SiC impingement plate 213 supported by a stainless steel holder 212. The dispenser 5-H2 is contained in the coating chamber 5-c in the narrow sense of the term, which shares the space with the group of means for high dispersion treatment of particles α. A coating space 5-L1 and the coating start region 5-L2 of the coating space are also contained in the coating chamber 5-c in the narrow sense of the term. The group of means α in the apparatus of Example 2 has such performance that the particles in a powder of core particles having an average diameter (D_{50}) of 1 μm and a frequency distribution by volume of ($[D_{50}/2, 3D_{50}/2]$, $\alpha \geq 90\%$) can be dispersed to a dispersity (β) of at least 70% (β ≥ 70%) right after impingement against the plate 213 in the dispenser 5-H2 at the last stage of dispersion treatment. Therefore, coating of the core particles starts with the dispersity β held at 70% and above.

A gas jet port 201 is provided on top of the plasma torch 5-A and an argon gas is supplied from a supply source 202 through this port at a rate of 20 L/min. The supplied argon gas is turned into a plasma by applied high-frequency waves, thereby forming a plasma flame in a plasma chamber 5-a within the plasma torch 5-A.

An alumina powder comprising particles with an average diameter of 2 μm serves as the feed of the coat forming substance and is supplied at a rate of 1.0 g/min from a feeder 315 equipped with a vessel for supplying the feed powder of the coat forming substance. The supplied alumina powder is carried by a carrier gas 203 flowing at a rate of 5 L/min so that it is introduced into the plasma flame via an inlet 304 of the feed of the coat forming substance provided at the bottom of the plasma torch 5-A. Being evaporated by the heat of the plasma flame, the alumina powder goes through the vapor phase to become the precursor of the coat forming substance in a coat forming substance precursor generating chamber 5-b.

The core particles of cubic boron nitride are supplied at a rate of 0.7 g/min from the feeder 214 equipped with a vessel for supplying the powder of core particles and they are dispersed with the agitating disperser 5-F1 while being carried by a carrier gas 206 that is supplied at a rate of 20 L/min. The particles then pass through the capillary dispenser 211 to enter the dispenser 5-H2 with the impingement plate in the coating chamber, in which they are dispersed in a gaseous atmosphere to a dispersity (β) of 82%.

The highly dispersed core particles of cubic boron nitride, entering the coating start region 5-L1 of the coating space 5-L2 start to contact and/or impinge against the precursor of the coat forming substance as they maintain the dispersity β of 82%.

Thus, the cubic boron nitride particles are given the coat of the coat forming substance on their surfaces and they descend down a coated particle cooling chamber 5-d together with the gas, eventually reaching a coated particle recovery section 5-G. The product comprising the coated cubic boron nitride particles is separated from the gas 209 by means of a filter 210 and collected for recovery. The recovered cubic boron nitride particles had alumina coats in an amount of 60% by volume.

The recovered fine cubic boron nitride particles having alumina coats on their surfaces were examined with a scanning electron microscope. All of those particles were uniformly coated with superfine alumina particles of about 0.005 μm in size.

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The resulting cubic boron nitride particles with 30 vol% alumina were stamped into a pellet having a diameter of 8 mm and a thickness of 6 mm. The pellet was put into a Pyrex glass capsule of generally the same design as in Example 1 and sealed after degassing by heating.

Thereafter, the encapsulated pellet was set in a ultrahigh pressure HIP apparatus and sintered by holding at a temperature of 1200°C and at a pressure of 1000 MPa for 3 h.

The sinter of Example 2 was checked for its crystalline phase by powder X-ray diffraction and no diffraction peaks were observed except for the cubic boron nitride and α-form alumina.

As Table 4 shows, the sinter was considerably dense, with a density of 98.8%, and at the same time it was superhard, with the Vickers microhardness being Hv (0.5/10) = 3750.

It should also be mentioned that the cubic boron nitride particles in the sinter were surrounded by alumina to provide a uniform distribution and a highly controlled microstructure.

Example 3

Quasi-fine diamond particles having an average diameter (DM) of 17 μm and a frequency distribution by volume of ((DM/2, 3DM/2), ≥ 80%) were coated with metallic zirconium.

The construction of the apparatus used is shown in Fig. 13 and Fig. 14 which is a partial enlarged view of Fig. 16; the apparatus is a specific example of the design shown in Fig. 5b.

As shown, the apparatus comprises: a plasma torch 6-A including a plasma chamber 6-a, a vessel 6-B for cooling a coat forming substance precursor generating chamber 6-b, a coating chamber cooling vessel 6-C in the narrow sense of the term, a coating chamber 6-c in the narrow sense of the term, a vessel 6-D for cooling a coated particle cooling chamber 6-d, a supply unit 6-E1 on the side where the feed of the coat forming substance is supplied, an agitating disperser 6-F1, a cyclone 6-I, a hopper 6-J and a rotary valve 6-K which are on the side where a powder of core particles is supplied, a pipe 307 inevitable to transport, and a coated particle recovery section 6-G. That part of this apparatus which was to generate the precursor of the coat forming substance was of the same design as in Example 1. The group of means for high dispersion treatment of particles α was composed of a feeder 312 equipped with a supply vessel, the agitating disperse 6-F1 which is dispersing means, and the cyclone 6-I which is means for selecting a mixture of a gas and the particles in a powder of highly dispersed core particles. The basic concept of the group α is shown in the Fig. 3b block diagram. The section of cyclone 6-I discharging the mixture of a gas and the particles in a powder of highly dispersed core particles is connected to the coating chamber 6-c in the narrow sense of the term by means of the pipe 307 inevitable to transport, whereas the section of cyclone 6-I discharging the powder portion consisting of less highly dispersed core particles is connected to the agitating disperser 6-F1 by means of a transport pipe 310 via the hopper 6-J and the rotary valve 6-K. Using the group of means α in the apparatus of Example 3, the particles in a powder of core particles having an average (DM) of 17 μm and a frequency distribution by volume of ((DM/2, 3DM), ≥ 80%) can be dispersed to a dispersity (β) of at least 85% in the section of cyclone 6-I (i.e., the means of final treatment) discharging the powder stream of highly dispersed particles. As shown, the coating space 6-L2 and the coating start region 6-L1 of this coating space are contained in the coating chamber 6-c in the narrow sense of the term. Because of the constraint by the flange portion coupling a coating chamber cooling vessel 6-C in the narrow sense of the term with the vessel 6-D for cooling the coated particle cooling chamber 6-d, the pipe 307 was indispensable to transport, but the resulting drop in dispersity β could be held at a reduced level. Hence, coating of the core particles starts with the dispersity β held at 80% and above in the coating start region.

A gas jet port 301 is provided on top of the plasma torch 6-A and an argon gas is supplied from a supply vessel 302 through this port at a rate of 40 L/min. The supplied argon gas is turned into a plasma by applied high-frequency waves, thereby forming a plasma flame in the plasma chamber 6-a within the plasma torch 6-A.

A zirconium powder serving as the feed of the coat forming substance is supplied at a rate of 0.8 g/min from a feeder 314 equipped with a vessel for supplying the feed powder of the coat forming substance. The supplied zirconia powder is carried by a carrier gas 303 flowing at a rate of 5 L/min so that it is introduced into the plasma flame via an inlet 304 the feed of the coat forming substance provided at the bottom of the plasma torch 6-A. Being evaporated by the heat of the plasma flame, the zirconia powder goes through the vapor phase to become the precursor of the coat forming substance in the coat forming substance precursor generating chamber 6-b.

Diamond core particles are supplied at a rate of 2.0 g/min from the feeder 313 equipped with a vessel for supplying the powder of core particles and they are dispersed with the agitating disperser 6-F1 while being carried by a carrier gas 305 that is supplied at a rate of 15 L/min. The particles then pass through a pipe 308 and thence enter the cyclone 6-I. The cyclone 6-I is adjusted in such a way that the fines produced will have a maximum particle diameter of 20 μm; a mixture of the gas and the particles in a powder of highly dispersed (β = 85%) core particles which are mostly single particles passes through the pipe 307 which is indispensable to transport, so that it is discharged into the coating chamber 6-c in the narrow sense of the term via a discharge port 306. On the other hand, the powder portion consisting of less highly dispersed core particles which have been selectively separated by cyclone 6-I passes through hopper 6-J and

rotary valve 6-K to be transported through a pipe 310 by a carrier gas 309 flowing at a rate of 10 L/min so that it is fed back to the agitating disperser 6-F1.

The highly dispersed diamond particles entering the coating start region 6-L1 of the coating space 6-L3 start to contact and/or impinge against the precursor of the coat forming substance as they maintain the dispersity (β) of 80%.

Thus, the diamond particles are given the coat of the coat forming substance on their surfaces and they descend down a coated particle cooling chamber 6-d together with the gas, eventually reaching a coated particle recovery section 6-O. The product comprising the coated diamond particles is separated from the gas 311 by means of a filter 312 and collected for recovery.

The recovered diamond particles having zirconium coats on the surfaces were examined with a scanning electron microscope. All of these particles were uniformly coated with superfine zirconium particles of about 0.005 μm in size. The zirconium coating weight was 10% by volume.

To provide a bonding material for binding the resulting zirconium-coated diamond particles, another lot of diamond particles were coated with titanium carbide under generally the same conditions as those used to produce the zirconium-coated diamond particles, thereby producing a coated diamond powder in which fine diamond particles of diameters ranging from 0.5 to 2 μm (average diameter = 1 μm) were coated with 15 vol% of titanium carbide. Thirty percent by volume of the previously prepared zirconium-coated diamond particles were wet mixed in acetone with seventy percent by volume of the thus prepared titanium carbide coated diamond particles. The mixture was stamped to a pellet having an outside diameter of 8 mm and a height of 2 mm. The pellet was buried in a pressure-transmitting medium surrounded by a shaped hexagonal boron nitride (h-BN) part and it was vacuum-dried overnight at 200°C and at 10^{-3} torr to remove low-boiling impurities. The pellet was then set in a cubic ultrahigh pressure apparatus, the pressure was raised to 5.5 GPa at room temperature, then the temperature was elevated to 1430°C, which was held for 30 min; thereafter, the temperature was lowered and the pressure reduced.

The resulting sinter was examined by X-ray diffractometry and only diamond, zirconium carbide and titanium carbide were found in it. The sinter was dense with a relative density of at least 99%. Quantification by X-ray analysis showed that the volume proportions of diamond, zirconium carbide and titanium carbide were about 86%, 3% and 11%, respectively.

The sinter was dense and rigid, forming a very highly controlled microstructure.

Example 4

Coated diamond particles were prepared by coating the bare diamond particles of Example 1 with various coat forming substances using apparatus of the same types as used in Examples 1 - 3. The names of the coat forming substances used to prepare the coated diamond particles and their coating weights (relative proportions by vol%), as well as the specific types of the apparatus used and the conditions for their use (coating operation) are shown in Table 1 together with the data for Example 1.

The coated diamond particles thus obtained were examined with a scanning electron microscope. All of the particles prepared in Runs 4-1 and 4-2 were uniformly covered with superfine particles of coat forming substances in sizes of about 0.005 μm.

The apparatus used in Run 4-1 was of the same type as used in Example 2, a metallic zirconium powder having an average diameter of 12 μm was supplied as a feed of coat forming substance from the feeder 215 at a rate of 0.6 g/min, and the core diamond particles were supplied from the feeder 214 at a rate of 1.2 g/min. The other conditions in Run 4-1 were the same as in Example 2.

The apparatus used in Run 4-2 was of the same type as used in Example 3, the group of means for high dispersion treatment of particles in this apparatus was to treat core diamond particles having an average diameter D_{sv} of 1 μm and a distribution by volume of ($D_{sv}/6 \leq SD_{sv} \leq 90\%$), so the particles in the powder of core particles could be dispersed to a dispersity of 75% or more (β ≥ 75%) at the discharge section of cyclone 6-I, making it possible to start the coating operation at a dispersity of 70% or more (β ≥ 70%) in the coating start region. In Run 4-2, a metallic titanium powder was supplied as a feed of coating forming substance from the feeder 314 at a rate of 0.9 g/min and the core diamond particles were supplied from the feeder 313 at a rate of 1.2 g/min; the cyclone 6-I was adjusted in such a way that the fines produced would have a maximum particle diameter of 1.5 μm and the dispersity that could be attained in the discharge section was 85% (β = 85%) whereas the dispersity attainable in the coating start region was 82% (β = 82%); the other conditions in Run 4-2 were the same as in Example 3.

The apparatus used in Runs 4-7 and 4-8 were both of the same type as used in Example 1 and operated under generally the same conditions as in Example 1 except that aluminum and metallic nickel were deposited in that order to give a total coating weight of 15% in Run 4-7 as calculated for NiAl (Al to Ni molar ratio being 1:1) and 20% in Run 4-8 for Ni_3Al (Al to Ni molar ratio being 1:3).

The thus prepared powders of coated diamond particles or mixtures thereof with silicon carbide whiskers (addition aids) were shaped as in Example 1 and set in a cubic ultrahigh pressure apparatus; the pressure was raised to 5.5 GPa

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at room temperature, then the temperature was elevated to 1450 - 1460°C, which was held for 30 min; thereafter, the temperature was lowered and the pressure reduced.

In Runs 4-9 and 4-10, 30% by volume of the titanium carbide and silicon carbide coated diamond particles prepared in Runs 4-4 and 4-6, respectively, were wet mixed in acetone with 10% by volume of silicon carbide whiskers as a fibrous substance (SiC: width = 0.3 μm; average length = 30 μm) and subsequently vacuum dried to produce dry blends.

Each of the sinters thus produced was entirely free from pores, could be densified to a relative density of 100% and had no portion left unsintered. As in Example 1, the surfaces of the sinters were polished with a diamond paste and their Vickers microhardness Hv (0.5/10) values were measured. The results are shown in Table 1 together with the sintering conditions including the data for Example 1.

The crystalline phases of the sinters were examined by powder X-ray diffraction. In Run 4-1, only diamond and ZrC were observed and quantification by X-ray analysis showed that the volume proportions of diamond and ZrC were about 78% and 22%, respectively. In Run 4-2, only diamond and TiC were observed and quantification by X-ray analysis showed that the volume proportions of diamond and TiC were about 76% and 24%. In Runs 4-3 to 4-8, only diamond and the coat forming substances used (TiC, TiC, SiC, SiC, NiAl and Ni_3Al, respectively) were observed. In Run 4-9, only diamond and TiC were observed in addition to SiC. In Run 4-10, only diamond and SiC were observed.

All of the sinters that produced were superhard, dense, uniform and had a uniform distribution of bonding materials to establish highly controlled microstructures. In particular, the composite sinters produced in Runs 4-9 and 4-10 had highly controlled microstructures in which the TiC bonding material (Run 4-9) or SiC bonding material (Run 4-10) and the SiC whiskers were dispersed uniformly between coated diamond particles.

Table 1

Run No.	Raw Material Composition			Coating Material		Coating Condition	Sintering Condition			Sinter		
	Core Particle Element	Core Forming Substance Base	Amount	Vol. % Water-soluble Substance	Coating Appearance Thickness Ex. or Com. No.	Coating Condition (Apparatus-related) Ex. or Com. No.	Atmosphere	Temperature °C	Time min	Mineral Square Cut	Density %	Hardness Hv
1	60	Si	20	-	1	1	O2	1400	30	CB	100	12000
A-1	60	Zr	20	-	2	A-1	O2	1400	30	CB	100	11500
A-2	60	Ti	20	-	2	A-2	O2	1400	30	CB	100	11500
A-3	60	SiC	10	-	1	1	O2	1400	30	CB	100	12000
A-4	60	SiC	20	-	1	A-3	O2	1400	30	CB	100	11800
A-5	60	SiC	20	-	1	A-4	O2	1400	30	CB	100	12000
A-6	10	SiC	30	-	2	A-5	O2	1400	30	CB	100	11000
A-7	60	BiAl	10	-	1	1	O2	1400	30	CB	100	1000
A-8	60	BiAl	20	-	1	A-2	O2	1400	20	CB	100	4300
A-9	72	SiC	18	10	1	A-4	O2	1400	20	CB	100	3650
A-10	72	SiC	18	10	1	A-6	O2	1400	20	CB	100	3400

CB: Cubic chamber pressure apparatus

Example 5

Using apparatus of the same types as used in Examples 1 - 3, high-pressure phase boron nitride powder particles, or cubic boron nitride (cBN) particles of the same type as used in Example 2 that had an average diameter (D_{50}) of 1 μm and a frequency distribution by volume of ($D_{90}/2$, $3D_{10}/2$), ≥ 90%) were covered with various coat forming substances. The names of the coat forming substances used to prepare the coated cubic boron nitride particles and their coating weights (relative proportions by vol%), as well as the specific types of the coating apparatus used and the conditions for their use (generally the same) are shown in Table 2.

The apparatus used in Run 5-1 was of the same type as used in Example 1; a metallic zirconium powder having an average diameter of 12 μm was supplied as a feed of coat forming substance from the feeder 112 at a rate of 0.8 g/min, and the cubic boron nitride core particles were supplied from the feeder 111 at a rate of 1.2 g/min. The other conditions in Run 5-1 were the same as in Example 1. The apparatus used in Run 5-2 was of the same type as used in Example 2; a metallic titanium powder having an average diameter of 25 μm was supplied as a feed of coat forming substance at a rate of 0.4 g/min whereas the core particles were supplied at a rate of 1.2 g/min; the other conditions in Run 5-2 were the same as in Example 2.

The apparatus used in Run 5-3 was of the same type as used in Example 3. Notwithstanding the difference in the core particles used (diamond or cubic boron nitride), the values of dispersity (β) attained in the discharge section of cyclone 6-1 and in the coating start region were the same as in Run 4-2 and titanium nitride as a feed of coat forming substance was supplied at a rate of 0.6 g/min whereas the core particles were supplied at a rate of 2.0 g/min. The other conditions in Run 5-3 were the same as in Example 3.

The coated cubic boron nitride particles thus prepared were examined with a scanning electron microscope. All of the particles prepared in Runs 5-1 to 5-3 were uniformly covered with superfine particles (ca. 0.005 μm) of coat forming substances as Fig. 15 shows (particularly for the case of Run 5-1).

The coating with NiAl in Run 5-14 and with Ni_3Al in Run 5-13, as well as the production of mixtures with SiC whiskers in Runs 5-15 and 5-16 were performed as in Runs 4-7 to 4-10, respectively.

The thus prepared powders of coated cubic boron nitride particles or mixtures thereof with SiC whiskers were shaped as in Example 1 and set in a cubic ultrahigh pressure apparatus; the pressure was raised to 5.1 - 5.6 GPa at room temperature, then the temperature was elevated to 1450 - 1500°C, which was held for 30 min; thereafter the temperature was lowered and the pressure reduced.

Each of the thus produced sinters was entirely free from pores, could be densified to a relative density of 100% and had no portion left unsintered. As in Example 1, the surfaces of the sinters were polished with a diamond paste and their Vickers microhardness Hv (0.5/10) values were measured. The results are shown in Table 2 together with the sintering conditions.

The crystalline phases of the sinters were examined by powder X-ray diffraction. In Run 5-1, Zr reacted with the cubic boron nitride, whereupon metallic Zr disappeared, changing to cubic boron nitride, ZrN and ZrB_2 which were found to be present in respective volume proportions of about 77%, 14% and 9% according to quantification by X-ray analysis. In the sinter of Run 5-2, only cubic boron nitride, TiN and TiB_2 were observed and their respective volume proportions were about 74%, 15% and 11%. In Runs 5-3 to 5-14, only the cubic boron nitride and the coating forming substances used (TiN, TiN, AlN, AlN, α- and β-Si_3N_4, α- and β-Si_3N_4, α-Al_2O_3, α-Al_2O_3, Co, Co, Ni_3Al and NiAl, respectively) were observed; in Runs 5-15 and 5-16, only TiN and α- and β-Si_3N_4, respectively, were observed in addition to the old SiC and the cubic boron nitride core particles. It should be noted that the sinter produced in Run 5-11 and 5-12 were entirely free from the peak of metallic Co.

Fig. 16 is a graph showing the Hv (0.5/10) value of the cubic boron nitride sinter of Run 5-1 in comparison with the hardness data of four typical commercial cutting tools made from cubic boron nitride sinters using ceramic bonding materials or sintering aids. The sinter produced in Run 5-1 was superhard with the Hv (0.5/10) being about 6500 and this value was comparable to or higher than those of the commercial cutting tools made of sintered cubic boron nitride. Furthermore, the hardness of the sinter of Run 5-1 was uniform over the entire surface subjected to the hardness measurement and it was substantially free from variations.

The polished surface of the sinter of Run 5-1 was coated with a gold film by a customary evaporation technique and examined with an electron microscope. The result is shown in the Fig. 17 electron micrograph (X5000), in which the dark area represents the cubic boron nitride and the light area the ZrN-ZrB_2 base substance. As one can see from Fig. 17, the sinter was entirely free from pores and could be sintered to a relative density of 100%. In addition, it had no portion left unsintered. In areas where cubic boron nitride particles contacted each other via a thin layer of the coat forming substance, the latter was bursted by the contiguous cubic boron nitride particles, which hence were coupled directly as a result of sintering. In all other areas, the ZrN-ZrB_2 base material was distributed to surround the cubic boron nitride particles, rendering the sinter to be characteristic in that it was dense, uniform and had the bonding material distributed to provide a highly controlled microstructure. It should also be noted that the cubic boron nitride particles in the sinter did not grow at all compared to the particles in the feed powder and that the coating on the cubic boron nitride was formed by chemical reaction between the cubic boron nitride and the metallic zirconium which served as the coat

forming substance; these factors combined together to offer the characteristic feature that the cubic boron nitride particles in the sinter were finer than those in the feed although they were coated zirconium. This is very favorable and, hence, ideal for the mechanical characteristics of the sinter. The differences can be clearly seen by comparing Fig. 17 with Fig. 18 which is an electron micrograph (X5000) of a polished surface of one of the above-mentioned commercial cutting tools that was made of sintered cubic boron nitride and which was coated with a gold film by a customary evaporation technique for examination. Obviously, the bonding material and the sintering aid were distributed irregularly in the cubic boron nitride sinter of the commercial cutting tool to form lumps in certain areas. On the other hand, the number of areas that were devoid of the necessary sintering aid and bonding material was by no means small and unsintered portions were found there. Another major difference from the sinter of Run 5-1 was that every cubic boron nitride particle in the cutting tool was coarse.

The cubic boron nitride is inherently very difficult to sinter. In spite of this fact, the coated particles in a cubic boron nitride powder that were produced in Example 5 in accordance with the invention behaved as if they were comparatively easy-to-sinter particles under industrially feasible ultrahigh pressures and elevated temperatures, thereby forming dense, rigid and superhard structures.

The sinters thus produced were similar to those produced in Examples 1 and 4 in that they were sinters or composite sinters that were superhard and which had dense, uniform and highly controlled microstructures with substantially uniform distributions of the bonding materials.

Table 2

Run No.	Relative proportions (vol %)				Coating apparatus Ex. No.	Coating Conditions (Approximate Ex. or Com. No.)	Sintering Conditions		Sintered Density %	Hardness Hv
	Core Particle	Coat Forming Substance		Moisture etc			Pressure atm	Temperature °C	Time min	Sintered appearance		
	CBN	Name	Amount									
S-1	60	ZrC	20	-	1	S-1	5.5	1450	30	CB	100	3500
S-2	60	TiC	20	-	2	S-2	5.1	1440	30	CB	100	3495
S-3	60	SiN	15	-	3	S-3	5.3	1480	30	CB	100	3480
S-4	60	SiN	30	-	1	S-1	5.2	1460	28	CB	100	3900
S-5	80	AlN	20	-	1	S-1	5.4	1490	30	CB	100	3800
S-6	70	AlN	30	-	1	S-3	5.4	1495	30	CB	100	3500
S-7	40	Si_3N_4	20	-	1	S-1	5.2	1430	30	CB	100	3300
S-8	20	Si_3N_4	30	-	1	S-7	5.3	1470	30	CB	100	3140
S-9	85	Al_2O_3	15	-	2	S-2	5.5	1480	30	CB	100	4141
S-10	80	Al_2O_3	20	-	2	S-3	5.4	1460	30	CB	100	3920
S-11	30	CB	10	-	1	S-1	5.3	1450	30	CB	100	3060
S-12	70	Co	30	-	1	S-1	5.5	1455	30	CB	100	2870
S-13	80	PtAl	18	-	1	S-1	5.1	1430	30	CB	100	4200
S-14	80	WAl	20	-	1	S-13	5.1	1460	30	CB	100	3950
S-15	72	TiN	18	10	1		5.3	1500	30	CB	100	3800
S-16	72	SiAl	18	10	1		5.3	1470	30	CB	100	3100

CB: Cubic ultrahigh pressure apparatus

Example 6

Coated diamond particles were prepared by coating the bare diamond particles of Example 1 with various coat forming substances using apparatus of the same types as used in Examples 1 - 3. The names of the coat forming substances used to prepare the coated diamond particles and their coating weights (relative proportions by vol%) are shown in Table 3.

The apparatus used in Run 6-1 was of the same type as used in Example 1; an alumina powder having an average diameter of 2 μm was supplied as a feed of coat forming substance from the feeder 112 at a rate of 1.5 g/min and the core diamond particles were supplied from the feeder 111 at a rate of 0.3 g/min. The other conditions in Run 6-1 were the same as in Example 1. The apparatus used in Run 6-2 was of the same type as used in Example 2; an alumina powder having an average diameter of 2 μm was supplied as a feed of coat forming substance at a rate of 1.0 g/min and the diamond particles were supplied at a rate of 0.7 g/min. The other conditions in Run 6-2 were the same as in Example 2. The apparatus used in Run 6-3 was of the same type as in Example 3; the values of dispersity (δ) attained in the discharge section of cyclone 6-1 and in the coating start region were the same as in Run 4-2 and an alumina powder was supplied at a rate of 0.6 g/min whereas the diamond particles were supplied at a rate of 1.5 g/min. The other conditions in Run 6-3 were the same as in Example 3.

The apparatus used in Run 6-4 was of the same type as used in Example 1 and operated to perform the coating operation under generally the same conditions as in Run 6-1. In Runs 6-5 to 6-9, inorganic salts consisting of KCl, $BaCl_2$ and NaF were mixed with TiO_2 and ferroalloy (Fe-Ti) to prepare an immersing bath. In this bath, diamond powder particles (up to 3 μm in diameter) were dipped for 1 h by the molten salt disproportionation method with the bath being melted at 900°C. As a result, the surfaces of the diamond powder particles were coated with TiC in such a way that the amount of internal addition would be 5% by volume. The surfaces of diamond particles were further coated with the apparatus of Example 1 under generally the same conditions as in Run 6-1.

The coated diamond particles thus prepared were examined with a scanning electron microscope. All of the particles prepared in Runs 6-1 to 6-3 were uniformly covered with superfine particles (ca. 0.005 μm) of coat forming substances as Fig. 19 shows (particularly for the case of Run 6-1).

In Runs 6-10 and 6-11, the powders of alumina-coated diamond particles prepared in Runs 6-1 and 6-2, respectively, were wet mixed with SiC whiskers at a volume ratio of 80:10 in acetone for 3 h in an alumina ball mill. In Run 6-12, a powder of diamond particles was coated with 32% by volume of alumina using the apparatus of Example 1 under generally the same conditions as in Run 6-1 and then wet mixed with SiC whiskers at a volume ratio of 85:15 in acetone for 3 h in an alumina ball mill. The SiC whiskers had their surfaces coated with the target TiC by sputtering in a coating weight of 33% by volume in terms of internal addition. The mixtures was vacuum dried at 10^{-4} torr at 800°C to make dry blends.

The coated diamond particles thus prepared or mixtures thereof with SiC whiskers were stamped into disks having a diameter of 16 mm and a thickness of 5 mm in Runs 6-1 and 6-10 and into disks having a diameter of 8 mm and a thickness of 5 mm in Runs 6-2 and 6-11. The disks were placed into Pyrex glass capsules packed with a h-BN powder, degassed to 10^{-6} torr at 400°C over 12 h and then sealed. The sealed capsules were set in a HIP or a ultrahigh pressure HIP apparatus using an argon gas as a pressure-transmitting medium and sintering was performed under the conditions shown in Table 3. In Runs 6-3 to 6-12, the coated diamond particles or mixtures thereof with whiskers were stamped into pellets having a diameter of 10 mm and a thickness of 8 mm and the pellets were buried in a pyrophyllite pressure-transmitting medium surrounded by a h-BN shaped part; the assemblies were set in a piston-cylinder (PC) ultrahigh pressure apparatus and sintering was performed under the conditions shown in Table 3. Following furnace cooling, the pressure was opened to atmosphere and the sinters were recovered.

The recovered sinters were measured for their density and Vickers microhardness Hv (0.5/10) as in Example 1 and the results are shown in Table 3.

The sinters of Runs 6-1 to 6-4 were checked for their crystalline phases by powder X-ray diffraction and no diffraction peaks were observed except for the diamond and α-form alumina. Similar results were obtained in the other runs: only diffraction peaks for diamond, titanium carbide and α-form alumina were observed in Runs 6-5 to 6-7; only diffraction peaks for diamond, titanium carbide and the monoclinic and tetragonal zirconium oxide each composing the PSZ were observed in Runs 6-8 and 6-9; only diffraction peaks for diamond, α-form alumina and silicon carbide were observed in Runs 6-10 and 6-11; and only diffraction peaks for diamond, α-form alumina, silicon carbide and titanium carbide were observed in Run 6-12. No other diffraction peaks were observed.

The surface of the sinter produced in Run 6-1 was polished with a diamond paste; the polished surface was coated with a gold film by a customary evaporation technique and examined with an electron microscope. The result is shown in the Fig. 20 micrograph (X5000), in which the dark area represents the diamond and the light area the alumina bonding material.

For comparison, bare diamond particles of the same type as used in Example 6 were mixed with an alumina powder at a ratio of 70:30 in volume percent and the mixture was sintered under the same conditions as in Example 6. The resulting sinter had a density of 92% and a Vickers microhardness of 2550. The difference is clearly visible by comparing

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Fig. 20 with Fig. 21 which is an electron micrograph (X3000) of a polished surface of the comparative sinter that was coated with a gold film by a customary evaporation technique for examination. Obviously, the bonding material and the sintering aid were distributed irregularly in the comparative sinter to form lumps in certain areas. On the other hand, the number of areas that were devoid of the necessary sintering aid and bonding material was by no means small and unsintered portions were found there. Thus, the sinter produced in Run 6-1 in accordance with the invention was significantly superior to the comparative sinter in terms of density, hardness and microstructure.

Each of the sinters produced in Runs 6-5 to 6-7 presented considerable difficulty in forming a polished surface for the hardness measurement and this fact shows that these sinters had high wear resistance, which was probably due to the formation of the titanium carbide coating film by dip coating.

The foregoing data, when considered with particular reference to Figs. 20 and 21, demonstrate that the sinters produced in Example 6 were entirely free from unsintered portions, pores, defects, etc., highly dense, superhard and comprised of highly controlled microstructures in which the diamond particles were surrounded by uniform distributions of the bonding material(s), i.e., α-form alumina, the combination of titanium carbide and α-form alumina, or the combination of titanium carbide and PSZ, or in which these coated diamond particles were further surrounded with a uniform dispersion of SiC.

Table 3

Run No.	Relative Proportions (vol%)						Sintering Conditions			Sintered Bodies	
	Coated Diamond Particles			Fibrous Substance							
	Diamond	Coat-Forming Substance		Whisker	Coating Substance	Pressure	Temperature	Time	Apparatus	Density	Hardness
		TiC	Al_2O_3	TaC	TiC	MPa	°C	hr.		%	Hs
E-1	20	-	20	-	-	150	1200	3	HIP1	96.0	2240
E-2	30	-	30	-	-	1000	1200	3	HIP2	99.0	2350
E-3	50	-	50	-	-	4800	1200	3	PC	98.2	3000
E-4	60	-	30	-	-	1500	1300	3	PC	99.5	2400
E-5	55	5	40	-	-	1800	1400	4	PC	94.6	2670
E-6	50	5	45	-	-	1800	1100	1	PC	95.0	3200
E-7	60	5	35	-	-	1500	1300	6	PC	99.6	2230
E-8	55	5	-	40	-	1500	1300	4	PC	99.3	2060
E-9	30	5	-	45	-	1900	1300	1	PC	99.4	1120
E-10	72	-	53	-	10	150	1200	3	HIP1	94.8	2080
E-11	45	-	45	-	10	1000	1200	3	HIP2	99.0	3220
E-12	40	-	48	-	10	4800	1300	3	PC	98.1	3170

HIP1: Hot apparatus incapable of producing ultrahigh pressures

HIP2: Especially pressure HIP apparatus

PC: Piston-cylinder (PC) ultrahigh pressure apparatus

Example 7

Cubic boron nitride (cBN) particles of the same type as used in Example 2 were coated with various coat forming substances using apparatus of the same types as used in Examples 1 - 3. The names of the coat forming substances used to prepare the coated cubic boron nitride particles and their coating weights (relative proportions by vol%) are shown in Table 4.

The apparatus used in Runs 7-1 and 7-2 were of the same types as used in Examples 1 and 3, respectively, an alumina powder having an average diameter of 2 µm was supplied as a feed of coat forming substance at respective rates of 1.5 and 8.1 g/min and the cubic boron nitride core particles were supplied at respective rates of 0.3 and 1.5 g/min. The other conditions in Runs 7-1 and 7-2 were the same as in Example 1 and Run 6-2, respectively. The apparatus used in Runs 7-3 and 7-4 was of the same type as used in Example 1 and the other conditions were the same as in Run 7-1. In Runs 7-5, 7-6 and 7-8, the procedures of Runs 6-6, 6-7 and 6-9 were repeated, except that the surfaces of cubic boron nitride particles were dip coated with the combination of titanium nitride (TiN) and titanium diboride (TiB_2) in place of titanium carbide and, further, alumina or PSZ coating was performed using the same apparatus as in Example 1 under generally the same conditions as in Run 7-1. In Run 7-7, the apparatus of Example 1 was used and coating was performed under generally the same conditions as in Run 7-1, except that alumina was replaced by PSZ. In Runs 7-9 and 7-10, the alumina coated cubic boron nitride particles as prepared in Run 7-1 and Example 2, respectively, were each mixed with SiC whiskers as in Runs 6-10 and 6-11. In Run 7-11, cubic boron nitride particles were coated with 93% alumina using the apparatus of Example 1 under generally the same conditions as in Run 7-1 and then mixed with TiC coated SiC whiskers that were prepared as in Run 6-12.

The coated cubic boron nitride particles thus prepared were examined with a scanning electron microscope. All of the particles prepared in Runs 7-1 to 7-3 were uniformly covered with superfine particles (ca. 0.005 µm) of coat forming substances as Fig. 22 shows (particularly for the case of Run 7-1).

The powders of coated cubic boron nitride particles thus prepared in Runs 7-1 to 7-8 or the mixtures thereof with SiC whiskers which were prepared in Runs 7-9 to 7-11 were shaped by stamping as in Runs 6-1, 6-3 to 6-12, respectively, and sintered with the sinter apparatus shown in Table 4 under the sintering conditions shown in Table 4.

The sinters thus produced were measured for their density and Vickers microhardness Hv (0.5/10) as in Example 1 and the results are shown in Table 4.

The crystalline phases of the sinters were examined by powder X-ray diffraction as in Runs 6-1 to 6-12, no diffraction peaks were observed except for the cubic boron nitride (core particles), the bonding materials or coat forming substances (α-alumina, titanium nitride, titanium diboride, as well as the PSZ composing monoclinic and tetragonal zirconium oxide), and whiskers (silicon carbide) and the titanium carbide coating thereon.

The polished surface of the sinter of Run 7-1 was coated with a gold film by a customary evaporation technique and examined with an electron microscope. The result is shown in the Fig. 23 electron micrograph (X5000), in which the dark area represents the cubic boron nitride and the light area the bonding material alumina. As one can see from Fig. 23, the sinter was entirely free from unsintered portions, pores, defects, etc. and it was comprised of a highly controlled microstructure in which the bonding material alumina was distributed uniformly around the cubic boron nitride particles.

For comparison, bare cubic boron nitride particles of the same type as used in Run 7-1 were mixed with an alumina powder at a ratio of 70:30 in volume percent and the mixture was sintered under the same conditions as in Run 7-1. The resulting sinter had a density of 94% and a Vickers microhardness of 2300. From an electron micrograph (X5000) of a polished surface of the comparative sinter that was coated with a gold film by a customary evaporation technique for examination, obviously, the bonding material and the sintering aid were distributed irregularly in the comparative sinter to form lumps in certain areas. On the other hand, in the comparative sinter, the number of areas that were devoid of the necessary sintering aid and bonding material was by no means small and unsintered portions were found there.

In Runs 7-5, 7-6 and 7-9, the TiN-TiB_2 coating film formed by dip coating were found to be as effective as the TiC coating film formed by dip coating in Runs 6-5 to 6-7.

Thus, the sinters produced in Example 7 were entirely free from unsintered portions, pores, defects, etc., highly dense, superhard and comprised of highly controlled microstructures in which the cubic boron nitride particles were surrounded by uniform distributions of the bonding material(s), i.e., α-alumina, PSZ, the combination of TiN-TiB_2 and α-alumina, or the combination of TiN-TiB_2 and PSZ, or in which these coated cubic boron nitride particles were further surrounded with a uniform dispersion of SiC as whiskers.

Table 4

Run No.	Relative Proportions (vol%)					Sintering Conditions			Sinter	
	Coated High-Pressure Boron Nitride Particles			Fibrous Substance	Cooling Substance	Pressure	Temperature	Time	Density	Hardness
	BBN	TiN-TiAlN	Al2O3	TiB2	Whisker	ZrO2	kg/cm2	°C	hr.	%	Hv
2	30	-	50	-	-	-	1000	1300	1	99.8	3230
2-1	30	-	70	-	-	-	100	1300	1	97.1	2900
2-2	30	-	30	-	-	-	1300	1300	1	99.8	3330
2-3	30	-	30	-	-	-	1500	1300	1	99.8	3370
2-4	60	-	10	-	-	-	1800	1300	1	99.2	4100
2-5	50	1	38	-	-	-	1500	1300	1	99.0	4030
2-6	60	1	38	-	-	-	1800	1300	1	98.5	4100
2-7	60	-	-	40	-	-	1500	1300	1	99.8	4300
2-8	50	1	2	46	-	-	1500	1300	1	99.8	4300
2-9	27	-	63	-	10	-	100	1300	1	97.1	2780
2-10	45	-	45	-	10	-	1000	1300	1	99.0	3540
2-11	60	-	40	-	10	1	1100	1300	1	98.2	3600

BBN: BBP sintered body capable of producing ultrahigh pressures

BBP: Sintered pressure BN sintered body

PB: Polycrystalline CBN ultrahigh pressure sintered body

Example 8

Coated diamond particles were prepared by coating the bare diamond particles of Example 1 with various coat forming substances using apparatus of the same type as used in Examples 1 - 3. The names of the coat forming substances used to prepare the coated diamond particles and their coating weights (relative proportions by vol%) are shown in Table 8.

The apparatus used in Runs 8-1, 8-2 and 8-4 were of the same types as used in Examples 1, 2 and 3, respectively; a titanium carbide powder having an average diameter of 2 µm was supplied as a feed of coat forming substance at a rate of 0.4 g/min and the core diamond particles were supplied at a rate of 2.0 g/min; the other conditions in Runs 8-1, 8-2 and 8-4 were generally the same as in Example 1 and Runs 4-1 and 4-2, respectively. In each of those runs, 10% titanium carbide coated diamond particles were obtained. In Runs 8-5 and 8-6, an apparatus of the same type as used in Example 1 was operated under generally the same conditions as in Run 8-1 to produce 10% titanium carbide coated diamond particles; in Runs 8-7 and 8-8, the same procedure was repeated to produce 10% metallic titanium coated diamond particles; in Runs 8-9 and 8-10, the same procedure was repeated to yield 20% titanium carbide coated diamond particles; in Run 8-3, the 10% titanium carbide coated particles prepared in Run 8-2 were used.

The coated diamond particles thus prepared were examined with a scanning electron microscope. All of the particles prepared in Runs 8-1 to 8-4 were uniformly covered with superfine particles (ca. 0.005 µm) of coat forming substances as Fig. 25 shows (particularly for the case of Run 8-1).

The thus prepared powders of coated diamond particles and powders of bonding materials having the various formulations shown in Table 8 were added in the various proportions shown in Table 8 and wet mixed in acetone for 2 h in an alumina ball mill. Thereafter, the mixtures were vacuum dried at 10^{-3} torr and at 200°C to produce dry blends. The bonding material powder used in Runs 8-1 to 8-4 was easily sinterable alumina of high purity (i.e., the alumina powder described in the official gazette of Unexamined Published Japanese Patent Application (kokai) Sho 63-151616); in Runs 8-5 and 8-6, PSZ was used as a bonding material powder, and in Runs 8-7 to 8-10, TiC was used. Take, for example, Run 8-1; in this run, 65 vol% of 10% TiC coated diamond particles were mixed with 43.4 vol% of easily sinterable alumina of high purity as a bonding material powder, and with sintering aids for the alumina powder which were 0.6 vol% and 1.0 vol%, respectively, of magnesia (MgO) and titania (TiO_x; x = 1 - 2).

In Runs 8-11 and 8-12, 90 vol% of the mixtures prepared in Runs 8-4 and 8-5, respectively, were mixed with 10 vol% of SiC whiskers and the mixtures thus prepared were used as sinter feeds.

The thus prepared mixtures of coated diamond particles with bonding material powders were stamped into disks having a diameter of 10 mm and a thickness of 6 mm in Runs 8-1, 8-7, 8-8 and 8-10. The disks were buried in a pyrophyllite pressure-transmitting medium surrounded by a h-BN shaped part and the assemblies were set in a piston-cylinder (PC) ultrahigh pressure apparatus, and sintering was performed under the conditions set forth in Table 8. In the other runs, the same mixtures of coated diamond particles with bonding material powders, or mixtures further containing SiC whiskers were stamped into disks having a diameter of 18 mm and a thickness of 6 mm. The disks were placed in a Pyrex glass capsule packed with a h-BN shaped part and the assemblies were degassed to 10^{-3} torr by heating at 400°C for 12 h and sealed. Thereafter, the capsules were set in a HIP or ultrahigh pressure HIP apparatus using an argon gas as a pressure-transmitting medium and sintering was performed under the conditions set forth in Table 8. Subsequently, the temperature was lowered and the pressure reduced for recovering the sinters.

The recovered sinters were measured for their density and Vickers microhardness Hv (0.5/10); the results are shown in Table 8 together with the sintering conditions and the types of sintering apparatus used.

The sinters were also checked for their crystalline phases by powder X-ray diffraction. In Example 8, no diffraction peaks were observed except for the diamond, coat forming substances (coating materials) and the bonding material powders or whisker SiC. Take, for example, Runs 8-1 to 8-4; in these cases, the only diffraction peaks observed were those for diamond, TiC and α-alumina. In the other runs, the only diffraction peaks observed were those for the following: diamond, TiC and PSZ composing monoclinic and tetragonal zirconium oxide (Runs 8-5 and 8-6); diamond, the TiC that was generated by the reaction between the coating metallic titanium and the diamond, as well as TiN (Runs 8-7 and 8-8); diamond and TiC (Run 8-9 and 8-10); diamond, TiC, α-alumina and SiC (Run 8-11); diamond, TiC, the PSZ composing monoclinic and tetragonal zirconium oxide, and SiC (Run 8-12).

The surface of the sinter produced in Run 8-1 was polished with a diamond paste and coated with a gold film by a customary evaporation technique and examined with an electron microscope. The result is shown in the Fig. 26 electron micrograph (X6000), in which the dark area represents the diamond and the light area the alumina-base bonding material.

As one can see from Fig. 26, all the sinters of Example 8 were entirely free from unsintered portions, pores, defects, etc., highly dense, superhard and comprised of highly controlled microstructures in which the diamond particles were surrounded by uniform distributions of the coating materials, the bonding materials (TiC, TiN, α-alumina and PSZ) and the SiC whiskers, if any.

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Table 5

Run No.	Relative Proportions (vol%)									Sintering Conditions				Binder	
	Diamond	Coating Material		Bonding Material Powder					Molybdenum	Pressure	Temperature	Time	Apparatus	Density	Hardness
		TiC	TiN	Si	WSi$_2$	TiN	Al$_2$O$_3$	MgO	ZrO$_2$	B4C	MPa	°C	hr		g/cm^3	Hv
E-1	50.0	3.0	-	-	-	-	43.4	0.6	3.0	-	1300	1200	1	HP	99.2	4110
E-2	30.0	3.0	-	-	-	-	63.4	0.6	3.0	-	130	1300	1	HP1	98.8	3130
E-3	40.0	4.0	-	-	-	-	51.4	0.6	4.0	-	1300	1300	1	HP2	99.4	3380
E-4	20.0	2.0	-	-	-	-	76.4	0.6	1.0	-	130	1300	1	HP1	97.8	2810
E-5	30.0	3.0	-	3	67.0	-	3	-	-	-	130	1200	1	HP1	99.5	1010
E-6	45.0	4.5	-	-	50.5	-	-	-	-	-	130	1300	1	HP1	98.8	1030
E-7	20.0	-	-	2.0	-	76.0	-	-	-	-	1300	1200	1	HP	99.1	2660
E-8	40.0	-	-	1.0	-	56.0	-	-	-	-	1300	1300	1	HP	97.8	3330
E-9	20.0	-	4.0	-	-	76.0	-	-	-	-	1300	1200	3	HP2	99.0	2640
E-10	40.0	-	8.0	-	-	52.0	-	-	-	-	1300	1300	3	HP	99.2	3400
E-11	18.0	1.8	-	-	-	-	68.9	0.3	0.9	10.0	130	1300	3	HP1	99.4	2880
E-12	27.0	2.7	-	-	60.3	-	-	-	-	10.0	130	1300	3	HP1	98.9	1880

HP1: Hot separate isostatic pressing with capsules

HP2: Superhigh pressure gas apparatus

HP: Piston-cylinder (DC) ultrahigh pressure apparatus

Example 9

Coated cubic boron nitride particles were prepared by coating the bare cubic boron nitride (cBN) particles of Example 1 with various coat forming substances using the apparatus employed in Examples 1 - 8. The names of the coat forming substances used to prepare those coated cubic boron nitride particles and their coating weights (relative proportions by vol%) are shown in Table 6.

The apparatus used in Runs 9-1, 9-2 and 9-3 were of the same types as used in Examples 1, 2 and 3, respectively; a titanium nitride powder having an average diameter of 2 µm was supplied as a feed of coat forming substance at a rate of 0.3 g/min and the cubic boron nitride core particles were supplied at a rate of 1.5 g/min; the other conditions in Runs 9-1, 9-2 and 9-3 were generally the same as in Example 1, Runs 4-2 and 4-3, respectively. In each of those runs, 10% titanium nitride coated cubic boron nitride particles were obtained. In Runs 9-4 and 9-5, an apparatus of the same type as used in Example 1 was operated under generally the same conditions as in Run 9-1 to produce 10% titanium nitride coated cubic boron nitride particles; in Runs 9-6 and 9-7, the same procedure was repeated to produce 10% metallic titanium coated cubic boron nitride particles; and in Runs 9-8 and 9-9, the same procedure was repeated to yield 20% titanium nitride coated cubic boron nitride particles.

The coated cubic boron nitride particles thus prepared were examined with a scanning electron microscope. All of the particles prepared in Runs 9-1 to 9-9 were uniformly covered with superfine particles (ca. 0.005 µm) of coat forming substances as Fig. 27 shows (particularly for the case of Run 9-1).

The thus prepared powders of coated cubic boron nitride particles and powders of bonding materials having the various formulations shown in Table 6 were added in the various proportions shown in Table 6. Those powders were wet mixed in acetone for 2 h in an alumina ball mill as in Example 8. Thereafter, the mixtures were vacuum dried at 10^{-3} torr and at 800°C to produce dry blends. The bonding material powders used in Example 9 were the same as those used in Example 8, i.e., easily sinterable alumina of high purity, PSZ, and TiN. As in Example 8, the use of easily sinterable high-purity alumina with an average particle diameter of 0.2 µm as a bonding material powder was accompanied by the use of sintering aids for the alumina powder which were magnesia (MgO) and titania (TiO_x; $x \approx 1 \sim 2$).

In Runs 9-10 and 9-11, 80 vol% of the mixtures prepared in Runs 9-3 and 9-4, respectively, were mixed with 10 vol% of SiC whiskers and the mixtures thus prepared were used as sinter feeds.

The thus prepared mixtures of coated cubic boron nitride particles with bonding material powders were stamped into disks in Runs 9-1, 9-6, 9-7 and 9-9 as in Run 8-1; in the other runs, the same mixtures of coated cubic boron nitride particles with bonding material powders, or mixtures further containing SiC whiskers were stamped into disks as in Run 8-2. The disks were then sintered with the sinter apparatus listed in Table 6 under the sinter conditions also set forth in Table 6. Thereafter, the temperature was lowered and the pressure reduced for recovering the sinters.

The recovered sinters were measured for their density and Vickers microhardness Hv (0.5/10); the results are shown in Table 6 together with the sintering conditions and the types of sintering apparatus used.

The sinters were also checked for their crystalline phases by powder X-ray diffraction. In Example 9, no diffraction peaks were observed except for the cubic boron nitride, coat forming substances (coating materials) and the bonding materials or whisker SiC. Take, for example, Runs 9-1 to 9-3; in these cases, the only diffraction peaks observed were those for cubic boron nitride, TiN and α-alumina. In the other runs, the only diffraction peaks observed were those for the following: cubic boron nitride, TiN and the PSZ composing monoclinic and tetragonal zirconium oxide (Runs 9-4 and 9-5); cubic boron nitride, the TiB_2 that was generated by the reaction between the coating metallic titanium and cubic boron nitride, as well as TiN (Runs 9-6 and 9-7); cubic boron nitride and TiN (Runs 9-8 and 9-9); cubic boron nitride, TiC, α-alumina and SiC (Run 9-10); and cubic boron nitride, TiC, the PSZ composing monoclinic and tetragonal zirconium oxide, and SiC (Run 9-11).

Quantification by X-ray analysis showed that the sinter of Run 9-6 consisted of cubic boron nitride, TiB_2 and TiN in proportions of 19.9%, 1.0% and 79.6%, respectively, by volume whereas the sinter of Run 9-7 had relative volume proportions of 30.1%, 2.0% and 68.0%.

The surface of the sinter produced in Run 9-1 was polished with a diamond paste and coated with a gold film by a customary evaporation technique and examined with an electron microscope. The result is shown in the Fig. 28 electron micrograph (X5000), in which the dark area represents the cubic boron nitride and the light area the alumina base bonding material.

As one can see from Fig. 28, all the sinters of Example 9 were entirely free from unsintered portions, pores, defects, etc., highly dense, superhard and comprised of highly controlled microstructures in which the cubic boron nitride particles were surrounded by uniform distributions of the coating materials, the bonding materials (TiN, TiB_2, α-alumina and PSZ) and the SiC whiskers, if any.

Table 6

Run No.	Relative Proportions (vol %)								Sintering Conditions				Sinter	
	cBN	Coating Material	Bonding Material Powder					Whisker SiC	Pressure MPa	Temperature °C	Time hr.	Apparatus	Density g/cm³	Hardness Hv
		TiN	Ti	Fe2	TiN	Al2O3	MgO	ZrO2						
2-1	10.0	2.0	-	-	-	42.4	0.6	1.0	-	1500	1300	3	EC	97.8	4030
2-2	40.0	4.0	-	-	-	34.4	0.6	1.0	-	1000	1300	3	KT22	98.1	3330
2-3	20.0	2.0	-	-	-	70.4	0.6	1.0	-	150	1300	3	KT21	98.3	2870
2-4	30.0	3.0	-	62.0	-	-	-	-	-	150	1300	3	KT21	98.6	1030
2-5	50.0	4.0	-	30.3	-	-	-	-	-	150	1300	3	KT21	98.0	2010
2-6	20.0	-	2.0	-	78.0	-	-	-	-	1500	1300	3	EC	98.3	2890
2-7	40.0	-	4.0	-	56.0	-	-	-	-	1500	1300	3	EC	98.0	3430
2-8	20.0	4.0	-	-	76.0	-	-	-	-	1000	1300	3	KT22	98.0	2900
2-9	40.0	6.0	-	-	54.0	-	-	-	-	1500	1300	3	EC	96.4	1310
2-10	10.0	1.6	-	-	-	46.8	0.3	0.8	10.0	150	1300	3	KT21	97.8	2790
2-11	22.0	2.2	-	60.3	-	-	-	-	10.0	150	1300	3	KT21	98.1	1940

KT21: KT apparatus incapable of producing ultrahigh pressures

KT22: Superhigh pressure ZTE apparatus

EC: Belt-cylinder (EC) ultrahigh pressure apparatus

Examples 10 and 11

Using the apparatus employed in Examples 1 - 3, pseudo-fine diamond particles (Example 10) or pseudo-fine particles of a high-pressure phase boron nitride powder (Example 11) were covered with various coat forming materials. The pseudo-fine diamond particles were the same as those used in Example 8; the pseudo-fine particles of a high-pressure phase boron powder were pseudo-fine cubic boron nitride (cBN) particles that had an average diameter (D_{50}) of 17 μm and a frequency distribution by volume of ($[D_{50}/2, 3D_{50}/2]$, ≥ 90%). The names of the coat forming substances used to prepare the pseudo-fine coated diamond and cubic boron nitride particles and their coating weights (relative proportions by vol%), as well as the specific types of the coating apparatus used and the conditions for their use (generally the same) are shown in Tables 7 and 8 together with the data for Example 3.

The apparatus used in Runs 10-1 and 11-1 was of the same type as used in Example 1; the group of means for high dispersion treatment of particles in this apparatus was to treat the pseudo-fine particles of either a diamond powder or a cubic boron nitride powder, in which the core particles had an average diameter of 17 μm and a frequency distribution by volume of ($[D_{50}/3, 5D_{50}]$, ≥ 90%) and, hence, said group of means was designed in such a way that it could realize an output dispersity (β) value of at least 80% (β ≥ 80%) for the core particles in those powders while, at the same time, the coating operation could be started with a dispersity (β) of at least 80% (β ≥ 80%) in the coating start region. A titanium carbide powder (Run 10-1) or a titanium nitride powder (Run 11-1) that were feeds for coat forming substances and which had an average particle diameter of 2 μm were supplied from the feeder 112 at a rate of 0.3 g/min whereas the pseudo-fine diamond or cubic boron nitride core particles were supplied from the feeder 111 at a rate of 3.0 g/min and introduced into the coating chamber after being dispersed to a dispersity (β) of 80%, and the dispersity (β) in the coating start region 3-L1 was 80%. The other conditions in Examples 10 and 11 were the same as in Example 1. The coating weights of titanium carbide and titanium nitride were each 5% by volume.

The apparatus used in Runs 10-2 and 11-2 was of the same type as used in Example 2; the dispersity (β) value attainable in these runs was the same as in Runs 10-1 and 11-1; a metallic titanium powder having an average particle diameter of 25 μm was supplied as a feed for coat forming substance from the feeder 215 at a rate of 0.5 g/min, and the pseudo-fine diamond or cubic boron nitride core particles were supplied from the feeder 214 at a rate of 2.5 g/min; the other conditions in Runs 10-2 and 11-2 were the same as in Example 2. The apparatus used in Run 11-3 was of the same type as used in Example 3; a zirconium powder having an average particle diameter of 28 μm was supplied as a feed for coat forming substance from the feeder 314 at a rate of 0.5 g/min and the pseudo-fine cubic boron nitride core particles were supplied from the feeder 313 at a rate of 2.0 g/min; the other conditions in Run 11-3 were the same as in Example 3. In Runs 10-2, 11-2 and 11-3, the coating weights of metallic titanium and zirconium were each 10% by volume.

The apparatus used in Runs 10-3, 10-4, 11-4 and 11-5 was of the same type as used in Example 1 and operated under generally the same conditions as in Example 1 to prepare the following in the respective runs: pseudo-fine diamond particles first coated with 6 vol% TiC, then with 50 vol% alumina; pseudo-fine diamond particles coated with 10 vol% TiC; pseudo-fine cubic boron nitride particles coated with 50 vol% alumina; and pseudo-fine cubic boron nitride particles coated with 10 vol% TiN.

The coated pseudo-fine diamond and cubic boron nitride particles were examined with a scanning electron microscope. All of the particles prepared in Examples 10 and 11 were uniformly covered with superfine particles (ca. 0.035 μm) of coat forming substances (TiC, TiN, Ti and Zr) as in Example 3 and as Fig. 29 (for the particular case of Run 10-1) and Fig. 30 (for the particular case of Run 11-1) show.

The thus prepared powders of pseudo-fine coated diamond or cubic boron nitride particles, or mixtures thereof with the bonding materials listed in Table 7 or 8 were shaped, set in the various types of sinter apparatus shown in Tables 7 and 8, and sintered under the various sintering conditions also set forth in Tables 7 and 8. Thereafter, the temperature was lowered and the pressure reduced for recovering the sinters. The specific procedures were as follows.

In Runs 10-1 and 10-2, 60% by volume of the powders of pseudo-fine diamond particles coated with TiC and Ti, respectively, were wet mixed in acetone with 40% by volume of bonding materials that were the powders of fine coated diamond particles as prepared by coating fine diamond particles of 0.5 - 2 μm in diameter (average diameter = 1 μm) with 15 vol% of TiC under generally the same conditions. In Runs 11-1, 11-2 and 11-3, 80% by volume of the powders of pseudo-fine cubic boron nitride particles coated with TiN, Ti and Zr, respectively, were wet mixed in acetone with 40% by volume of bonding materials that were the powders of fine coated cubic boron nitride particles as prepared by coating fine cubic boron nitride particles of 0.5 - 2 μm in diameter (average diameter = 1 μm) with 15 vol% of TiC under generally the same conditions. Thereafter, the resulting wet mixtures were shaped, vacuum dried and sintered with a cubic ultrahigh pressure apparatus as in Example 3.

In Runs 10-3 and 11-4, the powder of TiC and alumina coated pseudo-fine diamond particles and the powder of alumina coated fine cubic boron nitride particles, respectively, were stamped into disks having a diameter of 8 mm and a thickness of 5 mm; the disks were placed in Pyrex glass capsules packed with a h-BN powder. After degassing at 10^{-6} torr by heating at 600°C for 12 h, the capsules were sealed, set in a HIP apparatus using an argon gas as a pressure-transmitting medium and sintered under the conditions set forth in Tables 7 and 8.

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In Runs 10-4 and 11-5, the powder of TiC coated pseudo-fine diamond particles and the powder of TiN coated fine cubic boron nitride particles were respectively wet mixed in 40% by volume as in Run 9-2 with the associated bonding materials that were 83.4%, 0.8% and 1.0% by volume of the high-purity and easily sinterable alumina, magnesia (MgO) and titania (TiO_x; x = 1 ~ 8) which were used in Run 9-1. The mixtures were vacuum dried, shaped into disks, placed into capsules, degassed, sealed, set in a HIP apparatus and sintered under the conditions set forth in Tables 7 and 8.

The sinters thus produced were measured for their density and Vickers microhardness Hv (0.5/10). The results are shown in Tables 7 and 8 together with the sintering conditions and the types of sinter apparatus used.

As for the Vickers microhardness Hv (0.5/10) measurement of the sinters of the coated pseudo-fine particles, the hardness values obtained by the measurement were the values of the pseudo-fine particles and not the values of the sinters, since particles sizes of the pseudo-fine particles were larger than those of the fine particles and the hardness values of the sinters could not be measured. Therefore, in Tables 7 and 8 the Vickers microhardness values of the sinters were not shown.

The sinters were also checked for their crystalline phases by powder X-ray diffraction. In Examples 10 and 11, no diffraction peaks were observed except for the core particles (diamond or cubic boron nitride), coating materials (TiC, TiN and α-alumina), the products of reaction between the core particles and the metallic coating materials (TiC, ZrC, TiN, TiB_2, ZrN and ZrB_2) and the bonding materials (the diamond or cubic boron nitride in the core particles, TiC or TiN and α-alumina as the coating materials). Take, for example, Runs 10-1 and 10-2; in these cases, the only diffraction peaks observed were those for diamond and TiC. In the other runs, the only diffraction peaks observed were those for the following: diamond, TiC and α-alumina (Runs 10-3 and 10-4); cubic boron nitride and TiN (Run 11-1); cubic boron nitride, TiN and TiB_2 (Run 11-2); cubic boron nitride, TiN, ZrN and ZrB_2 (Run 11-3); cubic boron nitride and α-alumina (Run 11-4); and cubic boron nitride, TiN and α-alumina (Run 11-5). Quantification by X-ray analysis showed that the sinter of Run 10-3 consisted of 87% diamond and 13% TiC, the sinter of Run 11-2 consisted of cubic boron nitride, TiN and TiB_2 in proportions of about 87%, 10% and 3%, respectively, by volume; and the sinter of Run 11-3 consisted of cubic boron nitride, ZrN, ZrB_2 and TiN in proportions of about 88%, 8%, 1% and 11%, respectively, by volume.

The polished surface of the sinter of Run 10-1 was coated with a gold film by a customary evaporation technique and examined with an electron microscope. The result is shown in the Fig. 31 electron micrograph (X5000), in which the dark area represents the diamond and the light area TiC. As one can see from Fig. 31, the sinter was entirely free from pores and could be sintered to a relative density of 99%. In addition, it had no portion left unsintered. In areas where pseudo-fine diamond particles and/or fine diamond particles contacted each other via a thin layer of the coat forming substance, the latter was bursted by the contiguous pseudo-fine diamond particles and/or fine diamond particles, which hence were coupled directly as a result of sintering. In all other areas, TiC was distributed uniformly around the pseudo-fine diamond particles, rendering the sinter to be characteristic in that the coated pseudo-fine diamond particles had a dense, uniform and extremely highly controlled distribution. The diamond particles in the sinter were also characterized in that they did not grow at all compared to the particles in the feed powder. It has therefore been impossible to produce sinters having such an extremely highly controlled microstructure. Fig. 32 is an electron micrograph (X3000) showing a polished surface of the sinter of Run 11-1 and this was taken by the same method as used to prepare Fig. 31. Again, this sinter had established therein a microstructure in which pseudo-fine cubic boron nitride particles and/or fine cubic boron nitride particles and TiN as the coat forming substance were distributed uniformly, densely and in an extremely highly controlled manner.

Thus, in spite of the fact that diamonds and cubic boron nitrides are inherently very difficult to sinter, the coated pseudo-fine diamond and high-pressure phase boron nitride particles prepared in accordance with the invention behaved as if they were comparatively easy-to-sinter particles under industrially feasible ultrahigh pressures and elevated temperatures, thereby forming dense, rigid and superhard structures.

The sinters thus produced in Examples 10 and 11 were similar to that produced in Example 9 in that they were sinters or composite sinters that were superhard and which had dense, uniform and highly controlled microstructures with substantially uniform distributions of the bonding materials.

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Table 1

Ex. or Ref. No.	Relative Proportions (vol %)										Sintering Conditions			Sinter-ed Density g/cm³
	Core particle size	Coating Material				Bonding Material Powder					Pressure GPa	Temperature °C	Time min	Sintering Apparatus	
	Diamond	310	Zr	Al₂O₃	Diameter	Coating material	Al₂O₃							
1	22	-	1	-	10.2	10.1	-	-	-	-	5.5	1450	30	CB	3.99
10-1	62	-	-	-	31	1	-	-	-	-	5.5	1450	30	CB	3.99
10-2	34	-	6	-	11	2	-	-	-	-	5.5	1350	30	CB	3.99
10-3	19	-	-	50	-	-	-	-	-	-	6.15	1200	180	BEPA	3.99
10-4	38	4	-	-	-	-	33.1	0.411.6			6.15	1300	180	BEPA	3.99

Table 4

Run No.	Pelletizing Conditions (cont.)										Sintering Conditions				Others
	Core material	Coating material				Bonding material Powder					Over-score	Keeper-score	Time	Atmosphere	Density
	CBN	TiN	Ti	Si	Al_2O_3	Core material	Coating material	Al_2O_3	MnO/TiO		GPa	K	min		g
11-1	47	3	-	-	-	34	6	-	-	-	5.5	1350	20	Ar	3.09
11-2	51	-	4	-	-	24	6	-	-	-	5.5	1420	30	Ar	3.09
11-3	41	-	-	3	-	18.3	10.3	-	-	-	5.5	1450	30	Ar	3.09
11-4	40	-	-	-	30	-	-	-	-	-	6.13	1360	180	Air	3.04
11-5	30	4	-	-	-	-	-	33.3	6.67/3.3		5.16	1300	180	Air	3.05

Claims

1. A process for producing a diamond or high-pressure phase boron nitride sinter comprising the step of preparing coated diamond or high-pressure phase boron nitride particles and sintering said coated diamond or high-pressure phase boron nitride particles or mixtures containing such particles or mixtures of coated diamond or high-pressure phase boron nitride particles with bonding material,

sintering said coated diamond or high-pressure phase boron nitride particles or said mixtures containing such particles at pressures no less than 2000 MPa and at elevated temperatures, or

sintering said coated diamond or high-pressure phase boron nitride particles or said mixtures containing such particles at pressures of less than 2000 MPa and at temperatures not exceeding 1850°C under such sintering pressure and temperature conditions that diamond or high-pressure phase boron nitride is thermodynamically metastable if not stable; or

sintering said mixtures of coated diamond or high-pressure phase boron nitride particles with a bonding material in a volume ratio of (1 ~ 99):(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 1850°C under such conditions that diamond and/or high-pressure phase boron nitride particles are thermodynamically metastable, at pressures less than 2000 MPa and at temperatures not exceeding 1850°C under such sintering pressure and temperature conditions that diamond and/or high-pressure phase boron nitride is thermodynamically metastable if not stable,

wherein said coated diamond or high-pressure phase boron nitride particles are prepared by a method comprising the steps of charging a powder of diamond or high-pressure phase boron nitride core particles into a coating space and then permitting a precursor of a coat forming substance which is generated via the vapor phase and/or a precursor of a coat forming substance in a vapor-phase state to contact and/or impinge against the particles in said powder of core particles so that their surfaces are coated with the coat forming substance and

said coated diamond or high-pressure phase boron nitride particles are prepared by coating means comprising:

(A) a dispersing step in which the particles in the powder of core particles are dispersed by a group of means for high dispersion treatment of particles in a gaseous atmosphere to form a mixture of a gas and the particles in a powder of highly dispersed particles, the final treating means in said group of means for high dispersion treatment of particles being selected from between:

(a) dispersing means for dispersing the particles in the powder of core particles in a gaseous atmosphere; and

(b) means for selecting a mixture of a gas and the particles in a powder of highly dispersed particles which comprises:

(b-1) means for selecting a mixture of a gas and the particles in a powder of highly dispersed particles that separates a portion of the powder that is composed of less highly dispersed particles from the mixture of a gas and the particles in the powder of core particles in which the particles in the powder of core particles are dispersed in a gaseous atmosphere, thereby selecting a mixture of a gas and the particles in a powder of highly dispersed core particles in which the particles in the powder of core particles are present in the gaseous atmosphere mainly in a single-particle state; and

(b-2) feedback means by which the portion of the powder that is composed of less highly dispersed particles and which has been separated by said means for selecting a mixture of a gas and the particles in a powder of highly dispersed particles is transported to the final dispersing means among the dispersing means in the group of means for high dispersion treatment of particles and/or treating means upstream of the final dispersing means; and

(B) a coating step in which the particles in the powder of core particles that have been dispersed in the dispersing step (A) start to be coated by being allowed to contact and/or impinge against the precursor of the coat forming substance in the coating start region of the coating space as they are dispersed to satisfy the following conditions:

the dispersity β should be at least 70% if those particles have an average diameter of no more than 10 μm in the frequency distribution by volume;

the dispersity β should be at least 60% 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 50% 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 35% 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 600 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 600 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 3000 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 3000 MPa and at temperatures not exceeding 1850°C under such sintering pressure and temperature conditions that diamond or high-pressure phase boron nitride is thermodynamically metastable if not stable; or

sintering said mixtures of coated diamond or high-pressure phase boron nitride particles with a bonding material in a volume ratio of (1 - 90):(99 - 10), said bonding material being sintrable to a density of at least 95% at pressures less than 2000 MPa and at temperatures not exceeding 1850°C under such conditions that diamond and/or high-pressure phase boron nitride particles are thermodynamically metastable, at pressures less than 2000 MPa and at temperatures not exceeding 1850°C under such sintering pressure and temperature conditions that diamond and/or high-pressure phase boron nitride is thermodynamically metastable if not stable.

2. A process for producing a diamond or high-pressure phase boron nitride sinter according to claim 1 wherein said coated diamond or high-pressure phase boron nitride particles are prepared through the following additional step(s):

a step of disagglomeration or otherwise size reduction in which lumps of the coated diamond and/or high-pressure phase boron nitride particles that form as a result of association of several particles that contact one another via the coat forming substance on the coated particles are disagglomerated and/or otherwise reduced in size; and/or

a step of selective separation in which the associating lumps of the coated diamond or high-pressure phase boron nitride particles are selectively separated from the coated diamond or high-pressure phase boron nitride particles on a primary particle basis.

3. A process for producing a diamond or high-pressure phase boron nitride sinter according to claim 1 wherein the particles in the powder of diamond and/or high-pressure phase boron nitride core particles to be coated with the coat forming substance are coated by a dip method using a molten salt bath with one or more layers of the coat forming substance that derives from the dip method.

4. A process for producing a diamond or high-pressure phase boron nitride sinter according to claim 1, wherein the dispersing step is performed by:

a group of means for high dispersion treatment of particles that has such dispersing performance that a powder of core particles having an average diameter of no more than 10 μm in the frequency distribution by volume is dispersed in a gaseous atmosphere by the final treatment by said group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and that the dispersity β of the particles in said powder of core particles is adjusted to at least 70%;

a group of means for high dispersion treatment of particles that has such dispersing performance that a powder of core particles having an average diameter in excess of 10 μm but no more than 20 μm in the frequency distribution by volume is dispersed in a gaseous atmosphere by the final treatment by said group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and that the dispersity β of the particles in said powder of core particles is adjusted to at least 80%;

a group of means for high dispersion treatment of particles that has such dispersing performance that a powder of core particles having an average diameter in excess of 20 μm but no more than 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 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 30 μm but no more than 200 μm in the frequency distribution by volume is dispersed in a gaseous atmosphere by the final treatment by the group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and that the dispersity β of the particles in said powder of core particles is adjusted to at least 95%;

a group of means for high dispersion treatment of particles that has such dispersing performance that a

powder of core particles having an average diameter in excess of 300 μm but no more than 800 μm in the frequency distribution by volume is dispersed in a gaseous atmosphere by the final treatment by the group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and that the dispersity β of the particles in said powder of core particles is adjusted to at least 97%; and

a group of means for high dispersion treatment of particles that has such dispersing performance that a powder of core particles having an average diameter in excess of 800 μm in the frequency distribution by volume is dispersed in a gaseous atmosphere by the final treatment by the group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and that the dispersity β of the particles in said powder of core particles is adjusted to at least 99%; and

said coated diamond or high-pressure phase boron nitride particles being prepared by:

a method in which the mixture of a gas and the particles in the powder of highly dispersed core particles as formed by one of the means in said group of means for high dispersion treatment of particles is either discharged directly to the coating step or transported thereto via at least one member indispensable to transport that is selected from the group consisting of a hollow member, an intermediate member composed of a member that forms a hollow portion, and a pipe and which is provided between the dispersing and coating steps, as the mixture emerges from a discharge section that discharges said mixture of a gas and the particles; and/or

a method in which the mixture of a gas and the particles in the powder of highly dispersed core particles as formed by one of the means in said group of means for high dispersion treatment of particles is transported to the coating step via at least one means selected from the group consisting of means for maintaining the dispersion in a gaseous atmosphere of the highly dispersed core particles in the mixture of a gas with the core particles which have been dispersed in the gaseous atmosphere to attain one of the dispersities set forth above, means for enhancing the dispersion in a 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.

8. A process for producing a diamond or high-pressure phase boron nitride sinter according to claim 1 wherein the dispersing step is performed by:

a group of means for high dispersion treatment of particles that has such dispersing performance that a powder of core particles having an average diameter of no more than 10 μm in the frequency distribution by volume is dispersed in a gaseous atmosphere by the final treatment by said group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and that the dispersity β of the particles in said powder of core particles is adjusted to at least 70%;

a group of means for high dispersion treatment of particles that has such dispersing performance that a powder of core particles having an average diameter in excess of 10 μm but no more than 20 μm in the frequency distribution by volume is dispersed in a gaseous atmosphere by the final treatment by said group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and that the dispersity β of the particles in said powder of core particles is adjusted to at least 80%;

a group of means for high dispersion treatment of particles that has such dispersing performance that a powder of core particles having an average diameter in excess of 20 μm but no more than 50 μm in the frequency distribution by volume is dispersed in a gaseous atmosphere by the final treatment by the group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and that the dispersity β of the particles in said powder of core particles is adjusted to at least 90%;

a group of means for high dispersion treatment of particles that has such dispersing performance that a powder of core particles having an average diameter in excess of 50 μm but no more than 300 μm in the frequency distribution by volume is dispersed in a gaseous atmosphere by the final treatment by the group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and that the dispersity β of the particles in said powder of core particles is adjusted to at least 95%;

a group of means for high dispersion treatment of particles that has such dispersing performance that a powder of core particles having an average diameter in excess of 300 μm but no more than 800 μm in the frequency distribution by volume is dispersed in a gaseous atmosphere by the final treatment by the group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and that the dispersity β of the particles in said powder of core particles is adjusted to at least 97%; and

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.

8. A process for producing a diamond or high-pressure phase boron nitride sinter according to claim 1 wherein said coated diamond or high-pressure phase boron nitride particles are prepared with the coating start region of the coating space being located in either one of the following space regions that includes planes through which all particles in the powder of highly dispersed core particles as mixed with a gas will pass:

a space region in which a powder of core particles that have an average diameter of no more than 10 μm in the frequency distribution by volume is dispersed in a gaseous atmosphere by the final treatment by said group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and that the dispersity β of the particles in said powder of core particles is adjusted to at least 70%;

a space region in which a powder of core particles that have an average diameter in excess of 10 μm but no more than 20 μm in the frequency distribution by volume is dispersed in a gaseous atmosphere by the final treatment by said group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and that the dispersity β of the particles in said powder of core particles is adjusted to at least 80%;

a space region in which a powder of core particles that have an average diameter in excess of 20 μm but no more than 50 μm in the frequency distribution by volume is dispersed in a gaseous atmosphere by the final treatment by the group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and that the dispersity β of the particles in said powder of core particles is adjusted to at least 90%;

a space region in which a powder of core particles that have an average diameter in excess of 50 μm but no more than 300 μm in the frequency distribution by volume is dispersed in a gaseous atmosphere by the final treatment by the group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and that the dispersity β of the particles in said powder of core particles is adjusted to at least 95%;

a space region in which a powder of core particles that have an average diameter in excess of 300 μm but no more than 800 μm in the frequency distribution by volume is dispersed in a gaseous atmosphere by the final treatment by the group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and that the dispersity β of the particles in said powder of core particles is adjusted to at least 97%; and

a space region in which a powder of core particles that have an average diameter in excess of 800 μm in the frequency distribution by volume is dispersed in a gaseous atmosphere by the final treatment by the group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and that the dispersity β of the particles in said powder of core particles is adjusted to at least 99%, or wherein

said coated diamond or high-pressure phase boron nitride particles are prepared with the coating start region of the coating space being located in either one of the following space regions that includes planes through which all particles to be recovered into the recovery section of the recovery means will pass:

a space region in which a powder of core particles that have an average diameter of no more than 10 μm in the frequency distribution by volume is dispersed in a gaseous atmosphere by the final treatment by said group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and that the dispersity β of the particles in said powder of core particles is adjusted to at least 70%;

a space region in which a powder of core particles that have an average diameter in excess of 10 μm but no more than 20 μm in the frequency distribution by volume is dispersed in a gaseous atmosphere by the final treatment by said group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and that the dispersity β of the particles in said powder of core particles is adjusted to at least 80%;

a space region in which a powder of core particles that have an average diameter in excess of 20 μm but no more than 50 μm in the frequency distribution by volume is dispersed in a gaseous atmosphere by the final treatment by the group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles

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 500 μm in the frequency distribution by volume is dispersed in a gaseous atmosphere by the final treatment by the group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and that the dispersity β of the particles in said powder of core particles is adjusted to at least 95%;

a space region in which a powder of core particles that have an average diameter in excess of 300 μm but no more than 800 μm in the frequency distribution by volume is dispersed in a gaseous atmosphere by the final treatment by the group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and that the dispersity β of the particles in said powder of core particles is adjusted to at least 97%; and

a space region in which a powder of core particles that have an average diameter in excess of 800 μm in the frequency distribution by volume is dispersed in a gaseous atmosphere by the final treatment by the group of means for high dispersion treatment of particles, thereby forming a mixture of a gas and the particles in the powder of highly dispersed core particles and that the dispersity β of the particles in said powder of core particles is adjusted to at least 99%.

7. A process for producing a diamond or high-pressure phase boron nitride sinter according to claim 1 wherein the particles in the powder of core particles to be used have a size distribution of (D_{50}/3, 3D_{50}) ≥ 80%) in terms of frequency distribution by volume, where D_{50} represents the average particle diameter.

8. A diamond or high-pressure phase boron nitride sinter that is produced by either one of the processes for producing a diamond or high-pressure phase boron nitride sinter as recited in claims 1 - 7.

FIG.1

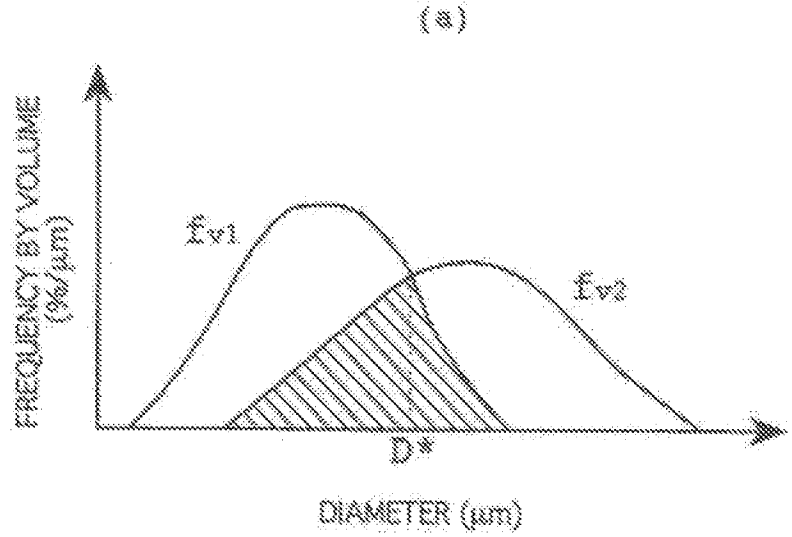
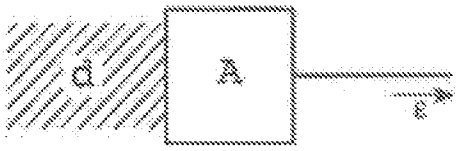
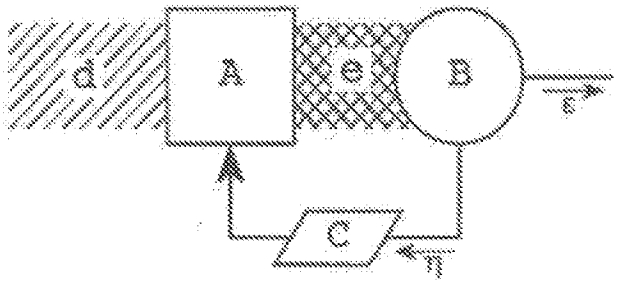

(a)
FREQUENCY BY VOLUME (%/μm)
f_{v1}
f_{v2}
D*
DIAMETER (μm)
(b)
FREQUENCY BY VOLUME (%/μm)
D_1
D_R
D_2
DIAMETER (μm)

FIG. 2

(a)



(b)



(c)

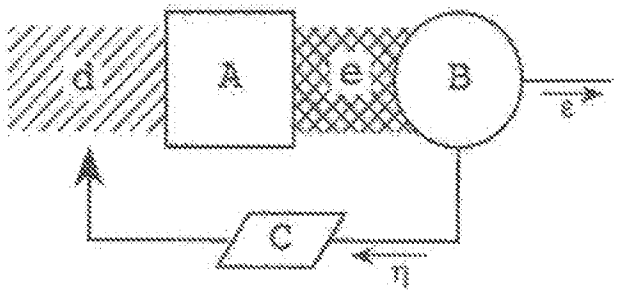


FIG. 3

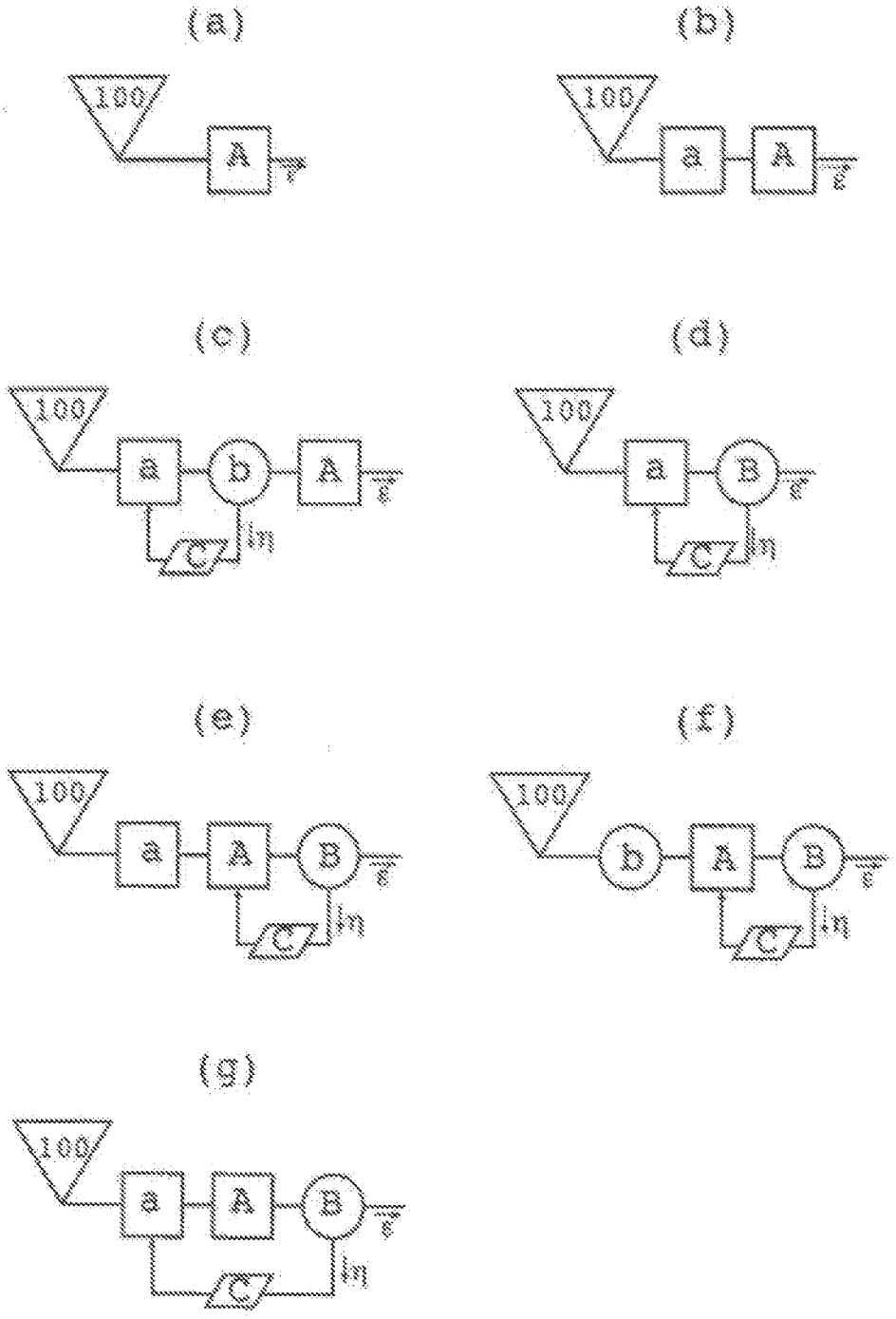

(a)
100
A
(b)
100
a
A
(c)
100
a
b
A
C
(d)
100
a
B
C
(e)
100
a
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C
(f)
100
b
A
B
C
(g)
100
a
A
B
C

FIG. 4

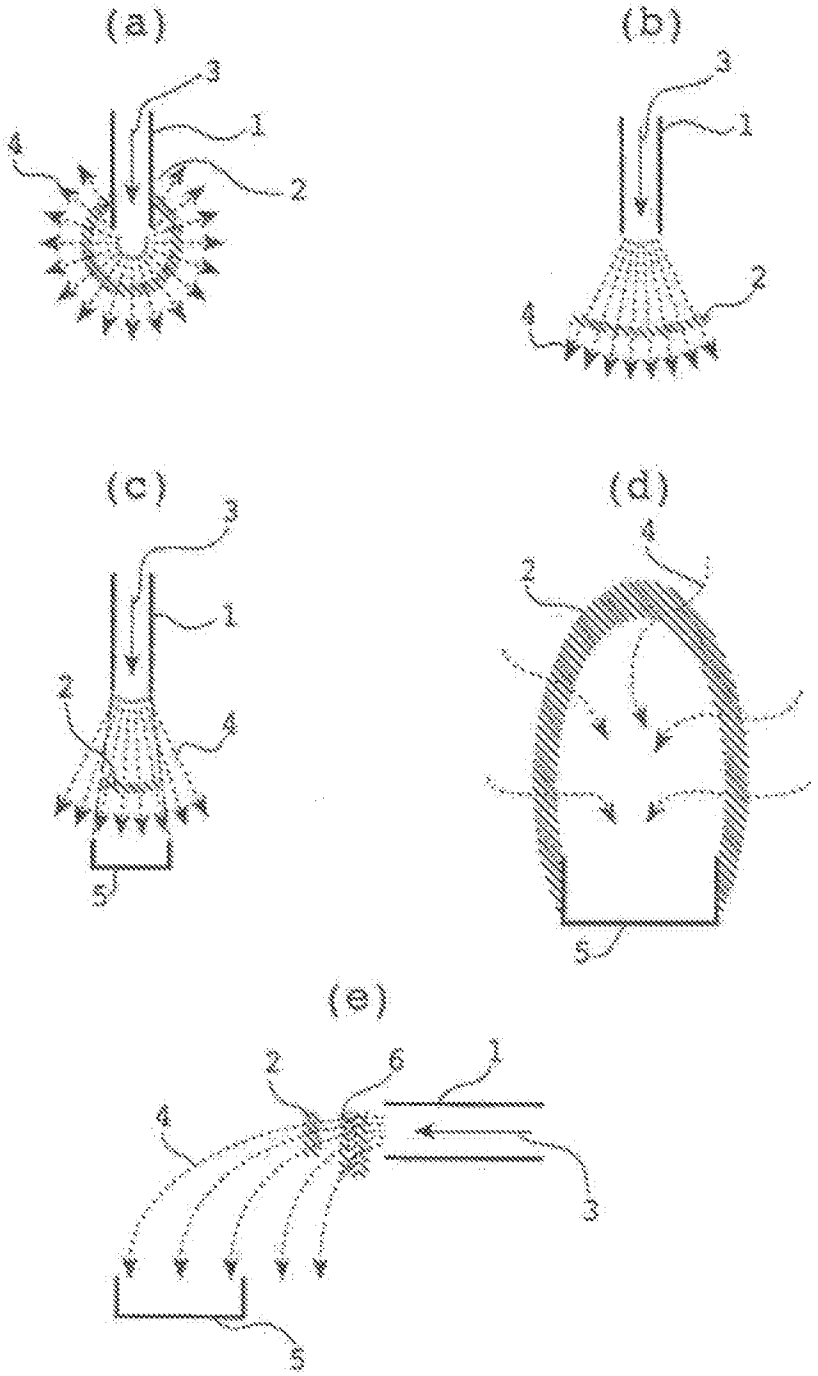
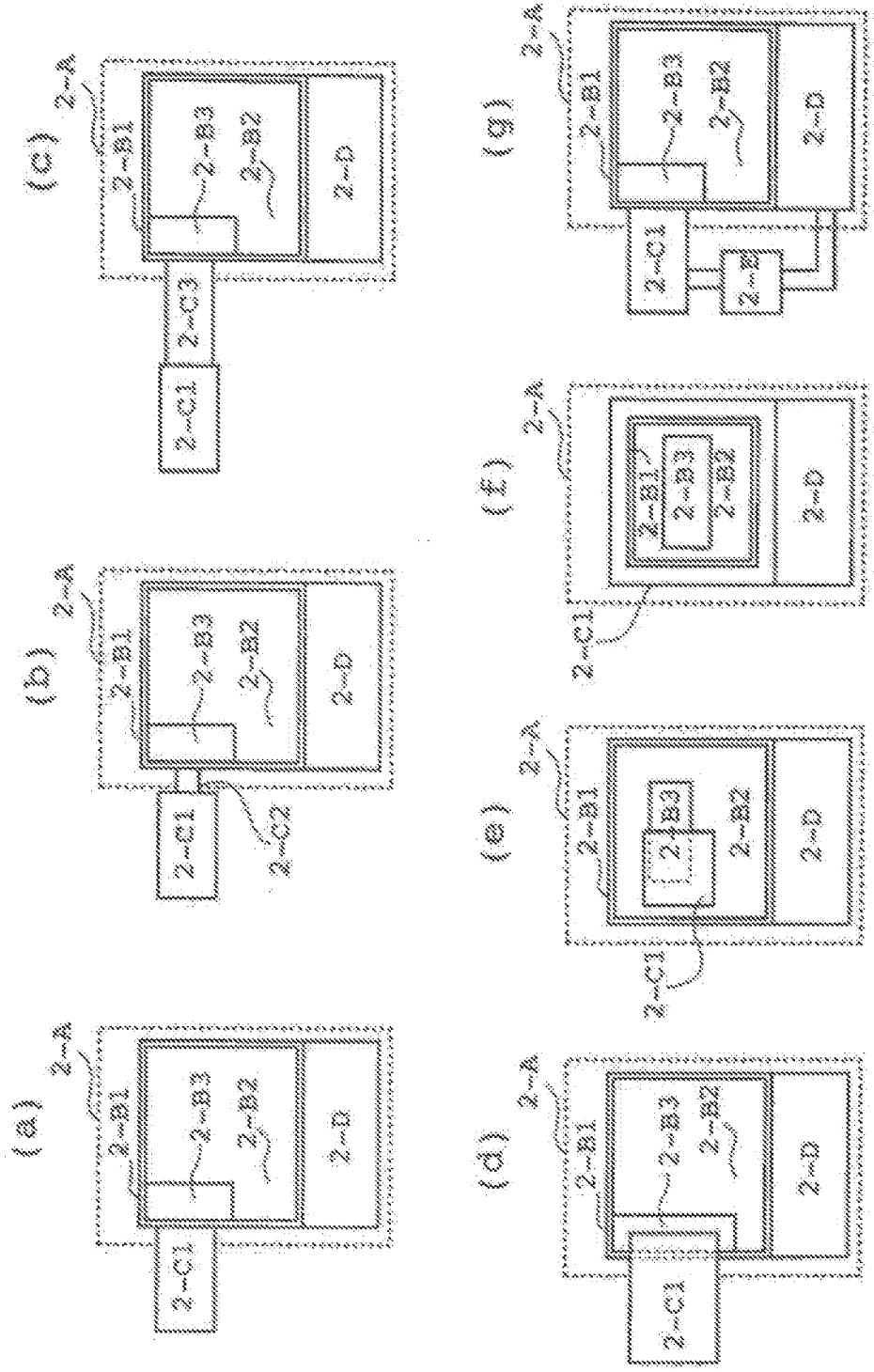


FIG. 5



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FIG. 6

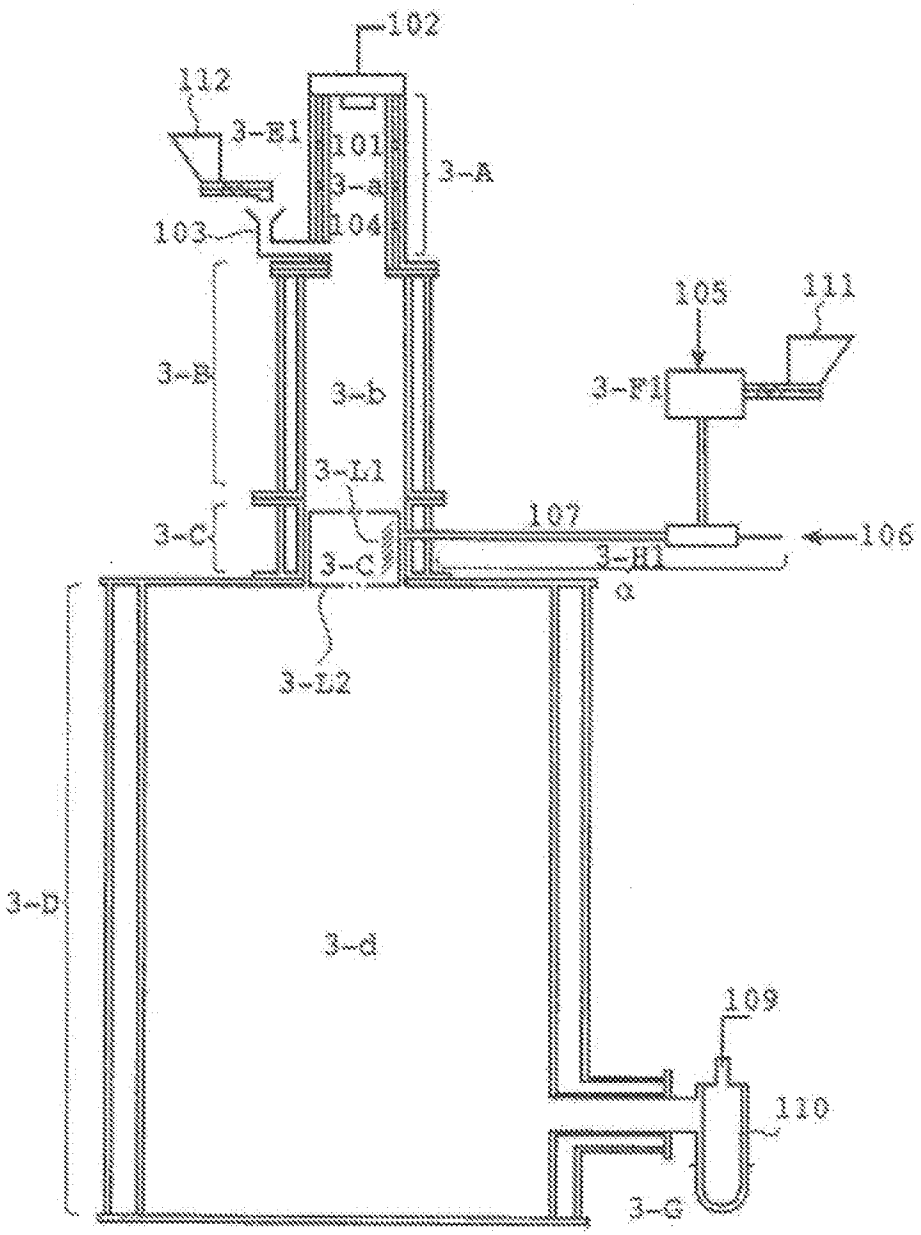

102
112
3-E1
101
3-a
104
3-A
103
105
111
3-B
3-b
3-F1
3-L1
107
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3-c
3-C
3-H1
Q
3-L2
3-D
3-d
109
110
3-G

FIG. 7

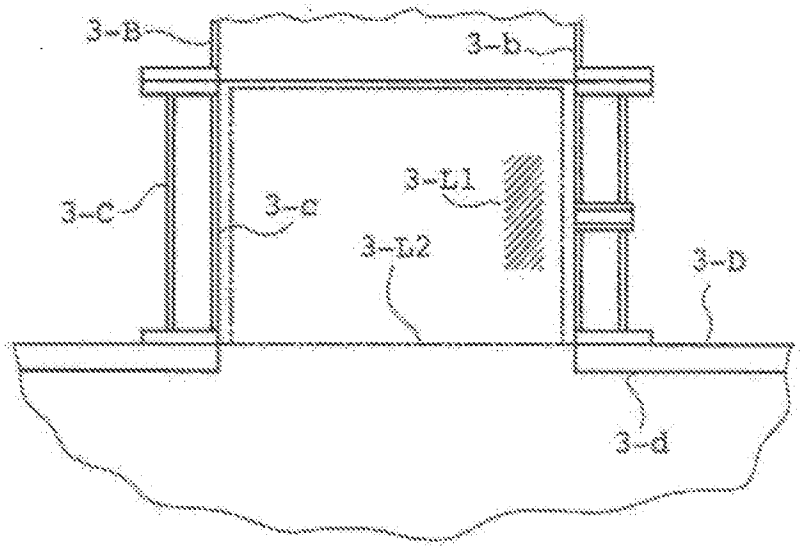


FIG. 8



FIG. 9

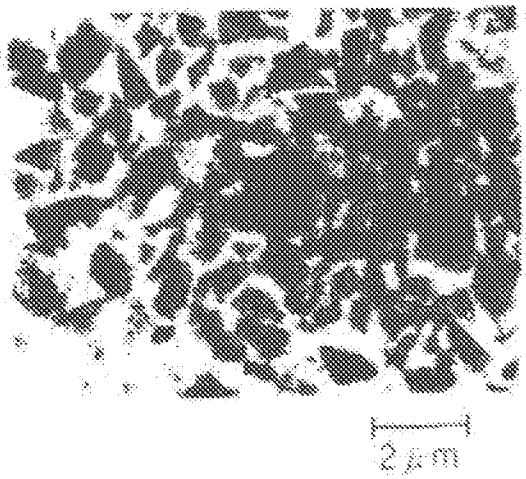


FIG. 10

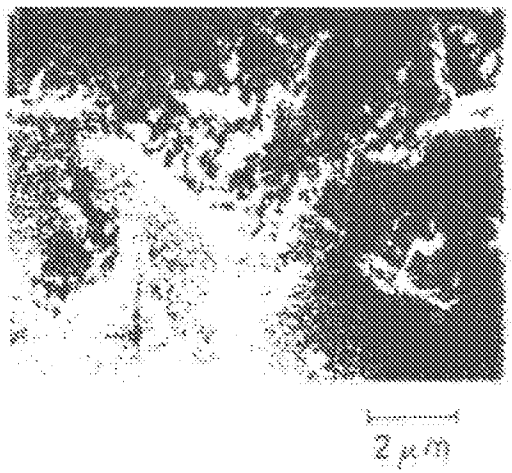


FIG. 11

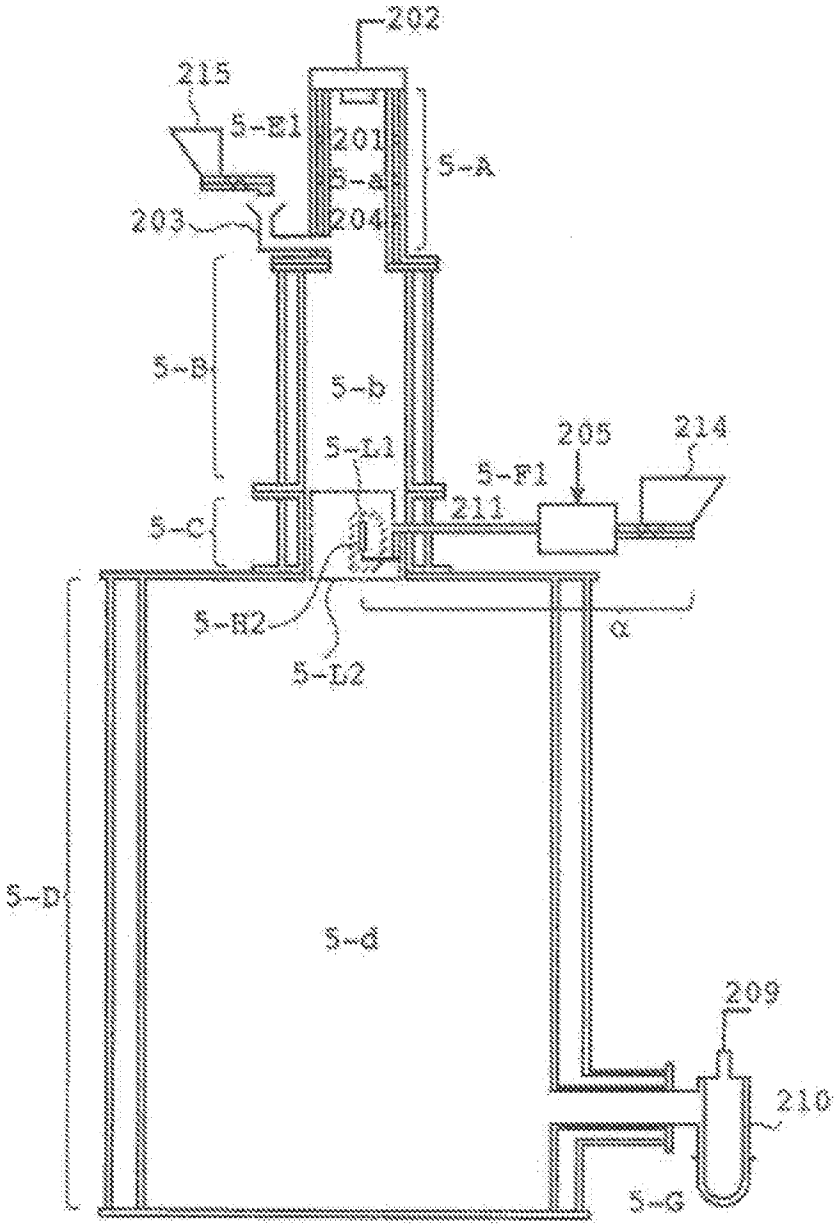

202
215
S-E1
201
S-A
203
204
S-B
S-b
205
214
S-F1
S-C
211
S-E2
S-E2
Q
S-D
S-d
209
210
S-G

FIG.12

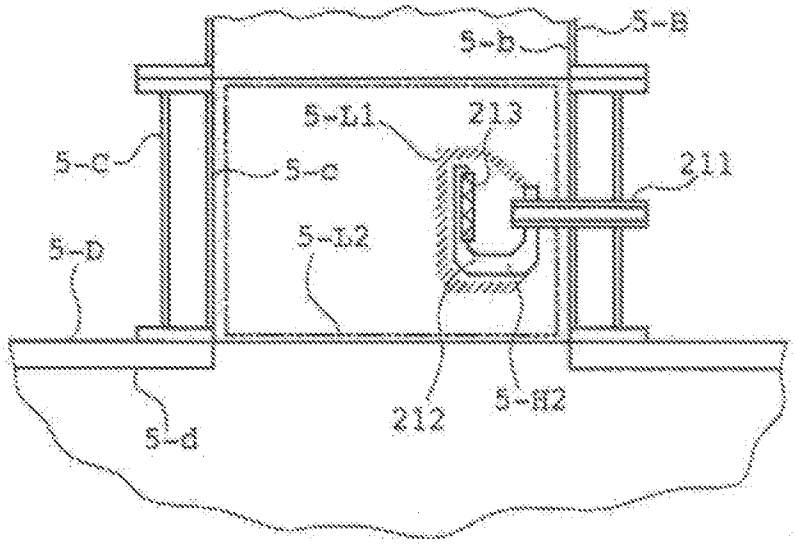


FIG. 13

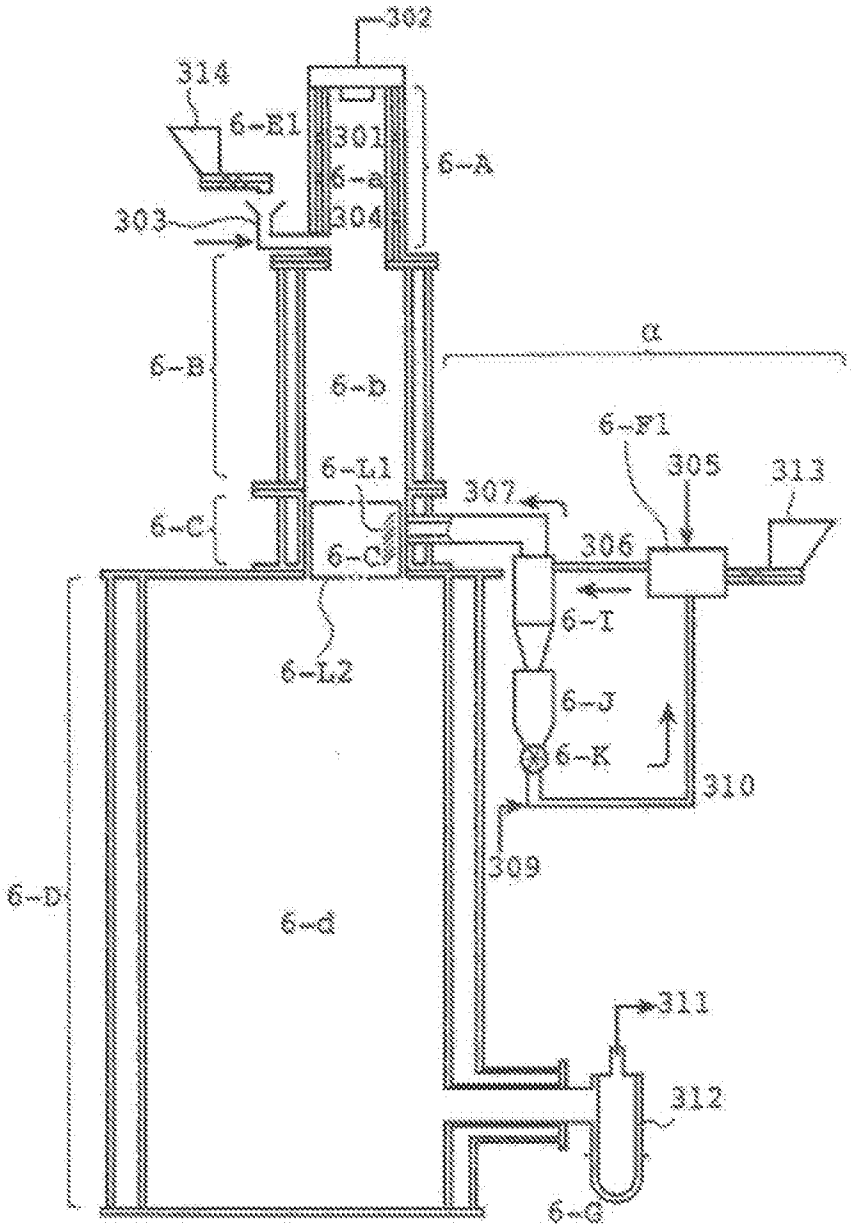

302
314
6-B1
301
6-A
303
304
α
6-B
6-b
6-F1
305
313
6-L1
307
6-C
6-c
306
6-I
6-L2
6-J
6-K
310
309
6-D
6-d
311
312
6-G

FIG. 14

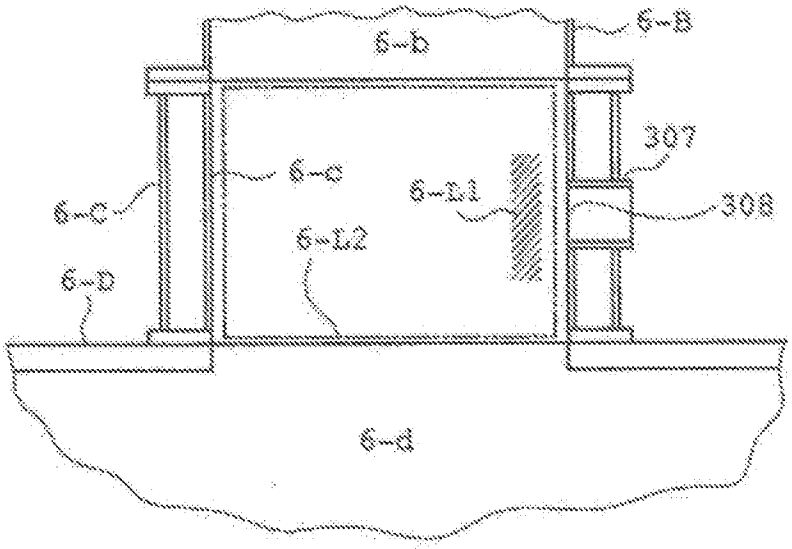


FIG. 15



FIG.16

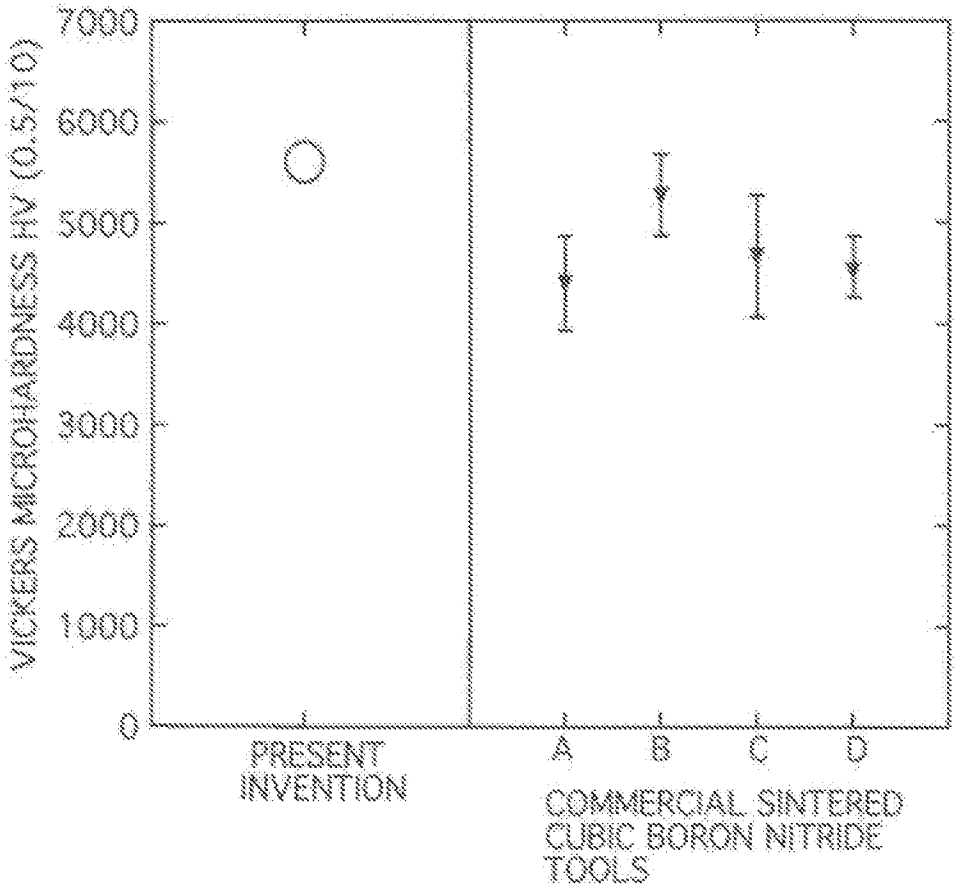

VICKERS MICROHARDNESS HV (0.5/10)
7000
6000
5000
4000
3000
2000
1000
0
PRESENT INVENTION
A
B
C
D
COMMERCIAL SINTERED CUBIC BORON NITRIDE TOOLS

FIG. 17

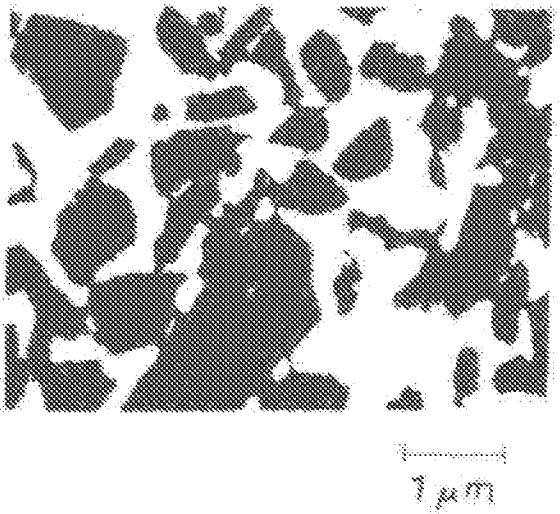


FIG. 18

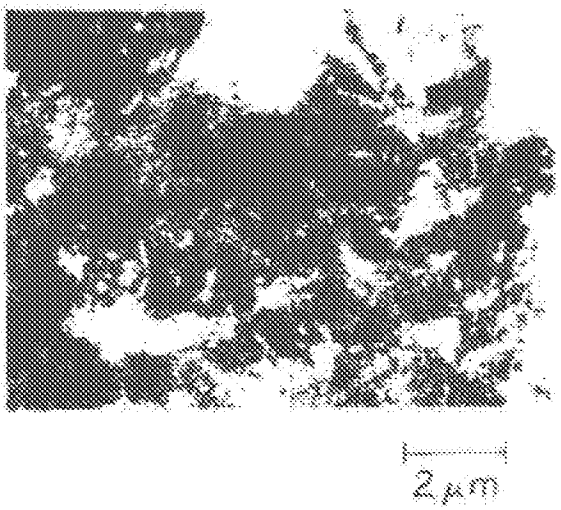


FIG.19



FIG.20

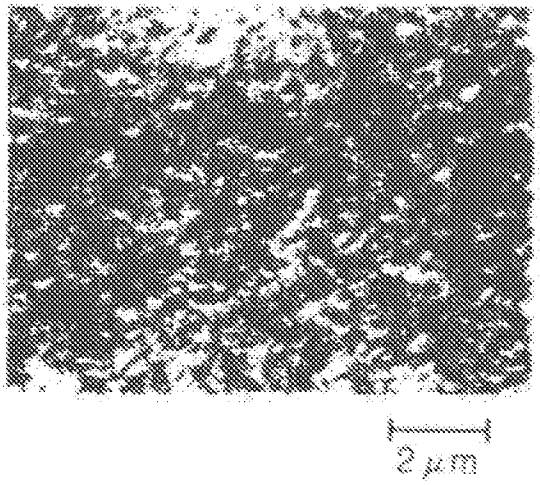


FIG.21

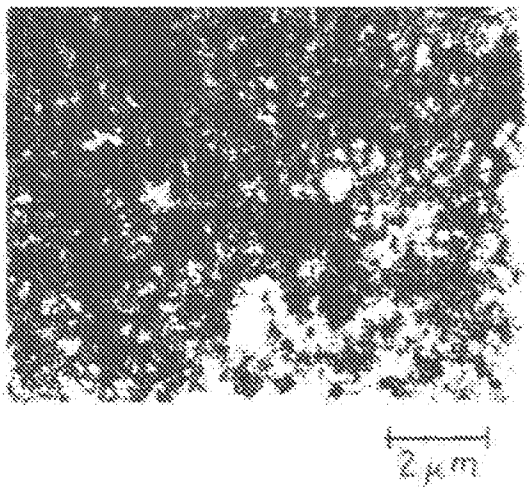


FIG. 22



FIG. 23

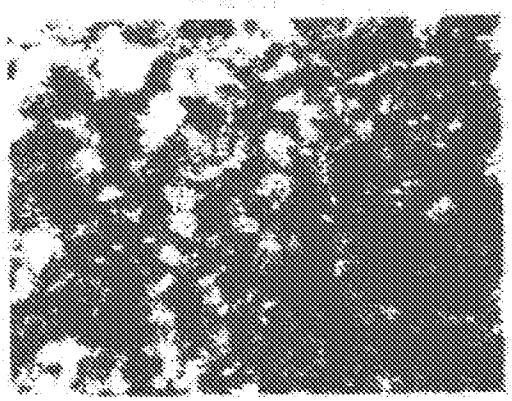


FIG. 24

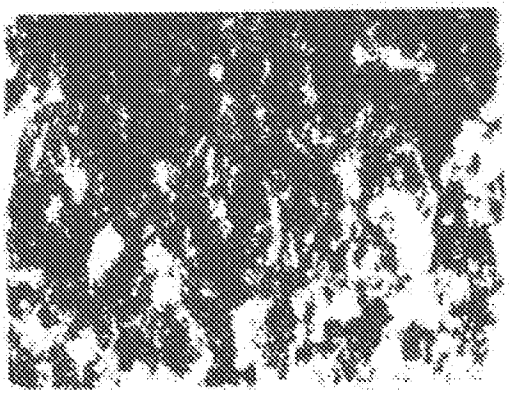


FIG.25

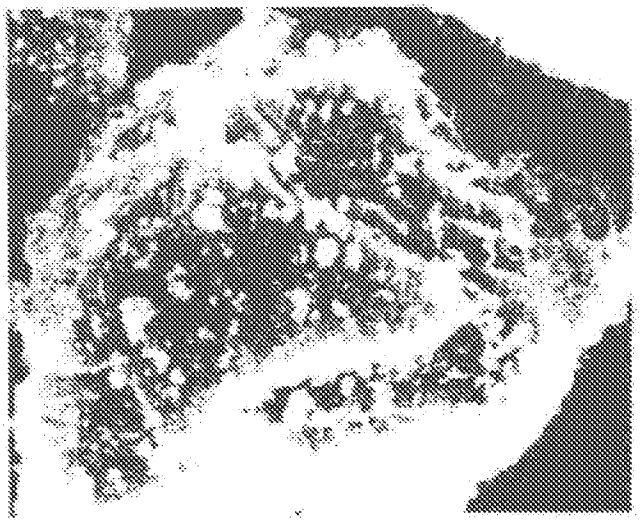


FIG.26

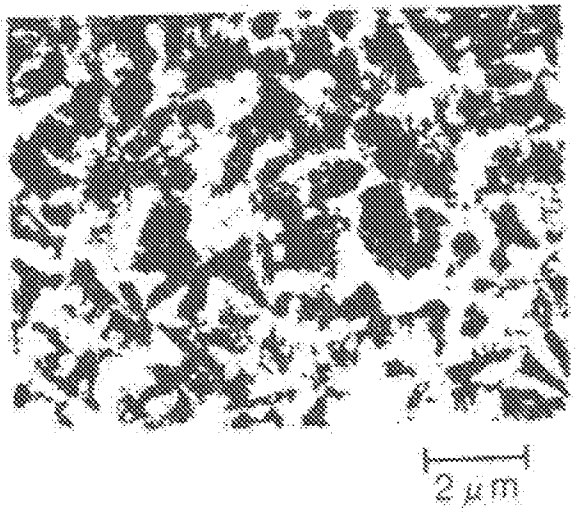


FIG.27



FIG.28

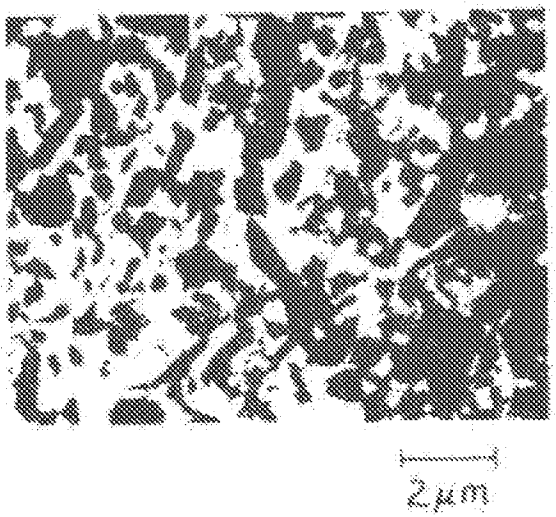


FIG. 29

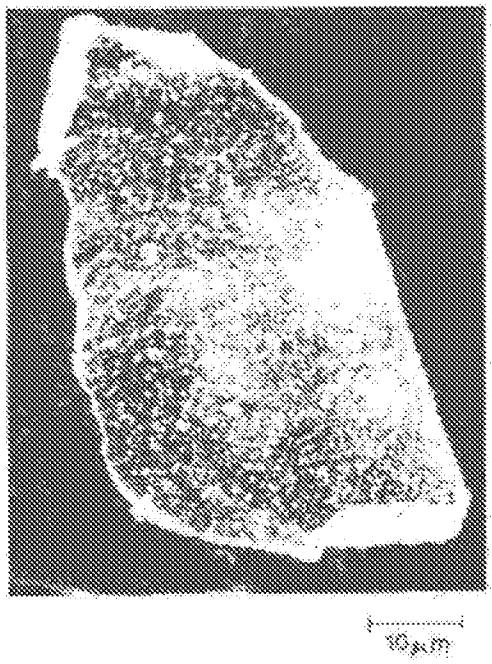


FIG. 30

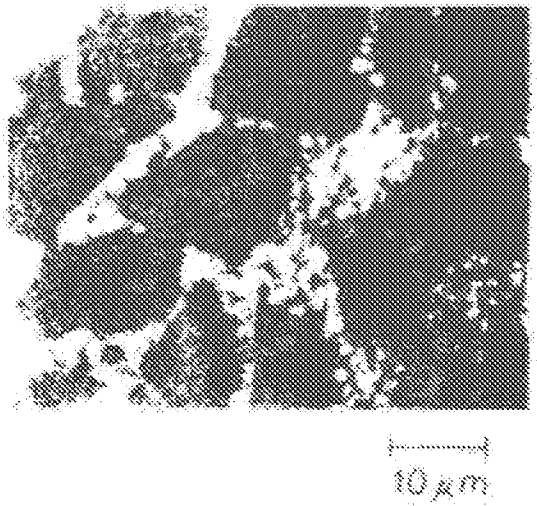


FIG. 31



FIG. 32

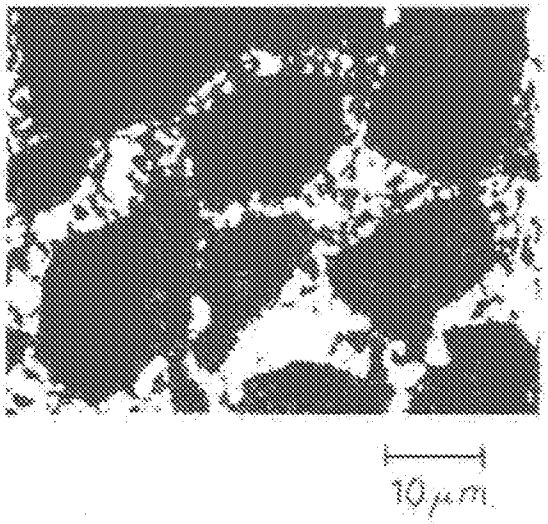


FIG.33a

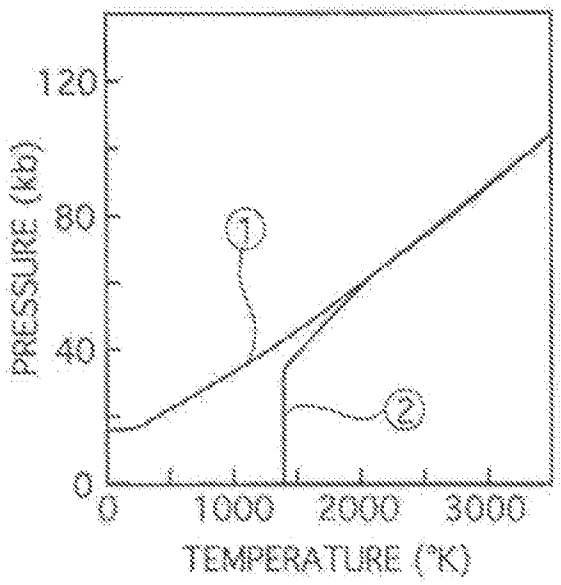
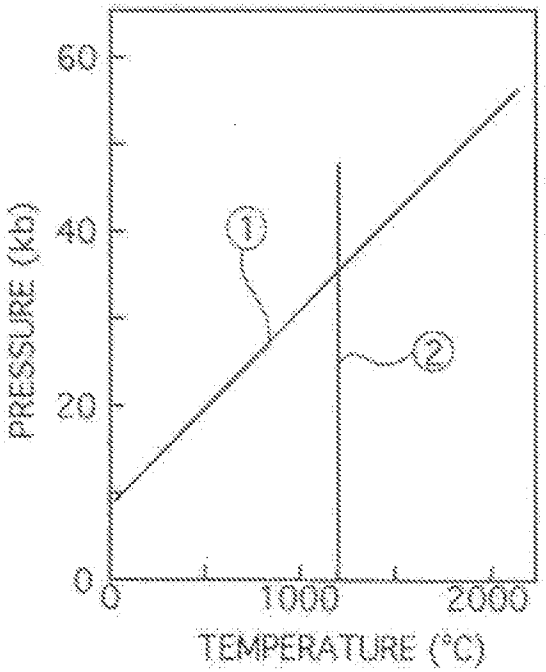


FIG.33b



European Patent Office

EUROPEAN SEARCH REPORT

Application Number
EP 94 11 8222

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int.Cl.6)
X	EP-A-0 273 573 (QA TECHNOLOGIES INC) 6 July 1988 * page 3, line 3 - line 29 *	1,2,4-8	C23C14/22
X	GB-A-2 214 195 (INCO LTD) 31 August 1989 * page 4, line 21 - page 5, line 31 *	1,2,4-8	
A,D	GB-A-2 242 443 (NISSHIN FLOUR MILLING CO) 2 October 1991 * example 4 *	1-8	
A,D	PATENT ABSTRACTS OF JAPAN vol. 014 no. 282 (C-0713) ,25 April 1990 & JP-A-02 083177 (SEIJI MOROOKA;OTHERS: 01) 13 February 1990, * abstract *	1-8	
			TECHNICAL FIELDS SEARCHED (Int.Cl.6) C23C

The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
THE HAGUE	26 April 1995	Schreurs, H

CATEGORY OF CITED DOCUMENTS

X : particularly relevant if taken alone
Y : particularly relevant if combined with another document of the same category
A : technological background
O : non-written disclosure
P : intermediate document

T : theory or principle underlying the invention
E : earlier patent document, but published on, or after the filing date
D : document cited in the application
L : document cited for other reasons

& : member of the same patent family, corresponding document

(12) UK Patent Application (19) GB (11) 2 239 011 (13) A

(43) Date of A publication 19.06.1991

(21) Application No 9026618.7

(22) Date of filing 06.12.1990

(30) Priority data
(31) 446468 (32) 11.12.1989 (33) US
538027 11.06.1990

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Jerome Johnson Thomas
Lawrence Rigio

(51) INT CL5
C01B 31/06

(52) UK CL (Edition K)
C1A A42

(56) Documents cited
EP 0288820 A

(58) Field of search
UK CL (Edition K) C1A A42
INT CL5 C01B
Online database: WPI; CLAIMS

(74) Agent and/or Address for Service
Paul M Turner
Urquhart - Dykes & Lord, 8 Lindsey Road, Poole, Dorset BH13 6AR, United Kingdom

(54) Single-crystal diamond of very high thermal conductivity

(57) Single-crystal diamond consisting of isotopically pure carbon-12 or carbon-13 has been found to have a thermal conductivity higher than that of any substance previously known, typically at least 40% higher than that of naturally occurring IIA diamond. It may be prepared by a method comprising comminution of diamond of high isotopic purity, such as that obtained by low pressure chemical vapor deposition employing an isotopically pure hydrocarbon in combination with hydrogen, followed by conversion of the comminuted diamond to single-crystal diamond by diffusion under high pressure through a metallic catalyst-solvent material to a region containing a diamond seed crystal.

GB 2 239 011 A

SINGLE-CRYSTAL DIAMOND OF VERY HIGH THERMAL CONDUCTIVITY

This invention relates to the preparation of single crystal diamond of extremely high thermal conductivity.

Diamond substances with high thermal conductivity are known in the art. For example, type IIA natural diamond, characterized by a very high degree of purity, has a thermal conductivity at 25°C (298°K) on the order of 21 watts/cm.°K, the highest of any material known prior to the present invention. Its electrical conductivity, on the other hand, is so low as to be negligible.

These properties make diamond an excellent material for conducting heat away from heat-generating objects or units. It may be used as a heat sink or as a conductor/spreader to conduct heat from the heat-generating object or unit to a heat sink of some other material.

Various areas exist in which heat conductors of very high thermal conductivity are necessary. One example is in repeating stations for fiber optic networks. Signals are transmitted by laser light over the fibers of such networks for very great distances. Since these signals decrease substantially in intensity over several kilometers, it is necessary to construct "repeating stations" periodically along the network, for the purpose of increasing the intensity of the light transmitted along the network. In a typical repeating station of this type, a photodetector is employed to convert the weakened signal transmitted by fiber optics to an electrical signal, which is then magnified, reconverted to a light signal by a light-emitting diode, and transmitted in turn along the next segment of the network.

In order to minimize the requisite number of repeating stations, optimum magnification of the signal in any station is desirable. However, the amount of radiant energy of any kind generated electrically is proportional to the fourth power of the current employed. While a portion of such radiant energy is in the form of light, the remainder thereof is lost as heat. In any individual station, therefore, very large amounts of heat are generated, requiring efficient heat conductors to maintain the operativeness of the repeating station.

The use of natural type IIA diamond as a conductor of heat in these areas has not been foreclosed despite its very high cost since the operative heat-conducting units are very small, typically about 1 mm. on a side. When larger heat-conducting elements are required, expense considerations become important and the use of natural diamond may accordingly be foreclosed.

Conventionally produced high-pressure synthetic diamond of gem quality is lower in cost than natural diamond. However, synthetic diamond of this type having high thermal conductivity cannot effectively be produced directly from graphite because a profound contraction in volume occurs in the conversion and introduces imperfections into the crystalline structure. For the most part, diamond prepared by low-pressure chemical vapor deposition (hereinafter sometimes "CVD") processes are not single crystal diamond and have substantially lower thermal conductivities, typically on the order of 12 watts/cm.°K at about 300°K (hereinafter sometimes "room temperature conductivity").

In U.S. Patent 3,895,313, there are disclosed various diamond materials which allegedly have very high thermal conductivities, and which allegedly are useful as optical elements for very high-power laser beams. In particular, it is stated that synthetic diamond grown from

isotopically pure carbon-12 or carbon-13 would be useful in this way, with room temperature conductivity values in the range of 10-20 watts/cm.°K being mentioned. This is, of course, the order of magnitude of natural type IIA diamond and also of diamond produced by chemical vapor deposition. It is further suggested that at a temperature of 70°K (-203°C), below the temperature of liquid nitrogen, a thermal conductivity exceeding 200 watts/cm.°K may be obtained, perhaps in diamond which has high isotopic purity and "relevant properties which closely approach the limits predicted by the theory of the perfect solid state" (i.e., are in single-crystal form). However, no methods for the preparation of such diamond are suggested, and the state of the art has hitherto been inadequate to put them in the possession of the public.

The present invention provides a method for preparing single-crystal diamond of very high chemical and isotopic purity. The raw material for such diamond is itself diamond, which eliminates the volume contraction encountered in the high-pressure conversion of graphite to diamond. The thermal conductivity of the diamond so produced has been found to be higher than that of any substance presently known including natural type IIA diamond, and also higher than the values in the aforementioned U.S. Patent 3,895,313. Accordingly, it is eminently suitable for use as a conductor of heat and also in numerous other areas.

In one of its aspects, the invention is a method for preparing single-crystal diamond of high isotopic purity which comprises the steps of:

(A) preparing diamond consisting of isotopically pure carbon-12 or carbon-13; and

(B) converting said diamond to single-crystal diamond by diffusion under high pressure through a metallic

catalyst-solvent material to a region containing a diamond seed crystal.

An essential feature of the method of this invention is the employment of isotopically pure carbon-12 or carbon-13. As explained hereinafter, it has been found that the increase in thermal conductivity resulting from the employment of chemically and isotopically pure carbon is vastly greater than would be expected based on theoretical considerations. In general, the isotopic purity of the hydrocarbon should be at least 99.2% by weight; that is, the other isotope should be present in a maximum amount of 8 parts per 1000. An isotopic purity of at least 99.9% by weight is preferred. The hydrocarbon should also have a high degree of chemical purity.

Various methods may be employed in step A for the preparation of diamond in isotopically pure form. For example, a gaseous carbon compound such as carbon monoxide may be separated into carbon-12 and carbon-13 species via differences in diffusivity and then converted to solid carbon by art-recognized means, such as combustion in a reducing flame in the case of carbon monoxide. The carbon thus formed may then be converted to diamond under conventional conditions, including high temperature and high pressure conditions or CVD conditions.

Alternatively, other methods may be employed including shock formation and CVD processes under conditions which produce a mixture of diamond and graphite. In processes of the latter type, the carbon-13 species will concentrate in the diamond phase and the carbon-12 species in the graphite phase. Other diamond precursors which may be employed in enriched form include pyrolytic graphite, amorphous or glassy carbon, liquid hydrocarbons and polymers.

It is usually found that conventional methods of CVD diamond formation are most convenient for the preparation of isotopically pure diamond. In such methods, a layer of diamond is deposited on at least one substrate. Any substrate material suitable for diamond deposition thereon may be employed; examples of such materials are boron, boron nitride, platinum, graphite, molybdenum, copper, aluminum nitride, silver, iron, nickel, silicon, alumina and silica, as well as combinations thereof. Metallic molybdenum substrates are particularly suitable under many conditions and are often preferred.

The method of chemical vapor deposition of diamond on a substrate is known, and the details need not be repeated herein. In brief, it requires high-energy activation of a mixture of hydrogen and a hydrocarbon, typically methane, whereupon the hydrogen gas is converted to atomic hydrogen which reacts with the hydrocarbon to form elemental carbon. Said carbon then deposits on the substrate in the form of diamond. Activation may be achieved by conventional means involving high-energy activation which produces atomic hydrogen from molecular hydrogen; such means include thermal means typically involving heated filaments, flame means, D.C. discharge means and radiation means involving microwave or radio-frequency radiation or the like.

Thermal and especially filament methods, employing one or more resistance heating units including heated wires or filaments, are often preferred for the purposes of this invention. In such methods, the filaments are typically of metallic tungsten, tantalum, molybdenum and rhenium; because of its relatively low cost and particular suitability, tungsten is often preferred. Filament diameters of about 0.2-1.0 mm. are typical, with about 0.8 mm. frequently being preferred. Distances from filaments to substrate(s) are generally on the order of 5-10 mm.

Said filaments are typically heated at temperatures of at least 2000°C and the optimum substrate temperature is in the range of 900-1000°C. The pressure in the deposition vessel is maintained up to about 760 torr, typically on the order of 10 torr. The hydrogen-hydrocarbon mixture generally contains hydrocarbon in an amount up to about 2% by volume based on total gases. For a description of illustrative CVD methods of diamond preparation, reference is made to copending, commonly owned applications U.S. Serial Nos. 07/389,210 and 07/389,212.

Isotopically pure hydrocarbon is employed in the CVD method, when used. In order to avoid contamination thereof, it is essential to employ equipment which does not contain natural carbon as an impurity. For this purpose, the CVD chamber should be constructed of materials substantially incapable of dissolving carbon. Typical materials of this type are quartz and copper.

As between carbon-12 and carbon-13, the former is usually preferred for various reasons. In the first place, carbon-12 is present in nature in much higher proportions than carbon-13, the latter typically occurring in amounts no higher than about 1% by weight; therefore, the use of carbon-12 involves minimum expense. In the second place, thermal conductivity is inversely proportional to the square of the mass number of the isotope, and diamond prepared from carbon-12 can therefore be expected to have a thermal conductivity about 17% greater than those prepared from carbon-13. In some applications, however, carbon-13 is preferred and its preparation and use are part of the invention.

The thickness of the CVD diamond layer deposited on the substrate is not critical. In general, it is convenient to deposit at least as much diamond as will be needed to produce a single crystal of the desired size. Of course, the

production of a larger amount of CVD diamond for use to make several crystals is also contemplated.

It is possible to convert the product of the CVD process directly to diamond of high thermal conductivity by high pressure means, as described hereinafter, employing the same in the form of a slab, sheet or broken pieces thereof. However, the method of this invention is most efficiently conducted if the isotopically pure diamond is first comminuted.

Comminution may be achieved by art-recognized means such as crushing and powdering. The particle size thereof is not critical so long as a sufficient degree of comminution is attained; the form known in the art as "grit diamond" is suitable.

Step B, the production of single crystal diamond, is conventional except that the isotopically pure diamond produced in step A is the raw material employed. Two things are achieved by using diamond rather than graphite or some other allotrope of carbon as the raw material: an easily obtained isotopically pure material may be employed, and the contraction in volume encountered in the conversion of graphite and other allotropes to diamond is avoided, permitting production of a single crystal of regular structure and high quality.

The process for producing single-crystal diamond under high pressure is also known in the art, and a detailed description thereof is not deemed necessary. Reference is made, for example, to Encyclopedia of Physical Science & Technology, vol. 6, pp. 492-506 (Academic Press, Inc., 1987); Strong, The Physics Teacher, January 1975, pp. 7-13; and U.S. Patents 4,073,380 and 4,082,185, for general descriptions of the process. It generally involves diffusion of the carbon employed as a source material through a liquid bath of a metallic catalyst-solvent material, at pressures on the order

of 50,000-60,000 atmospheres and temperatures in the range of about 1300-1500°C. A negative temperature gradient, typically of about 50°C, is preferably maintained between the material being converted and the deposition region, which contains a diamond seed on which crystal growth can begin.

Catalyst-solvent materials useful in step B are known in the art. They include, for example, iron; mixtures thereof with nickel, aluminum, nickel and cobalt, nickel and aluminum, and nickel, cobalt and aluminum; and mixtures of nickel and aluminum. Iron-aluminum mixtures are frequently preferred for the production of single-crystal diamond, with a material consisting of 95% (by weight) iron and 5% aluminum being particularly preferred for the purposes of the invention.

Following preparation of the single-crystal diamond by the method of this invention, it is often preferred to remove the portion attributable to the seed crystal by polishing. This is particularly true if the seed crystal is not isotopically pure.

Studies on single-crystal diamond prepared according to the invention and having various degrees of isotopic purity have shown that at carbon-12 purity levels of 99.2%, 99.5% and 99.9% by weight, the room temperature conductivities are, respectively, 10%, 25% and 60% greater than that of natural type IIA diamond. At lower temperatures, even higher differences in thermal conductivity are to be expected. Such thermal conductivities are higher than those of any material previously known. Single-crystal diamond of this type is another aspect of the invention, as is diamond prepared by the method described herein.

The reasons for the extremely high thermal conductivity of the single-crystal diamond of this invention are not fully understood. It is assumed, however, that the phenomenon is principally a function of the mean free path of

the phonons (i.e., lattice vibration modes) in the diamond crystals. Thermal conductivity is directly proportional to specific heat, sound velocity in and phonon mean free path of the crystal, and isotope effects on specific heat and sound velocity are negligible.

In a simplified calculation, the reciprocal of the phonon mean free path can be considered as equal to the sums of the reciprocals of the mean free paths attributable to phonon-phonon scattering and to isotope effects. It has been calculated that the isotope-related mean free path is 34,000 Angstroms and the phonon scattering-related path is 1900 Angstroms; consequently, the isotope effect would account for a decrease in mean free path on the order of only about 5.2%. One possible reason for the substantially greater observed isotope effect on thermal conductivity is that contrary to theory, the isotopic constitution of the diamond has a direct effect on the mean free path attributable to phonon-phonon scattering. This effect has apparently not been previously recognized.

By reason of the aforementioned high thermal conductivity value and essentially non-existent electrical conductivity, the isotopically pure single crystal diamond of the present invention is singularly useful as a conductor of heat from electronic devices and similar heat-generating sources. Articles comprising such a source in contact with said diamond as a thermal conductor are another aspect of the invention.

A further aspect is abrasive articles comprising said diamond. Such articles can be expected to have extraordinarily long life by reason of their ability to dissipate frictional heat generated during their use. Typical areas of application include abrasive grit, diamond compacts, wire drawing dies, saw blades, scribing tools, drills, tool sharpeners, and polishing tools for optical

items, stone and precious stones including diamonds and articles made therefrom.

Still another aspect is light-filtering articles comprising said diamond with a pinhole aperture therein. They are useful, for example, as spatial filters for laser beams and the like. Articles of this type fabricated from natural diamond are subject to radiation-induced damage, probably thermal in nature. The substantially higher thermal conductivity of the diamond of this invention may be expected to greatly minimize damage of this kind.

The invention is illustrated by an example in which a layer of CVD diamond was first deposited on a molybdenum substrate in a chamber constructed of quartz and copper, neither of which dissolves substantial amounts of carbon. The substrate was vertically disposed in a plane parallel to and 8-9 mm. distant from the plane of a tungsten filament about 0.8 mm. in diameter. The vessel was evacuated to a pressure of about 10 torr, the filament was heated to about 2000°C by passage of an electric current and a mixture of 98.5% (by volume) hydrogen and 1.5% methane was passed into the vessel. The methane employed was substantially impurity-free and 99.9% thereof contained the carbon-12 isotope. Upon removal and mass spectroscopic analysis of the diamond thus obtained, it was found that 99.91% of the carbon therein was carbon-12.

Thermal conductivity of the isotopically pure CVD diamond was measured by the mirage detection of thermal waves generated by a modulated argon-ion beam impinging on the diamond crystals, in accordance with a conventional method. The room temperature conductivity was shown to be about 12 watts/cm.°K. A control sample of similar CVD diamond prepared from methane having the naturally occurring isotope distribution (98.96% C-12, 1.04% C-13) had essentially the same conductivity.

The isotopically pure CVD diamond was crushed and powdered, and was used as a source of carbon for the growth of a single-crystal diamond under high pressure and high temperature conditions. Specifically, a conventional belt apparatus was employed at 52,000 atmospheres and 1400°C, employing a catalyst-solvent mixture of 95% (by weight) iron and 5% aluminum. A small (0.005 carat) single-crystal diamond seed of normal isotopic distribution was used to initiate growth, and a negative temperature gradient of about 50°C was maintained between the CVD diamond and the seed crystal. The process was continued until a single crystal of 0.95 carat had been produced. It was shown by analysis that 99.93% of the carbon therein was the C-12 isotope.

The diamond was polished on a standard diamond scaife to remove the seed crystal, and its room temperature conductivity was compared with those of several other materials, including a control single-crystal diamond prepared from the CVD diamond with normal isotope distribution. The results were as follows, all values being in watts/cm.°K:

Isotopically pure C-12 diamond (this invention)	31.8
Control	21.18
Naturally occurring type IIA diamond	21.2
CVD diamond	12.0
Cubic boron nitride	7.8
Silicon carbide	4.9
Copper	4.0
Beryllium oxide	3.7
Boron phosphide	3.6
Aluminum nitride	3.2
Silicon	1.6
Aluminum oxide	0.8

Thus, the room temperature conductivity of the diamond of this invention is 48.7% greater than that of the control. It is also much higher than the room temperature conductivity of any other diamond or non-diamond material which has been measured.

At 70°K, below the boiling point of liquid nitrogen, the diamond of the present invention is theoretically predicted to have a conductivity of about 2675 watts/cm.°K, more than 13 times the minimum value predicted in the aforementioned U.S. Patent 3,895,313.

CLAIMS

1. A method for preparing single-crystal diamond of high isotopic purity which comprises the steps of:

(A) preparing diamond consisting of isotopically pure carbon-12 or carbon-13; and

(B) converting said diamond to single-crystal diamond by diffusion under high pressure through a metallic catalyst-solvent material to a region containing a diamond seed crystal.

2. A method as claimed in claim 1 wherein the carbon in the hydrocarbon is carbon-12.

3. A method as claimed in claim 1 or claim 2 wherein the product of step A is comminuted.

4. A method as claimed in any of the preceding claims wherein the carbon in the hydrocarbon is at least 99.2% isotopically pure.

5. A method as claimed in any of the preceding claims wherein the diamond of step A is prepared by chemical vapor deposition.

6. A method as claimed in any of the preceding claims wherein the equipment employed in step A is constructed of materials substantially incapable of dissolving carbon.

7. A method as claimed in claim 4 wherein the particle size achieved by comminution is that of grit diamond.

8. A method as claimed in claim 7 or claim 6 wherein step A is conducted at a filament temperature of at least 2000°C, a substrate temperature in the range of 900-1000°C and a pressure of about 10 torr.

9. A method as claimed in claim 7 or claim 8 wherein the filaments are of tungsten and the substrate is molybdenum.

10. A method as claimed in claim 6 wherein the carbon in the hydrocarbon is at least 99.9% isotopically pure.

11. A method asclaimed in claim 10 wherein the catalyst-solvent material employed in step B is an iron-aluminum mixture.

12. A method as claimed in claim 11 wherein a negative temperature gradient is maintained in step B between the material being converted and the deposition region.

13. A method as claimed in claim 12 wherein the catalyst-solvent material employed in step B is a mixture of 95% iron and 5% aluminum by weight.

14. A method as claimed in claim 13 wherein the temperature gradient is about 50°C.

15. A method as claimed in claim 12 wherein the pressure in step B is in the range of 50,000-60,000

atmospheres and the temperature is in the range of 1300-1500°C.

16. A method as claimed in claim 13 wherein the seed crystal is single-crystal diamond of normal isotopic distribution.

17. A method as claimed in claim 16 wherein the portion of the product diamond attributable to the seed crystal is removed by polishing.

18. Single-crystal diamond consisting of at least 99.2% by weight isotopically pure carbon-12 or carbon-13, said diamond having a thermal conductivity at 300°K at least 10% greater than that of natural type IIA diamond.

19. Diamond as claimed in claim 18 which consists of carbon-12.

20. An article comprising a heat-generating source in contact with the diamond of claim 18 as a thermal conductor.

21. An abrasive article comprising the diamond of claim 18.

22. A light-filtering article comprising the diamond of claim 18 with a pinhole aperture therein.

23. Single-crystal diamond consisting of at least 99.5% by weight isotopically pure carbon-12 or carbon-13, said diamond having a thermal conductivity at 300°K at

least 25% greater than that of natural type IIA diamond.

26. Single-crystal diamond consisting of at least 99.9% by weight isotopically pure carbon-12 or carbon-13, said diamond having a thermal conductivity at 300°K at least 40% greater than that of natural type IIA diamond.

29. Single-crystal diamond prepared by the method of claim 1.

28. Single-crystal diamond prepared by the method of claim 2.

27. Single-crystal diamond prepared by the method of claim 4.

28. Single-crystal diamond prepared by the method of claim 10.

29. Single-crystal diamond prepared by the method of claim 17.

30. A method as claimed in claim 1 substantially as hereinbefore described in any one of the examples.

31. A single crystal diamond as claimed in claim 18 substantially as hereinbefore described in any one of the examples.

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B3D DMN
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U1S S1578 S1645 S1652 S1660

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JP580090465 A & Japio abstract of JP580090465 A

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UK CL (Edition R) **B3D DEN DMN , B5A AB18 AB19 ANA ATXP AT3P , C7D DB702 DB704 DB706 DB708 , C7F FHB FHE FHX**
INT CL[7] **B22F 7/04 , B24B 53/12 , B29C 33/42 39/02 39/10 41/08 41/20 45/37 , B29D 31/00 , C23C 16/00 16/27 16/44**
Online: WPI, EPODOC, JAPIO

(54) Abstract Title
Cast diamond tools and their formation by chemical vapor deposition; diamond hoses

(57) A method and design of cast diamond tools includes a mold that contains a negative body shape and surface finish of an intended diamond tool. Diamond deposited by a vapor phase, such as by chemical vapor deposition, is applied to the mold. After depositing diamond to a certain thickness, the mold is dissolved in acid or another suitable solvent, leaving behind the diamond cast that duplicates the intended body shape and surface finish. The diamond cast is then used to fabricate suitable tools, such as a cutting insert, or a wire-drawing die. In addition pad conditioners for polishing silicon wafers may be manufactured using this process, where the soluble mold is formed of silicon. In a variation a diamond hose is formed by overcoating a twisted wire with diamond after which the central metal core is dissolved leaving a hollow diamond hose which is then cast in an epoxy resin.

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FIG. 1a

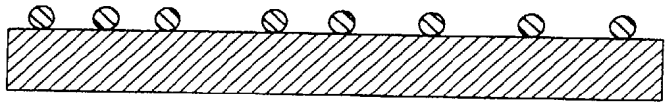


FIG. 1b

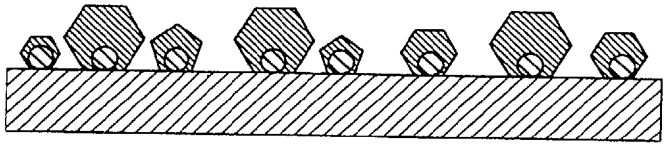
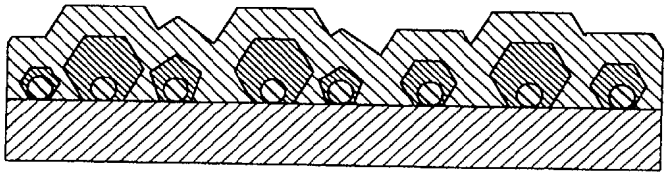


FIG. 1c



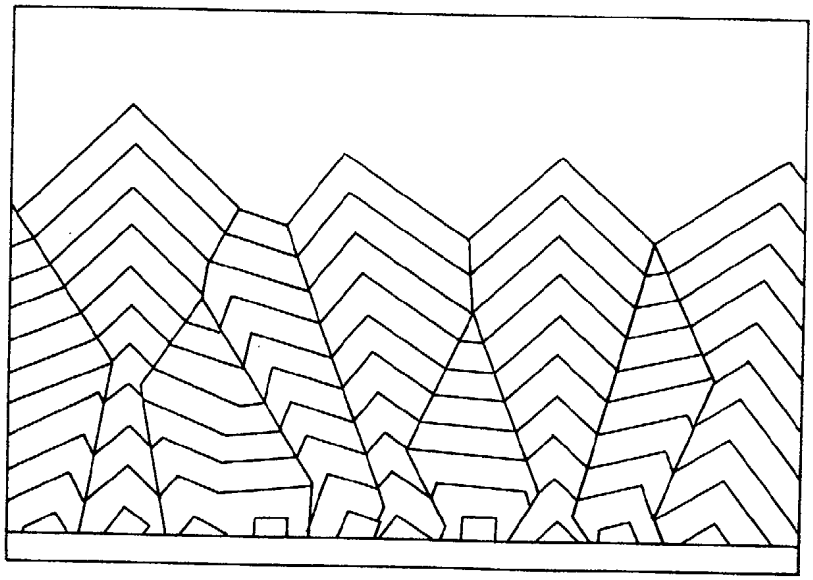


FIG. 1d

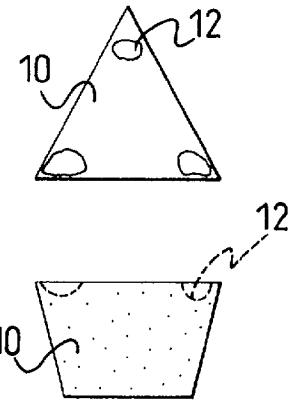


FIG. 2
PRIOR ART

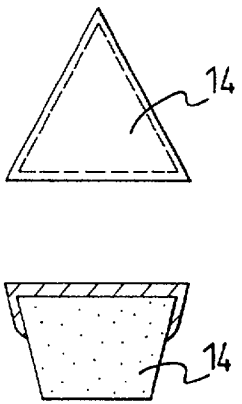


FIG. 3
PRIOR ART

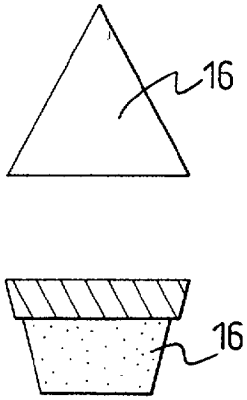


FIG. 4
PRIOR ART

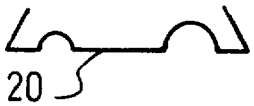


FIG. 5

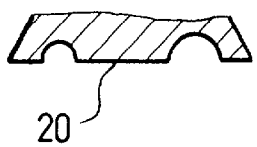


FIG. 6

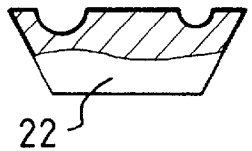


FIG. 7

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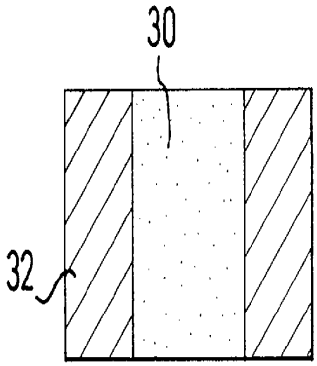


FIG. 8
PRIOR ART

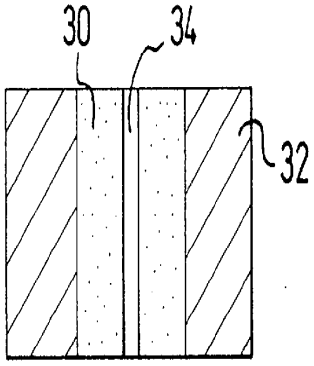


FIG. 9
PRIOR ART

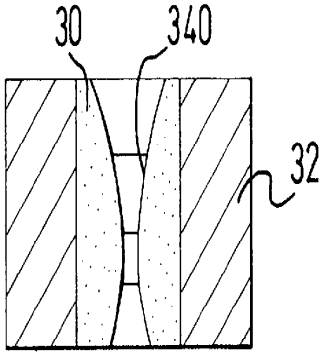


FIG.10
PRIOR ART

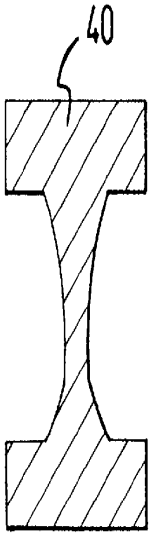


FIG.11

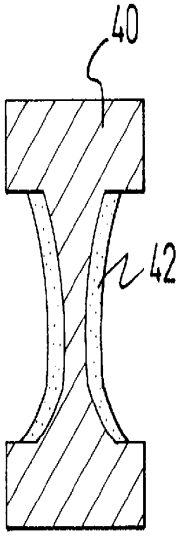


FIG.12

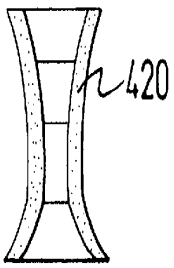


FIG.13

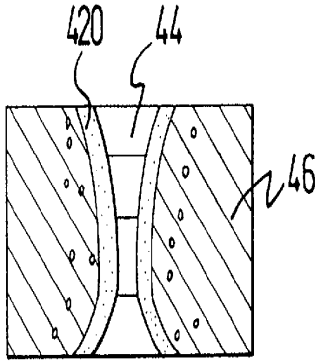


FIG.14

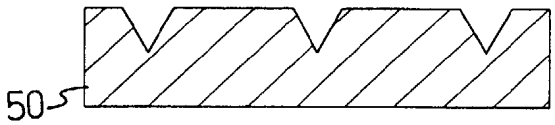


FIG. 15

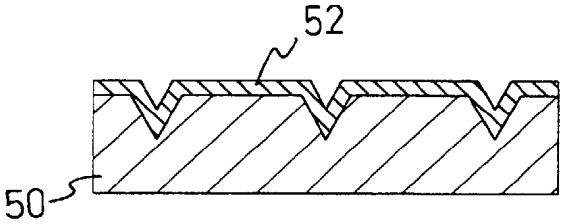


FIG. 16

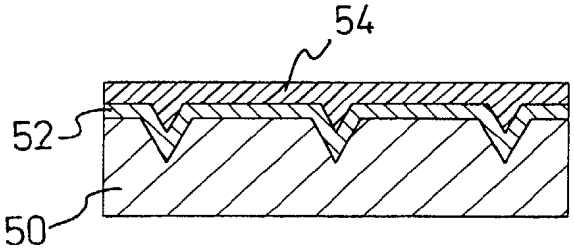


FIG. 17

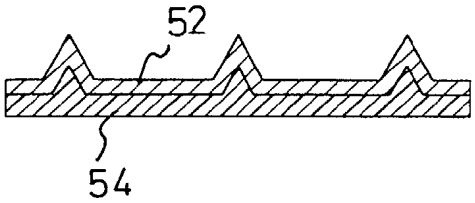


FIG. 18

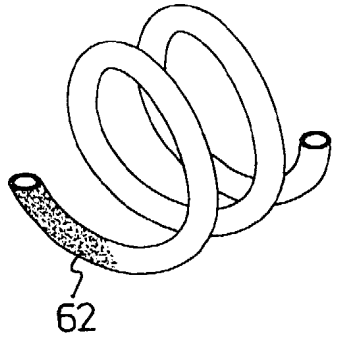


FIG. 21

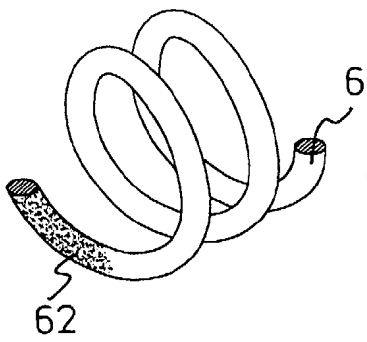


FIG. 20

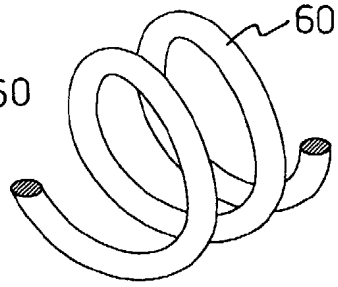


FIG. 19

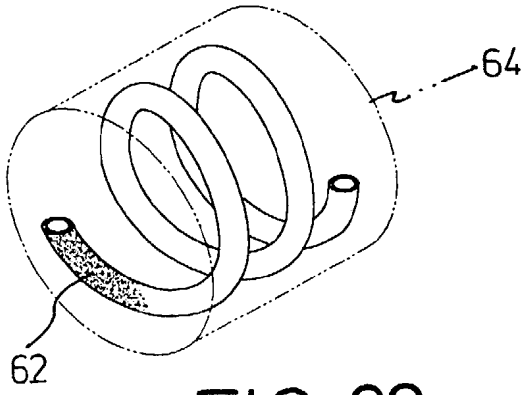


FIG. 22

CAST DIAMOND TOOLS AND THEIR FORMATION BY CHEMICAL VAPOR DEPOSITION

1. Field of the Invention

The present invention relates to a manufacturing method and design of cast diamond tools.

2. Description of Related Art

As a material, diamond has many extreme properties, such as high wear resistance, high thermal conductivity, rapid transmission of sound waves, and corrosion inertness. These superior properties have made diamond the ideal material for many advanced applications. For example, diamonds have been used widely as superabrasives, heat spreaders, acoustic devices, and chemical barriers. Conventionally, diamonds are synthesized under high pressure (more than fifty times atmospheric pressure), but such diamonds are typically in grit form. Although they are ideal for superabrasives (e.g., for diamond saws), they are not suitable for non-mechanical applications (e.g., heat spreaders).

In recent years, diamond films up to 1mm in thickness have also been deposited commercially. These films are deposited by CVD methods using gas reactants. The gases included typically a small amount (<5%) carbonaceous material, e.g., methane (CH4), that is diluted in an ample amount of

hydrogen (H2). During the process, the gases are heated to a high temperature so the carbonaceous gas will decompose to free up carbon atoms. At the same time, hydrogen molecules will dissociate to form atoms. Normally, carbon atoms will deposit as either amorphous carbon or graphite, but with the surrounding of hydrogen atoms, they will maintain the diamond structure (sp3 bond), as in the case of methane, and precipitate out as diamond. Even with the formation of non-diamond carbon, the ready presence of hydrogen will convert the carbon back to methane. Hence, hydrogen atoms play a key role in catalyzing the formation of diamond. Hence, the higher the concentration of hydrogen atoms, the better the quality of diamond that is formed.

There are various ways to heat the gas mixture, among them hot filament (e.g., using tungsten), microwave agitation, oxyacetylene flame, and direct current (DC) arc are commonly used. Although the temperature for diamond deposition is typically in the range of 800 ℃ to 900 ℃, the reaction temperature for the gases is much higher. In fact, the higher the reaction temperature is, the more complete the decomposition of the gases to form carbon and hydrogen atoms, and the faster the deposition rate of the diamond. Thus, the hot filament method can only reach 2000 ℃ or slightly over, so its

deposition is the slowest (about 1 micron per hour). The microwave agitation can heat the gases to a higher temperature, so the deposition rate is intermediate (about 10 microns per hour). The oxyacetylene can attain an even higher plasma temperature, so its deposition rate is quite high (e.g., 30 microns per hour). The DC arc method can surpass the gases to the highest temperature (about 6000 ℃) and hence it can deposit diamond at the highest rate (e.g., 50 microns per hour) of the above methods. However, the higher the deposition rate, the smaller the area of the deposition, and the less uniform the diamond film produced. Hence, there is a tradeoff between deposition rate and deposition area.

CVD diamond films contain polycrystalline diamond grains. Diamond nuclei are first formed on the surface of the substrate, and these nuclei will grow with ever increasing coarseness, as shown in Figs. 1a to 1d. Hence, the film on the nucleation side tends to show the same surface finish as the substrate material, but the growing side becomes ever-increasingly rough with the coarsening of the grain. For most applications (e.g., heat spreader), the rough surface must be ground and polished. However, as diamond is the hardest material known, the machining of the coarse diamond is extremely difficult and tedious. As a result, the cost associated with the finishing of a

diamond film is often higher than that for its deposition.Fig. 1 shows the nucleation and growth of a diamond film. Note the nucleation surface is a negative of the substrate surface, and the growing surface is becoming more and more rough with the deposition time.

Moreover, certain machining procedures, such as to cut a curved groove on the diamond surface, or to drill a square hole through diamond are almost impossible to achieve. Such limitations have severely constrained diamond films' potential applications.

For example, CVD diamond films have been used as a premium cutting insert. When it is used to cut ductile material (e.g., copper alloy), the long cutting thread must be interrupted from time to time. This is conveniently done by providing a trough near the cutting edge, as commonly done with other cutting tools, to bend and break the material being removed during the thread cutting operation. However, it is not possible to make such a trough on the flat diamond film. Hence, CVD diamond cutters, although extremely durable, are not suitable to cut ductile materials.

As another example, many advanced applications, e.g., circuitry in the electronics industry, would require using triangular, square, or irregularly-shaped shaped metal wires.

These wires cannot be drawn with conventional diamond wiredrawing dies that contain only circular holes. Hence, there is no suitable product to serve this significant demand from the market.

Furthermore, most applications use the surface (e.g., an extruding die) of diamond films, hence the property of the bulk body may not be critical. However, the conventional CVD method must produce the diamond film from the bottom up. Hence, if the top surface is required, the entire film must be grown slowly to reach the top portion. Such a tedious growth process can be very costly.

Example 1- Cutting Inserts Made of Diamond

Conventional diamond cutting inserts include two main types. The first type is to coat a WC insert with thin diamond film (e.g., 30 microns). The problem of this design is that the deposited diamond surface is rough, so the cutting surface does not have a fine finish. Alternatively, a thick diamond film (e.g., 300 microns) can be grown. The diamond film can then be ground and polished by a laser to form a triangular cutting tip. This tip is then brazed onto a WC insert. This method is tedious and expensive. In addition to the above described drawbacks, these diamond film cutting inserts suffer another deficiency which is that their top faces are always flat. Hence,

they do not possess chip breakers for efficiently cutting ductile materials.

Fig. 2 shows a conventional WC insert (10) with a depressed chip breaker (12) near each cutting corner. Fig. 3 shows a diamond coated WC insert (14) without a chip breaker. Fig. 4 illustrates a thick diamond brazed onto a WC insert (16), again without a chip breaker.

Example 2 - Wire Drawing Dies Made of Diamond

Many metal wires are drawn by using polycrystalline diamond (PCD) tools. The PCD (30) is enclosed by WC (32) (Fig. 8), and a hole (34) is drilled through the PCD (30) (Fig. 9), typically by a laser. The hole (34) is then enlarged to form a flare (340), as shown in Fig. 10, and the surface is polished by using fine diamond paste. Although there is much laborious finishing work involved, such PCD dies are still the best tools used for drawing metal wires. Even so, PCD dies contain only round holes, hence, it is not possible to draw polygonal wires, such as those with square cross-sections.

All PCDs contain more than 10% cobalt. As cobalt can reconvert diamond to amorphous carbon or graphite, so the PCD must be kept below a temperature of about 700 ℃. Hence, the lubrication and cooling by water or other liquid is critical.

However, during the drawing process, the metal wire tends to press against the PCD and it may weld with cobalt embedded in the PCD. As a result, excessive heating is inevitable and the PCD may deteriorate rapidly due to the reconversion to amorphopus carbon or graphite. Moreover, the dragging may damage the surface of the metal wire by causing weld spots and burn marks.

Example 3 - Pad Conditioners Made of Diamond

Chemical Mechanical Polishing (CMP) is an essential step for manufacture silicon chips with sophisticated architecture. During the CMP process, silicon wafers are pressed against a rotating pad typically made of polyurethane. The table is fed with a stream of slurry that contains ultrafine (less than 0.2 microns) abrasive (e.g., silica or alumina). The wafers are therefore polished by the abrasive to reach a certain flatness and smoothness. However, the polished debris can coat the pad and make the latter unusable, so a diamond conditioner is required to scrape off the deposited debris from time to time.

The pad conditioner contains a plurality of diamond grits that are attached to a metal substrate (e.g., stainless steel). These grits are used to rake the pad to prevent its top from glazing. The effectiveness in cleaning the pad, and the durability of the conditioner are dependent on two critical

factors: the separation of diamond grits, and the leveling of their tops. These two factors are notoriously difficult to control. Hence, almost all diamond conditioners used commercially contain randomly distributed diamond grits with a high variability of the top height.

The object of this invention to make diamond film products that either eliminate or minimize the expensive post-synthesis operations.

It is another object of this invention to produce diamond film products with irregular and novel body shapes and surface finishes.

It is yet another object of this invention to simplify the diamond manufacture process by casting only the critical part of the diamond film, and then filling in the non-critical part with fast grown diamond or other material.

In the drawings

Figs. 1 shows stages of diamond nuclei formed on a surface of a substrate;

Fig. 2 shows a conventional tungsten carbide tool insert;

Fig. 3 shows a conventional diamond coated tungssten carbide tool insert without a chip breaker;

Fig. 4 shows conventional tungsten carbide with a thick diamond film but without a chip breaker;

Fig. 5 shows a thin metal mold for use in the present invention;

Fig. 6 shows the mold of Fig. 5 filled with diamond film deposition, in accordance with the present invention;

Fig.7 shows the mold of Fig. 5 dissolved away;

Fig. 8 shows a prior art polycrystalline diamond (PCD) enclosed by tungsten carbide;

Fig. 9 shows the PCD of Fig. 8 with a hole drilled therethrough;

Fig. 10 shows the PCD of Fig. 9 with the hole enlarged;

Fig. 11 shows a mold for use with the present invention;

Fig. 12 shows the mold of Fig. 11with diamond deposition ;

Fig. 13 shows the diamond deposition of Fig. 12 formed as a die, with the mold dissolved away;

Fig. 14 shows the diamond of Fig. 13 with the diamond die encased by a metal;

Fig. 15 shows a silicon wafer in accordance with the present invention;

Fig. 16 shows the silicon wafer of Fig. 15 cast with chemical vapor deposition (CVD) diamond;

Fig. 17 shows the silicon wafer of Fig. 16 with an epoxy resin layer;

Fig. 18 shows a disk with diamond pyramids after the the

silicon has been dissolved away;

Fig. 19 shows a wire having been twisted, in accordance with the present invention;

Fig. 20 shows the wire of Fig. 19 coated with chemical vapor deposition diamond;

Fig. 21 shows the wire of Fig. 19 dissolved away, leaving a diamond hose; and

Fig. 22 shows the diamond hose of Fig. 21 encased in resin.

This invention reverses the common practice of growing diamond film to form a body and then machining it to a required shape. Instead, a diamond film is cast in a mold to form the body shape and surface finish it in a single process. A remaining portion can then be filled in at a later stage using a much less expensive material or method. This novel concept can produce the cast that duplicates the body shape and surface finish of the mold, hence, expensive post-synthesis machining works are eliminated. Moreover, the complicated body shape or delicate surface finish can both be achieved from the mold. As a result, diamond film products with novel and unique geometries can now be produced for the first time.

A key ingredient of this invention is the ability to reproduce a surface finish of the mold to the deposited diamond cast and experiments have been performed to test this

feasibility. Many materials with different surface finishes have been used as the substrate for depositing CVD diamond. The CVD reactor used is a large production system manufactured by sp3, Inc. of California. The reactor is a hot filament system with a deposition area of 30 cm x 40 cm. It has been discovered that if the substrate material is suitable (e.g., copper, tungsten carbide), and the surface treatment is proper (i.e., thoroughly cleaned), diamond films so deposited can faithfully reproduce the surface feature of the substrate. Hence, the concept of reverse deposition was proven.

Conventional diamond films have their growing sides used as appropriate tool surfaces. This invention relies on using a nucleation side for the application. The nucleation side contains diamond microcrystals that have been formed on the substrate. These newly formed nuclei may not be chemically pure, nor structurally sound. Moreover, amorphous carbon or pyrolitic graphite may be co-deposited there. As the nucleation side will be used as the tool surface, it is critical to make this side contain as much diamond as possible.

Several techniques have been applied to improve the diamond quality of the nucleation side. For example, during the early phase of diamond deposition, the methane flow rate is reduced and the gas pressure is increased. In this way, the

decomposition rate of carbon will decrease, but the concentration of hydrogen atoms will increase. The slow deposition rate coupled with the increased amount of catalyst can assure that diamond nuclei formed are of high quality.

Moreover, the nucleation rate must be increased to fill up the minute crevices of the substrate. The nucleation rate can be effectively boosted (up to one million times) by applying a negative bias (e.g., 100 V) on the substrate. Alternatively, the substrate can be polished by using a fine diamond paste. After polishing, the embedded diamond micron powders can serve as effective seeds for diamond nucleation. If the nucleation rate can be raised, the diamond quality on the nucleation side will increase, and the substrate finish can be faithfully translated onto the nucleated surface.

Certain metals, e.g., iron, cobalt, nickel and their alloys, can catalyze at a high temperature (>700 degrees Celsius) diamond back to amorphous carbon or graphite. It is important that the substrate material contains as little as possible of such metals. For example, a good substrate material for diamond deposition is cobalt cemented tungsten carbide (WC). If WC is used for diamond deposition, the mount of cobalt should be limited to 4% or less. In recent years, binder free WC materials have been available. These materials are suitable to

make molds (e.g., the hole for a wire-drawing die). If the WC grain is ultrafine (submicron), the diamond nucleation will further increase. The result is a very smooth surface of high diamond content, which is ideal for many applications.

Fig. 5 shows a thin metal mold (20) that has the negative geometry of a conventional cutting insert with chip breakers. The metal can be W, Mo, Ta, Zr, Ti, Cr, V, Cu, Si or other suitable material. Fig. 6 shows that this metal mold (20) is filled up by the deposition of diamond film. Fig. 7 shows that the metal mold (20) is dissolved away, e.g., by soaking in acid. A bottom part (22) of the remaining diamond film contains the same geometrical form of the metal mold (20). A top part is then packed with WC particles or other refractory grains (e.g., SiC) and infiltrated with an alloy (e.g., copper-manganese alloy). The solidified alloy can then serve as the substrate of the cutting insert as shown in Fig. 7. This diamond insert contains the original surface of the metal mold (20), including the chip breaker. This diamond cutting tool does not require expensive mechanical finishing work.

This invention can solve all the above mentioned problems of the prior art.

Referring to Fig. 11, a center column (40) made of a binderless WC (or W metal) is first fabricated with the

appropriate geometry and surface finish. As shown in Fig. 12, this center column (40) is then coated with CVD diamond (42) to a thickness of 50 to 100 microns. The center column (40) is subsequently dissolved in acid, leaving a diamond tube cast in the same geometry of the center column (40), as seen in Fig. 13. This diamond tube is placed at the center of a stainless steel ring (46), and a space between them is packed with WC particles or SiC grains. A pile of copper-manganese-nickel alloy is placed on top of these particles. The set up is next placed in a vacuum furnace and heated to cause the melting of the copper alloy. The copper alloy will then infiltrate into the powder and consolidated the assembly. This rigid assembly contains a stainless steel outer ring and a diamond inner tube (44). Moreover, the diamond tube is compressed by the shrinking copper alloy that reduces the volume significantly during the solidification process. This compression is highly desirable as it will protect the tube from expansion due to the outward pushing force exerted by the drawing wire. Furthermore, this compressional force is adjustable in that may be varied to suit the type of wire to be drawn (e.g., low for drawing copper and high for drawing tungsten). The compressional force can be varied simply by adjusting the powder/metal ration. the more metal used, the higher the

compression force is.

The above described novel technology not only eliminates the expensive finishing operation, and hence greatly reduces the production cost, but also it improves the quality of the wire drawing die in many aspects. For example, unlike PCD that contains less than 90% in volume of diamond, CVDD is made entirely of diamond, hence, its wear life can be much higher than PCD (the service life is not linearly proportional to the diamond content, but is an exponential function).

CVDD is much more slippery than PCD that contains metal adhering cobalt, hence the heat generated during wire drawing would be significantly less. Moreover, CVDD can stand a temperature of 1200 ℃ that is much higher than 700 ℃ for PCD. Hence, the use of liquid lubrication may not be necessary. The elimination of liquid lubricant not only pays for the cost of the wire drawing die itself, but also it avoids environmental contamination associated with the lubricant. Concern about protecting the environment is forcing the machining industry to operate "dry", so this invention anticipates such a world trend.

Furthermore, the elimination of cobalt in the contact surface can improve greatly the surface finish of the wire, e.g., to make it have a reflective finish. Such a high quality wire is

highly welcomed by the electronics industry (e.g., contamination-free copper wires) and the jewelry business (e.g., shinning gold wires).

An even more significant breakthrough based on this invention is that wire drawing with non-circular holes can be performed as easily as circular holes. Hence, wires with triangular or square cross sections can be produced readily. The introduction of non-circular wires currently unavailable can allow new designs of products for certain fields (e.g., in the electronics industry), hence it may spur the creation of a new industry.

Using the cast diamond concept as taught by this invention, an extremely high quality diamond conditioner can be fabricated with strict control of diamond separation and top height. Firstly, a silicon wafer (50) is machined with impressions of identical pyramids that are placed in specific locations (e.g., with a fixed distance from the neighbor) as shown in Fig. 15. Secondly, the engraved silicon wafer is cast with CVD diamond (52), as shown in Fig. 16. Thirdly, an epoxy resin (54) is then attached on the top, as shown in Fig. 17. The entire assembly is then dipped in hydrofluoric acid to dissolve the silicon substrate. The remaining disk, when inverted, will show identical diamond pyramids protruding to the same height,

as shown in Fig. 18. Such a diamond layout is ideal for making a CMP pad conditioner. This method can also allow new designs of pad conditioners. For example, the distribution of the pyramids may be such that they follow a spiral pattern. In this way, the slurry may be guided to follow the pattern so as to improve the distribution of the abrasive. The result can be an increased polishing rate of the wafer and a reduced consumption of the expensive slurry. Moreover, the uniformity and flatness of the wafer may also be improved.

Diamond Hose

This invention can allow the manufacture of novel and unique diamond devices, such as a diamond hose that can be used to transmit corrosive fluid. The manufacturing steps follow the same procedures as described above. Firstly, the hose is fabricated using a metal (e.g., by twisting a W wire (60)), as shown in Fig. 19. Secondly, the twisted wire (60) is overcoated by CVDD (62), as shown in Fig. 20, to form a center metal core. Thirdly, the center metal core is dissolved, leaving a hollow diamond hose, as shown in Fig. 21. Finally, this diamond hose is cast in an epoxy resin (64) to form an easily handled tool, as shown in Fig 22.

The same idea may be applied to manufacture of other novel objects, such as a diamond flask or a diamond gear.

There is no limitation of the type of shape that may be produced by CVD diamond.

The above few examples are merely used to illustrate the flexibility of applying this invention.

It is to be understood, however, that even though numerous characteristics and advantages of the present invention have been set forth in the foregoing description, together with details of the structure and function of the invention, the disclosure is illustrative only, and changes may be made in detail, especially in matters of shape, size, and arrangement of parts within the principles of the invention to the full extent indicated by the broad general meaning of the terms in which the appended claims are expressed.

CLAIMS:

1. A CVD diamond product cast by a mold with the surface features determined by contact interface of the mold.

2. The product as described in Claim 1, wherein the mold is made of metal.

3. The product as described in Claim 2, wherein the metal is essentially any metal selected from the group of W, Mo, Ta, Ti, Zr, V, Cr, or any of its carbides, and Cu.

4. The product as described in Claim 1, wherein the mold has a concave shape.

5. The product as described in Claim 4, wherein the concave shape is a cup.

6. The product as described in Claim 1, wherein the mold has a convex shape.

7. The product as described in Claim 6, wherein the convex shape is cylindrical.

8. The product as described in Claim 1, wherein the CVD diamond has a thickness of 30 to 200 microns.

9. The product as described in Claim 1, wherein the product is one of a cutting tool made of diamond, a wire drawing die made of diamond, a pad conditioner made of diamond, a diamond diaphragm, or a diamond pipe.

10. The product as described in Claim 9, wherein the

cutting tool contains chip breakers.

11. The product as described in claim 9, wherein the wire drawing die has a nonspherical hole.

12. The product as described in Claim 9, wherein the wire drawing die is produced by infiltrating molten alloy around a CVD diamond tube.

13. A method to make a CVD diamond product, comprising the following steps:

a. making a negative mold that contains a shape and a surface of an intended product;

b. depositing a CVD diamond to a desired thickness at an interface of the mold:

c. dissolving the mold by acid or another solvent; and

d. mounting a remaining diamond object in a suitable holder.

14. The method according to Claim 12, wherein the CVD diamond is deposited by a method using hot filament, microwave plasma, oxyacetylene flame, or DC arc to heat gas used therein.

15. The method according to Claim 13, wherein the gas contains methane and hydrogen.

Application No: GB 0022947.6 **Examiner:** Pete Beddoe
Claims searched: 1-15 **Date of search:** 19 December 2000

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Search Report under Section 17

Databases searched:

UK Patent Office collections, including GB, EP, WO & US patent specifications, in:

UK Cl (Ed.R): C7F (FHB, FHE, FHX); B5A (AB18, AB19, ANA, AT3P, ATXP); C7D (DB702, DB704, DB706, DB708); B3D (DEN, DMN)

Int Cl (Ed.7): C23C (16/00, 16/27, 16/44); B29C (33/42, 39/02, 39/10, 41/08, 41/20, 45/37); B29D 31/00; B22F 7/04; B24B 53/12

Other: Online: WPI, EPODOC, JAPIO

Documents considered to be relevant:

Category	Identity of document and relevant passage	Relevant to claims
A	US 5507987 (SAINT GOBAIN) see whole doc	
X	US 4774991 (SMIT) see esp col2 line 52 - col3 line 39 & figs	1,13 at least
X	CH 684249 A & WPI Accession no 94-249735 (REISHAUER) see esp figs & abstract	1,13 at least
X	JP 2256464 A & WPI Accession no 90-357429 (ASAHI) see esp figs & abstract	1,13 at least
X	JP 5890465 A & Japio abstract of JP 5890465 A (OOSAKA) see esp figs & abstract	1,13 at least

X	Document indicating lack of novelty or inventive step	A	Document indicating technological background and/or state of the art.
Y	Document indicating lack of inventive step if combined with one or more other documents of same category.	P	Document published on or after the declared priority date but before the filing date of this invention.
&	Member of the same patent family	E	Patent document published on or after, but with priority date earlier than, the filing date of this application.

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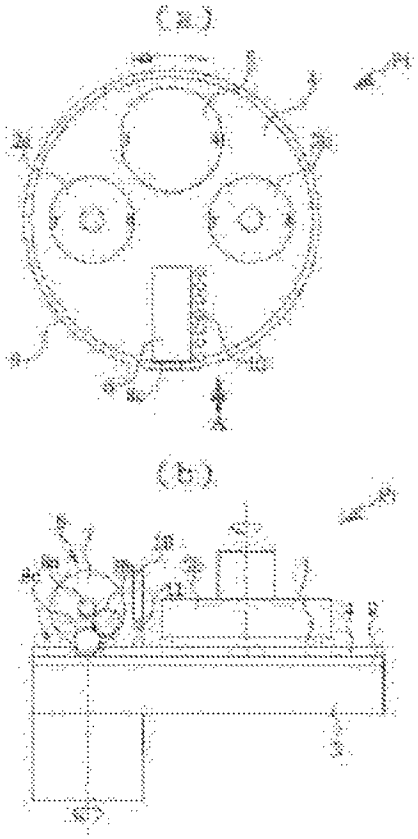
CHEMICAL-MECHANICAL POLISHING DEVICE, AND MANUFACTURE OF SEMICONDUCTOR INTEGRATED CIRCUIT DEVICE USING SAME

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Classification:	- **International:**	***B24B37/00; H01L21/304;*** (IPC1-7): B24B37/00; H01L21/304
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Application number:	JP19970208874 19970804	
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Abstract of JP11048122 (A)

PROBLEM TO BE SOLVED: To provide a polishing pad of long life in a chemical-mechanical polishing(CMP) technique, as well as to polish uniformly a whole surface of a polished film on a semiconductor wafer at stable polishing speed. SOLUTION: After a surface of a polishing pad 4 is ground by the first dressor 5 with embedded diamond particles to achieve flatness, a surface of a polished film on a semiconductor wafer 1 is polished, and a sodding grain on a surface of the polishing pad 4 is set up simultaneously by the second dressor 6 composed of a cylindrical brush 7 while supplying a polishing abrasive solution through a supplying nozzle 10 so as to restore an original coarse sodding grain.



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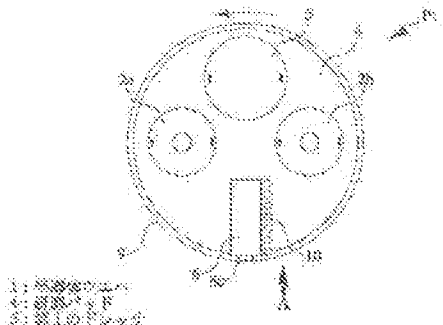
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(54)【発明の名称】　化学的機械研磨装置およびこれを用いた半導体装置の製造方法

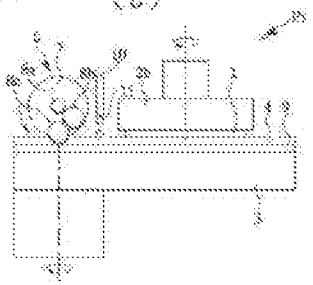
(57)【要約】
【課題】　CMP技術において、寿命の長い研磨パッドを得ると同時に、安定した研磨速度で半導体ウエハ上の絶縁膜の全面を均一に研磨する。
【解決手段】　ダイヤモンド粒子が埋め込まれた第1のドレッサ5で研磨パッド4の表面を切削して平坦度を出した後、半導体ウエハ1上の絶縁膜の研磨を開始すると同時に、供給ノズル10から研磨砥粒液11を供給しながら円盤状のブラシによって構成される第2のドレッサ6で研磨パッド4の表面の溝を立たせて、元の高い溝を復元する。

図 1

(a)



(b)



【特許請求の範囲】
【請求項１】　研磨定盤上に貼り付けられた研磨パッドに半導体ウエハを押し付けて前記半導体ウエハ上に設けられた各種膜の表面を研磨する化学的機械研磨装置であって、前記研磨パッドの表面を整形するドレッサが、少なくとも２種類設けられていることを特徴とする化学的機械研磨装置。
【請求項２】　請求項１記載の化学的機械研磨装置において、前記研磨パッドの表面を整形するドレッサとして、少なくとも前記研磨パッドの表面を平坦化する第１のドレッサと、前記研磨パッドの表面の荒さを復元させる第２のドレッサとが設けられていることを特徴とする化学的機械研磨装置。
【請求項３】　請求項２記載の化学的機械研磨装置において、前記第１のドレッサはダイヤモンド粒子が埋め込まれたドレッサであり、前記第２のドレッサはポリマー繊維からなるブラシによって構成されたドレッサであることを特徴とする化学的機械研磨装置。
【請求項４】　請求項３記載の化学的機械研磨装置において、前記半導体ウエハ上に設けられた各種膜の表面を研磨すると同時に、前記研磨パッドの表面に研磨砥粒液を供給しながら、前記第２のドレッサを前記研磨パッドの表面に押さえ付けて間欠的または連続的に回転させることを特徴とした化学的機械研磨装置。
【請求項５】　請求項４記載の化学的機械研磨装置において、前記第２のドレッサの前記研磨パッドに対する回転方向が、少なくとも前記半導体ウエハの回転方向とはほぼ逆方向の成分を有していることを特徴とする化学的機械研磨装置。
【請求項６】　請求項４記載の化学的機械研磨装置において、前記研磨砥粒液の供給口が前記第２のドレッサに内蔵されていることを特徴とする化学的機械研磨装置。
【請求項７】　請求項２～６のいずれか１項に記載の化学的機械研磨装置において、前記第２のドレッサが前記研磨パッドの研磨面に対して平行な回転軸を有する円筒状のドレッサであることを特徴とする化学的機械研磨装置。
【請求項８】　請求項７記載の化学的機械研磨装置において、前記第２のドレッサが強制的に回転されることを特徴とする化学的研磨装置。
【請求項９】　請求項７又は８に記載の化学的機械研磨装置において、前記第２のドレッサの円周速度は、前記研磨パッドが貼り付けられた研磨定盤の最外周部の円周速度よりも速いことを特徴とする化学的機械研磨装置。
【請求項１０】　請求項１～９のいずれか１項に記載の化学的機械研磨装置を用いた半導体集積回路装置の製造方法であって、前記第１のドレッサおよび前記第２のドレッサで整形される前記研磨パッドによって、半導体ウエハ上に設けられた各種膜の表面を平坦に加工する工程を有することを特徴とする半導体集積回路装置の製造方法。
【発明の詳細な説明】
【０００１】
【発明の属する技術分野】本発明は、半導体集積回路装置の製造技術に関し、特に、半導体ウエハ上に堆積された絶縁膜または金属膜の表面の凹凸を化学的機械研磨（Chemical Mechanical Polishing ；CMP）装置を用いて平坦に加工する技術に関するものである。
【０００２】
【従来の技術】従来のCMP装置P_1の要部断面の模式図を図５に示す。図において、５１は半導体ウエハ、５２は剛性板、５３は弾性材の合成樹脂から成る加圧用パッド、５４は剛性板５２にその厚み方向に貫通設けられた通気孔、５５は弾性板５３に設けられた通気孔５４と一致する如く加圧用パッド５３に配列された通気孔、５６は加圧ヘッド、５７は研磨パッド、５８は研磨パッドが貼り付けられた研磨定盤、５９は半導体ウエハ５１が研磨中に加圧ヘッド５６から外れないように設けられたストッパである。
【０００３】半導体ウエハ５１は、剛性板５２および加圧用パッド５３にそれぞれ設けられた通気孔５４，５５からの真空吸引により加圧ヘッド５６に装着された後、研磨パッド５７上に押し付けられる。研磨時の圧力は加圧ヘッド５６、剛性板５２、加圧用パッド５３を介して半導体ウエハ５１に加えられる。半導体ウエハ５１は加圧ヘッド５６と共に回転し、同じく回転する研磨定盤５８に貼り付けられた研磨パッド５７に押さえつけながら研磨される。
【０００４】次に、前記CMP装置P_1を用い、半導体ウエハ上に堆積された絶縁膜または金属膜の表面の凹凸を平坦化する製造工程の一例を図６（ａ）～（ｆ）を用いて説明する。
【０００５】これらの図において、６０は半導体基板、６１は第１層目の配線、６２は層間絶縁膜、６３は層間絶縁膜６２に開孔されたスルーホール、６４は金属膜、６５は第２層目の配線である。
【０００６】まず、半導体基板６０上に、例えばトランジスタ等の半導体素子、電荷蓄積用容量素子または抵抗素子（いずれも図示せず）などを形成した後、これらを互いに電気的に接続する第１層目の配線６１を形成する（工程（ａ））。この際、半導体基板６０の表面には第１層目の配線６１の厚み分の凹部が生ずる。
【０００７】次に、ＣＶＤ（Chemical Vapor Deposition）法を用いて酸化シリコン膜または窒化シリコン膜などによって構成される層間絶縁膜６２を半導体基板６０上に堆積させて、上記第１層目の配線６１を層間絶縁膜６２で覆う（工程（ｂ））。この際、ほぼ前記第１層目の配線６１の厚み分の段差が層間絶縁膜６２の表面に残存する。次いで、前記CMP装置P_1を用いて、層間絶

絶縁膜６２の表面を研磨して平坦に加工する（工程（ｃ））。

【０００８】次に、第１層目の配線６１に達するスルーホール６３を層間絶縁膜６２に設けた後、ＣＶＤ法またはスパッタリング法などによって半導体基板６０上に金属膜６４を堆積し（工程（ｄ））、次いで、前記ＣＭＰ装置Ｐ１を用いて層間絶縁膜６２の表面に堆積された金属膜６４を研磨除去する。これによって、スルーホール６３に金属膜６４を埋め込む（工程（ｅ））。

【０００９】次に、半導体基板６０上に金属膜（図示せず）を堆積した後、この金属膜をパターニングすることによって第２層目の配線６５を形成する（工程（ｆ））。第１層目の配線６１と第２層目の配線６５とはスルーホール６３に埋め込まれた金属膜６４を介して電気的に接続される。また、上記工程（ａ）～（ｆ）を同様に繰り返すことにより所望の多層配線を有する半導体集積回路装置を形成することができる。

【００１０】ところで、上記工程（ｃ）での層間絶縁膜６２の表面の平坦化および上記工程（ｅ）での金属膜６４の表面の平坦化で要求される研磨技術の基本的性能としては、被研磨膜の凹凸の平坦度、研磨量の均一性、スループットおよび研磨速度の安定性が挙げられる。これらのうち、被研磨膜の凹凸の平坦度は使用する研磨パッドの機械的材料、研磨量の均一性はウエハ加圧方式によってほぼ決定されるのに対し、スループットおよび研磨速度の安定性は、研磨砥粒の作用、加圧力または回転速度などの研磨条件に加えて、研磨パッドの表面状態に強く依存する。

【００１１】特に、本発明者の実験により、研磨速度の安定性は研磨パッドの表面の荒さの変化と強い相関関係があることが明らかとなった。図７は、本発明者の実験結果の一例であり、半導体ウエハの処理枚数に対する半導体ウエハ上の被研磨膜の研磨速度の変化および研磨パッドの表面の荒さの変化を示すグラフ図である。

【００１２】図に示すように、半導体ウエハの処理枚数が増加するに従い、研磨速度は初期に急激に低下し、その後、なだらかに低下した後、ほぼ一定の速度に安定する。また、研磨速度の変化と同様に、研磨パッドの表面の荒さも半導体ウエハの処理枚数が増加するに従い減少した後、ほぼ一定の荒さに落ち着く。

【００１３】この理由は、研磨パッドの表面の荒さが減少すると研磨剤（液）の保持力が低下して研磨パッドの表面に供給された研磨砥粒液が半導体ウエハの回転により半導体ウエハの外周部で掻き出されてしまい、半導体ウエハ上の研磨面と研磨パッド面との間に存在する研磨砥粒の量が減少して、研磨速度が低下するものと考えられる。なお、研磨パッドの表面の荒さが減少する理由は、研磨パッドの表面の荒の構造が、研磨中に繰り返し受ける半導体ウエハへの研磨圧力によって潰れてしまうためである。

【００１４】そこで、研磨中における研磨パッドの表面状態の経時変化を防ぎ、半導体ウエハの表面の研磨速度を一定に保つために、半導体ウエハ上の被研磨膜を研磨する加圧ヘッドとは別に、ダイヤモンド粒子を埋め込んだ円盤（以下、ドレッサと称す）を研磨作業後または研磨作業中に研磨パッド上に摺動させて、研磨パッドの表面を整形するドレッシングが研磨パッドに施されている。

【００１５】図８および図９に従来の研磨パッドのドレッシング方法の概略図を示す。図８に示すドレッシング方法は、大型のドレッサ６６に回転と揺動を加えながら研磨パッド５７の表面を整形する方法であり、図９に示すドレッシング方法は、アーム揺動６７を用いて小型のドレッサ６８を研磨パッド５７の全面に均等に揺動させて、研磨パッド５７の表面を整形する方法である。これらのドレッシング方法には、半導体ウエハ上の被研磨膜の研磨と研磨パッドの表面のドレッシングとを同時に行う同時ドレッシング、または半導体ウエハ上の被研磨膜の研磨前後の半導体ウエハを搬送する空き時間に研磨パッドの表面のドレッシングを行う間欠ドレッシングがある。

【００１６】なお、ＣＭＰ装置のドレッシング方法については、例えばソリッド・ステート・テクノロジ（Solid State Technology, 日本版,Dec. 1994, Iqbal Ali et al., "層間絶縁膜の化学的機械研磨に関する概観"）などに記載されている。

【００１７】

【発明が解決しようとする課題】しかしながら、前記ドレッシング方法には、以下の問題点があることを本発明者は見いだした。

【００１８】すなわち、まず、同時ドレッシングでは、研磨パッドの表面が常にドレッシングされるので安定な研磨速度が得られるが、研磨パッドの表面が常にドレッサによって削り取られるため研磨パッドの寿命が著しく低下する。また、間欠ドレッシングでは、研磨パッドの寿命は長いが、半導体ウエハ上の被研磨膜の研磨中に研磨速度が低下して安定な研磨速度が得られず、さらに、ドレッシングに要する時間が単独で必要なためにＣＭＰ装置のスループットが低下する。

【００１９】これらの問題を解決する方法の一つとして、研磨パッドに対するドレッサの押圧力を低下させる方法がある。この方法を採用すれば、研磨速度をある程度安定化させ、研磨パッドの寿命を長くすることができる。

【００２０】しかし、ドレッシングには、研磨パッドの表面の荒さを一定に保ち研磨速度を安定化させる目的の他に、研磨パッドの全面をドレッシングでの切削によって整形し、研磨パッドの表面の平坦度を得ることにより半導体ウエハ上の被研磨膜の全面の均一性を確保する目的があり、研磨パッドに対するドレッサの押圧力を低下

させると研磨パッドの表面の平坦度を維持することが難しく、半導体ウエハ上の被研磨膜の全面を均一に研磨することができない問題がある。

【００２１】本発明の目的は、ＣＭＰ技術において、寿命の長い研磨パッドを得ると同時に、安定した研磨速度で半導体ウエハ上の被研磨膜の全面を均一に研磨することができる技術を提供することにある。

【００２２】本発明の前記ならびにその他の目的と新規な特徴は、本明細書の記述および添付図面から明らかになるであろう。

【００２３】

【課題を解決するための手段】本願において開示される発明のうち、代表的なものの概要を簡単に説明すれば、次のとおりである。

【００２４】すなわち、本発明のＣＭＰ装置は、研磨定盤上に貼り付けられた研磨パッドの表面を整形するドレッサとして、少なくとも、ダイヤモンド粒子が埋め込まれ、研磨パッドの表面を平坦化する第１のドレッサと、ポリマ繊維からなるブラシによって掃かれ、研磨パッドの表面の荒さを復元させる第２のドレッサとを有しており、上記第１のドレッサにより研磨パッドの表面を切削した後、第１のドレッサを後退させ、次いで、加圧ヘッドに装着された半導体ウエハを研磨パッドの表面に押さえ付けて半導体ウエハ上の被研磨膜の表面を研磨すると同時に、研磨パッドの表面に研磨剤供給液を供給しながら上記第２のドレッサを断続的または連続的に研磨パッドの表面に押さえ付けて、研磨パッドの表面の荒めを立たせる方向に揺動させるものである。

【００２５】上記した手段によれば、第２のドレッサによるドレッシングによって、研磨パッドの表面を切削することなく、半導体ウエハ上の被研磨膜の表面を研磨することによって倒れた研磨パッドの表面の荒めを立たせて、比較的容易に元の荒い荒めに復元できるので、研磨パッドの寿命を大幅に長くできると同時に、半導体ウエハ上の被研磨膜の研磨速度を安定化することができる。さらに、研磨パッドに対する第１のドレッサの押圧力を低下させる必要がないので、研磨パッドの表面の平坦度が確保できて、半導体ウエハ上の被研磨膜の全面を均一に研磨することができる。

【００２６】

【発明の実施の形態】以下、本発明の実施の形態を図面に基づいて詳細に説明する。

【００２７】なお、実施の形態を説明するための全図において同一機能を有するものは同一の符号を付し、その繰り返しの説明は省略する。

【００２８】本発明の一実施の形態であるＣＭＰ装置Ｐ$_1$が有する研磨パッドのドレッシング方法を図１に示す模式図を用いて説明する。図１（ａ）は、ＣＭＰ装置Ｐ$_1$の上面図であり、図１（ｂ）は、図１（ａ）中に記載のＡ方向から見たＣＭＰ装置Ｐ$_1$の側面図である。１は半導体ウエハ、２ａ，２ｂは加圧ヘッド、３は研磨定盤、４は研磨パッド、５は第１のドレッサ、６は第２のドレッサ、７はブラシ、８ａ，８ｂ，８ｃは歯車、９はギア、１０は供給ノズル、１１は研磨剤供給装置である。

【００２９】被研磨材料である半導体ウエハ１は、加圧ヘッド２ａ，２ｂに装着され、回転する研磨定盤３上に張り付けられた研磨パッド４の表面に加圧ヘッド２ａ，２ｂを介して押さえ付けられて、その表面は研磨される。なお、研磨時の加圧ヘッド２ａ，２ｂおよび研磨定盤３の回転数は、例えば共に３０回／分であり、半導体ウエハ１および研磨定盤３の直径は、例えばそれぞれ８インチおよび６００ｍｍである。半導体ウエハ１は、例えば３００ｇｒ／ｃｍ^{2}の圧力で研磨パッド４の表面に押さえ付けられている。

【００３０】研磨パッド４の表面は、第１のドレッサ５および第２のドレッサ６を用いてドレッシングされる。第１のドレッサ５にはダイヤモンド粒子が埋め込まれており、第１のドレッサ５は研磨パッド４の表面を切削して平坦度を出すために用いられる。第２のドレッサ６は研磨パッドの研磨面に対して平行な回転軸を有する円筒状のブラシ７と歯車８ａ，８ｂ，８ｃとによって構成されており、研磨パッド４の表面の荒めを立たせて元の荒い荒めを復元するために用いられる。

【００３１】第２のドレッサ６の歯車８ｃを研磨定盤３の外周端面に形成したギアと噛み合わせて研磨定盤３を回転させることにより、歯車８ｂを介して歯車８ａと一体のブラシ７を強制的に回転駆動させる。この際、第２のドレッサ６の研磨パッド４に対する揺動方向は、半導体ウエハ１の揺動方向とは逆方向の成分を有している。また、ブラシ７の円周速度は研磨定盤３の最外周部の円周速度より約１．５倍速くなるように、歯車８ｃのギア比とブラシ７の径は選ばれる。ブラシ７には、例えば線径１００μｍ、長さ５ｍｍのナイロンブラシを使用した。

【００３２】まず、第１のドレッサ５を用いて研磨パッド４の表面を切削した後、第１のドレッサ５を後退させ、次いで、半導体ウエハ１上の被研磨膜の表面を研磨すると同時に、第２のドレッサ６を断続的または連続的に研磨パッド４上で揺動させて、研磨パッド４の表面を整形する。この際、第２のドレッサ６の近傍に設置した供給ノズル１０から研磨剤供給装置１１が約２００ｍｌ／分の速度で研磨パッド４上に供給される。研磨剤供給装置１１の供給口は第２のドレッサ６に内蔵してもよい。

【００３３】図２に、本実施の形態である前記ＣＭＰ装置Ｐ$_1$を用いて、半導体ウエハ上にプラズマＣＶＤ法で堆積された酸化シリコン膜の表面を研磨した際の酸化シリコン膜の研磨速度と半導体ウエハの研磨枚数との関係を示す。図において、四角印は前記本実施の形態のドレッシング、白丸印は従来の同時ドレッシング、黒丸印は従来の間欠ドレッシングを採用した際のそれぞれの酸化

シリコン膜の研磨速度を示す。

【００３４】いずれの場合も一つの研磨定盤に対して２つの加圧ヘッドを有し、２枚の半導体ウエハを同時に研磨した結果であり、図中には２枚の半導体ウエハで得られたそれぞれの酸化シリコン膜の研磨速度の平均値を１点で示してある。半導体ウエハ上の酸化シリコン膜の研磨時間は５分である。

【００３５】本実施の形態のドレッシングでは、半導体ウエハ１上の酸化シリコン膜の研磨前の第１のドレッサ５によるドレッシング時間は３分であり、半導体ウエハ１上の酸化シリコン膜の研磨中は第２のドレッサ６でドレッシングを行っている。研磨パッドには厚さが１．２ｍｍの発泡ポリウレタン性のＩＣ１０００（米国ロデール社）を用いた。なお、従来の間欠ドレッシングのドレッシング時間は２分である。

【００３６】図から明らかなように、従来の間欠ドレッシングの研磨速度は他のドレッシングの研磨速度と比べて約１０％低く、また、±５％以上の研磨速度の変動がある。しかし、本実施の形態のドレッシングでは半導体ウエハの研磨枚数が２５枚まで従来の同時ドレッシングとほぼ同じ研磨速度が得られ、また、±２％以下の研磨量の均一性（図示せず）が得られる。

【００３７】図３に、本実施の形態である前記ＣＭＰ装置Ｐ，を用いて、半導体ウエハ上に堆積された酸化シリコン膜を研磨した際の研磨パッドの厚さと半導体ウエハの研磨枚数との関係を示す。図において、四角印は前記本実施の形態のドレッシング、白丸印は従来の同時ドレッシング、黒丸印は従来の間欠ドレッシングを採用した際のそれぞれの研磨パッドの厚さを示す。

【００３８】なお、本発明者の実験から、研磨パッドをその厚さが初期の厚さ（例えば１．２ｍｍ）の半分になるまで研磨すると、半導体ウエハ上の被研磨膜の研磨速度の安定性または半導体ウエハ上の被研磨膜の研磨量均一性に顕著な劣化が認められることが明らかになっており、このことから、研磨パッドの厚さが半分となった時点を研磨パッドの寿命と称す。

【００３９】図に示すように、従来の同時ドレッシングでは約１００枚の半導体ウエハを研磨するだけで研磨パッドは寿命に達し、従来の間欠ドレッシングでも約２５０枚の半導体ウエハを研磨すると研磨パッドは寿命に達する。これに対し、本実施の形態のドレッシングでは、約４０００枚の半導体ウエハを研磨すると研磨パッドが寿命に達する。

【００４０】次に、本実施の形態の前記ＣＭＰ装置Ｐ，を用いたＤＲＡＭ（Dynamic Random Access Memory）のメモリ部の製造方法を図４を用いて説明する。なお、ＤＲＡＭの周辺回路部はｎチャネル型ＭＩＳＦＥＴ（Metal Insulator Semiconductor Field Effect Transistor）Ｑｎの製造方法のみを記載する。

【００４１】まず、ｐ－型シリコン単結晶からなる半導体基板１２の主面上に周知の方法でｐ型ウエル１３、フィールド絶縁膜１４およびゲート絶縁膜１５を順次形成する。

【００４２】次に、図示はしないが、半導体基板１２上に堆積された窒化シリコン膜、タングステンシリサイド（ＷＳｉ$_2$）膜および多結晶シリコン膜からなる積層膜を順次エッチングすることにより、ＷＳｉ$_2$膜および多結晶シリコン膜からなるメモリセルのメモリセル選択用ＭＩＳＦＥＴのゲート電極１６および周辺回路部のｎチャネル型ＭＩＳＦＥＴＱ$_n$のゲート電極１６を形成する。

【００４３】なお、上記ゲート電極１６を構成するメタルシリサイド膜にＷＳｉ$_2$膜を用いたが、その他のメタルシリサイド膜、例えばモリブデンシリサイド（ＭｏＳｉ$_2$）膜、チタンシリサイド（ＴｉＳｉ$_2$）膜、タンタルシリサイド（ＴａＳｉ$_2$）膜などを用いてもよい。

【００４４】次に、レジストパターンならびに上記窒化シリコン膜、ＷＳｉ$_2$膜および多結晶シリコン膜からなる積層膜をマスクにして、周辺回路部のｐ型ウエル１３にｎ型不純物、例えばリン（Ｐ）をイオン注入し、ｎチャネル型ＭＩＳＦＥＴＱ$_n$のｎ型半導体領域（ソース領域、ドレイン領域）１７を、ゲート電極１６に対して自己整合で形成する。

【００４５】その後、半導体基板１２上に堆積された窒化シリコン膜をＲＩＥ（Reactive Ion Etching）法などの異方性エッチングで加工することによって、ゲート電極１６の側壁にサイドウォールスペーサを形成し、ゲート電極１６を窒化シリコン膜からなる絶縁膜１８で覆う。

【００４６】なお、上記サイドウォールスペーサを形成した後、周辺回路部のｐ型ウエル１３に高濃度のｎ型不純物、例えば砒素（Ａｓ）をイオン注入することにより、ｎチャネル型ＭＩＳＦＥＴＱ$_n$のソース領域、ドレイン領域をＬＤＤ（Lightly Doped Drain）構造としてもよい。

【００４７】次に、半導体基板１２上に酸化シリコン膜１９および第１のＢＰＳＧ（Boron-doped Phospho Silicate Glass）膜２０をＣＶＤ法によって堆積した後、９００～９５０℃のリフロー処理により上記第１のＢＰＳＧ膜２０の表面を平坦化する。

【００４８】その後、レジストパターンをマスクにして第１のＢＰＳＧ膜２０、酸化シリコン膜１９およびゲート絶縁膜１５と同一層の絶縁膜を順次エッチングすることにより、メモリセル選択用ＭＩＳＦＥＴの一方の後に形成されるｎ型半導体領域２１上に第１のコンタクトホール２２を形成する。

【００４９】次いで、上記第１のコンタクトホール２２内にＰが導入された多結晶シリコン膜２３からなる第１プラグ電極を形成する。なお、この多結晶シリコン膜２３に導入されたＰの拡散によってメモリセル選択用ＭＩ

ＳＦＥＴの一方のｎ型半導体領域２１が形成される。

【００５０】次に、半導体基板１２上に酸化シリコン膜２４をＣＶＤ法によって堆積する。次いで、図示はしないが、レジストパターンをマスクにして酸化シリコン膜２３、第１のＢＰＳＧ膜２０、酸化シリコン膜１９およびゲート絶縁膜１５と同一層の絶縁膜を順次エッチングすることにより、メモリセル選択用ＭＩＳＦＥＴの他方の後に形成されるｎ型半導体領域上に第２のコンタクトホールを形成する。この際、後に形成されるビット線を周辺回路部に延在し、周辺回路部の半導体基板１２に接続するための第２のコンタクトホール（周辺回路部）２５を形成してもよい。

【００５１】次いで、半導体基板１２上にＰが導入された多結晶シリコン膜２６およびWSi_2膜２７をＣＶＤ法によって順次堆積した後、レジストパターンをマスクにしてWSi_2膜２７および多結晶シリコン膜２６を順次エッチングすることにより、WSi_2膜２７および多結晶シリコン膜２６からなるビット線を形成する。

【００５２】また、多結晶シリコン膜２６に導入されたＰの拡散によってメモリセル選択用ＭＩＳＦＥＴの他方のｎ型半導体領域（図示せず）は形成され、ビット線は第２のコンタクトホールを通して、このメモリセル選択用ＭＩＳＦＥＴの他方のｎ型半導体領域に接続される。この際、多結晶シリコン膜２６に導入されたＰの拡散によって周辺回路部のｐ型ウエル１３にもｎ型半導体領域（周辺回路部）２８が形成され、第２のコンタクトホール（周辺回路部）２５を通して、ビット線が上記ｎ型半導体領域（周辺回路部）２８に接続される。

【００５３】次に、半導体基板１２上に酸化シリコン膜２９、窒化シリコン膜３０および第２のＢＰＳＧ膜（図示せず）をＣＶＤ法によって順次堆積した後、９００～９５０℃のリフロー処理により上記第２のＢＰＳＧ膜の表面を平坦化する。

【００５４】次に、半導体基板１２上にＰが導入された多結晶シリコン膜３１をＣＶＤ法によって堆積した後、レジストパターンをマスクにして多結晶シリコン膜３１をエッチングする。次いで、半導体基板１２上にＣＶＤ法によって堆積されたＰが導入された多結晶シリコン膜３２をＲＩＥ法などの異方性エッチングによって加工し、上記多結晶シリコン膜３１の側壁に多結晶シリコン膜３２からなるサイドウォールスペーサを形成する。

【００５５】次いで、レジストパターンをマスクにしてメモリセルの第２のＢＰＳＧ膜、窒化シリコン膜３０、酸化シリコン膜２９および酸化シリコン膜２４を順次エッチングすることにより、第１のコンタクトホール２２内に設けられた第１プラグ電極上に第３のコンタクトホール３３を形成した後、半導体基板１２上にＰが導入された多結晶シリコン膜３４および第３のＢＰＳＧ膜（図示せず）をＣＶＤ法によって順次堆積する。

【００５６】次に、レジストパターンをマスクにして上記第３のＢＰＳＧ膜および多結晶シリコン膜３４、３１を順次エッチングした後、半導体基板１２上にＰが導入された多結晶シリコン膜３５をＣＶＤ法によって堆積する。次いで、この多結晶シリコン膜３５をＲＩＥ法などの異方性エッチングによって加工し、メモリセルの第３のＢＰＳＧ膜および多結晶シリコン膜３４、３１の側壁に多結晶シリコン膜３５を残す。

【００５７】次に、例えば、フッ酸溶液を用いたウエットエッチングによって、第３のＢＰＳＧ膜および第２のＢＰＳＧ膜を除去し、メモリセルに多結晶シリコン膜３１、３２、３４、３５からなる円筒型の蓄積電極を形成する。

【００５８】次に、半導体基板１２上に厚さ約２０ｎｍの酸化シリコン膜（図示せず）をＣＶＤ法によって堆積し、続いて、厚さ約３０ｎｍの非晶質の酸化タンタル（Ta_2O_5）膜（図示せず）をＣＶＤ法によって堆積した後、半導体基板１２に酸化処理を施すことによって、上記Ta_2O_5膜を結晶化する。その後、半導体基板１２上にＴｉＮ膜３６をＣＶＤ法によって堆積し、次いで、フォトレジストをマスクにしてこのＴｉＮ膜３６をエッチングすることにより、ＴｉＮ膜３６からなるプレート電極を形成する。

【００５９】なお、容量絶縁膜にTa_2O_5膜を用いたが、その他の酸化メタル膜（例えば、（Ｂａ，Ｓｒ）ＴｉＯ膜またはＰｂ（Ｚｒ，Ｔｉ）O_3膜）などを用いてもよく、また、上記プレート電極にＴｉＮ膜を用いたが、その他のメタルナイトライド膜（例えば、ＷＮ膜）またはメタル膜（例えば、Ｗ膜）などを用いてもよい。

【００６０】次に、半導体基板１２上に酸化シリコン膜３７および第４のＢＰＳＧ膜３８をＣＶＤ法によって順次堆積した後、９００～９５０℃のリフロー処理により上記第４のＢＰＳＧ膜３８の表面を平坦化する。

【００６１】次いで、レジストパターンをマスクにして第４のＢＰＳＧ膜３８および酸化シリコン膜３７を順次エッチングすることにより、ＴｉＮ膜３６からなるプレート電極上に第４のコンタクトホール３９ａを形成し、同時に、第４のＢＰＳＧ膜３８、酸化シリコン膜３７および酸化シリコン膜２９を順次エッチングすることにより、WSi_2膜２７および多結晶シリコン膜２６からなるビット線上に第４のコンタクトホール３９ｂを形成する。

【００６２】さらに、同時に、第４のＢＰＳＧ膜３８、酸化シリコン膜３７、酸化シリコン膜２９、酸化シリコン膜２４、第１のＢＰＳＧ膜２０、酸化シリコン膜１９およびゲート絶縁膜１５と同一層の絶縁膜を順次エッチングすることにより、ｎチャネル型ＭＩＳＦＥＴQ_3のｎ型半導体領域１７上に第４のコンタクトホール３９ｃを形成する。

【００６３】次に、半導体基板１２上に金属膜（図示せず）を堆積した後、レジストパターンをマスクにして上

記金属膜をエッチングすることにより、第１層目のメタル配線M_1が形成される。次いで、半導体基板１２上にＥＣＲ（Electron Cyclotron Resonance：電子サイクロトロン共鳴）プラズマＣＶＤ法によって酸化シリコン膜を堆積した後、この酸化シリコン膜の表面を本実施の形態である前記ＣＭＰ装置P_1を用いて研磨することによって、その表面が平坦化された酸化シリコン膜によって構成される第１の層間絶縁膜４０を設ける。

【００６４】次に、レジストパターンをマスクにして上記第１の層間絶縁膜４０をエッチングすることにより、第１層目のメタル配線M_1に達するスルーホール４１を形成した後、半導体基板１２上に金属膜（図示せず）を堆積し、次いで、この金属膜をレジストパターンをマスクにしてエッチングすることにより、第２層目のメタル配線M_2を形成する。

【００６５】ここで、第１の層間絶縁膜４０の表面が平坦化されているので、第２層目のメタル配線M_2を形成する際のフォトリソグラフィ工程において、マスクパターン転写における焦点深度に余裕が生じ、高解像度のレジストパターンが形成される。これによって、レジストパターンをマスクとして加工、形成される第２層目のメタル配線M_2の欠損または隣接する第２層目のメタル配線M_2間の短絡を防ぐことができる。

【００６６】さらに、上記第１の層間絶縁膜４０と同様に、半導体基板１２上にＥＣＲプラズマＣＶＤ法によって酸化シリコン膜を堆積した後、この酸化シリコン膜の表面を本実施の形態である前記ＣＭＰ装置P_1を用いて研磨することによって、その表面が平坦化された酸化シリコン膜によって構成される第２の層間絶縁膜４３を設ける。

【００６７】次に、レジストパターンをマスクにして上記第２の層間絶縁膜４２をエッチングすることにより、第２層目のメタル配線M_2に達するスルーホール４２を形成した後、半導体基板１２上に金属膜（図示せず）を堆積し、次いで、この金属膜をレジストパターンをマスクにしてエッチングすることにより、第３層目のメタル配線M_3を形成する。

【００６８】最後に、半導体基板１２の表面をパッシベーション（図示せず）で被覆することにより、本実施の形態のＣＭＰ装置P_1を用いて加工された第１の層間絶縁膜４０および第２の層間絶縁膜４２を有するＤＲＡＭが完成する。

【００６９】このように、本実施の形態では、研磨パッド４の寿命が、従来の同時ドレッシングを採用した研磨パッドの寿命の約１０倍以上、また、従来の間欠ドレッシングを採用した研磨パッドの寿命の約１６倍以上長くなり、例えば、従来の同時ドレッシングでは４～５時間毎に必要があった研磨パッドの交換を週に１度の頻度とすることができる。さらに、第２のドレッサ６のプランジャを研磨パッド４の表面の窪みを立たせる方向に駆動させることによって、研磨パッド４の表面の倒れた毛のを立たせて、比較的容易に元の荒い状態に復元することができるので、研磨速度を安定化することができる。さらに、研磨パッド４に対する第１のドレッサ５の押圧力を低下させる必要がないので、研磨パッド４の表面の平坦度が確保できて、半導体ウエハ１上の被研磨膜の全面を均一に研磨することができる。

【００７０】以上、本発明者によってなされた発明を発明の実施の形態に基づき具体的に説明したが、本発明は前記実施の形態に限定されるものではなく、その要旨を逸脱しない範囲で種々変更可能であることはいうまでもない。

【００７１】

【発明の効果】本願によって開示される発明のうち、代表的なものによって得られる効果を簡単に説明すれば、以下のとおりである。

【００７２】本発明によれば、プランジャによって揺動される第２のドレッサによるドレッシングによって、研磨パッドの表面を切削することなく、比較的容易に研磨パッドの表面の窪みを荒い毛並みに復元することができるので、研磨パッドの寿命を大幅に長くできると同時に、半導体ウエハ上の被研磨膜の表面の研磨速度を安定化することができ、さらに、ダイヤモンド粒子を埋め込んだ第１のドレッサの研磨パッドに対する押圧力を低下させる必要がないので、研磨パットの表面の平坦度が確保できて、半導体ウエハ上の被研磨膜の全面を均一に研磨することができる。

【００７３】また、本発明によれば、研磨パッドの寿命が長くなるので、半導体製造装置としての生産能力、稼働率が大幅に向上し、また、高価な研磨パッドの消費量が減少する。

【図面の簡単な説明】

【図１】本発明の一実施の形態であるＣＭＰ装置の概式図であり、（ａ）は上面図、（ｂ）は要部断面図である。

【図２】本発明の一実施の形態であるドレッシング、従来の同時ドレッシングまたは従来の間欠ドレッシングをそれぞれ採用した研磨パッドを有するＣＭＰ装置を用いて、半導体ウエハ上の酸化シリコン膜の表面を研磨した際の酸化シリコン膜の研磨速度の経時変化を示すグラフ図である。

【図３】本発明の一実施の形態であるドレッシング、従来の同時ドレッシングまたは従来の間欠ドレッシングをそれぞれ採用した研磨パッドを有するＣＭＰ装置を用いて、半導体ウエハ上の酸化シリコン膜の表面を研磨した際の研磨パッドの厚さの経時変化を示すグラフ図である。

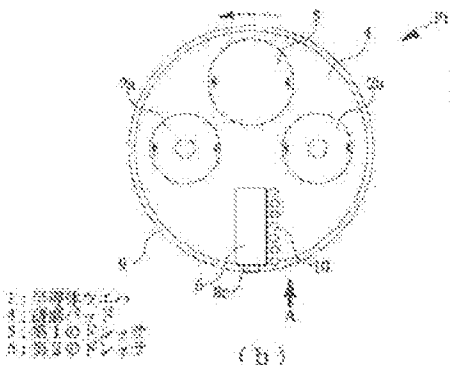
【図４】本発明の一実施の形態であるドレッシングを採用した研磨パッドを有するＣＭＰ装置を用いて平坦化される層間絶縁膜を有するＤＲＡＭを示す半導体基板の要

断面図である。
【図５】従来のＣＭＰ装置の要部構成の模式図である。
【図６】（ａ）～（ｆ）はそれぞれ従来のドレッシングを採用した研磨パッドを有するＣＭＰ装置を用いて平坦化された絶縁膜または金属膜を有する半導体基板の要部断面図である。
【図７】従来のドレッシングを採用した研磨パッドを有するＣＭＰ装置を用いて研磨された半導体ウエハ上の被研磨膜の研磨速度と研磨パッドの高さの経時変化を示すグラフ図である。
【図８】従来の研磨パッドのドレッシング方法を説明するための概略図である。
【図９】従来の研磨パッドのドレッシング方法を説明するための概略図である。
【符号の説明】
１　半導体ウエハ
２ａ　加圧ヘッド
２ｂ　加圧ヘッド
３　研磨定盤
４　研磨パッド
５　第１のドレッサ
６　第２のドレッサ
７　ブラシ
８ａ　歯車
８ｂ　歯車
８ｃ　歯車
９　ギア
１０　供給ノズル
１１　研磨電解溶液
１２　半導体基板
１３　ｐ型ウエル
１４　フィールド絶縁膜
１５　ゲート絶縁膜
１６　ゲート電極
１７　ｎ型半導体領域（ソース領域、ドレイン領域）
１８　絶縁膜
１９　酸化シリコン膜
２０　第１のＢＰＳＧ膜
２１　ｎ型半導体領域
２２　第１のコンタクトホール
２３　多結晶シリコン膜
２４　酸化シリコン膜
２５　第２のコンタクトホール（周辺回路部）
２６　多結晶シリコン膜
２７　タングステンシリサイド膜
２８　ｎ型半導体領域（周辺回路部）
２９　酸化シリコン膜
３０　窒化シリコン膜
３１　多結晶シリコン膜
３２　多結晶シリコン膜
３３　第３のコンタクトホール
３４　多結晶シリコン膜
３５　多結晶シリコン膜
３６　窒化チタン膜
３７　酸化シリコン膜
３８　第４のＢＰＳＧ膜
３９ａ　第４のコンタクトホール
３９ｂ　第４のコンタクトホール
３９ｃ　第４のコンタクトホール
４０　第１の層間絶縁膜
４１　スルーホール
４２　第２の層間絶縁膜
４３　スルーホール
５１　半導体ウエハ
５２　弾性板
５３　加圧用パッド
５４　通気孔
５５　通気孔
５６　加圧ヘッド
５７　研磨パッド
５８　研磨定盤
５９　ノズル
６０　半導体基板
６１　第１層目の配線
６２　層間絶縁膜
６３　スルーホール
６４　金属膜
６５　第２層目の配線
６６　大型のドレッサ
６７　アーム機構
６８　小型のドレッサ
P_1　本発明の実施の形態のＣＭＰ装置
P_2　従来のＣＭＰ装置
Q_n　ｎチャネル型ＭＩＳＦＥＴ
M_1　第１層目のメタル配線
M_2　第２層目のメタル配線
M_3　第３層目のメタル配線

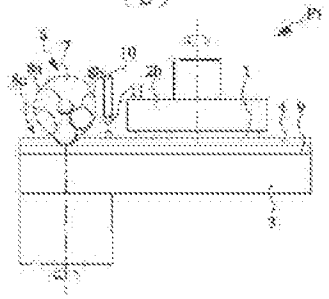
【図１】

図　１

（ａ）

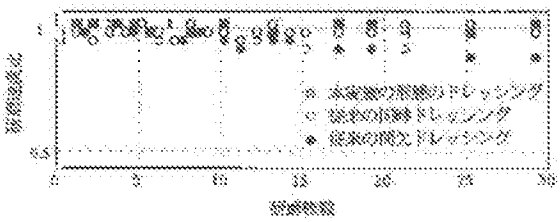


（ｂ）



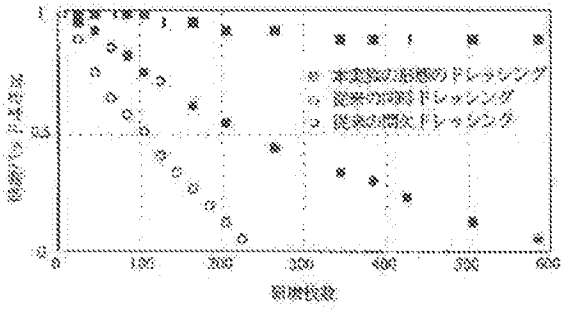
【図２】

図　２



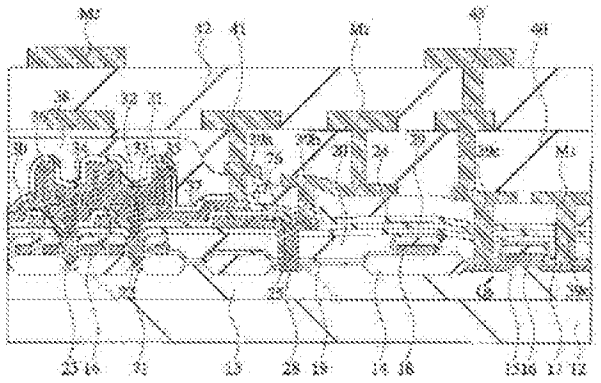
【図３】

図　３



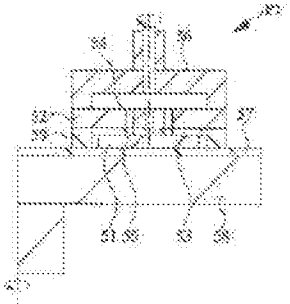
【図４】

図　４



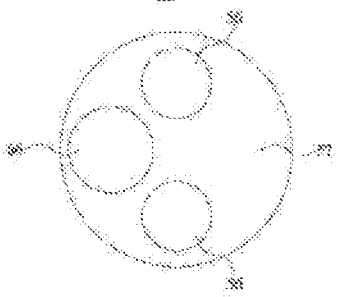
【図５】

図　５



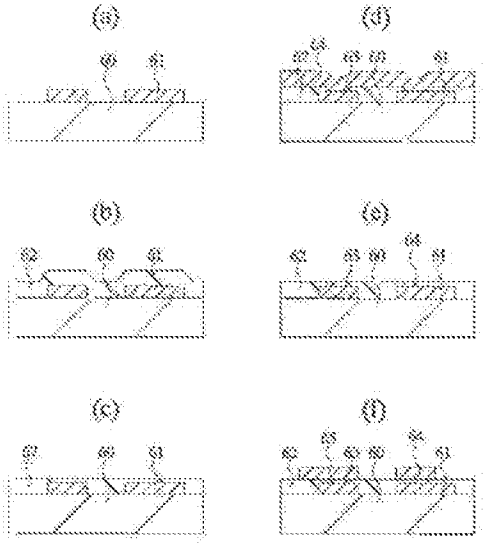
【図６】

図　６



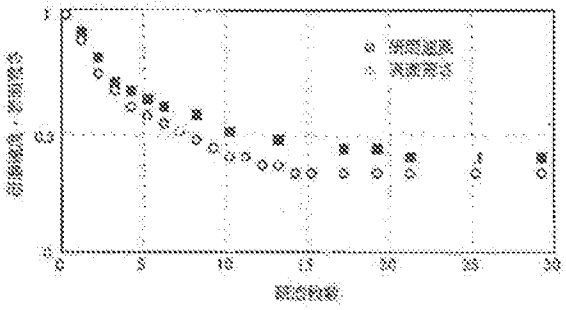
【図6】

図 6



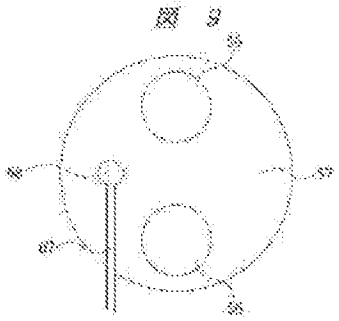
【図7】

図 7



【図9】

図 9



フロントページの続き

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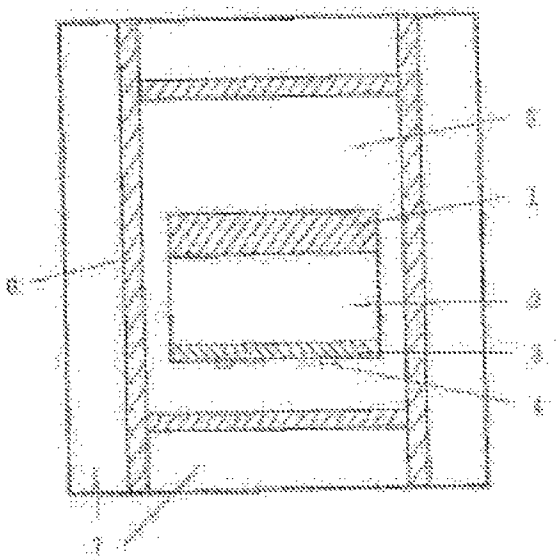
Bibliographic data: JP6182184 (A) — 1994-07-05

SYNTHESIS OF SINGLE CRYSTAL DIAMOND

Inventor(s):	SUMIYA HITOSHI; SATO SHUICHI +
Applicant(s):	SUMITOMO ELECTRIC INDUSTRIES +
Classification:	- **International:** *B01J3/06; C01B31/06; C30B29/04;* (IPC1-7): B01J3/06 - **European:**
Application number:	JP19920341940 19921222
Priority number (s):	JP19920341940 19921222
Also published as:	JP3259384 (B2)

Abstract of JP6182184 (A)

PURPOSE:To advantageously produce a colorless, transparent high-quality single-crystal diamond in the synthesis thereof employing temp. difference process by a method where Ti, etc., are added to a solvent metal as nitrogen getter, a metal plate such as made of Cu is interposed between the solvent metal and seed crystal and, in this condition, the synthesis of the diamond is started. CONSTITUTION:In the synthesis of a single crystal diamond employing temp. difference process, one or more metals selected as nitrogen getter from among Ti, Zr, Hf, V, Nb and Ta are added to a solvent metal 2, a metal plate 3 consisting of one or more metals selected from among Cu, Ag, Au, Zn and Cd is interposed between the solvent metal 2 and the seed crystal 4 and, in this condition, the diamond synthesis is started.; Since, in this way, a colorless, transparent high quality IIa type diamond almost free from inclusion can be synthesized inexpensively and stably, a synthetic diamond useable in accessory or optical part application can be produced advantageously.



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特開平6－182184
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(51)Int.Cl.'	識別記号	庁内整理番号	FI	技術表示箇所
B01J 3/06	R			
	S			

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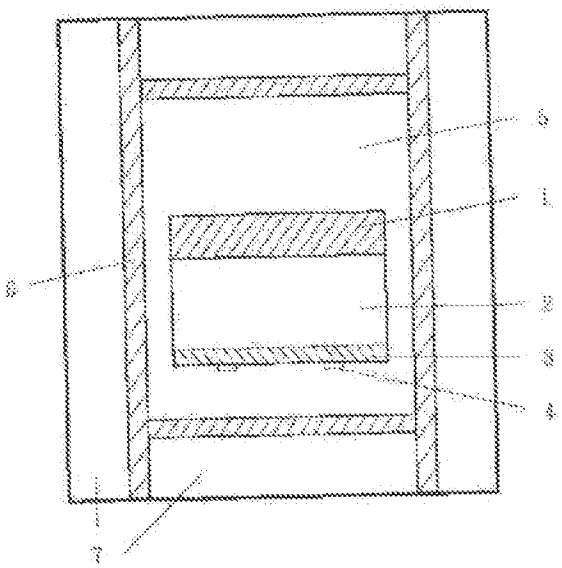
(21)出願番号　特願平4－349140

(22)出願日　平成4年(1992)12月22日

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(54)【発明の名称】　ダイヤモンド単結晶の合成方法

(57)【要約】
【目的】　装飾用途や光学部品などに用いることができる無色透明で良質なダイヤモンド単結晶の新規な合成方法を提供する。
【構成】　温度差法によるダイヤモンド単結晶合成において、溶媒金属に窒素ゲッターとしてTi，Zr，Hf，V，Nb，Taから選ばれる一種もしくは二種以上の金属を添加し、かつ、前記溶媒金属と融結晶との間にCu，Ag，Au，Zn，Cdから選ばれる一種もしくは二種以上からなる金属板を配置した状態でダイヤモンドの合成を開始することを特徴とする。これにより、無色透明でインクルージョンの発生しない良質なIIa型ダイヤモンド結晶を安価に且つ安定に合成できるので、装飾用途や光学部品に供し得る合成ダイヤモンドの有利な製法である。



【特許請求の範囲】
【請求項１】　温度差法によるダイヤモンド結晶合成において、溶媒金属に窒素ゲッターとしてＴｉ，Ｚｒ，Ｈｆ，Ｖ，Ｎｂ，Ｔａから選ばれる一種もしくは二種以上の金属を添加し、かつ、前記溶媒金属と種結晶との間にＣｕ，Ａｇ，Ａｕ，Ｚｎ，Ｃｄから選ばれる一種もしくは二種以上からなる金属板を配置した状態でダイヤモンドの合成を開始することを特徴とするダイヤモンド単結晶の合成方法。
【請求項２】　前記の溶媒金属と種結晶との間に配置される金属板の厚みは０．０１ｍｍ以上、１ｍｍ以下であることを特徴とする請求項１記載のダイヤモンド単結晶の合成方法。
【請求項３】　前記窒素ゲッターとして用いられるＴｉ，Ｚｒ，Ｈｆ，Ｖ，Ｎｂ，Ｔａから選ばれる一種もしくは二種以上の金属の含有量が、溶媒金属に対して０．２～１０重量％であることを特徴とする請求項１または２記載のダイヤモンド単結晶の合成方法。
【請求項４】　前記溶媒金属は、Ｆｅ，Ｃｏ，Ｎｉ，Ｍｎ，Ｃｒの中から選ばれる一種もしくは二種以上からなり、且つ０．１～５．０重量％の炭素を含むことを特徴とする請求項１または２または３記載のダイヤモンド単結晶の合成方法。
【発明の詳細な説明】
【０００１】
【産業上の利用分野】本発明は装飾用途や光学部品などに用いられる無色で透明なダイヤモンド単結晶の合成方法に関するものである。
【０００２】
【従来の技術】現在市販されている装飾用ダイヤモンドは、主に南アフリカ、ロシアより産出されるものの中から、無色透明で内部欠陥の少ないものを選別して用いられている。天然装飾用ダイヤモンドは宝石の中でも最も販売量が多い。また、ダイヤモンドを用いた光学部品として、レーザー窓やＩＲアンビルセルなどがあるが、いずれも天然原石の中から赤外領域に光の吸収のない透明なダイヤモンド（ＩＩａ型と呼ばれる）が選ばれて用いられている。しかし、透明無色な原石の産出は極めて少なく、安定供給や価格に問題がある。一方、人工合成によるダイヤモンドは通常、超高圧高温下で合成する際に溶媒中の窒素が結晶格子内に取り込まれるために黄色く着色してしまうが、溶媒中に窒素ゲッターを添加することで無色透明のダイヤモンドを得ることができる。この窒素ゲッターとしては、たとえば、The Journal of Physical Chemistry, vol.79, No.12 (1975) p1338 に示されているように、Ａｌがよく知られている。具体的には、米国特許第４０３４０６６号明細書には、Ｆｅ溶媒にＡｌを３～５重量％添加することにより宝石級の無色透明なダイヤモンド単結晶が得られると記載されている。Ａｌ以外の窒素ゲッターを用いた例として、たとえば無機材質研究所研究報告書第３９号、ｐ１６～にＴｉやＺｒを溶媒金属に添加することで結晶中の窒素が除去されたという報告がある。
【０００３】
【発明が解決しようとする課題】しかし、特に無色透明の合成ダイヤモンドは合成コストが天然ダイヤモンド上りはるかに高くなるため工業生産は行われていない。この理由は、合成には高価で特殊な装置が必要である上に、Ａｌなどを窒素ゲッターとして添加した場合、その添加量の増加に従って窒素が結晶中に取り込まれ（以下インクルージョンと呼ぶ）て、不良結晶となることが多くなるため、良質な結晶とするためには成長速度を大幅に下げる必要があるからである。とくにＴｉやＺｒを窒素ゲッターとして用いた場合は合成中に溶媒中に生成したＴｉＣやＺｒＣなどの炭化物が原因でより多くのインクルージョンが結晶中に取り込まれるようになる。
【０００４】本発明者らが行った実験による結果では、窒素ゲッターとしてＡｌを用い、溶媒金属に均一混合した場合、無色透明なダイヤモンド結晶を合成するためには、その添加量は溶媒に対し少なくとも４重量％（約１２体積％）必要であるが、この場合インクルージョンの巻き込みなしに結晶成長させるためには成長速度を１ｍｇ／ｈｒ以下にする必要があった。この成長速度では、たとえば１カラット（２００ｍｇ）の結晶を合成するには２００時間以上の合成時間を要し、製造コストは膨大なものとなる。また、Ｔｉ，Ｚｒなど、Ａｌより窒素との反応性の高い物質を窒素ゲッターとして溶媒に均一添加した場合、添加量は１重量％でも無色透明な結晶となる。しかし、これらは炭化物を形成しやすく、成長速度を大幅に低下させたとしてもＴｉＣやＺｒＣなどの炭化物の影響で、良質な結晶は殆ど得られない。本発明はかかる問題点を解決し、無色透明でインクルージョンのほとんどない結晶を、安価にしかも安定して合成できる方法を提供し、人工合成ダイヤモンドの装飾用途又は光学部品用途への使用を可能とするものである。
【０００５】
【課題を解決するための手段】本発明者らは、ＴｉやＺｒを窒素ゲッターとして溶媒に添加して成長させたダイヤモンド結晶を観察したところ、インクルージョンのほとんどは種結晶を起点として連続して結晶中に取り込まれていることがわかった。さらに、詳細に調べると成長初期に種結晶直上に生成したＴｉＣやＺｒＣなどの炭化物がインクルージョン起点となっていることがわかった。そこで本発明者らは、成長初期に種結晶上に炭化物生成を防ぐため溶媒と種結晶の間にあらかじめＣｕ，Ａｇ，Ａｕ，Ｚｎ，Ｃｄなど、それ自体炭化物を作らず、加えて、ＴｉＣやＺｒＣやＮｂＣなどのＩＶａ族もしくはＶａ族元素の炭化物を分解する働きのある金属箔を配置したところ、種結晶上にほとんど炭化物を生成しないことを見いだした。その結果、炭化物を起点としたインクル

ージョンの巻き込みを防ぐことができ、このため、従来の２倍以上の早い成長速度でも、良質なＩＩａ結晶が得られることを確認した。

【０００６】本発明は温度差法によるダイヤモンド結晶合成において、溶媒金属に、窒素ゲッターとしてＴｉ、Ｚｒ、Ｈｆ、Ｖ、Ｎｂ、Ｔａから選ばれる一種もしくは二種以上の金属を添加し、さらに前記溶媒金属と種結晶との間にＣｕ、Ａｇ、Ａｕ、Ｚｎ、Ｃｄから選ばれる一種もしくは二種以上からなる金属板を配置した状態でダイヤモンドの合成を開始することを特徴とするものである。本発明において溶媒金属と種結晶との間に配置される金属板の厚みは０．０１ｍｍ以上、１ｍｍ以下であることが好ましい。また、窒素ゲッターとして用いられるＴｉ、Ｚｒ、Ｈｆ、Ｖ、Ｎｂ、Ｔａから選ばれる一種もしくは二種以上の金属の含有量は、溶媒金属に対して０．２～１０重量％であることが好ましい。窒素ゲッターとしてのこれら金属は、他の金属、例えば、Ａｌ、Ｃｕ、Ｓｉ、Ｓｎ等との合金の形で添加することもできる。また、溶媒金属は、Ｆｅ、Ｃｏ、Ｎｉ、Ｍｎ、Ｃｒの中から選ばれる一種もしくは二種以上からなり、且つ０．１～６．０重量％の炭素を含むことが好ましい。

【０００７】図１は本発明の一具体例であってダイヤモンド結晶合成用の試料室構成を示す縦断面図である。溶媒金属２には窒素ゲッターとしてＴｉ、Ｚｒ、Ｈｆ、Ｖ、Ｎｂ、Ｔａが添加され、この溶媒金属と種結晶４の間にＣｕ、Ａｇ、Ａｕ、Ｚｎ、Ｃｄから選ばれる金属板３が配置される。１は炭素源、５は絶縁体、６は黒鉛ヒーター、７は圧力媒体である。この金属板３の厚みは０．０１ｍｍ未満では効果は不十分で、１ｍｍをこえるとＣｕ、Ａｇ、Ａｕ、Ｚｎ、Ｃｄなどの金属がダイヤモンドの結晶成長に悪影響を及ぼし、結晶が多結晶化したり、煤の残しのある不良結晶となってしまう。また溶媒２に窒素ゲッターとして添加するＴｉ、Ｚｒ、Ｈｆ、Ｖ、Ｎｂ、Ｔａの添加量としては、０．２重量％未満では窒素は十分に除去されず、合成されたダイヤモンドは黄色み帯びた結晶となる。一方、１０重量％をこえると多結晶化や自発核発生が多くなり、良質なダイヤモンド結晶が得られなくなる。

【０００８】ここで、２の溶媒金属はＦｅ、Ｃｏ、Ｎｉ、Ｍｎ、Ｃｒの中から選ばれる一種もしくは二種以上からなる金属であり、種結晶溶解防止のための０．１～６．０重量％の炭素をあらかじめ添加しておくのが好ましい。炭素添加量が０．１重量％未満もしくは炭素を含まない溶媒金属を用いた場合、種結晶上にＰｔなどの種結晶溶解防止材を配置する必要があるが、このような種結晶溶解防止材を配置することは、多結晶化やインクルージョンの巻き込みの原因となり好ましくない。また、炭素添加量が６重量％をこえると、自然核発生がおこりやすくなり、種結晶以外の部分より結晶成長するため結晶同士が干渉し、良質の結晶が得られなくなる。

【０００９】【作用】本発明ではＴｉ、Ｚｒ、Ｈｆ、Ｖ、Ｎｂ、Ｔａなど、窒素との反応性の高い元素を窒素ゲッターとするため、少量の添加でほとんど窒素を含まない無色透明なＩＩａ型のダイヤモンド結晶が得られ、また、溶媒と種結晶の間に、炭化物生成防止のために、Ｃｕ、Ａｇ、Ａｕ、Ｚｎ、Ｃｄの金属板をあらかじめ配置した状態で合成を開始する構成になっているため、インクルージョンの混入を大幅に抑えることができる。その結果、従来よりかなり速い成長速度でも良質なＩＩａ型ダイヤモンド結晶が得られるようになる。また、本発明に用いる種結晶、炭素源等はこの種の技術分野で公知のものを用いることができる。また、温度条件による合成の条件等は適宜選択することができる。

【００１０】【実施例】以下実施例により本発明をさらに詳細に説明する。

実施例１

溶媒の原料として粒径５０～１００ミクロンの高純度Ｆｅ粉末、Ｃｏ粉末、グラファイト粉末を用い、

Ｆｅ：Ｃｏ：Ｃ＝６０：４０：４．５（重量比）

となるように配合した。これに、さらに窒素ゲッターとして平均粒径５０ミクロンのＴｉ粉末を１．５重量％添加し、十分に混合した。この混合粉末を型押し成形し、脱ガス、焼成したもの（直径２０ｍｍ、厚み１０ｍｍ）を溶媒とした。図１に示す試料室構成で炭素源１にはダイヤモンドの粉末、種結晶４には直径５００ミクロンのダイヤモンド結晶３個を用いた。種結晶４と溶媒２の間に配置する金属板３としては厚み０．３ｍｍのＣｕ板を用いた。そして、炭素源１と種結晶４部に約３０℃の温度差がつくように加熱ヒーター６内にセットした。これを超高圧発生装置を用いて、圧力５．５ＧＰａ、温度１３００℃で７０時間保持し、ダイヤモンドの合成を行った。その結果０．７～０．９カラットの無色透明な、インクルージョンのほとんどない良質なＩＩａ型のダイヤモンド結晶が得られた。ＥＳＲにより、結晶中の窒素濃度を測定するといずれも０．１ｐｐｍ以下であった。磁気天秤によりインクルージョン量を測定するといずれも０．３重量％以下であった。

【００１１】実施例２

種結晶４と溶媒２の間に配置する金属板３に厚み０．５ｍｍのＣｕ板を用いた他は実施例１と同様にしてダイヤモンドの合成を行った。その結果、実施例１とほとんど同じ良質なＩＩａ型ダイヤモンド結晶が得られた。

実施例３

種結晶４と溶媒２の間に配置する金属板３に厚み０．２ｍｍのＡｇ板を用いた他は実施例１と同様にしてダイヤモンドの合成を行った。その結果、実施例１とほとんど同じ良質なＩＩａ型ダイヤモンド結晶が得られた。

実施例４

種結晶４と溶媒３の間に配置する金属板３に厚み０．３ｍｍのＡｕ板を用いた他は実施例１と同様にしてダイヤモンドの合成を行った。その結果、実施例１とほとんど同じ良質なＩＩａ型ダイヤモンド結晶が得られた。

【００１２】実施例５

種結晶４と溶媒３の間に配置する金属板３に厚み０．０５ｍｍのＡｕ板を用いた他は実施例１と同様にしてダイヤモンドの合成を行った。その結果、実施例１とほとんど同じ良質なＩＩａ型ダイヤモンド結晶が得られた。

実施例６

種結晶４と溶媒３の間に配置する金属板３に厚み０．２ｍｍのＺｎ板を用いた他は実施例１と同様にしてダイヤモンドの合成を行った。その結果、実施例１とほとんど同じ良質なＩＩａ型ダイヤモンド結晶が得られた。

実施例７

溶媒２に添加する窒素ゲッターに粒径４４ミクロン以下（平均１０ミクロン）のＺｒ粉末を溶媒に対し３重量％添加した他は実施例１と同様にしてダイヤモンドの合成を行った。その結果、実施例１とほとんど同じ良質なＩＩａ型ダイヤモンド結晶が得られた。

【００１３】実施例８

溶媒２に添加する窒素ゲッターに粒径４４ミクロン以下（平均１０ミクロン）のＡｌＴｉ合金粉末を溶媒に対し３重量％添加した他は実施例１と同様にしてダイヤモンドの合成を行った。その結果、実施例１とほとんど同じ良質なＩＩａ型ダイヤモンド結晶が得られた。

実施例９

溶媒２に添加する窒素ゲッターとしてＡｌＴｉ合金粉末の代わりに、ＣｕＴｉ、ＳｎＴｉ又はSi_3Ti_5合金粉末を用いた他は実施例８と同様にしてダイヤモンドの合成を行った。その結果、いずれの場合も、実施例１とほとんど同じ良質なＩＩａ型ダイヤモンド結晶が得られた。

実施例１０

溶媒の原料として粒径５０～１００ミクロンの高純度Ｆｅ粉末、Ｎｉ粉末、Ｃｏ粉末、グラファイト粉末を用い、

Ｆｅ：Ｎｉ：Ｃｏ：Ｃ＝６０：３０：１０：４．２（重量比）

となるように配合した他は実施例１と同様にしてダイヤモンドの合成を行った。その結果、実施例１とほとんど同じ良質なＩＩａ型ダイヤモンド結晶が得られた。

【００１４】実施例１１

溶媒の原料として粒径５０～１００ミクロンの高純度Ｆｅ粉末、Ｎｉ粉末、Ｍｎ粉末、グラファイト粉末を用い、

Ｆｅ：Ｎｉ：Ｍｎ：Ｃ＝６０：３０：１０：４（重量比）

となるように配合した他は実施例１と同様にしてダイヤモンドの合成を行った。その結果、実施例１とほとんど同じ良質なＩＩａ型ダイヤモンド結晶が得られた。

実施例１２

溶媒の原料として粒径５０～１００ミクロンの高純度Ｆｅ粉末、Ｎｉ粉末、グラファイト粉末を用い、

Ｆｅ：Ｎｉ：Ｃ＝７０：３０：３．５（重量比）

となるように配合した他は実施例１と同様にしてダイヤモンドの合成を行った。その結果、実施例１とほとんど同じ良質なＩＩａ型ダイヤモンド結晶が得られた。

実施例１３

溶媒の原料として粒径５０～１００ミクロンの高純度Ｃｏ粉末、グラファイト粉末を用い、

Ｃｏ：Ｃ＝１００：４．７（重量比）

となるように配合した他は実施例１と同様にしてダイヤモンドの合成を行った。その結果、実施例１とほとんど同じ良質なＩＩａ型ダイヤモンド結晶が得られた。

【００１５】実施例１４

溶媒の原料として粒径５０～１００ミクロンの高純度Ｎｉ粉末、グラファイト粉末を用い、

Ｎｉ：Ｃ＝１００：４．２（重量比）

となるように配合した他は実施例１と同様にしてダイヤモンドの合成を行った。その結果、実施例１とほとんど同じ良質なＩＩａ型ダイヤモンド結晶が得られた。

【００１６】比較例１

溶媒と種結晶の間に金属板を配置せずに、他は実施例１と同様にダイヤモンドの合成を試みた。窒素含有量の少ない（約０．１ｐｐｍ）結晶が得られたが、インクルージョンの巻き込み量は約１．５重量％と多く、良質な結晶は得られなかった。

実施例１５

溶媒と種結晶の間に配置するＣｕ板の厚みを２ｍｍとした他は実施例１と同様にダイヤモンドの合成を試みた。成長したダイヤモンド結晶は表面に溶け残しのない単品状となった。

実施例１６

窒素ゲッターとして溶媒に添加するＴｉの量を１５重量％とした他は実施例１と同様にダイヤモンドの合成を試みた。種結晶から成長した結晶は多結晶化していた。

【００１７】実施例１７

溶媒の原料として粒径５０～１００ミクロンの高純度Ｆｅ粉末、Ｎｉ粉末、Ｃｏ粉末を用い、

Ｆｅ：Ｎｉ：Ｃｏ＝６０：３０：１０（重量比）

となるように配合し、グラファイトを添加しなかった他は実施例１と同様にしてダイヤモンドの合成を行った。その結果、種結晶は溶媒中に完全に溶解してしまい、ダイヤモンドの成長は不充分であった。

実施例１８

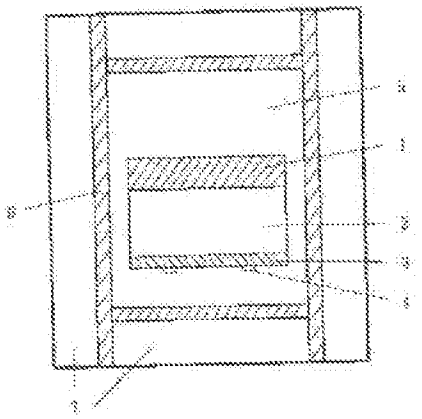
溶媒の原料として粒径５０～１００ミクロンの高純度Ｆｅ粉末、Ｎｉ粉末、Ｃｏ粉末、グラファイト粉末を用い、

Ｆｅ：Ｎｉ：Ｃｏ：Ｃ＝６０：３０：１０：７（重量

比）
となる上うに配合した他は実施例１と同様にしてダイヤモンドの合成を行った。その結果、種結晶以外の所よりダイヤモンドが多数自然発生し、このため結晶同士が干渉し、良質なダイヤモンド結晶はほとんど得られなかった。
【００１８】
【発明の効果】以上説明したように、本発明によれば、無色透明でインクルージョンのほとんどないダイヤモンド結晶を、安価に安定して合成できる。本方法によって合成ダイヤモンドを装飾用途、光学部品などに利用することが可能なものとなる。

【図面の簡単な説明】
【図１】図１は本発明の一具体例であって、結晶合成用の試料室構成を示す概略断面図である。
【符号の説明】
１：炭素源
２：溶媒金属（窒素ゲッター含む）
３：金属板
４：種結晶
５：絶縁体
６：黒鉛ヒーター
７：圧力媒体

【図１】



Espacenet

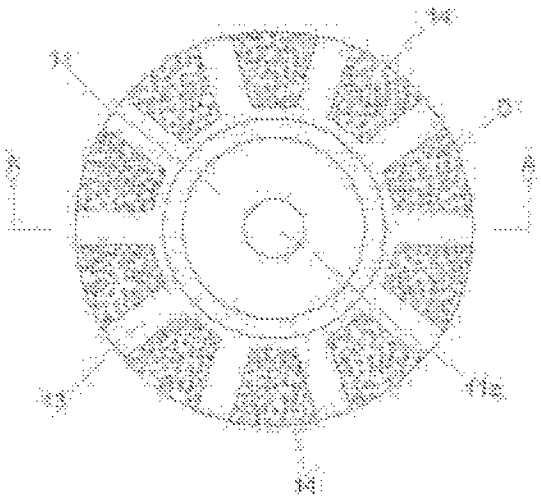
Bibliographic data: JP2004025401 (A) — 2004-01-29

DISC-SHAPED DIAMOND GRINDING WHEEL

Inventor(s):	NAGAYA AKIHIKO; KOTANI NORIOMI ±	
Applicant(s):	AIRTEC JAPAN KK; GOEI SEISAKUSHO KK ±	
Classification:	**- International:**	***B24D3/00; B24D3/06; B24D7/06;*** (IPC1-7): B24D3/00; B24D3/06; B24D7/06
	- European:	
Application number:	JP20020188194 20020627	
Priority number (s):	JP20020188194 20020627	

Abstract of JP2004025401 (A)

PROBLEM TO BE SOLVED: To provide a disc-shaped diamond grinding wheel hardly causing loading and having flexibility. ; SOLUTION: A base plate 11 of the disc diamond grinding wheel is made into a cup form of 10 cm in diameter having flexibility, and a jig mounting hole 11a to mount the base plate 11 on a grinder, etc. is provided on a central part of the base plate 11. Additionally, a rubber sheet 13 is adhered on a do-nut shaped part on a flat surface of a peripheral part of the base plate 11 , and a plurality of polishing chips 14 are adhered along a circumference under a deposition method coating diamond abrasive grains D on its upper layer. As they are supported by the base plate 11 excellent in flexibility, strength, etc. and the rubber sheet 13 transmits its elasticity to the polishing chips 14, the polishing chips 14 respectively and independently polish a polishing article along its surface and also polish the small area part and the curved surface. ; COPYRIGHT: (C)2004,JPO



Last updated: 14.03.2012 Worldwide Database 5.7.38; 93p

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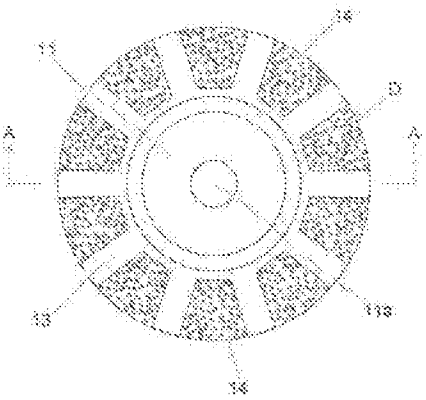
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Fターム(参考)　3C063 AA02 AB05 BA03 BB02 BC02 BF02 BG01 BG07 CC02 EE26

(54)【発明の名称】ディスク状ダイヤモンド砥石

(57)【要約】
【課題】目詰まりの起こし難く、可撓性を有するディスク状ダイヤモンド砥石を得る。
【解決手段】ディスク状ダイヤモンド砥石の基板１１は可撓性を有する直径１０cmのカップ状とされ、基板１１の中心部には基板１１をグラインダ等に取り付けるための治具取付孔１１ａが設けられている。また、基板１１の開口部の平面上のドーナツ状部分にはゴムシート１３が接着され、更にその上層にダイヤモンド砥粒Dを硬化した溶着法による複数個の研磨チップ１４が円周に沿って接着されている。可撓性、弾度等に優れた基板１１に支持され、ゴムシート１３がその弾力性を研磨チップ１４に伝えるため、研磨チップ１４はそれぞれ独立に被研磨物の面に沿って研磨でき、小断面部や曲面も研磨することができる。
【選択図】　図１



【特許請求の範囲】
【請求項１】
ダイヤモンド砥粒を塗布した複数個の金属片から成る研磨チップを可撓性材料から成るディスク状基板上の外周予定部全面に配列したことを特徴とするディスク状ダイヤモンド砥石。
【請求項２】
前記研磨チップは前記ディスク状基板の円周に沿って等間隔に配列したことを特徴とするディスク状ダイヤモンド砥石。
【請求項３】
前記研磨チップは前記ディスク状基板上に積層したゴム層上に接着した請求項１に記載のディスク状ダイヤモンド砥石。
【請求項４】
前記ダイヤモンド砥粒は溶着により前記研磨チップに塗布した請求項１に記載のディスク状ダイヤモンド砥石。

【発明の詳細な説明】
【０００１】
【発明に属する技術分野】
本発明は、グラインダ等の回転工具に取り付け、硬質材料表面に塗装された表面塗膜を剥離したり、金属表面の錆を除去したり、或いは研磨をするディスク状ダイヤモンド砥石に関するものである。
【０００２】
【従来の技術】
例えば、既設の橋梁や工場の煙突、タンク等の保守に際して、表面塗膜の補修をする場合において、それらの表面に塗装されている重防食系塗料の剥離がなかなか困難であり、二度、三度の手間を要する厄介事である。
【０００３】
通常では、グラインダ等の電動工具の回転により金属片を表面塗膜に打ち付けることにより面を荒らし、その後にディスク状サンドペーパによって塗膜を剥離する。
【０００４】
【発明が解決しようとする課題】
しかしながら上述の従来例においては、金属片を打ち付ける際に、その母材の鉄やコンクリートの打撃音や振動が激しく、母材を痛めることもある。また、ディスク状サンドペーパも重防食塗料により瞬時で目詰まりを起こしてしまい、交換頻度も多く、作業効率も極めて悪い。
【０００５】
通常の建物の外壁塗膜の剥離においても、塗料の違いこそあれ、目詰まりによる交換頻度を生ずる。目詰まりさえしなければ非常に長い砥粒が耐久であることは明白であるが、実際には砥粒は脱落しておらず研磨性能は十分あっても、目詰まりで砥石が使用できなくなるという問題がある。
【０００６】
現在、ダイヤモンド砥石を使用した研磨材としては、図４に模式的に示すような金属粉末を用いてダイヤモンド砥粒を混入して固めたメタルボンドと称される研磨剤１と、図５に示すように表面にメッキによる電着法により製造された研磨剤２が知られている。
【０００７】
しかしながら、前述のメタルボンド製の研磨剤１は、ダイヤモンド砥粒Ｄの突き出しが少なく、目詰まりするため塗膜剥離には不適である。後者の電着製の研磨剤２は砥粒の突き出しは多いが、ダイヤモンド砥粒Ｄとメッキの間に隙間が発生してメッキが剥離し易く、ダイヤモンド砥粒Ｄの脱落が多く目詰まりを生じ易い。
【０００８】
また、現状のディスク状ダイヤモンド砥石としては、カップ型のものが多く用いられてい

るが、金属製の台形状板の平坦部にスクルポント製ダイヤモンドを下記含有のブロックにして取り付けたものが多く、騒音、振動共に非常に激しい。

【０００９】

本発明の目的は、上述の問題点を解消し、金属片を打ち付ける作業を不要とし、目詰まりが少なく長時間の作業に耐え、作業音や振動が静かな可能性を有し、小面積部や曲面をも研磨することのできるディスク状ダイヤモンド砥石を提供することにある。

【００１０】

【課題を解決するための手段】

上記目的を達成するための本発明に係るディスク状ダイヤモンド砥石は、ダイヤモンド砥粒を含有した複数個の金属片から成る研磨チップを可撓性材料から成るディスク状基板上の外周下近傍表面に配列したことを特徴とする。

【００１１】

【発明の実施の形態】

本発明を図１～図３に図示の実施の形態に基づいて詳細に説明する。

図１は本実施の形態におけるディスク状ダイヤモンド砥石の平面図、図２は図１における Ａ－Ａ線に沿った断面図を示している。基板１１は例えばエポキシ樹脂材に繊維資料としてガラスファイバ材料を混合し、例えば直径１０ｃｍの可撓性を有するカップ状とされ、基板１１の中心部には基板１１をグラインダ等に取り付けるための取付孔１１ａが設けられている。また、基板１１の周辺部の平面状のドーナツ状部分１２にはゴムシート１３が接着され、更にその上層にダイヤモンド砥粒Ｄを含有した例えば１０個の研磨チップ１４が接着されている。

【００１２】

研磨チップ１４としては、特に含浸方法を用いてダイヤモンド砥粒Ｄを含有したものが好適である。含浸方法は自溶性合金タイプとブロンズタイプの２種があり、自溶性合金タイプはＮｉ－Ｃｒ系合金粉末、ブロンズタイプはブロンズ系粉末及びＴｉ等の活性金属粉末を、市販の真空用バインダと混合することにより、先ずペーストを作製する。続いて、所定の形状、例えば本実施の形態では、周辺の基板１１と同曲率にして展伏に打ち抜いた金属片上に、厚みが一定になるようにすり切り治具を使用して上述のペーストを塗布する。

【００１３】

ペーストを塗布すり切りした後に、塗布したペースト上に砥粒散等を用い、ダイヤモンド砥粒を散布する。なお、自溶性合金タイプの場合には市販の真空用バインダを金属片上に均一に塗布し、ダイヤモンド砥粒Ｄを散布した後に、Ｎｉ－Ｃｒ系合金粉末を振りかける方法でも同様なものが作製できる。

【００１４】

次に、ダイヤモンド砥粒Ｄを散布した金属片を乾燥機に入れ、２４時間程乾燥することにより、バインダに含まれている揮発成分を十分に除去する。続いて、この乾燥処理を行った金属片を真空炉にセットし、真空度が１０－２以上の高真空とした後に、自溶性合金タイプは１０００～１１００℃、ブロンズタイプは８００～９５０℃の温度で焼成を行う。これらの温度で焼成を行うことによりダイヤモンド砥粒Ｄをニッケル系合金、活性金属含有ブロンズ系合金等でそれぞれ良好に固着することができる。

【００１５】

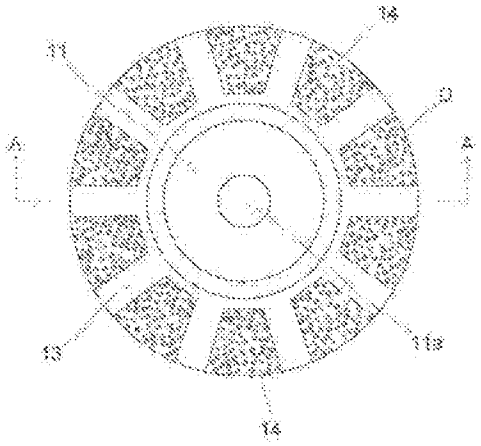
固着されたダイヤモンド砥粒Ｄは、図３の模式図に示すように活性金属と反応層Ｒを形成し母相と金属結合することにより、研磨チップ１４上からのダイヤモンド砥粒Ｄの突き出しが大きい。そして、研磨チップ１４は真空炉内の温度が室温に下った後に取り出す。

【００１６】

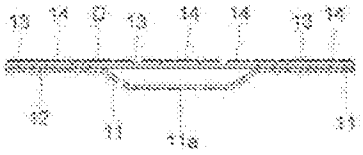
なお、研磨チップ１４のダイヤモンド砥粒Ｄの接着や電着等の塗布方式では、被研磨物に上って耐久性上問題があることもあり、基板１１にダイヤモンド砥粒Ｄを混入したメタルボンド製のブロックを、円弧や波状等に成形し貼り付けたものでも、振動や騒音を格段に緩和することができる。

【００１７】
ディスク状ダイヤモンド砥石を被研磨物の研磨に用いる際には、基板１１をグラインダ等の回転工具に取り付けて回転させ、被研磨物に研磨チップ１４を押し当てる。この際に、基板１１は柔軟性、屈曲性、強度等に優れた基板１１に支持されつつ、ゴムシート１３がその弾力性を研磨チップ１４に伝えるため、研磨チップ１４はそれぞれ独立に被研磨物の面に沿って研磨できるため、小径穴部や曲面においても、優れた研磨を行うことができる。
【００１８】
なお、本実施の形態においては研磨チップ１４はゴム層１３を介して取り付けたが、ゴム層１３を省略して基板１１に直接取り付けてもよい。また、研磨チップ１４は薄板の金属片上にダイヤモンドを散布したが、研磨チップ１４の形状は扇状に限らず、円形やその他の形状でも支障はない。
【００１９】
【発明の効果】
以上説明したように本発明に係るディスク状ダイヤモンド砥石は、特に、金属の面取りや硬質磁性鋼、塗料等の研磨・研削に有効であり、均一な広い面積や狭い幅をも容易に得ることができると共に、打撃音や振動を押さえ、作業者の労務上も、周囲への騒音問題についても、問題が発生することは少ない。
【００２０】
また、可撓性を有するための研磨チップが被研磨面に良く密着し、効率良く研磨作業をうことができる。更に、ダイヤモンド砥粒を電着法により被着すれば、電着法のように弾性があり、メタルボンド法に近い耐久性のあるものとなる。
【図面の簡単な説明】
【図１】ディスク状ダイヤモンド砥石の平面図である。
【図２】図１のＡ－Ａ線に沿った断面図である。
【図３】焼結による研磨チップの模式的断面図である。
【図４】メタルボンドによる研磨チップの模式的断面図である。
【図５】電着法による研磨チップの模式的断面図である。
【符号の説明】
１１　基板
１２　ドーナツ状部分
１３　ゴムシート
１４　研磨チップ
Ｄ　ダイヤモンド砥粒
Ｂ　反応層

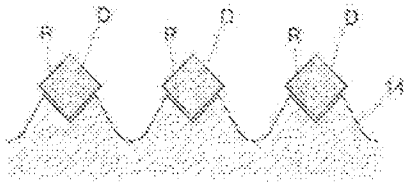
【図1】


14
11
D
A
A
13
11a
14

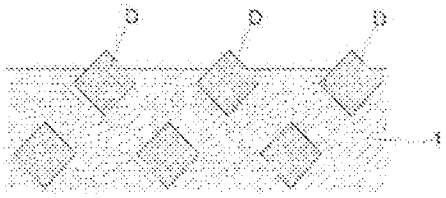
【図2】


13
14
D
13
14
14
13
14
12
11
11a
11

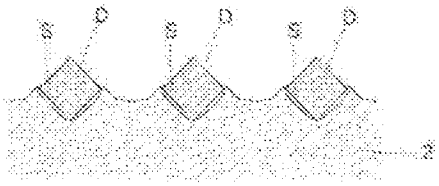
【図3】


R
D
R
D
R
D
14

【図4】


D
D
D
1

【図5】


S
D
S
D
S
D
2

Espacenet

Bibliographic data: JP2000343436 (A) — 2000-12-12

GRINDING WHEEL AND MANUFACTURE THEREOF

Inventor(s): TOGE NAOKI; NONOSHITA TETSUYA ± (TOGE NAOKI, ; NONOSHITA TETSUYA)

Applicant(s): NORITAKE DIAMOND IND; NORITAKE CO LTD ± (NORITAKE DIAMOND IND CO LTD, ; NORITAKE CO LTD)

Classification: **- international:** ***B24D3/00; B24D3/02; B24D3/06; B24D3/28; B24D5/00;*** (IPC1-7): B24D3/00; B24D3/02; B24D3/06; B24D3/28; B24D5/00

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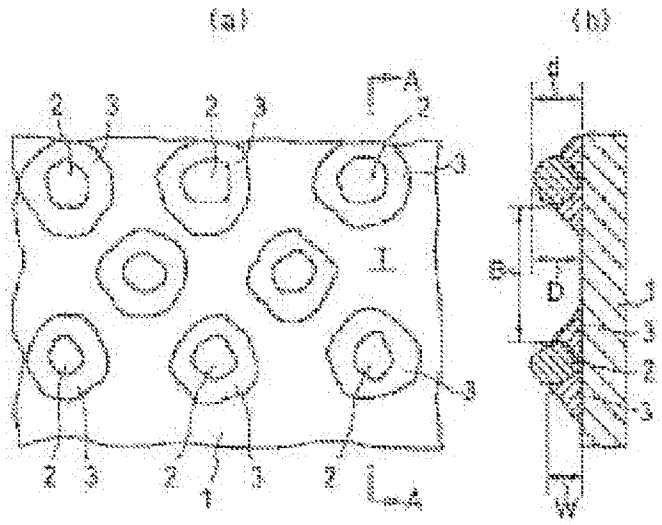
Application number: JP19990150470 19990528

Priority number(s): JP19990150470 19990528

Also published as: JP3387851 (B2)

Abstract of JP2000343436 (A)

PROBLEM TO BE SOLVED: To obtain long-term stable cutting quality through the excellent supply of a polishing solution and discharge of chips while obtaining the holding force and necessary protruding height of abrasive grains by setting wax material thickness to be specific times as large as an abrasive grain diameter, and forming a chip pocket with base metal matrix exposed with a width specific times as large as the abrasive grain diameter between the abrasive grains. SOLUTION: In arranging abrasive grains 2 in one layer on a base metal 2 for brazing, the abrasive grains 2 are held with a wax material thickness W of about 1/3-1/2 times as large as an abrasive grain diameter(d),and a chip pocket T with the matrix of the base metal 1 exposed with a width about 2/3-2.5 times as large as the abrasive grain diameter(d) between the adjacent abrasive grains 2 is regularly formed.; Such a wax material thickness W can ensure the protruding height of the abrasive grains 2 required to exhibit excellent cutting quality while ensuring the holding force of the abrasive grains 2, and the regular formation of the chip pocket T with the exposed matrix of the base metal 1 permits the uniform distribution of the abrasive grains 2 and excellent supply of a polishing solution and discharge of chips.


(a)
(b)
A
A
I
D
W

Espacenet

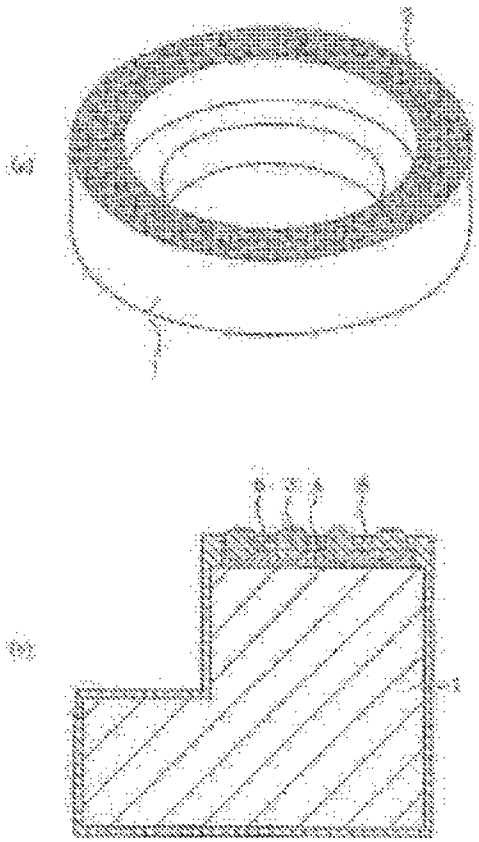
Bibliographic data: JP11077536 (A) — 1999-03-23

CONDITIONER FOR CMP AND ITS MANUFACTURE

Inventor(s):	HARA TOMOYOSHI ±
Applicant(s):	ASAHI DIAMOND IND ±
Classification:	- **International:** ***B24B53/12;*** (IPC1-7): B24B53/12 - **European:**
Application number:	JP19970255987 19970904
Priority number(s):	JP19970255987 19970904
Also published as:	JP3895840 (B2)

Abstract of JP11077536 (A)

PROBLEM TO BE SOLVED: To prevent a plated layer fixing diamond abrasive grains from being corroded even by highly acidic polishing liquid and to prevent contamination caused by metallic elution, and falling off of diamond abrasive grains. SOLUTION: This CPM conditioner has diamond abrasive grains 3 fixed by a plated layer 4, and is applied with nitric acid resistant plating to the plated layer 4; coated its surface with a synthetic resin, and having action tip parts of the diamond abrasive grains projected out of the synthetic resin layer 6. The CPM conditioner is manufactured by fixing the diamond abrasive grains 3 to an action surface by an electrodeposition or a reversible electroforming followed by nitric acid resistant plating 5 and covering with synthetic resin its action surface.



Last updated: 14.03.2012 Worldwide Database 6.7.38; 93p

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B24B 53/12		B24B 53/12	Z

審査請求　未請求　請求項の数3　FD　(全 9 頁)

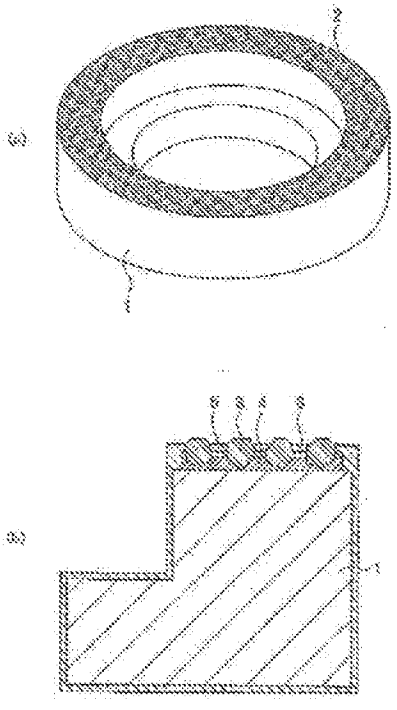
(21)出願番号　特願平9－255387

(22)出願日　平成9年(1997)9月4日

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(54)【発明の名称】　CMP用コンディショナ及びその製造方法

(57)【要約】
【課題】半導体ウェーハ研磨用のポリッシングマットのコンディショニングにおいて、親性の高い研磨液によってもダイヤモンド砥粒を固着するメッキ層が侵されるおそれがなく、金属の溶出によるコンタミネーションやダイヤモンド砥粒の脱落を生じないCMP用コンディショナ及びその製造方法を提供する。
【解決手段】ダイヤモンド砥粒がメッキ層により固着された本コンディショナにおいて、メッキ層上に耐酸性のメッキが施され、その表面がさらに合成樹脂により被覆され、ダイヤモンド砥粒の先端作用部が合成樹脂層より突出してなることを特徴とするCMP用コンディショナ、及び、作用面にダイヤモンド砥粒を電着法又は反転電着法によって固着したのち、さらに耐酸性のメッキを施し、その表面を合成樹脂により被覆することを特徴とする該CMP用コンディショナの製造方法。



【特許請求の範囲】
【請求項１】ダイヤモンド砥粒がメッキ層により固着された定盤において、メッキ層上に耐腐食性のメッキが施され、その表面がさらに合成樹脂により被覆され、ダイヤモンド砥粒の先端作用部が合成樹脂層より突出してなることを特徴とするＣＭＰ用コンディショナ。
【請求項２】耐腐食性のメッキが、アルミニウム、クロム、金、ロジウム、銀又はこれらの合金のメッキである請求項１記載のＣＭＰ用コンディショナ。
【請求項３】台用面にダイヤモンド砥粒を電着法又は反転電鋳法によって固着したのち、さらに耐腐食性のメッキを施し、その表面を合成樹脂により被覆することを特徴とする請求項１又は請求項２記載のＣＭＰ用コンディショナの製造方法。
【発明の詳細な説明】
【０００１】
【発明の属する技術分野】本発明は、ＣＭＰ用コンディショナ及びその製造方法に関する。さらに詳しくは、本発明は、半導体ウェーハ研磨用のポリッシングマットのコンディショニングにおいて、酸性の高い研磨液によってもダイヤモンド砥粒を固着するメッキ層が侵されず、ダイヤモンド砥粒が脱落するおそれのないＣＭＰ用コンディショナ及びその製造方法に関する。
【０００２】
【従来の技術】一般に半導体ウェーハの表面を研磨するウェーハ加工装置では、円盤状の定盤の上に研磨用ポリッシングマットを貼り付け、定盤上面に１枚又は複数枚のウェーハを載置し、これらウェーハをポリッシングマット上でキャリアにより強制回転させつつポリッシングマットとウェーハの間に微細な研磨粒子と研磨液を供給して、界面の化学的機械的作用によりケミカルメカニカルポリッシング（ＣＭＰ）を行っている。ポリッシングマットとしては、ポリエステル不織布にポリウレタン樹脂を含浸させたベロアタイプマット、ポリエステル不織布を素材としてその上に発泡ポリウレタン層を形成したスエードタイプマット、あるいは独立気泡を有する発泡ポリウレタンのマットなどや、さらにこれらの多層構造体などが使用されている。また、研磨粒子としては、酸化鉄、アルミナ、炭酸バリウム、酸化セリウム、コロイダルシリカなどが用いられ、研磨液には水酸化カリウム溶液、希硫酸、過酸化水素水、硝酸鉄水溶液など、それぞれの場合に応じて使い分けられる。このようなウェーハの研磨を繰り返すうちに、研磨粒子や研磨屑などがポリッシングマットの微細な孔に入り込んで目詰まりを起こしたり、研磨粒子と研磨液の化学反応によってポリッシングマットの表面が硬化して、研磨速度が低下してしまう。このため、ポリッシングマットの表面を再生して研磨速度を回復させる、いわゆるコンディショニングと呼ばれる操作を常時又は定期的に行う必要があり、このような操作にはＣＭＰ用コンディショナと呼ばれる工具が使用される。ダイヤモンド砥粒を埋めたコンディショニング材料であり、ダイヤモンド砥粒を利用した半導体ウェーハ研磨用のポリッシングマットのコンディショニングが検討されている。例えば、特開昭６４－７１６６１号公報には、ダイヤモンド砥粒と合金粉末を混合し、加熱焼結したダイヤモンドペレットを台面に貼り付けるか、あるいは、台面にダイヤモンド砥粒を均一に分布するように載せて電着した修正リングを用い、ポリッシングマットと修正リングを相対移動させることによりポリッシングマットの表面を研削して平坦度を高める方法が提案されている。しかし、合金粉末を用いて焼結したダイヤモンドペレットからは、コンディショニングの際に合金が溶出してウェーハの研磨時にウェーハを汚染するおそれがある。また、特開平４－３６４７３０号公報には、ウェーハ研磨装置の定盤に貼り付けられたポリッシングマットのコンディショニングに、ダイヤモンド砥粒をエポキシ樹脂に電着したペレットを用いる方法が提案されている。しかし、ペレットがメタルボンドの場合、金属が研磨液によって溶解され、半導体ウェーハに汚染して悪影響を及ぼすおそれがある。特に、一般にメタルボンドの主成分として使用される銅は悪影響が著しい。本発明者らは、先に半導体デバイスの層間絶縁膜及び金属配線などのＣＭＰ用のポリッシングマットのコンディショニングに際して、金属分による汚染を生じないコンディショナとして、台金作用面にメッキにより固着されたダイヤモンド砥粒層を有し、メッキされた金属が合成樹脂層により被覆され、ダイヤモンド砥粒の先端作用部が合成樹脂層より突出してなるコンディショナを提案した。このコンディショナは、ＣＭＰ用ポリッシングマットのコンディショニングに好適に使用されているが、さらに強い酸性条件下でのＣＭＰ加工においても、金属の溶出やダイヤモンド砥粒の脱落のおそれのないＣＭＰ用コンディショナが求められるようになった。
【０００３】
【発明が解決しようとする課題】本発明は、半導体ウェーハ研磨用のポリッシングマットのコンディショニングにおいて、酸性の高い研磨液によってもダイヤモンド砥粒を固着するメッキ層が侵されるおそれがなく、金属の溶出によるコンタミネーションやダイヤモンド砥粒の脱落を生じないＣＭＰ用コンディショナ及びその製造方法を提供することを目的としてなされたものである。
【０００４】
【課題を解決するための手段】本発明者は、上記の課題を解決すべく鋭意研究を重ねた結果、ダイヤモンド砥粒がメッキ層により固着されたコンディショナにおいて、メッキ層上に耐腐食性のメッキを施し、さらにその表面を合成樹脂で被覆することにより、強い酸性条件下でのＣＭＰ加工の際にも、金属の溶出によるコンタミネーションやダイヤモンド砥粒の脱落を生ずることがなく、ま

た。このようなコンディショナは、作用面にダイヤモンド砥粒を電着法又は反転電着法によって固着したのち、その表面に耐酸性のメッキを施し、さらに合成樹脂で被覆することにより、容易に製造し得ることを見いだし、この知見に基づいて本発明を完成するに至った。すなわち、本発明は、（１）ダイヤモンド砥粒がメッキ層により固着されたコンディショナにおいて、メッキ層上に耐酸性のメッキが施され、その表面がさらに合成樹脂により被覆され、ダイヤモンド砥粒の先端作用部が合成樹脂層より突出してなることを特徴とするＣＭＰ用コンディショナ、（２）耐酸性のメッキが、アルミニウム、クロム、金、ロジウム、銀又はこれらの合金のメッキである第（１）項記載のＣＭＰ用コンディショナ、及び、（３）作用面にダイヤモンド砥粒を電着法又は反転電着法によって固着したのち、さらに耐酸性のメッキを施し、その表面を合成樹脂により被覆することを特徴とする第（１）項又は第（２）項記載のＣＭＰ用コンディショナの製造方法、を提供するものである。さらに、本発明の好ましい態様として、（４）耐酸性のメッキが、金メッキである第（２）項記載のＣＭＰ用コンディショナ、（５）金メッキの厚さが２～１０μｍである第（４）項記載のＣＭＰ用コンディショナ、を挙げることができる。

【０００５】

【発明の実施の形態】本発明のＣＭＰ用コンディショナは、台金作用面にダイヤモンド砥粒がメッキ層により固着され、メッキ層上に耐酸性のメッキが施され、その表面がさらに合成樹脂層により被覆され、ダイヤモンド砥粒の先端作用部が合成樹脂層より突出してなるものである。図１(ａ)は、本発明のＣＭＰ用コンディショナの一態様の斜視図であり、図１(ｂ)は、図１(ａ)のコンディショナを中心線を通る平面で切断したときの切断部端面図である。図１に示すＣＭＰ用コンディショナは、カップ型の台金１の作用面にダイヤモンド砥粒がメッキにより固着されて、ダイヤモンド砥粒層２を形成している。ダイヤモンド砥粒３は、メッキ層４により台金１に固着され、メッキ層上に耐酸性のメッキ５が施され、その表面が合成樹脂層６により被覆され、ダイヤモンド砥粒の先端作用部が合成樹脂層より突出している。本発明において、耐酸性のメッキとは、２０㎜×２０㎜×０.２～３㎜の金属試験片を５重量％硝酸５０ml に２４時間浸漬したのち、ＪＩＳ Ｋ ０１２１にしたがって試液をフレーム原子吸光分析して、金属の溶出量が５μｇ／ml以下である金属のメッキをいう。このような金属としては、例えば、アルミニウム、クロム、金、ロジウム、銀などや、これらの金属の合金を挙げることができる。本発明のコンディショナは、ダイヤモンド砥粒がすべてメッキにより台金に強固に固着され、メッキ層上に耐酸性のメッキが施され、その表面がさらに合成樹脂層により被覆されているので、pHが２以下であるような強い酸性の研磨液を用いた場合でも、研磨液が合成樹脂層に浸透してダイヤモンド砥粒を固着しているメッキ層と直接接触することがなく、金属分の溶出やダイヤモンド砥粒の脱落を生ずるおそれがない。そのため、本発明のＣＭＰ用コンディショナは、半導体デバイスの層間絶縁膜及び金属配線などのＣＭＰ用のポリッシングマットのコンディショニングに使用したとき、脱落したダイヤモンド砥粒によるウェーハの傷発生することがなく、ＣＭＰ加工後のウェーハの洗浄工程を簡略化することができる。

【０００６】図２及び図３は、本発明のＣＭＰ用コンディショナの製造方法の一態様の説明図である。本態様においては、台金作用面にダイヤモンド砥粒をメッキにより仮固定し、ついでダイヤモンド砥粒をメッキ層により固着してダイヤモンド砥粒層を形成し、メッキ層上に耐酸性のメッキを施し、その表面をさらに合成樹脂により被覆し、ダイヤモンド砥粒の先端作用部を突出させる。ダイヤモンド砥粒をメッキにより仮固定する場合は、図２(ａ)に示すように、台金１のダイヤモンド砥粒固定面７を残して、絶縁性のマスキング材８で被覆する。台金をメッキ浴に浸漬し、ダイヤモンド砥粒固定面にダイヤモンド砥粒３を載置し、台金に陰極を接続し、メッキ浴に陽極を接続して、電気メッキを行う。使用するダイヤモンド砥粒に特に制限はないが、ＪＩＳ Ｂ ４１３０に規定する粒度１００／１２０～３０／４０の粒径を有する砥粒であることが好ましく、粒度６０／８０程度の粒径を有する砥粒であることがより好ましい。ダイヤモンド砥粒の粒径が、粒度１００／１２０に相当する粒径未満であると、被覆する合成樹脂層の厚さが薄くなって、耐酸性のメッキの表面を完全に被覆しきれなくなるおそれがある。ダイヤモンド砥粒の粒径が、粒度３０／４０に相当する粒径を超えると、ダイヤモンド砥粒が高価になって経済的に不利であるばかりでなく、コンディショニングの際にポリッシングマットが損傷するおそれがある。メッキする金属は、ダイヤモンド砥粒を仮固定することができるものであれば特に制限はなく、例えば、ニッケル、クロムなどを前後に使用することができる。ダイヤモンド砥粒が仮固定され、ダイヤモンド砥粒固定面から脱落しない状態になれば、余剰のダイヤモンド砥粒を除去する。図２(ｂ)は、ダイヤモンド砥粒が、ダイヤモンド砥粒固定面に仮固定された状態を示す断面図である。ダイヤモンド砥粒を仮固定したとき、大部分のダイヤモンド砥粒はその一部が台金のダイヤモンド砥粒固定面に接した状態で仮固定されるが、台金のダイヤモンド砥粒固定面に接しない状態で付着し、浮き石９となっているダイヤモンド砥粒が存在する場合もあるので、仮固定を終了したのち、ダイヤモンド砥粒層の表面をアルミナ砥石、シリコンカーバイド砥石などを用いて軽く研磨することにより、浮き石を除去することが好ましい。図２(ｃ)は、浮き石を除去した状態を

示す断面図である。

【０００７】洗浄を終了したのち、台金をふたたびメッキ浴に浸漬し、台金に陰極を接続し、メッキ液に陽極を接続して、ダイヤモンド砥粒固定面のメッキを行い、ダイヤモンド砥粒をメッキ層４により固着してダイヤモンド砥粒層を形成し、図２(d)に示す状態とする。メッキする金属は、ダイヤモンド砥粒を固定することができるものであれば特に制限はなく、例えば、ニッケル、クロムなどを使用することができるが、特にニッケルが好ましい。ニッケルメッキによりダイヤモンド砥粒を固着する場合、添加剤を加えたスルファミン酸ニッケル浴を使用すると、ニッケルの硬度はＨＶ４００～６００、伸び率は１～５％となり、ニッケル固着層は十分な靭性を有する。ダイヤモンド砥粒を固着するメッキ層の厚さは、ダイヤモンド砥粒の粒径の４０％以上であることが好ましく、５０％以上であることがより好ましい。メッキ層の厚さがダイヤモンド砥粒の粒径の４０％未満であると、ダイヤモンド砥粒を保持する力が不足するおそれがある。ダイヤモンド砥粒をメッキ層により固着してダイヤモンド砥粒層を形成したのち、さらにメッキ層上に耐硝酸性のメッキを施す。耐硝酸性のメッキを施す方法には特に制限はなく、メッキする金属に応じてそれぞれ公知の方法を適宜選択することができる。例えば、耐硝酸性のメッキが金メッキである場合は、金シアン化カリウムを用いるシアン化物メッキ浴法や塩化金を用いるリン酸塩浴法などを挙げることができる。金は最も貴な金属であるので、台金をメッキ浴に浸漬するだけで置換によって表面に金が析出する。このような析出物は素地金属との密着力が弱く剥離しやすいので、台金に陰極を空気中で接続し、通電を始めてから手早くメッキ浴に浸漬することが好ましい。使用する陽極の材質には特に制限はなく、例えば、純質炭素、ステンレス鋼、ニクロム鋼などを使用することができる。金のメッキを続けると、時間の経過にしたがって析出結晶の粗大になるので、時々台金をメッキ浴から取り出して綿布で摩擦し、ふたたびメッキを繰り返すことにより、平滑なメッキ面を得ることができる。金メッキの厚さは、２～１０μｍであることが好ましく、３～６μｍであることがより好ましい。金メッキの厚さが２μｍ未満であると、強い靭性条件下でダイヤモンド砥粒を固着するメッキ層が侵されるおそれがある。金メッキの厚さは１０μｍで十分であり、１０μｍを超える金メッキは経済的は必要はない。図３(a)は、メッキ層上に耐硝酸性のメッキ５が施された状態を示す断面図である。メッキ層上に耐硝酸性のメッキを施したのち、台金よりマスキング材を外して洗浄、乾燥し、合成樹脂層により耐硝酸性のメッキの表面を被覆する。使用する合成樹脂には特に制限はなく、熱硬化性樹脂及び熱可塑性樹脂のいずれをも使用することができる。熱硬化性樹脂としては、例えば、エポキシ樹脂、ポリウレタン樹脂、不飽和ポリエステル樹脂、フッ素樹脂などを挙げることができる。熱可塑性樹脂としては、例えば、フッ素樹脂、ポリアミド樹脂、ポリイミド樹脂などを挙げることができる。また、本発明においては、樹脂の機械的強度を向上させるために、非導電性の無機化合物粒子やファイバーを加えることもできる。熱硬化性樹脂としては、加熱硬化型のほかに常温硬化型の樹脂も使用することができ、さらに、熱硬化性樹脂に代えて光硬化性樹脂も使用することができる。

【０００８】図３(b)は、耐硝酸性のメッキの表面を、さらに合成樹脂層で被覆した状態を示す断面図である。合成樹脂層もで被覆したのち合成樹脂層の表面部分を除去することにより、ダイヤモンド砥粒３の先端作用部を突出させる。ダイヤモンド砥粒の先端作用部を突出させる方法に特に制限はなく、例えば、ダイヤモンド砥粒の最突出部が見えるまで回転盤などを用いて合成樹脂層を除去したのち、一般砥石などによるドレッシング、鏡面などの定盤上でのシリコンカーバイドやアルミナなどの遊離砥粒を用いたドレッシング、ショットブラストなどにより、ダイヤモンド砥粒の突出量を容易に所定の値とすることができる。ダイヤモンド砥粒は、メッキによって強固に台金に固着されているので、ダイヤモンド砥粒に強い力がかかっても、コンディショナからダイヤモンド砥粒が脱落するおそれがない。図３(c)は、合成樹脂層の表面部分を除去してダイヤモンド砥粒の先端作用部が合成樹脂層６より突出し、かつ耐硝酸性のメッキの表面が合成樹脂層に覆われて露出しない状態を示す断面図である。

【０００９】図４及び図５は、本発明のＣＭＰ用コンディショナの製造方法の他の態様の説明図である。本態様においては、反転型のダイヤモンド砥粒固定面にダイヤモンド砥粒をメッキにより固定し、さらにダイヤモンド砥粒をメッキ層により埋め込んだのち、必要に応じてメッキ層を平坦化し台金の作用面に接合する。その後、反転型を除去し、メッキ層の表面部分を除去してダイヤモンド砥粒を露出させ、メッキ層上に耐硝酸性のメッキを施し、その表面をさらに合成樹脂により被覆し、ダイヤモンド砥粒の先端作用部を突出させる。ダイヤモンド砥粒をメッキにより固定する場合は、図４(a)に示すように、反転型１０のダイヤモンド砥粒固定面７を残して、絶縁性のマスキング材９で被覆する。反転型をメッキ浴に浸漬し、ダイヤモンド砥粒固定面にダイヤモンド砥粒３を装着し、反転型に陰極を接続し、メッキ液に陽極を接続して、電気メッキを行う。使用するダイヤモンド砥粒に特に制限はないが、ＪＩＳ Ｂ ４１３０に規定する粒度１００／１２０～５０／６０の粒径を有する砥粒であることが好ましく、粒度６０／８０程度の粒径を有する砥粒であることがより好ましい。ダイヤモンド砥粒の粒径が、粒度１００／１２０に相当する粒径より小さいと、被覆する合成樹脂層の厚さが薄くなって、耐硝酸性のメッキの表面を完全に被覆しきれなくなるおそれ

がある。ダイヤモンド砥粒の粒径が、粒度３０／４０に相当する粒径より大きいと、ダイヤモンド砥粒が急峻になって経済的に不利であるばかりでなく、コンディショニングの際にポリッシングマットが損傷するおそれがある。メッキする金属は、ダイヤモンド砥粒を仮固定することができるものであれば特に制限はなく、例えば、ニッケル、クロムなどを好適に使用することができる。ダイヤモンド砥粒が仮固定され、ダイヤモンド砥粒固定面から脱落しない状態になれば、余剰のダイヤモンド砥粒を除去する。図４（ｂ）は、ダイヤモンド砥粒が、ダイヤモンド砥粒固定面に仮固定された状態を示す断面図である。

【００１０】余剰のダイヤモンド砥粒を除去したのち、反転型をふたたびメッキ液に浸漬し、反転型に陰極を接続し、メッキ液に陽極を接続して、ダイヤモンド砥粒固定面のメッキを行い、ダイヤモンド砥粒をメッキ層４により埋め込んでダイヤモンド砥粒層を形成し、図４（ｃ）に示す状態とする。メッキする金属は、ダイヤモンド砥粒を埋め込んで固着することができるものであれば特に制限はなく、例えば、ニッケル、クロムなどを使用することができるが、特にニッケルが好ましい。ニッケルメッキによりダイヤモンド砥粒を埋め込む場合、添加剤を加えたスルファミン酸ニッケル浴を使用すると、ニッケルの硬度はＨＶ４００～６００、伸び率は１～５％となり、ニッケル固着層は十分な靭性を有する。ダイヤモンド砥粒をメッキにより埋め込んでダイヤモンド砥粒層を形成した反転型は、マスキング材を外して洗浄、乾燥し、必要に応じて、反転型上のメッキ層の表面を平坦に加工する。次いで、図４（ｄ）に示すように、反転型１０上のメッキ層４を台金１に接合する。反転型上のメッキ層４若しくは台金１のダイヤモンド砥粒層接合面又はその双方に接着剤を塗付し、メッキ層とダイヤモンド砥粒層接合面を合わせて固定し、メッキ層を台金に接合する。使用する接着剤は、コンディショナの使用に十分な強度を有するものであれば特に制限はなく、例えば、エポキシ接着剤などを好適に使用することができる。必要に応じて、接着剤に無機充填材、例えば、アルミニウム粉末などを混合することができる。メッキ層４を台金１に接合したのち、反転型１０を除去する。反転型を除去する方法には特に制限はなく、例えば、研磨加工、フライス盤加工などを挙げることができるが、研磨加工を特に好適に使用することができる。図５（ａ）は、メッキ層４を台金１に接合し、反転型を除去した状態を示す。

【００１１】台金に接合したメッキ層は、金属の表面部分を除去してダイヤモンド砥粒を露出させる。ダイヤモンド砥粒を露出させる方法には特に制限はなく、例えば、一般砥石などによるドレッシング、研磨などの定盤上でのシリコンカーバイドやアルミナなどの遊離砥粒によるドレッシング、ショットブラスト、金属溶解液による化学エッチング、電解エッチングなどにより行うことができる。化学エッチング処理は、台金及びダイヤモンド砥粒層を形成する材料のうち、メッキされた金属のみを溶解する薬剤に浸漬することにより行うものである。このような薬剤としては、金属がニッケルである場合はエンストリップＮＰ［メルテックス(株)製］、金属がクロムである場合はエンストリップＣＲ－５［メルテックス(株)製］などの市販されている薬剤を使用することができる。図５（ｂ）は、メッキ層４の表面部分を除去して、ダイヤモンド砥粒３を露出させた状態を示す。ダイヤモンド砥粒を露出させたメッキ層は、次いでその上に耐硝酸性のメッキを施す。耐硝酸性のメッキを必要としない台金の部分は、あらかじめマスキング材により保護することができる。耐硝酸性のメッキを施す方法には特に制限はなく、メッキする金属に応じてそれぞれ公知の方法を適宜選択することができる。例えば、耐硝酸性のメッキが金メッキである場合は、金シアン化カリウムを用いるシアン化物メッキ法や塩化金を用いるリン酸塩浴法などを挙げることができる。金は最も貴な金属であるので、台金をメッキ液に浸漬するだけで置換によって表面に金が析出する。このような析出物は素地金属との密着力が弱く剥離しやすいので、台金に陽極を常気中で接続し、通電を始めてから手早くメッキ液に浸漬することが好ましい。使用する陽極の材質には特に制限はなく、例えば、炭素鋼系、ステンレス鋼、ニクロム鋼などを使用することができる。金のメッキを続けると、時間の経過にしたがって析出結晶が粗雑になるので、時々台金をメッキ液から取り出して刷毛で擦過し、ふたたびメッキを繰り返すことにより、平滑なメッキ面を得ることができる。金メッキの厚さは、２～１０μｍであることが好ましく、３～６μｍであることがより好ましい。金メッキの厚さが２μｍ未満であると、強い酸性条件下でダイヤモンド砥粒を固着するメッキ層が侵されるおそれがある。金メッキの厚さは１０μｍで十分であり、１０μｍを超える金メッキは通常は必要はない。図５（ｃ）は、メッキ層４上に耐硝酸性のメッキ５が施された状態を示す断面図である。メッキ層上に耐硝酸性のメッキを施したのち、洗浄、乾燥し、合成樹脂層により耐硝酸性のメッキの表面を被覆する。使用する合成樹脂には特に制限はなく、熱硬化性樹脂及び熱可塑性樹脂のいずれをも使用することができる。熱硬化性樹脂としては、例えば、エポキシ樹脂、ポリウレタン樹脂、不飽和ポリエステル樹脂、フッ素樹脂などを挙げることができる。熱可塑性樹脂としては、例えば、フッ素樹脂、ポリアミド樹脂、ポリイミド樹脂などを挙げることができる。また、本発明においては、樹脂の機械的強度を向上させるために、非導電性の無機化合物粒子やファイバーを加えることもできる。熱硬化性樹脂としては、加熱硬化型のほかに常温硬化型の樹脂も使用することができ、さらに、熱硬化性樹脂に代えて光硬化性樹脂も使用することができる。

【００１２】図５(d)は、導電性のメッキの表面を、さらに合成樹脂層で被覆した状態を示す断面図である。合成樹脂層で被覆したのち合成樹脂層の表面部分を除去することにより、ダイヤモンド砥粒の先端作用部を突出させる。ダイヤモンド砥粒の先端作用部を突出させる方法に特に制限はなく、例えば、ダイヤモンド砥粒の最突出部が見えるまで研削盤などを用いて合成樹脂層を除去したのち、一般砥石などによるドレッシング、鏡研などの定盤上でのシリコンカーバイドやアルミナなどの遊離砥粒を用いたドレッシング、ショットブラストなどにより、ダイヤモンド砥粒の突出量を容易に所定の値とすることができる。ダイヤモンド砥粒は、メッキによって強固に固着されているので、ダイヤモンド砥粒に強い力がかかっても、コンディショナからダイヤモンド砥粒が脱落するおそれがない。図５(e)は、合成樹脂層の表面部分を除去してダイヤモンド砥粒の先端作用部を合成樹脂層より突出し、かつ導電性のメッキの表面が合成樹脂層に覆われて露出しない状態を示す断面図である。本発明により製造されたコンディショナは、ダイヤモンド砥粒の仮固定の際に、ダイヤモンド砥粒が反転型のダイヤモンド砥粒固定面に倣って固定され、ダイヤモンド砥粒層を台金に接合したとき、ダイヤモンド砥粒の最突出部は反転型のダイヤモンド砥粒固定面に接した部分であるので、コンディショナにおいて、各ダイヤモンド砥粒の最突出部が完全に同一平面上に存在する構造となる。また、ダイヤモンド砥粒層と台金の接合後の研削回転等により、ダイヤモンド砥粒層面の振れ精度は数μｍである。ニッケルの電鋳によりダイヤモンド砥粒を固着する場合、添加剤を加えたスルファミン酸ニッケル浴を使用すると、ニッケルの硬度はＨＶ４００～６００、伸び率は１～５％となり、ニッケル固着層は十分な靭性を有する。このために、ポリッシングマットのコンディショニングを精度よく能率よく行うことができ、ダイヤモンド砥粒の脱落が長期にわたって生じない。

【００１３】合成樹脂によりダイヤモンド砥粒層を覆う方法によれば、数ミリ程度の厚さの合成樹脂層も短時間で形成することができ、研削盤や超音波槽による合成樹脂の除去も短時間で精度よく行うことができる。本発明のＣＭＰ用コンディショナにおいて、ダイヤモンド砥粒の突出量は、ダイヤモンド砥粒の平均粒径の５～５０％であることが好ましく、平均粒径の２０～３０％であることがより好ましい。ダイヤモンド砥粒の突出量が平均粒径の５％未満であると、コンディショナのコンディショニング作用が不充分になるおそれがある。ダイヤモンド砥粒の突出量が平均粒径の５０％を超えると、コンディショニング作用が強すぎてポリッシングマットを損傷し、ダイヤモンド砥粒が脱落するおそれがある。本発明方法においては、必要に応じてさらにラップ加工を行うことができる。ラップ加工は、ダイヤモンド砥石を用いて突出したダイヤモンド砥粒の先端部をカットするものであり、ラップ加工を行うことにより、コンディショナに、使用当初から安定した優れた性能を発揮させることができる。本発明のＣＭＰ用コンディショナによれば、導電性のメッキがバリアとなって、ｐＨの低い酸性の強い研磨液を用いても、メッキ層の金属と研磨液が接触することがないので、ダイヤモンド砥粒が脱落してウェーハを傷つけたり、金属分が溶出してウェーハを汚染することがない。さらに、本発明のコンディショナはダイヤモンド砥粒の最突出部の平坦性に優れるので、ポリッシングマットの平坦度が向上し、ポリッシングマットの切れ味が向上する。

【００１４】

【実施例】以下に、実施例をあげて本発明をさらに詳細に説明するが、本発明はこれらの実施例により何ら限定されるものではない。

実施例１

寸法が１２７Ｄ－５Ｗ－２０Ｔ－３０Ｈの台金を、ステンレス鋼（ＳＵＳ３０４）を旋盤加工することにより作製した。次に、ダイヤモンド砥粒固定面を残して、表面を絶縁テープ及び塗料によりマスキングした。台金のアルカリ脱脂処理を行い、塩化ニッケル２４０ｇ／リットル及び硫酸１００ｇ／リットルを含有する前処理液に浸漬し、電流密度１０Ａ／ｄｍ²で常温にて陰極に台金をセットし２分間電解エッチングしたのち、陰極に台金をセットしてストライクメッキを３分間行った。次いで、スルファミン酸ニッケルメッキ浴で電流密度１Ａ／ｄｍ²で１５分間メッキを行い、下地メッキ層を３μｍ形成した。次に、ダイヤモンド砥粒固定面に粒度＃６０／８０、平均粒径２５０μｍのダイヤモンド砥粒を載置し、メッキ盛りと硬度調節のための添加剤を加えたスルファミン酸ニッケルメッキ浴を用い、電流密度０．５Ａ／ｄｍ²で３時間メッキを行い、ダイヤモンド砥粒一層分を仮固定した。台紙のダイヤモンド砥粒を拭い落とし、電流密度１Ａ／ｄｍ²で１時間埋め込みメッキを行ったのち、＃１００アルミナ砥石を当て、研き石となっているダイヤモンド砥粒を除去した。次いで、塩化ニッケル２４０ｇ／リットル及び硫酸１００ｇ／リットルを含有する前処理液に浸漬して陰極に台金をセットし、電流密度１０Ａ／ｄｍ²で常温にて３０秒電解エッチングしたのち、陰極に台金をセットしてストライクメッキを２分間行った。次に、スルファミン酸ニッケルメッキ浴で電流密度１Ａ／ｄｍ²で１５分間メッキを行い、さらに同じスルファミン酸ニッケルメッキ浴で、電流密度１Ａ／ｄｍ²で５時間メッキを行い、ダイヤモンド砥粒を固着するよう厚さ約１２５μｍのメッキを施した。ダイヤモンド砥粒固定面及び台金を洗浄したのち、シアン化金２．５ｇ／リットル、シアン化カリウム３０．０ｇ／リットル、第二リン酸ナトリウム１５．０ｇ／リットル及びシアン化コバルト１０．０ｇ／リットルを含むメッキ浴に浸漬し、ステンレス鋼を陰極とし、浴温５０℃で、電流密

度０．２Ａ／ｄｍ²、電圧２Ｖの条件で２０時間メッキを行い、厚さ４μｍの金合金層を形成した。金合金の組成は、金９９．７重量％、コバルト０．３重量％であった。マスキングをすべて外し、ダイヤモンド電着層が形成された台金を洗浄、乾燥したのち、フッ素樹脂塗料［日鍍硬質工業(株)、ＰＦＡ］を、硬膜厚さが１００μｍとなるように塗付して、３９０℃で３０分間焼き付けた。その後、アルミナ砥粒＃８００を用いてスエードタイプの研磨マットで、ダイヤモンド砥粒先端部が５０μｍ露出するまでラップ加工を行った。得られたＣＭＰ用コンディショナを５重量％硝酸に２４時間浸漬したが、金属分の溶出は全く認められなかった。また、このＣＭＰ用コンディショナを研磨マット（ＩＣ１０００）を用い、スラリー（硝酸鉄＋硝酸＋アルミナ）でＣＭＰ加工を行ったところ、金属膜ウェーハの研磨レート１，８７０Å／分という良好な結果が得られた。

実施例２

寸法が２８０Ｄ－６Ｗ－２０Ｔ－１４０Ｈの反転型（ＳＵＳ３０４）を、旋盤加工により作製した。ダイヤモンド砥粒固定面をマスキングテープで囲い、さらにその他のダイヤモンド砥粒層非接触部分のマスキング処理を行った。アルカリ脱脂処理したのち、反転型を、メッキ応力と硬度調節のための添加剤を加えたスルファミン酸ニッケル浴に浸め、粒度＃６０／８０のダイヤモンド砥粒をダイヤモンド砥粒固定面に載置した。電流密度１Ａ／ｄｍ²で２時間メッキを行い、ダイヤモンド砥粒の仮固定を行ったのち、余剰のダイヤモンド砥粒を除去し、さらに電流密度２Ａ／ｄｍ²で９６時間メッキを行い、３ｍｍの厚みのニッケル固着層を形成してダイヤモンド砥粒を完全に埋め込んだ。次いで、反転型上のニッケル固着層面を、旋盤加工により平坦に加工した。寸法２８６Ｄ－１２Ｗ－３５Ｔ－１６０Ｈの台金（ＳＵＳ３０４）を、旋盤加工により作製した。２液型エポキシ接着剤１０重量部にアルミニウム粉末５重量部を混合し、反転型上のニッケル固着層面と台金のダイヤモンド砥粒層接着部分に塗布し接合した。接合層が十分に硬化したのち、旋盤で反転型を正にして台金の裏面を切削して基準面を出した。さらに、台金を旋盤にくわえ、反転型を切除した。その後、ニッケル剥離液エンストリップＮＰ［メルテックス(株)］を用いて、ダイヤモンド電着層面のニッケルを約１２５μｍ溶解除去した。ダイヤモンド砥粒層が形成された台金を洗浄、乾燥したのち、ダイヤモンド砥粒層以外の部分をマスキング処理し、シアン化金２．５ｇ／リットル、シアン化カリウム３０．０ｇ／リットル、第二リン酸ナトリウム１５．０ｇ／リットル及びシアン化カリウム１０．０ｇ／リットルを含むメッキ浴に浸漬し、ステンレス鋼を陽極とし、浴温５０℃、電流密度０．２Ａ／ｄｍ²、電圧２Ｖの条件で３５時間メッキを行い、厚さ５μｍの金合金層を形成した。金合金の組成は、金９９．７重量％、コバルト０．３重量％であった。次いで、エポキシ塗料を硬膜厚さが１１０μｍとなるように塗付し、焼き付けた。その後、シリコンカーバイド＃８００を用いてエポキシ樹脂層約５０μｍを削り出し、ダイヤモンド砥粒の先端作用部をエポキシ樹脂層から突出させて、ＣＭＰ用コンディショナを得た。得られたＣＭＰ用コンディショナを５重量％硝酸に２４時間浸漬したが、金属分の溶出は全く認められなかった。また、このＣＭＰ用コンディショナを用いて、ポリエステル不織布を基剤とし、その上に発泡ポリウレタン層を形成したスエードタイプのポリッシングマットのコンディショニングを行った。平坦性に優れたコンディショニングを、効率よく行うことができた。

【００１５】

【発明の効果】本発明のＣＭＰ用コンディショナは、ｐＨ２以下のような強い酸性条件下でのＣＭＰ加工に使用しても、メッキ層から金属が溶出したり、ダイヤモンド砥粒が脱落するおそれがなく、安定してコンディショニングを行うことができる。

【図面の簡単な説明】

【図１】図１は、本発明のＣＭＰ用コンディショナの一態様の斜視図及び断面端面図である。

【図２】図２は、本発明のＣＭＰ用コンディショナの製造方法の一態様の説明図である。

【図３】図３は、本発明のＣＭＰ用コンディショナの製造方法の一態様の説明図である。

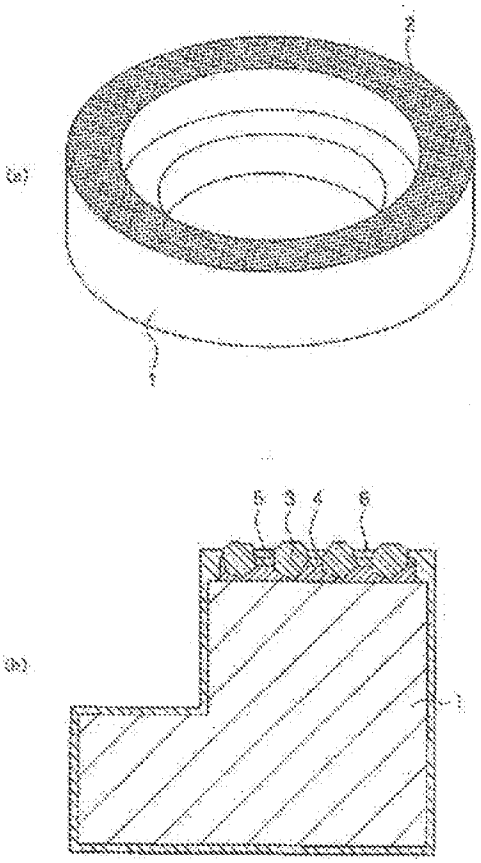
【図４】図４は、本発明のＣＭＰ用コンディショナの製造方法の他の態様の説明図である。

【図５】図５は、本発明のＣＭＰ用コンディショナの製造方法の他の態様の説明図である。

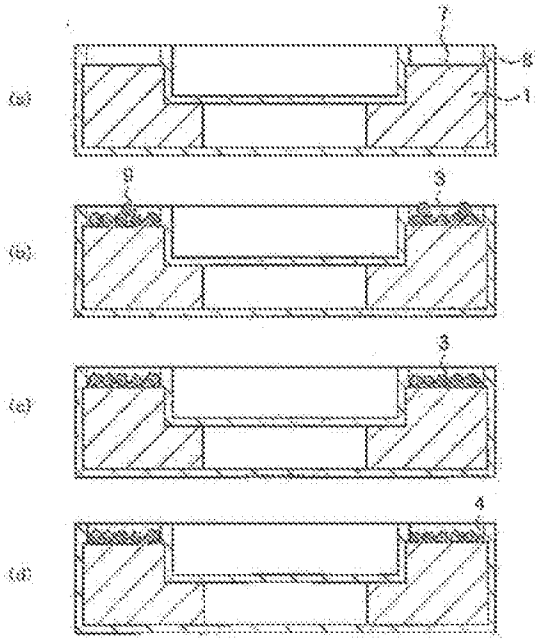
【符号の説明】

１　台金
２　ダイヤモンド砥粒層
３　ダイヤモンド砥粒
４　メッキ層
５　耐腐蝕性のメッキ
６　合成樹脂層
７　ダイヤモンド砥粒固定面
８　マスキング材
９　捨て石
１０　反転型

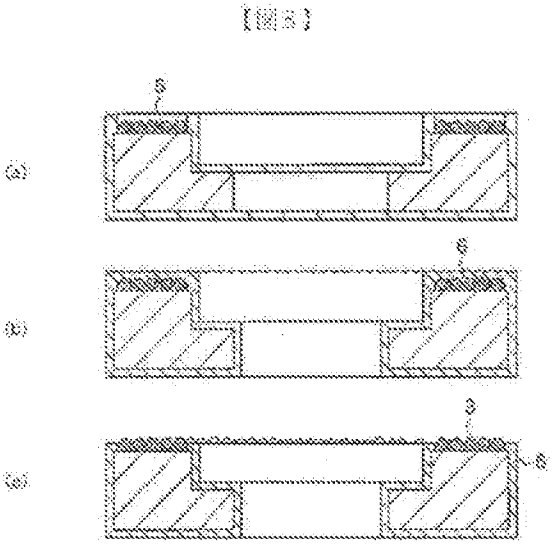
【図1】



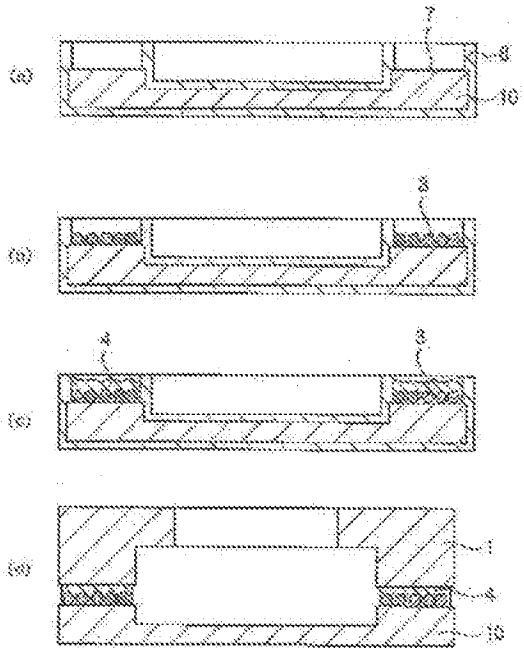
【図2】



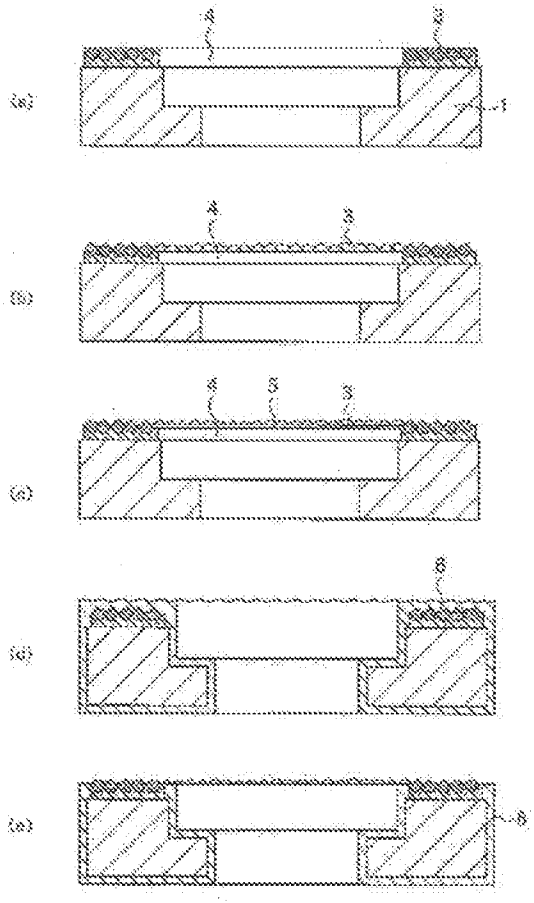
【図3】



【図4】



【図5】



Espacenet

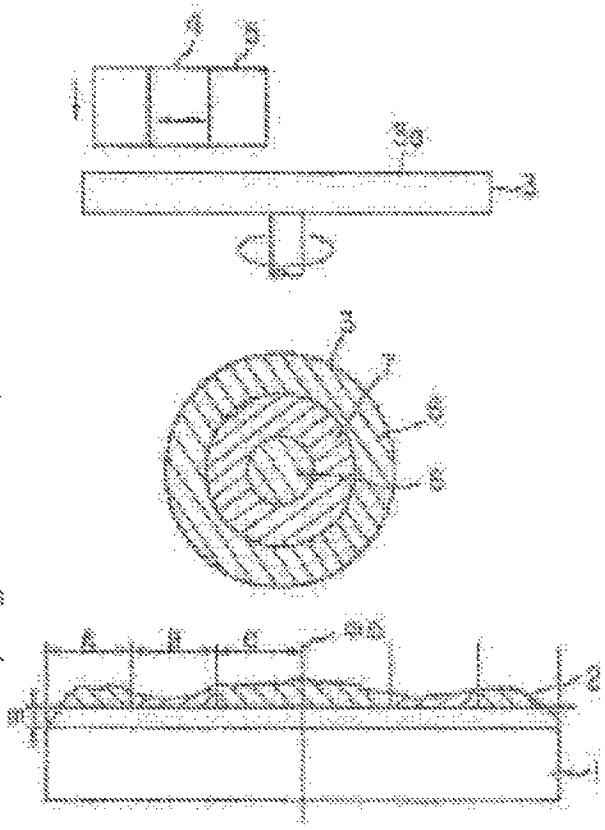
Bibliographic data: JP10180618 (A) — 1998-07-07

GRINDING PAD ADJUSTING METHOD FOR CMP DEVICE

Inventor(s):	TAKEUCHI NOBUYOSHI ±
Applicant(s):	NIPPON KOKAN KK ±

Classification:	- ***B24B37/00; H01L21/304;*** (IPC1-7): B24B37/00; H01L21/304 **- International:** **- European:**
Application number:	JP19960343567 19961224
Priority number (s):	JP19960343567 19961224

Abstract of JP10180618 (A)



PROBLEM TO BE SOLVED: To maintain the flatness of the device surface of a semiconductor substrate to be ground by dividing the surface of a pad according to the film thickness distribution of the device surface before grinding where the pad surface of the grinding pad is pressed, and adjusting each surface roughness of the pad surface to each different roughness with a plurality of grinding wheels different in hardness. SOLUTION: The film thickness distribution of a film 2 deposited on the device surface of a semiconductor substrate 1 takes a shape having a doughnut- like recess. According to this film thickness distribution, the inside of a pad surface 3a is divided into, for instance areas A, B and C. A grinding pad 3 needs to adjust surface roughness such that an outer peripheral pad surface part 6 and a center pad surface part 8 for grinding the areas A and C have grinding amounts larger than that of an inner peripheral pad surface part 7 for grinding the area B. Accordingly, a diamond grinding wheel 4 having hardness for reducing a grinding amount more than that of a diamond grinding wheel 5 is loaded, pressed to the pad surface 3a and, then by rotating the grinding pad 3, surface roughness is formed on the pad surface 3a.

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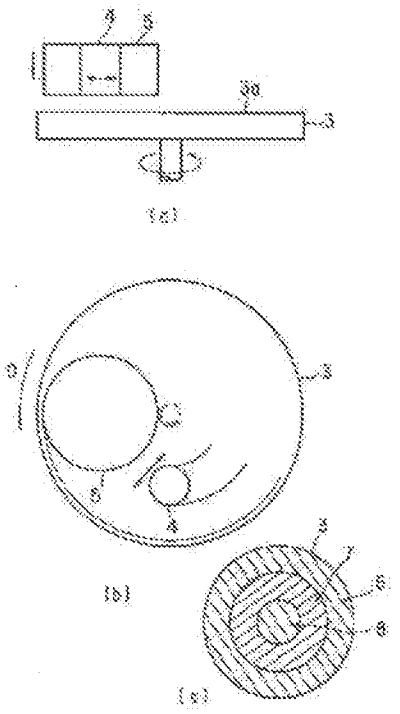
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(54)【発明の名称】　CMP装置の研磨パッドの調整方法

(57)【要約】
【課題】従来の研磨パッドの調整は、パッド面に単一的な表面荒れを形成していたため、半導体装置のデバイス面の凸部に押し付けられたパッド面が削られ、平坦性がなくなり、表面荒れに研磨の能力の差が生じていた。
【解決手段】本発明は、研磨する半導体基板のデバイス面の膜厚分布を研磨前に測定し、その膜厚分布に対応して、硬度が異なる複数のダイヤモンド砥石を用いて、パッド面内を区分して表面荒れの粗さをそれぞれ調整し、パッド面内での研磨量を変化させることにより、研磨パッドの平坦性を維持させ、研磨される半導体基板のデバイス面の平坦化するCMP装置の研磨パッドの調整方法である。



【特許請求の範囲】
【請求項１】　半導体基板の回路素子を形成するデバイス面を平坦化する化学的機械研磨（ＣＭＰ）装置に用いる研磨パッドの研磨を行うパッド面の調整方法において、
前記研磨パッドのパッド面を研磨前のデバイス面の段差分布に応じて、該パッド面内を区分して、硬度の異なる複数の砥石で、該パッド面の表面荒れを異なる粗さにそれぞれ調整することを特徴とするＣＭＰ装置の研磨パッドの調整方法。
【請求項２】　前記ＣＭＰ装置の研磨パッドの調整方法において、
前記デバイス面が平坦化されるまでの研磨量に比例して、そのデバイス面部分に押し付けるパッド面部分の表面荒れが粗くなるように調整することを特徴とする請求項１記載のＣＭＰ装置の研磨パッドの調整方法。
【請求項３】　前記ＣＭＰ装置の研磨パッドの調整方法において、
硬度が異なる砥石を回転する研磨パッドの動径方向にそれぞれ配置して、前記パッド面に押し付け、パッド面を区分してそれぞれ研磨し、異なる粗さの表面荒れに調整することを特徴とする請求項１記載のＣＭＰ装置の研磨パッドの調整方法。
【発明の詳細な説明】
【０００１】
【発明の属する技術分野】本発明は、半導体デバイスの製造工程において、半導体基板表面の平坦化を行う化学的機械研磨（ＣＭＰ：Chemical Mechanical Polishing）技術に係り、特にＣＭＰ装置における研磨パッドの調整方法に関する。
【０００２】
【従来の技術】近年、処理時間の高速化や微細化の要求に伴い半導体デバイスの回路素子は積み上げられて多層化し、配線層も多層化されている。
【０００３】このような回路素子等の多層化は、半導体基板上で回路素子を形成した領域と未形成の領域との間に段差を生じさせ、さらに積み上げる毎にそれらの段差が加算される。この結果、上層膜では、フォトリソグラフィ工程のマスク露光の際に、焦点深度を越えたり、エッチング困難な場合が生じる。さらに、例えばビアホールにメタル配線を形成した場合、段差によりビアホールの深さに違いが生じ、ビアホール抵抗のばらつきの原因にもなっている。
【０００４】そこで、多層に回路素子を形成する際に、半導体基板表面の平坦化を図る技術が求められていた。
【０００５】従来の平坦化技術としては、エッチバック技術やＳＯＧ（Spin On Glass）技術が知られている。これらの技術は、段差近傍のローカルな平坦化には有用であった。しかし、半導体デバイスの多層化や大型化が進むと半導体基板全体を平坦化する技術が必要である。
【０００６】エッチバック技術による平坦化は、オーバーエッチングの管理が難しく、半導体基板のデバイス面全体を平坦化させるには、容易でなかった。さらに、層間絶縁膜のような同一材料が全表面を覆った面を平坦化するには問題がないが、例えばＡｌ等を含有する配線のようにエッチングされにくい材料を使用した配線等が含まれる構成であれば、エッチングにより平坦化することは難しい。
【０００７】また、ＳＯＧ技術においては、ＳＯＧ膜をスピンコーティングして塗布し、段差の平坦化を実現していたが、ＳＯＧ膜自体のクラック耐性が乏しく、半導体基板全体を平坦化させる膜厚までは形成できない問題があり、さらに塗布により平坦化させるためには、回路素子の表面形状や配置に工夫を施さねばならなかった。このため、多層化された回路素子や多層化配線には、十分な平坦化を提供できる技術ではなくなった。
【０００８】そこで新たな平坦化技術として、インゴットからスライスカットされたシリコンウエハへの平面研磨を行うポリッシング研磨技術と化学研磨技術の特徴を併せ持たせ、半導体デバイスの平坦化に応用したＣＭＰ技術が注目されている。
【０００９】このＣＭＰ技術は、図４に示すように、ポリッシング研磨における研磨剤として、アルミナやシリカ等の研磨粒子に過酸化水素水等の化学物質を併せた研磨剤（スラリー液）２１を用いて、定盤２２上に軟質または、粘弾性を有する材料からなる研磨パッド２３を張り付け、化学反応を伴う機械研磨によりデバイス面を機械的に研磨して段差を無くし、表面を平坦化するものである。
【００１０】この機械において、駆動可能なテーブル２４に真空吸着により固定された半導体基板２５は、そのデバイス面２５ａを対向し回転する研磨パッド２３に押しつけられ、研磨剤２１が注ぎ込まれて研磨される。デバイス面が平坦化される。このＣＭＰ技術は、研磨レートが小さいため高精度の研磨が可能であり、研磨された面に加工変質を残すことがわずかしか考えない特徴がある。
【００１１】
【発明が解決しようとする課題】前述したＣＭＰ技術において、研磨に用いられる研磨パッドの表面は、砥石例えば、ダイヤモンド砥石で削られ、適当な表面荒れが設けられている。この研磨パッドの表面荒れにより、デバイス面上に積層形成された絶縁膜又は金属膜を研磨を行っているが、ダイヤモンド砥石自らの表面も削られるため、表面荒れの削り取られて研磨能力が劣化する。
【００１２】そこで、研磨パッドは、予め定めた処理回数（割った量）毎に、その表面を再度、ダイヤモンド砥石で削り直し、適当な表面荒れが保たれるように調整を行っている。
【００１３】通常、研磨前のデバイス面は、いわゆるａｓ ｄｅｐｏの状態にあり、その膜厚分布は下地の平坦さによ

る影響と、成膜装置の特性に依存する面内膜厚分布の凸凹傾向があり、ＣＭＰ技術において研磨パッドによる研磨を行った場合、研磨後の研磨パッドもこの凸凹形状に依存して平坦な表面の形態が変化する。つまり、膨らみに削れていくのである。

【００１４】例えば、図５に示すようなデバイス面では、凸形状の部分２５ｂに押しつけられた研磨パッド部分２３ｂは多く削れ、表面荒れの部分も削り取られる。また、凹形状の部分２５ｃを押し付けられた研磨パッド部分２３ｃはあまり削れないため、表面荒れの部分も残っている。

【００１５】この削れ具合の差により、研磨後の研磨パッド表面は、膨らんだ凸形状に変化し、且つ研磨パッド内で研磨の能力の差が生じている。従って、このように表面形状が変化していく研磨パッドで、引き続きデバイス面を研磨すると、半導体基板の表面も平坦にならない場合が生じる。

【００１６】また従来の研磨パッドの調整は、研磨パッド表面の平坦化維持を考慮したものではなく、単一的な表面荒れを形成することを目的としているため、研磨パッドの表面全体を１つのダイヤモンド砥石で均一的に削るだけであった。

【００１７】よって、研磨パッドの表面荒れの均一性を持たせる管理を難しくして、研磨パッドのパッド面の平坦性を維持させ、研磨された半導体基板のデバイス面の平坦化を維持させるために、研磨パッドの交換回数や調整回数が多くなり、作業効率の低下を招き、結果的にコストアップとなる。

【００１８】そこで本発明は、半導体基板のデバイス面の膜厚分布に応じて、研磨パッドのパッド面内を区分して表面荒れの粗さをそれぞれ調整し、研磨量の平坦性と研磨された半導体基板のデバイス面の平坦化を維持させるＣＭＰ研磨パッドの調整方法を提供することを目的とする。

【００１９】

【課題を解決するための手段】本発明は上記目的を達成するために、半導体基板の回路素子を形成するデバイス面を平坦化する化学的機械研磨（ＣＭＰ）装置に用いる研磨パッドの研磨を行うパッド面の調整方法において、前記研磨パッドのパッド面を押し付ける研磨前のデバイス面の膜厚分布に応じて、該パッド面内を区分して、硬度の異なる複数の砥石で、該パッド面の表面荒れを異なる粗さにそれぞれ調整するＣＭＰ装置の研磨パッドの調整方法を提供する。

【００２０】以上のようなＣＭＰ研磨パッドの調整方法は、研磨前の半導体基板のデバイス面の膜厚分布に対応して、硬度が異なる複数の砥石により、デバイス面の膜の研磨量に比例して、そのデバイス面に押し付けられるパッド面の表面荒れが粗くなるように研磨して、パッド面内での研磨量を変化させることにより、研磨される半導体基板のデバイス面の平坦化を維持する。

【００２１】

【発明の実施の形態】以下、図面を参照して本発明の実施の形について詳細に説明する。

【００２２】まず、本発明によるＣＭＰの研磨パッドの調整方法の概要について説明する。図２（ａ）には、半導体基板上に回路素子を形成するための絶縁膜や金属膜をＣＶＤ（Chemical Vapor Deposition）装置やスパッタリング装置を用いて半導体基板上に形成した場合の膜厚分布の一例を示す。

【００２３】この半導体基板１のデバイス面上に堆積された絶縁膜２の膜厚分布の形状は、同心円状で、半導体基板１の中心で厚く、エッヂ方向に向かって一旦薄くなるが、次第に厚くなり、再度エッヂで薄くなる。すなわち、ドーナツ形状の凹みを有した形状である。

【００２４】このような膜厚分布を持つ半導体基板をＣＭＰ技術により平坦化させる場合、図２（ｂ）に示すように、斜線で示すＡ領域及びＣ領域の膜厚が近似し、仮に膜厚ｍに平坦化するものとすると、Ａ領域及びＣ領域は、研磨量を多くし、Ｂ領域は、研磨量を少なくすることにより、効率的に平坦化できる。

【００２５】従って、半導体基板１のデバイス面に押し付けられ研磨を行う研磨パッドのパッド面（研磨面）は、膜厚分布と逆の凸凹具合の調整が必要となる。つまり後述する図１（ｃ）に示すように、堆積膜の厚いＡ領域及びＣ領域に押し付けられるパッド面部分５及びパッド面部分８は、表面荒れを粗くして研磨レートを上げるように調整し、堆積膜が薄く研磨量が少ないＢ領域に押し付けられるパッド面部分７は、その表面荒れを研磨パッド部分６に比べて、粗くせずに研磨レートを下げるように調整する。

【００２６】このような調整を行うためには研磨パッドのパッド面内を区分して、それぞれの表面荒れの粗さに調整する必要がある。

【００２７】図１（ａ），（ｂ）に示すように、図２（ｂ）に示したような膜厚分布に対応する、少なくとも硬度が異なる２つのダイヤモンド砥石４、５を研磨パッド３の円周方向の上に配置して、研磨パッドのパッド面３ａの表面荒れを調整する。少なくともダイヤモンド砥石４は浮動可能に備えられている。

【００２８】この膜厚分布は、予め成膜装置毎に膜厚分布の形状に関するデータを取って、パーソナルコンピュータ等に記憶させておき、研磨の際に読み出して利用してもよいし、その都度、半導体基板のデバイス面の膜厚分布を計測してもよい。

【００２９】本実施形態では、図２（ｂ）に示した堆積膜の膜厚分布を例とすると、研磨パッド３は、図１（ｃ）に示すように半導体基板１のＡ領域及びＣ領域を研磨する外周部のパッド面部分６及び中心部のパッド面部分８が、Ｂ領域を研磨する内周部のパッド面部分７よ

りも研磨量が多くなるように、表面荒れの粗さを調整する必要がある。

【００３０】その調整方法は、ダイヤモンド砥石５に比べて、研磨量が低い硬度のダイヤモンド砥石４を装着してパッド面３ａに押し付け、研磨パッド３を回転させて、パッド面３ａに表面荒れを形成する。

【００３１】本実施形態の研磨パッドの調整方法によれば、ＣＭＰ装置に装着して、半導体基板のデバイス面を平坦化する研磨パッド内の表面荒れの粗さを、デバイス面の膜厚分布（研磨すべき量）に基づき、研磨量の多い部分ほど粗くなるように調整することにより、パッド面の平坦化を維持し、且つ同時に膜厚分布の傾向を持つデバイス面を研磨する処理回数（研磨量）を増やすことができる。

【００３２】また、表面荒れの粗さは、研磨量や研磨剤や研磨速度及び研磨を行う絶縁膜の材質や成膜方法により異なるため、経験的にあらかじめ決定する。

【００３３】次に図３を参照して、本発明による第２の実施形態としてのＣＭＰ研磨パッドの調整方法について説明する。

【００３４】前述した第１の実施形態では、硬度が異なる２つのダイヤモンド砥石により、研磨パッドを調整したため、２種類の粗さの表面荒れが形成されたが、図２（ｂ）に示した絶縁膜２の膜厚分布で、例えば、Ｃ領域の膜厚がＡ領域の膜厚より、さらに厚かった場合には、Ｃ領域の絶縁膜の研磨量をＡ領域よりも多くする必要がある。そこで、図３（ａ）、（ｂ）に示すように、硬度が異なる３つのダイヤモンド砥石１１，１２，１３を研磨パッド３の半径方向に配置し、研磨パッド３の表面荒れの調整を行う。このダイヤモンド砥石１１、１２、１３の取り付け機構においては、砥石の支持や研磨パッドに対する研磨位置の移動が可能な構成になっているものとする。

【００３５】この調整は、図３（ｃ）に示すような半導体基板１の膜厚分布において、絶縁膜２を膜厚ｂに平坦化する際に、Ｃ’領域の研磨量ｑがＡ’領域の研磨量ｐよりも２倍程度ある場合など、半導体基板の外周部と中心部の膜厚に差があり、前述した第１の実施形態による硬度が２種類のダイヤモンド砥石では、十分な平坦化が実現されない膜厚分布に対応する。このような調整において、砥石の配置は、ダイヤモンド砥石１２、ダイヤモンド砥石１１、ダイヤモンド砥石１３の順に研磨量が多くなる硬度ものを配置する。

【００３６】以上のことから本実施形態は、第１の実施形態に比べて、パッド面内の表面荒れの粗さをさらに調整でき、研磨する絶縁膜の膜厚分布に依らず、平坦化が実現でき、第１の実施形態と同様な効果が得られる。

【００３７】本実施形態では、硬度が異なる２若しくは３個のダイヤモンド砥石を用いて説明したが、これらに限定されるものではなく、半導体基板の径やデバイス面の膜厚分布、膜の材質により、自由に硬度の選択と配置をしてもよく、また、砥石側を回転させてパッド面を押し付け研磨してもよい。

【００３８】尚、従来、ＣＭＰ装置の研磨機構では研磨パッドだけでなく、研磨を施す半導体基板側も回転させ、且つ半導体基板支持体を揺動方向に揺動する研磨機構もあり、半導体基板のエッヂ領域近傍は、研磨パッドの外周側と内周側とで研磨されるが半導体基板中心部は揺動中心近くで研磨されている。このような研磨装置では、半導体基板の外周部が中心部分よりも、研磨量が多くなっていたが、本実施形態の調整方法を利用して、研磨パッドの表面荒れを調整し、中心部分の研磨量を多くすることにより、この問題を解決できる。

【００３９】

【発明の効果】以上詳述したように本発明によれば、半導体基板のデバイス面の膜厚分布に応じて、研磨パッドのパッド面内を区分して表面荒れの粗さをそれぞれ調整し、研磨面の平坦性と研磨された半導体基板のデバイス面の平坦化を維持させるＣＭＰ研磨パッドの調整方法を提供することができる。

【図面の簡単な説明】

【図１】本発明による第１の実施形態としてのＣＭＰ研磨パッドの調整方法を実施するための研磨パッドと砥石の配置の構成例を示す図である。

【図２】成膜装置を用いて形成した時の絶縁膜の膜厚分布の一例を示す図である。

【図３】本発明による第２の実施形態としてのＣＭＰ研磨パッドの調整方法を実施するための研磨パッドと砥石の配置の構成例を示す図である。

【図４】ＣＭＰ装置の概略的な構成を示す図である。

【図５】従来の半導体基板と研磨パッドの研磨状態を説明するための図である。

【符号の説明】

１…半導体基板

２…絶縁膜

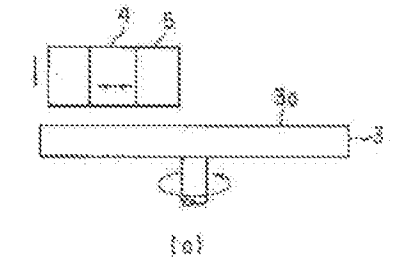
３…研磨パッド

３ａ…パッド面

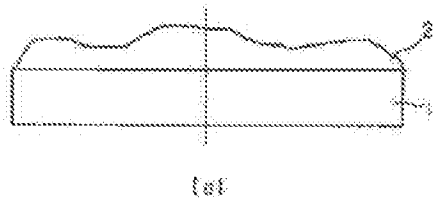
４，５…ダイヤモンド砥石

６，７，８…研磨パッド部分

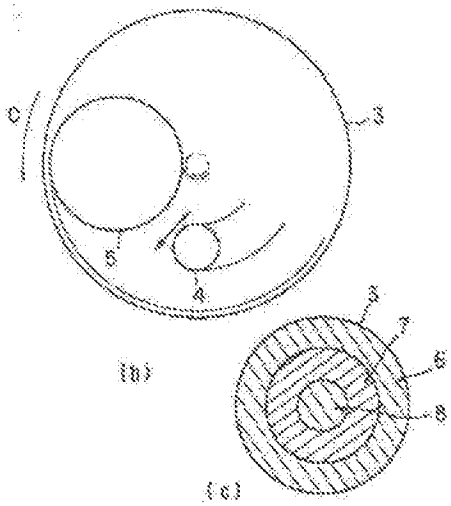
【図1】

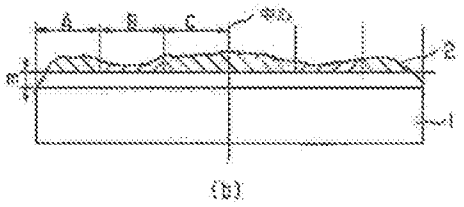


【図2】

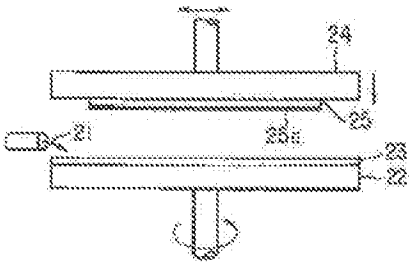


【図3】

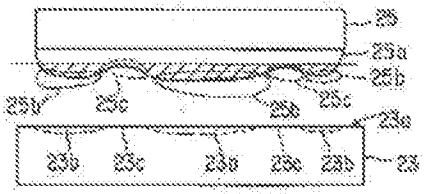




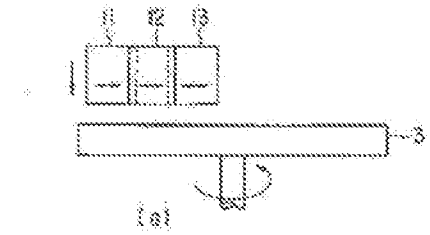
【図4】

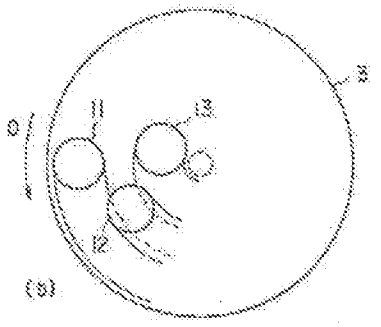


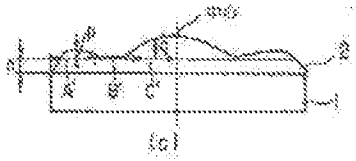
【図5】



【図6】







Espacenet

Bibliographic data: JP10128654 (A) — 1998-05-19

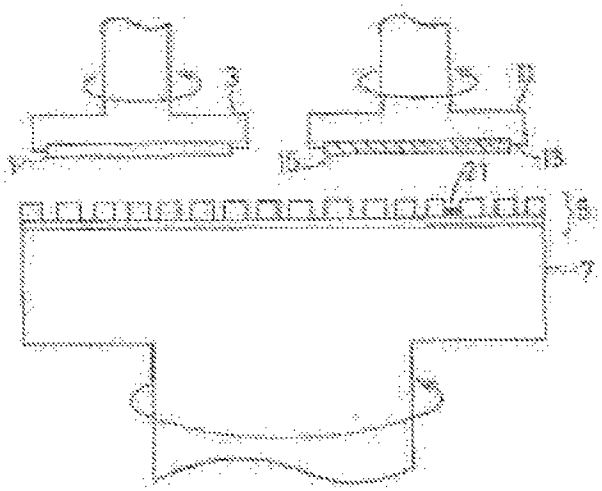
CMP DEVICE AND ABRASIVE CLOTH CAPABLE OF BEING USED IN THIS CMP DEVICE

Inventor(s):	KATO NOBUHIRO; MURAKAMI SATOSHI; WATANABE TOMOJI ±	
Applicant(s):	TOSHIBA CORP ±	
Classification:	**- International:**	***B24B37/00; B24B37/04; B24B49/18; B24B53/007; H01L21/304;*** (IPC1-7): B24B37/00; H01L21/304
	- European:	B24B49/18; B24B53/017
Application number:	JP19960290397 19961031	
Priority number (s):	JP19960290397 19961031	
Also published as:	US6059921 (A) TW423043 (B)	

Abstract of JP10128654 (A)

PROBLEM TO BE SOLVED: To provide a CMP device and an abrasive cloth capable of being used in the CMP device, which can suppress deterioration of polishing performance of the abrasive cloth and easily detect a limit of use. SOLUTION: In a CMP device polishing a semiconductor substrate 1, a diamond dresser 11 is provided with a material 15 capable of mechanically removing a diamond abrasive grain coming off onto an abrasive cloth 5 inside a diamond abrasive grain electrocoating surface 13, and, by the material 15, simultaneously with dressing, the diamond abrasive grain on the cloth 5 is removed. Further, the cloth 5 is provided with a use limit detection part 21 with a work amount of dimple work or the like less than of the other part, and, by polishing the semiconductor substrate 1, the point of time exposing a surface of the use limit detection part 21 is detected as a use limit.

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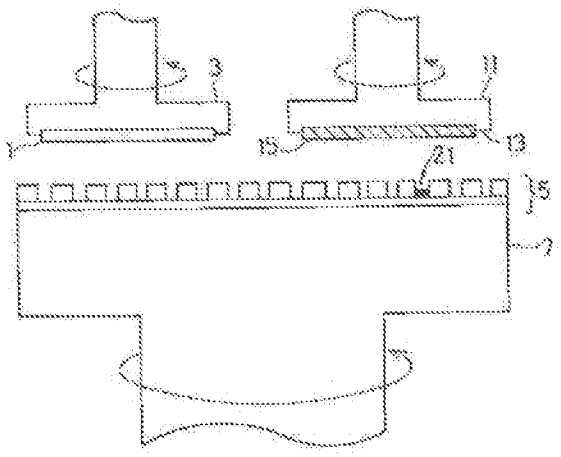
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(54)【発明の名称】　ＣＭＰ装置及び該ＣＭＰ装置に用いることのできる研磨布

(57)【要約】
【課題】　研磨布の研磨性能の劣化を抑制し、かつ、使用限界を容易に検出することができるＣＭＰ装置及び該ＣＭＰ装置に用いることのできる研磨布を提供することを課題とする。
【解決手段】　半導体基板１の研磨を行うＣＭＰ装置で、ダイヤモンドドレッサー１１はダイヤモンド砥粒電着面１３の内側に研磨布５上に散布したダイヤモンド砥粒を機械的に除去することが可能な材料１５を備え、材料１５によりドレッシングを同時に研磨布５上のダイヤモンド砥粒を除去する。さらに、研磨布５は、ティンプル加工等の加工量が他の部分よりも多い使用限界検出部２１を備え、半導体基板１の研磨により使用限界検出部２１が表面に露出した時点を使用限界として検出する。



【特許請求の範囲】
【請求項１】　底面開口部に設けられたダイヤモンド砥粒電着面により研磨布表面のドレッシングを行うダイヤモンドドレッサーを有するＣＭＰ装置において、
前記ダイヤモンドドレッサーがドレッシングの際に前記ダイヤモンド砥粒電着面から前記研磨布表面に脱落するダイヤモンド砥粒を除去する手段を具備することを特徴とするＣＭＰ装置。
【請求項２】　底面開口部に設けられたダイヤモンド砥粒電着面により研磨布表面のドレッシングを行うダイヤモンドドレッサーを有するＣＭＰ装置において、
前記ダイヤモンドドレッサーは、前記ダイヤモンド砥粒電着面の内側に前記研磨布上に脱落したダイヤモンド砥粒を積極的に除去することが可能の材料を備え、前記材料によりドレッシングと同時に該ドレッシングの際に前記ダイヤモンド砥粒電着面から前記研磨布上に脱落した前記ダイヤモンド砥粒を除去することを特徴とするＣＭＰ装置。
【請求項３】　前記材料は、ナイロンブラシであることを特徴とする請求項２記載のＣＭＰ装置。
【請求項４】　前記材料は、スポンジであることを特徴とする請求項２記載のＣＭＰ装置。
【請求項５】　前記材料は、モヘアブラシであることを特徴とする請求項２記載のＣＭＰ装置。
【請求項６】　表面に凹部が形成された研磨布により基板の研磨を行うＣＭＰ装置において、
前記研磨布の該研磨布の使用限界を目視により確認することができる使用限界検出手段を具備することを特徴とするＣＭＰ装置。
【請求項７】　表面に凹部が形成された研磨布により基板の研磨を行うＣＭＰ装置において、
前記研磨布は、前記凹部内に使用限界検出部を備え、前記基板の研磨により前記使用限界検出部が前記研磨布表面に露出した時点を使用限界として検出することを特徴とするＣＭＰ装置。
【請求項８】　前記使用限界検出部は、前記凹部内で他の部分よりも浅く加工された部分であることを特徴とする請求項７記載のＣＭＰ装置。
【請求項９】　前記凹部を通して研磨布全面に供給されることを特徴とする請求項６乃至請求項８のいずれか１項に記載のＣＭＰ装置。
【請求項１０】　表面に凹部が形成され、基板の研磨に用いられる研磨布において、
前記凹部内に他の部分よりも浅く加工された部分を使用限界検出部として備え、前記基板の研磨により前記使用限界検出部が前記研磨布表面に露出した時点を使用限界と検出することを特徴とする研磨布。
【請求項１１】　前記凹部は、研磨布全面に純水を供給するために設けられたものであることを特徴とする請求項１０記載の研磨布。

【発明の詳細な説明】
【０００１】
【発明の属する技術分野】本発明はＬＳＩ製造工程における平坦化技術として用いられるＣＭＰ装置及び該ＣＭＰ装置に用いることのできる研磨布に関し、特に、研磨布の研磨特性の劣化を抑制し、かつ、使用限界を容易に検出することができるＣＭＰ装置及び該ＣＭＰ装置に用いることのできる研磨布に関する。
【０００２】
【従来の技術】近年、ＬＳＩ（Large Scale Integrated Circuit）の高集積化が多層化するのに伴い、層間絶縁膜の平坦化技術が非常に重要となってきているが、その技術として現在最も注目されているものが化学的機械研磨（chemical mechanical polishing ：ＣＭＰ）技術である。
【０００３】図５は、上記ＣＭＰ技術を実現する一般的なＣＭＰ装置の構成を示す図である。同図に示すように、このＣＭＰ装置は半導体基板１を固定し、半導体基板１を回転させるトップリング３と、半導体基板１の表面を研磨する研磨布５と、研磨布５を固定し、研磨布５を回転させるターンテーブル７とを少なくとも有し、所定の製造工程を経て凹凸状態となっている半導体基板１の表面を研磨布５に押し付け、研磨剤９を供給しながらトップリング３とターンテーブル７をそれぞれ回転させ、化学反応を生じさせながら機械的に半導体基板１表面を研磨布５で研磨することにより半導体基板１表面の平坦化を図るものである。
【０００４】上述したようなＣＭＰ装置においては、研磨布の研磨性能維持のためにドレッシング（dressing）と呼ばれる作業が必要である。ドレッシングとは硬くなった研磨布の目を再び鋭くする目立のことであり、半導体基板の処理枚数の増加に伴って鋭くなっていく研磨布の目を再び鋭くすることにより、研磨布の研磨性能を維持するのである。このドレッシングは例えば次のようなダイヤモンドドレッサーにより実施される。図６は、ドレッシングを行う際のダイヤモンドドレッサーと研磨布を示す図であり、ドレッシングはダイヤモンドドレッサー１１をターンテーブル７上に固定された研磨布５の表面に押し付け、ダイヤモンドドレッサー１１とターンテーブル７をそれぞれを回転させることにより行われる。また、ダイヤモンドドレッサー１１自体を揺動させる場合もある。図７は、上記ダイヤモンドドレッサー１１の拡大図であり、ダイヤモンドドレッサー１１の底面周辺にはダイヤモンド砥粒を電着して形成したダイヤモンド砥粒電着面１３が設けられ、このダイヤモンド砥粒電着面１３を研磨布表面に当てることにより上記ドレッシングが行われる。なお、ドレッシングは上述した半導体基板の研磨と同時に、または、その研磨前、研磨後に行われる。
【０００５】また、図８は、図５と同様に一般的なＣＭ

Ｐ装置の構成を示す図であるが、図８に示すように、ＣＭＰ装置に用いられる研磨布５の表面は実際には紙ヤスリの研磨布５全面に行き渡るようにディンプル加工や格子状の溝加工が施されている。この研磨布５により上述したように半導体基板１の表面を研磨した場合、半導体基板１の処理枚数の増加に伴って研磨布５は磨耗し易くなるが、研磨布５の使用限界を越えた場合には研磨速度や研磨量の面内均一性の値が悪化してしまう。そのため、実際に上記ＣＭＰ装置により半導体基板１を研磨する場合には、その直前に予め測定された使用限界検出用のテスト基板を予め研磨し、そのテスト基板を評価することにより研磨速度や研磨量の面内均一性の値を算出し、その算出結果に基づいて研磨布５が使用限界に達しているか否かを判断し、使用限界に達していないと判断した場合にのみ本来の半導体基板１の研磨を行い、使用限界に達していると判断した場合には研磨布５の交換が行われる。

【０００６】

【発明が解決しようとする課題】しかしながら、従来のＣＭＰ装置には次のような問題があった。

【０００７】それは、上述したドレッシングの際に、ダイヤモンド砥粒電着面からダイヤモンド砥粒が研磨布表面に脱落し、その脱落したダイヤモンド砥粒がその後の研磨の時に半導体基板表面にキズをつけてしまうということである。図９は、図７のダイヤモンド砥粒電着面１３の断面図であるが、図９（ａ）に示すように、ダイヤモンド砥粒電着面１３は、ダイヤモンド砥粒１７をニッケル層１９に埋め込み、露出しているダイヤモンド砥粒１７により研磨布のドレッシングを行うものであるが、同図（ｂ）に示すように、ドレッシング時の研磨布との摩擦によりニッケル層１９からダイヤモンド砥粒１７が脱落し、研磨布に脱落してしまうのである。さらに、ドレッサー１１の使用と共にニッケル層１９が磨耗し、新たにニッケル層１９表面に露出するダイヤモンド砥粒１７やニッケル層１９への埋まり具合が浅くなってくるものもあり、上述したダイヤモンド砥粒１７の脱落は次々と起こり得るのである。

【０００８】また、研磨布の使用限界を検出するために半導体基板の研磨装置にテスト研磨が行われるが、その時用いられるテスト基板は再利用することはできず、廃棄しなければならない。一方、半導体基板の径は、そのサイズの拡大と共に徐々に上昇している。従って、廃棄コストの増大を招くのは必至である。さらに、テスト研磨及びテスト基板の評価に要する時間は量産ラインにおいて生産効率を向上させる上で非常に問題である。

【０００９】本発明は上記事情に鑑みて成されたものであり、その目的は、ドレッシングの際にダイヤモンド砥粒電着面から研磨布表面に脱落したダイヤモンド砥粒をその後の研磨の時までに除去することにより半導体基板表面にキズをつけることのないＣＭＰ装置を提供することにある。また、その他の目的は、研磨布の使用限界をテスト研磨することなく容易に判断することができるＣＭＰ装置及び研磨布を提供することにある。

【００１０】

【課題を解決するための手段】上記の目的を達成するために本発明の第１の特徴は、底面周辺部に設けられたダイヤモンド砥粒電着面により研磨布表面のドレッシングを行うダイヤモンドドレッサーを有するＣＭＰ装置において、前記ダイヤモンドドレッサーがドレッシングの際に前記ダイヤモンド砥粒電着面から前記研磨布表面に脱落するダイヤモンド砥粒を除去する手段を具備することである。

【００１１】ここで、前記手段は、具体的には、前記ダイヤモンド砥粒電着面の内側に配置される前記研磨布上に摺接したダイヤモンド砥粒を機械的に除去することが可能な材料であり、例えば、ナイロンブラシ、スポンジである。また、ナイロンブラシに比べて毛先の柔らかいモヘアブラシを用いれば、押し付け圧にあまり影響されることなく良好な除去効果を得ることができる。なお、上記材料の外径は、少なくともＣＭＰ処理を行う基板の外径以上であることが必要である。さらに、ドレッシングの際にダイヤモンドドレッサー自体を揺動させる場合には、少なくとも、研磨布表面の内で研磨に使用される領域全体に上記材料が接触することが可能な外径であることが必要である。

【００１２】上記構成によれば、ダイヤモンドドレッサーとナイロンブラシ等の材料を一体化させることにより、ドレッシングの際にダイヤモンド砥粒電着面から研磨布上に脱落するダイヤモンド砥粒の除去をドレッシングと同時に行うことができる。従って、その後の研磨においてダイヤモンド砥粒による基板表面のキズを減らすことができる。

【００１３】本発明の第２の特徴は、表面に凹部が形成された研磨布により基板の研磨を行うＣＭＰ装置において、前記研磨布が前記研磨布の使用限界を目視により確認することができる使用限界検出手段を具備することである。

【００１４】ここで、前記使用限界検出手段は、好ましくは、前記凹部の加工量を調節してその他の部分よりも浅く加工された部分であり、前記研磨布の摩耗により前記浅く加工された部分が表面に露出した時点を使用限界と検出するものである。なお、前記浅く加工された部分の深みは研磨の基板の研磨速度と研磨量の面内均一性の値が悪化する時期に研磨布表面に露出して目視で確認することができるよう設定する。

【００１５】上記構成によれば、使用限界検出手段が研磨布の表面に露出し目視で確認できた時点を研磨布の交換時期とするので、従来のようなテスト研磨を行う必要が無くなり、また、その際無駄に消費されていたテスト基板も不要となる。従って、コストの低減を図ることが

できる。

【００１６】

【発明の実施の形態】以下、本発明の実施の形態について図面を用いて説明する。図１は、本発明の実施の形態に係るＣＭＰ装置の構成を示す図である。なお、従来例と同一部分には同一符号が付してある。

【００１７】同図に示すように、このＣＭＰ装置は半導体基板１を固定し、半導体基板１を回転させるトップリング３と、半導体基板１の表面を研磨する研磨布５と、研磨布５を固定し、研磨布５を回転させるターンテーブル７とを有し、所定の製造工程を経て凹凸状態となっている半導体基板１の表面を研磨布５に押し付け、砥液Ｑを供給しながらトップリング３とターンテーブル７をそれぞれ回転させ、化学反応を生じさせながら機械的に半導体基板１表面を研磨布５で研磨することにより半導体基板１表面の平坦化を図るものである。また、ドレッシングを行うダイヤモンドドレッサー１１を有し、ダイヤモンドドレッサー１１をターンテーブル７に固定された研磨布５の表面に押し付け、ダイヤモンドドレッサー１１とターンテーブル７それぞれを回転させることにより、半導体基板１の処理枚数の増加に伴って鋭くなっていく研磨布５の目を再び鋭くし、その研磨性能を維持するものである。

【００１８】図２は、図１に示すダイヤモンドドレッサー１１の拡大図である。なお、従来例と同一部分には同一符号が付してある。

【００１９】図２に示すように、本実施の形態に係るダイヤモンドドレッサー１１は、ダイヤモンド電着電着面１３の内縁に、研磨布上に脱落したダイヤモンド砥粒を機械的に除去することが可能な材料、例えば、ナイロンブラシを配置することにより、ドレッシング時に研磨布表面上に脱落するダイヤモンド砥粒を研磨布表面から除去し、あるいは、研磨布表面の内で半導体基板の研磨に使用されない領域へ除外することにより従来では問題となったダイヤモンド砥粒による半導体基板表面のキズを無くすことができる。また、かかるダイヤモンド砥粒を上記ナイロンブラシに取り込むことにより除去することも可能である。

【００２０】さらに、ダイヤモンドドレッサー自体にナイロンブラシ等の材料が内蔵されているので、従来と同じドレッシング時間だけでドレッシングと同時にダイヤモンド砥粒の除去も行うことができる。従って、全体の処理時間を短くすることができる。

【００２１】ここで、研磨布上に脱落したダイヤモンド砥粒を機械的に除去することが可能な材料としては、ナイロンブラシ以外にスポンジでも同様な効果を得ることができる。また、ナイロンブラシに比べて毛先の柔らかいモヘアブラシを用いることにより、押し付け圧により影響されることなく良好な除去効果を得ることができる。また、上記材料の外径は、少なくともＣＭＰ処理を行う半導体基板の外径以上であることが必要である。さらに、ドレッシングの際にダイヤモンドドレッサー自体を揺動させる場合には、少なくとも、研磨布表面の内で研磨に使用される領域全体に上記材料が接触することが可能な外径であることが必要である。

【００２２】図３は、図１に示す研磨布５の拡大図であり、図３（ａ）がその平面図、図３（ｂ）がそのＡ－Ａ´における断面図である。なお、従来例と同一部分には同一符号が付してある。

【００２３】図３に示すように、本実施の形態に係る研磨布５は、例えば従来例と同様にディンプル加工あるいは格子状の溝加工をすることで表面に凹部が形成されているが、さらに、その加工量を測定してその他の部分よりも深く加工された部分を形成し、その深く加工された部分を研磨布５の使用限界サイン２１として用いる構成としたものである。このような構成とすることにより、この研磨布５で半導体基板１の表面を研磨した場合に半導体基板１の処理枚数の増加に伴って研磨布５は摩耗し薄くなると使用限界サイン２１が徐々に表面に現れることとなるが、その出現度合によって研磨布５の使用限界を容易に検知することができるのである。従って、従来では研磨布５の使用限界を検知するために必要であったテスト研磨が不要となり、その際無駄に消費されていたテスト基板も必要なくなる。なお、使用限界サイン２１は研磨布５の円部内に形成されてもよいが、上述した通り円部内で深く加工された部分を使用限界サイン２１として用いることで使用限界サイン２１を研磨布５の素材と同材質から簡易に得ることができ、好ましい。

【００２４】図４は、図３に示す研磨布５を用いて実際に半導体基板を研磨した結果を示す図であり、図４（ａ）が半導体基板の酸化膜を０．６μｍ堆積したシリコン基板である場合、図４（ｂ）がポリシリコン膜を０．５μｍ堆積したシリコン基板である場合である。また、図４（ａ）及び（ｂ）それぞれの横軸は研磨した半導体基板の枚数であり、縦軸は研磨速度及び研磨量の面内均一性である。なお、ここで用いた研磨布５はポリウレタン材料の研磨布であり、その厚みを約１．３μｍ、使用限界サイン２１の厚みを０．５μｍとしたものである。

【００２５】図４（ａ）に示すように、酸化膜を０．６μｍ堆積したシリコン基板を研磨した場合には、約６００枚研磨した辺りで急激に研磨速度は減少すると共に研磨量の面内均一性が悪化していることが分かる。この時、研磨布５表面には使用限界サイン２１が目視で確認され、それにより、容易に研磨布５が交換時期であることを検知することができた。また、図４（ｂ）に示すように、ポリシリコン膜を０．５μｍ堆積したシリコン基板を研磨した場合には、約９００枚研磨した辺りで急激に研磨速度は減少すると共に研磨量の面内均一性が悪化していることが分かる。この時も同様に、研磨布５表面

には使用限界サイン２１が目視で確認され、それにより、容易に研磨布５が交換時期であることを検知することができた。

【００２６】ここで、研磨布５の材料としてポリウレタンを用いて説明したが、サイロン、レーヨン等の半導体基板を研磨するために用いられる研磨布材料全てに適用することが可能である。また、使用限界サインの厚みは所望の半導体基板の研磨速度と研磨量の値等の一定の値が変化する時期に研磨布表面に現れて使用限界サインを目視で確認することができるような厚さに適宜設定する。なお、研磨する半導体基板として酸化膜、ポリシリコン膜を成膜したシリコン基板について説明したが、本実施の形態はこれらの研磨に使用されるのみではなく、一般的にＬＳＩ製造工程で用いられる成膜材料。例えば、高融点金属（Ｓｉ、Ｍｏ、Ｗ、Ｔｉ、Ｔａ等）並びにそれら高融点金属の窒化物、登化物及びシリサイド、金属配線用材料（Ａｌ、Ｃｕ、Ａｌ－Ｓｉ－Ｃｕ、Ａｌ－Ｃｕ等）の研磨においても適用され得る。

【００２７】

【発明の効果】以上説明したように本発明によれば、ドレッシングを行う際同時に研磨布上に付着したダイヤモンド砥粒を除去することができるので、従来問題であった付着したダイヤモンド砥粒による基板表面のキズを無くすことができる。また、ダイヤモンドドレッサーに研磨布上に付着したダイヤモンド砥粒を除去するサイロンブラシ等の材料を一体化させることにより、ドレッシングとダイヤモンド砥粒の除去を同時に行うことができるので、処理時間の低減を図ることができ、さらに、装置の点からコストの低減を行うことができる。

【００２８】また、研磨布の使用限界を目視で容易に検出することができるので、従来必要であったテスト研磨が不要となり、事前に準備されていたテスト基板を無くすことができる。従って、全体の処理時間は短縮され、コストの低減にもつながる。

【図面の簡単な説明】

【図１】本発明の実施の形態に係るＣＭＰ装置の構成を示す図である。

【図２】図１に示すダイヤモンドドレッサーの拡大図である。

【図３】図１に示す研磨布の拡大図である。

【図４】図３に示す研磨布を用いて実際に半導体基板を研磨した結果を示す図である。

【図５】一般的なＣＭＰ装置の構成を示す図である。

【図６】ドレッシングを行う際のダイヤモンドドレッサーと研磨布を示す図である。

【図７】図６に示すダイヤモンドドレッサーの拡大図である。

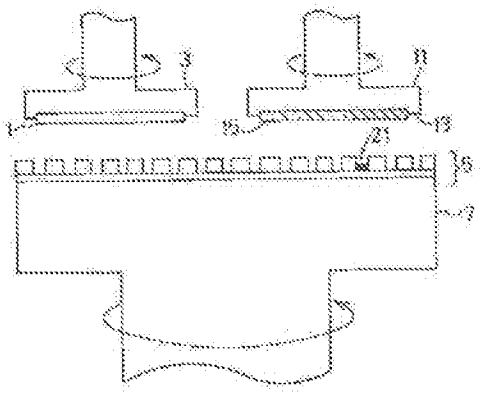
【図８】他の一般的なＣＭＰ装置の構成を示す図である。

【図９】図７に示すダイヤモンド砥粒電着面の断面図である。

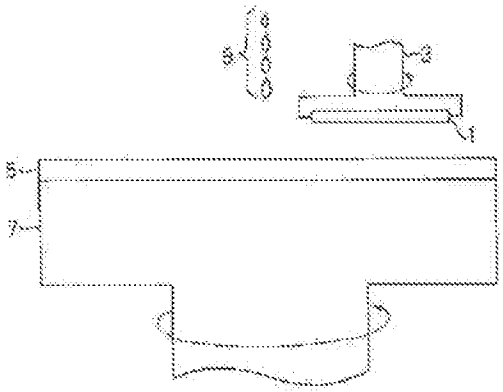
【符号の説明】

１　半導体基板
３　トップリング
５　研磨布
７　ターンテーブル
９　純水
１１　ダイヤモンドドレッサー
１３　ダイヤモンド砥粒電着面
１５　サイロンブラシ
１７　ダイヤモンド砥粒
１９　ニッケル膜
２１　使用限界サイン

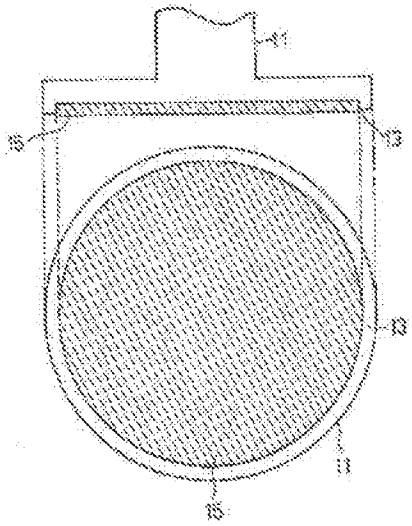
【図１】



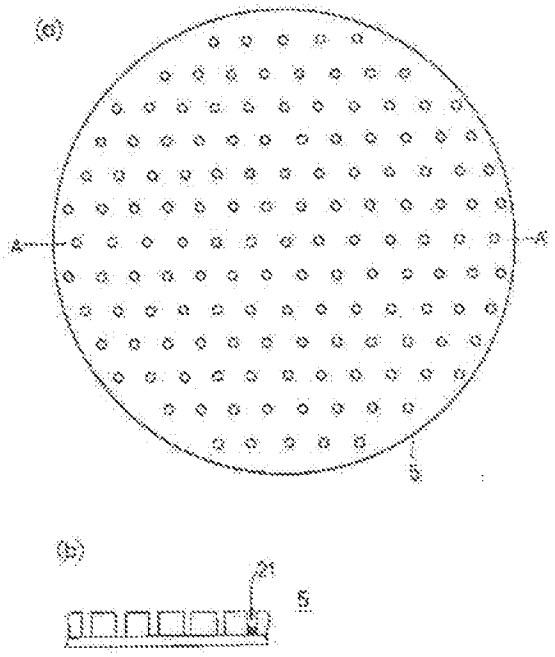
【図５】



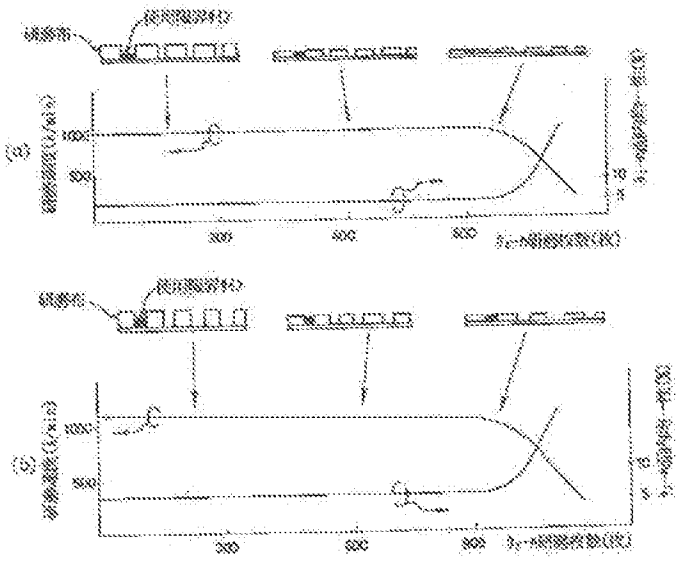
【図2】



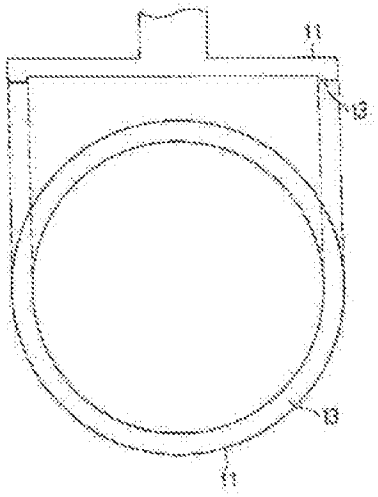
【図3】



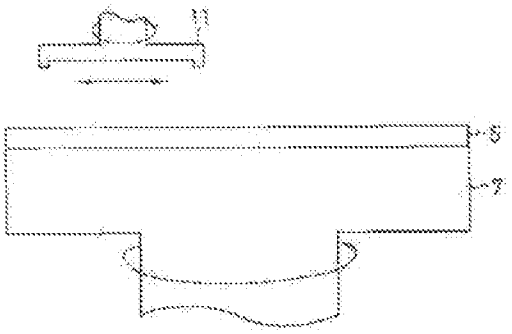
【図4】



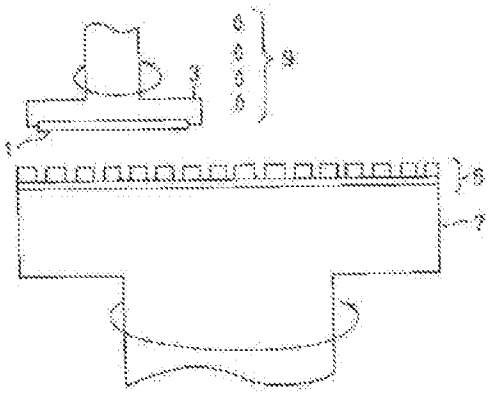
【図7】



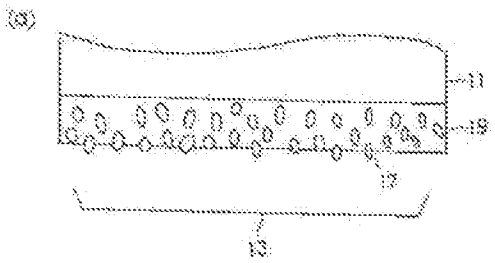
【図６】

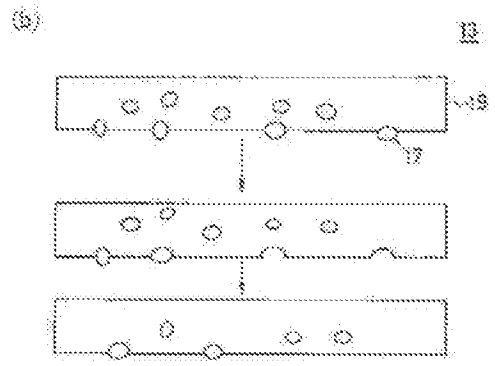


【図８】



【図９】





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Bibliographic data: JP2003071718 (A) — 2003-03-12

CMP CONDITIONER, METHOD FOR ARRANGING HARD ABRASIVE GRAIN USED IN CMP CONDITIONER AND METHOD FOR MANUFACTURING CMP CONDITIONER

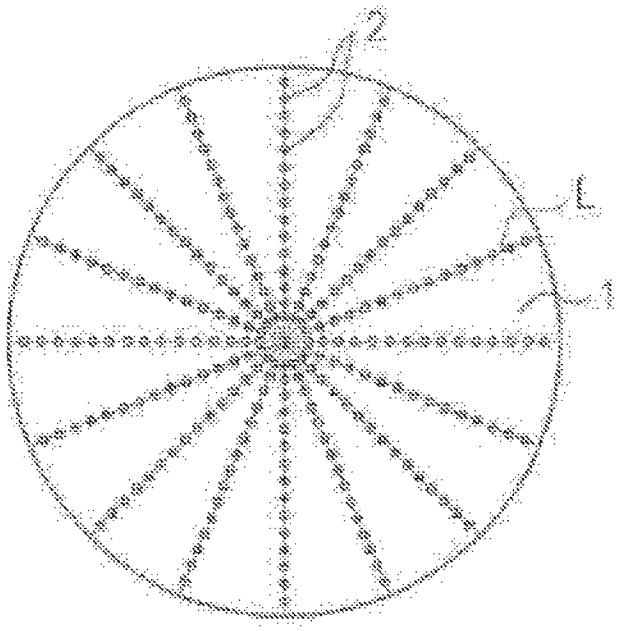
Inventor(s):	HASHINO HIDEJI; KINOSHITA TOSHIYA; SATO SETSUO; ARAKI RYUICHI ±
Applicant(s):	NIPPON STEEL CORP ±

Classification:	- **International:**	***B24B37/00; B24B53/12; B24D3/00; B24D7/18; H01L21/304;*** (IPC1-7): B24B37/00; B24B53/12; B24D3/00; B24D7/18; H01L21/304
	- **European:**	
Application number:	JP20010262167 20010830	
Priority number (s):	JP20010262167 20010830	

Abstract of JP2003071718 (A)

PROBLEM TO BE SOLVED: To provide stable CMP conditioner characteristics and to relieve slurry or the like in polishing without forming relief groove or the like on a support member. SOLUTION: This CMP conditioner of polishing cloth for a semiconductor substrate is composed of the support member 1 and plural diamond grains 2 brazed on a surface of the supporting member 1. Plural straight lines or curved lines radially extending from a center of the support member 1 are laid out on the surface of the disk-shaped support member 1 and the diamond grains 2 are arranged on the straight lines or the curved lines. The diamond grains 2 are arranged in such a manner that density of the diamond grains 2 at edge parts is smaller than that at a center par of the support member 1. Since area where no diamond grain 2 exists is secured in radial shape on the surface of the support member 1, slurry can be relieved outward from the support member 1 in polishing.

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（P2003－71718A）
(43)公開日　平成15年3月12日（2003.3.12）

(51)Int.Cl.'		識別記号	FI		ﾃｰﾏｺｰﾄﾞ(参考)	
B24B	53/12		B24B	53/12	Z	3C047
	37/00			37/00	A	3C058
B24D	3/00	310	B24D	3/00	310D	3C063
	7/18			7/18	Z	
H01L	21/304	622	H01L	21/304	622M	

審査請求　未請求　請求項の数13　OL　（全 7 頁）

(21)出願番号　特願2001－262157(P2001－262157)

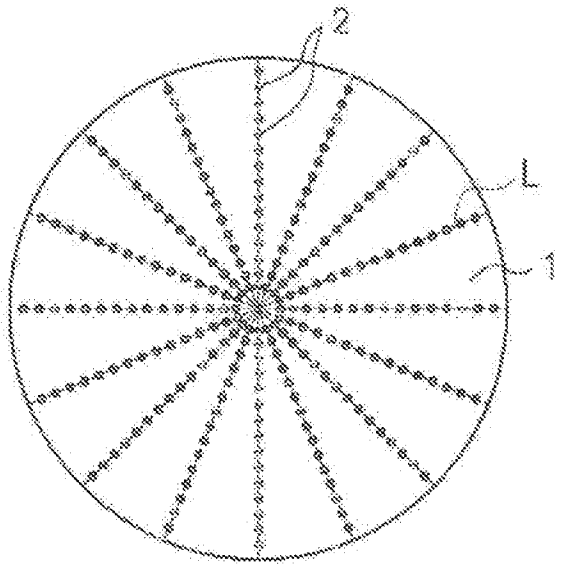
(22)出願日　平成13年8月30日(2001.8.30)

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最終頁に続く

(54)【発明の名称】　ＣＭＰコンディショナー、ＣＭＰコンディショナーに使用する被覆砥粒の選別方法、及びＣＭＰコンディショナー製造方法

(57)【要約】
【課題】　安定したＣＭＰコンディショナー特性が得られるとともに、支持部材に逃し溝等を形成しなくとも研磨時にスラリー等を逃すことができるようにする。
【解決手段】　半導体基盤用研磨布のＣＭＰコンディショナーは、支持部材１と、支持部材１の面上にろう付けされた被覆のダイヤモンド粒２とからなり、円板状の支持部材１の表面において、支持部材１の中心から放射状に伸びる複数の直線もしくは曲線を考え、それら直線もしくは曲線上にダイヤモンド粒２を配置するようにしている。このようにしたＣＭＰコンディショナーでは、ダイヤモンド粒２が支持部材１の中央部分に比べて縁部分における密度が小さくなるよう配列されており、支持部材１の表面上には、ダイヤモンド粒２が存在しない領域が放射状に確保されることになるので、研磨時にスラリーを支持部材１の外側に向けて逃すことができる。



【特許請求の範囲】
【請求項１】　支持部材と、前記支持部材の面上に設けられた複数の硬質砥粒とを備えたＣＭＰコンディショナーであって、
前記支持部材の面上に、前記複数の硬質砥粒を規則的に、かつ、前記支持部材の内側から外側にかけて密度が減少するように配列させたことを特徴とするＣＭＰコンディショナー。
【請求項２】　前記硬質砥粒を、前記支持部材の中心から放射状に配列させていることを特徴とする請求項１に記載のＣＭＰコンディショナー。
【請求項３】　支持部材と、前記支持部材の面上に設けられた複数の硬質砥粒とを備えたＣＭＰコンディショナーであって、
前記支持部材の面上に、前記複数の硬質砥粒が存在しない領域を放射状に確保していることを特徴とするＣＭＰコンディショナー。
【請求項４】　前記硬質砥粒はダイヤモンド粒であることを特徴とする請求項１～３のいずれか１項に記載のＣＭＰコンディショナー。
【請求項５】　モリブデン、クロム、又はシリコンウムより選ばれた１種以上を０．５～２９ｗｔ％含む融点６５０℃～１２００℃の合金を用いて、前記ダイヤモンド粒を金属及び／又は合金からなる前記支持部材に、埋着、ろう付けし、前記ダイヤモンド粒と前記合金との界面にモリブデン、クロム、又はシリコンウムより選ばれた金属の炭化物層が形成されていることを特徴とする請求項４に記載のＣＭＰコンディショナー。
【請求項６】　前記融点６５０℃～１２００℃の合金がニッケル基合金であることを特徴とする請求項５に記載のＣＭＰコンディショナー。
【請求項７】　規則的に、かつ、内側から外側にかけて密度が減少するように配列させた複数の貫通穴が形成された薄板状の配列部材を被配列面上に位置させる手順と、
前記配列部材の各貫通穴に硬質砥粒を入れ込む手順とを有することを特徴とするＣＭＰコンディショナーに使用する硬質砥粒の配列方法。
【請求項８】　複数の貫通穴の存在しない領域が放射状に確保された薄板状の配列部材を被配列面上に位置させる手順と、
前記配列部材の各貫通穴に硬質砥粒を入れ込む手順とを有することを特徴とするＣＭＰコンディショナーに使用する硬質砥粒の配列方法。
【請求項９】　前記被配列面は、ＣＭＰコンディショナーを構成する支持部材の表面であることを特徴とする請求項７又は８に記載のＣＭＰコンディショナーに使用する硬質砥粒の配列方法。
【請求項１０】　複数の硬質砥粒を規則的に、かつ、内側から外側にかけて密度が減少するように配列させた状態で保持部材に保持する手順と、
前記保持部材により保持された硬質砥粒を、ＣＭＰコンディショナーを構成する支持部材の表面に転写する手順とを有することを特徴とするＣＭＰコンディショナーに使用する硬質砥粒の配列方法。
【請求項１１】　複数の硬質砥粒の存在しない領域が放射状に確保された状態で前記複数の硬質砥粒を保持部材に保持する手順と、
前記保持部材により保持された硬質砥粒を、ＣＭＰコンディショナーを構成する支持部材の表面に転写する手順とを有することを特徴とするＣＭＰコンディショナーに使用する硬質砥粒の配列方法。
【請求項１２】　前記保持部材には前記硬質砥粒を保持するための第１の接着手段を設け、前記支持部材の表面には第２の接着手段を設け、これら第１、２の接着手段の性質に差を持たせたことを特徴とする請求項１０又は１１に記載のＣＭＰコンディショナーに使用する硬質砥粒の配列方法。
【請求項１３】　請求項７～１２のいずれか１項に記載のＣＭＰコンディショナーに使用する硬質砥粒の配列方法を利用して前記硬質砥粒を前記支持部材の表面上に配列させた後、前記硬質砥粒を前記支持部材の表面に固着することを特徴とするＣＭＰコンディショナー製造方法。
【発明の詳細な説明】
【０００１】
【発明の属する技術分野】本発明は、半導体基板用の研磨布の目詰まりを解消し、異物を除去するのに使用されるＣＭＰコンディショナー、ＣＭＰコンディショナーに使用する硬質砥粒の配列方法、及びＣＭＰコンディショナー製造方法に関する。
【０００２】
【従来の技術】ウェハのポリッシングにおいては、ＣＭＰ(Chmical Mechanical Polishing)と呼ばれる研磨方法が採用されている。ＣＭＰは、砥粒の機械作用に化学的研磨作用を重畳して働かせることにより、研磨速度の確保と被研磨材が無欠陥であることの両立を可能としたものであり、シリコンウェハの仕上げポリッシング工程で広く使用されている。
【０００３】また、近年ではデバイスの高集積化に伴い、集積回路を製造する所定の段階で、ウェハ表面やウェハ表面に導電体・誘電体膜が形成された半導体基板表面を研磨することが必要になってきた。半導体基板は、研磨されて、高い段差、引っかき傷、曇り等の表面欠陥が除去される。通常、この工程は、ウェハ上に種々の素子及び集積回路を形成する間に行われる。この研磨工程では、シリコンウェハの仕上げポリッシング工程と同様に、研磨速度と無欠陥であることの両立が必要である。化学スラリーを導入することにより、半導体表面により大きな研磨除去速度及び無欠陥性が与えられる化学的か

つ機械的平坦化が行われる。

【０００４】ＣＭＰ工程の一例としては、図８に示すように、例えば５～３００ｎｍ程度の粒径を有するシリカ粒子を苛性ソーダ、アンモニア及びアミン等のアルカリ溶液に懸濁させてＰＨ９～１２程度に化学スラリー１０１と、ポリウレタン樹脂等からなる研磨布１０２とが用いられる。研磨時には、化学スラリー１０１を流出しながら、半導体基板１０３を研磨布１０２に適当な圧力で当接させ、同図の矢印に示すように相対回転させることにより研磨が行われる。

【０００５】そして、前記研磨布１０２のコンディショニング法としては、研磨布１０２に水又は化学スラリー１０１を流しながら、ＣＭＰコンディショナーを用いたコンディショニングを行って、研磨布１０２の目詰まりを解消し、異物を除去していた。ＣＭＰコンディショナーを用いたコンディショニングは、半導体基板１０３の研磨が行われた後に、ＣＭＰコンディショナーを研磨布１０２に当接させるか、或いは、半導体基板１０３の研磨と同時に、半導体基板１０３が当接する位置とは別の位置でＣＭＰコンディショナーを研磨布１０２に当接させるかして行われる。

【０００６】

【発明が解決しようとする課題】従来の研磨布のコンディショニングに用いられるＣＭＰコンディショナーでは、図９に示すように、円板状の支持部材９０１の表面に、硬質砥粒としてダイヤモンド粒２０２を人手で撒く等して適当に均一に分布させた後、これらダイヤモンド粒２０２を固着させていた。しかし、この場合、いかに丁寧にダイヤモンド粒２０２を散布したとしても、ダイヤモンド粒２０２は平均一化分布し、ＣＭＰコンディショナー個体間での製造の原因となり、安定したＣＭＰコンディショナー特性の発現が妨げられていた。

【０００７】また、従来のＣＭＰコンディショナーでは、スラリーの逃げが悪いため、マイクロスクラッチが多くなった。また、スラリーの逃げを改善するためには、図１０に示すように、支持部材２０１に化学スラリー１０１を逃すための逃し溝２０３等を形成しておき、研磨時に、この逃し溝２０３を介して化学スラリー１０１を逃すことが公知されていた。しかし、支持部材２０１に逃し溝２０３を形成するのでは、ＣＭＰコンディショナー特性に悪影響を与えるおそれがあり、また、その逃し溝の加工に手間がかかり、コストアップの原因となってしまう。

【０００８】本発明は前記のような点に鑑みてなされたものであり、安定したＣＭＰコンディショナー特性が得られるとともに、逃し溝等を形成しなくとも研磨時にスラリー等を逃すことができ、マイクロスクラッチを減らすようにすることを目的とする。

【０００９】

【課題を解決するための手段】本発明のＣＭＰコンディショナーは、支持部材と、前記支持部材の面上に設けられた複数の硬質砥粒とを備えたＣＭＰコンディショナーであって、前記支持部材の面上に、前記複数の硬質砥粒を規則的に、かつ、前記支持部材の内側から外側にかけて密度が減少するように配列させた点に特徴を有する。

【００１０】本発明の他のＣＭＰコンディショナーは、支持部材と、前記支持部材の面上に設けられた複数の硬質砥粒とを備えたＣＭＰコンディショナーであって、前記支持部材の面上に、前記複数の硬質砥粒が存在しない領域を略放射状に確保している点に特徴を有する。

【００１１】本発明のＣＭＰコンディショナーに使用する硬質砥粒の配列方法は、規則的に、かつ、内側から外側にかけて密度が減少するように配列させた複数の貫通穴が形成された薄板状の配列部材を被配列面上に位置させる手順と、前記配列部材の各貫通穴に硬質砥粒を入れ込む手順とを有する点に特徴を有する。

【００１２】本発明の他のＣＭＰコンディショナーに使用する硬質砥粒の配列方法は、複数の貫通穴の存在しない領域が略放射状に確保された薄板状の配列部材を被配列面上に位置させる手順と、前記配列部材の各貫通穴に硬質砥粒を入れ込む手順とを有する点に特徴を有する。

【００１３】本発明の他のＣＭＰコンディショナーに使用する硬質砥粒の配列方法は、複数の硬質砥粒を規則的に、かつ、内側から外側にかけて密度が減少するように配列させた状態で保持部材に保持する手順と、前記保持部材により保持された硬質砥粒を、ＣＭＰコンディショナーを構成する支持部材の表面に転写する手順とを有する点に特徴を有する。

【００１４】本発明の他のＣＭＰコンディショナーに使用する硬質砥粒の配列方法は、複数の硬質砥粒の存在しない領域が略放射状に確保された状態で前記複数の硬質砥粒を保持部材に保持する手順と、前記保持部材により保持された硬質砥粒を、ＣＭＰコンディショナーを構成する支持部材の表面に転写する手順とを有する点に特徴を有する。

【００１５】本発明のＣＭＰコンディショナー製造方法は、前記ＣＭＰコンディショナーに使用する硬質砥粒の配列方法を利用して前記硬質砥粒を前記支持部材の表面上に配列させた後、前記硬質砥粒を前記支持部材の表面に固着する点に特徴を有する。

【００１６】

【発明の実施の形態】以下、図面を参照して、本発明の半導体基板用研磨布のＣＭＰコンディショナー、半導体基板用研磨布のＣＭＰコンディショナーに使用する硬質砥粒の配列方法、及びＣＭＰコンディショナー製造方法の実施の形態について説明する。

【００１７】図１を用いて、ＣＭＰコンディショナーについて説明する。同図に示すように、ステンレス鋼等からなる円板状の支持部材１の表面には、硬質砥粒としてダイヤモンド砥粒２が固着されている。

【００１８】図２、３には、支持部材１の表面におけるダイヤモンド粒２の配列の概要を示す。図２に示す例は、円板状の支持部材１の中心から放射状に伸びる複数の直線（一点鎖線Ｌ）を考え、それら直線上にダイヤモンド粒２を配置したものである。このようにしたＣＭＰコンディショナーでは、ダイヤモンド粒２が支持部材１の内側から外側にかけて密度が小さくなるように配列されており、支持部材１の表面上には、ダイヤモンド粒２の存在しない領域が放射状に複数形成されることになる。

【００１９】また、図３に示す例は、円板状の支持部材１の中心から放射状に伸びる複数の曲線（一点鎖線Ｌ）を考え、それら曲線上にダイヤモンド粒２を配置したものである。このようにしたＣＭＰコンディショナーでは、ダイヤモンド粒２が支持部材１の内側から外側にかけて密度が小さくなるように配列されており、支持部材１の表面上には、ダイヤモンド粒２が存在しない領域が放射状に複数形成されることになる。本発明でいう略放射状とは、図２に示すように直線的に放射する場合だけでなく、図３に示すように曲線的に放射する場合も含むものとする。

【００２０】なお、実際のダイヤモンド粒２は、支持部材１に比べて非常に小さなものであるが、図１や後述する図２、３では、説明を簡単にするためダイヤモンド粒２を大きく図示する。また、直線や曲線の数についても、より密な状態で放射させるようにするが、図２、３では簡単に図示する。

【００２１】以下、図４～７を参照して、ダイヤモンド粒２の配列方法について説明する。本実施の形態では、次の２通りの方法により、ダイヤモンド粒３を配列させている。

【００２２】第１の方法では、図４に示すように、ろう材３が設けられた支持部材１の表面に、粘着剤４を塗布しておく。そして、塗着剤４を塗布した支持部材１の表面上に配列板５を載置して、マスキングする。

【００２３】図５に示すように、配列板５には、ダイヤモンド粒２を配列させるための貫通穴６が形成されている。すなわち、配列板５には、図２や図３に示す配列と同様に貫通穴６を配列させている。貫通穴６の口径Ｘは、ダイヤモンド粒２のサイズＤに対して、１．０Ｄ＜Ｘ＜２．０Ｄとなっており、１つの貫通穴６に１個以上のダイヤモンド粒２が同時に入り込まないようにしている。なお、配列板５の周囲には、位置決め用穴５ａが設けられている。

【００２４】図４に示すように、前記配列板５を支持部材１の表面に載置した状態で、配列板５上にダイヤモンド粒２を散布する。このとき、配列板５に適当な振動を加えるなどして、ダイヤモンド粒２が全ての貫通穴６に入り込むようにする。全ての貫通穴６にダイヤモンド粒２が入り込んだならば、配列板５上の余分なダイヤモンド粒２をはけ等を用いて取り除く。その後、配列板５を支持部材１の表面から取り外せば、ダイヤモンド粒２は、図２や図３に示すように配列された状態で支持部材１の表面上に残ることになる。

【００２５】以上述べたようにして支持部材１の表面にダイヤモンド粒２を配列させたならば、半田、ろう付けを行い、ダイヤモンド粒２を固定する。このろう付けの際に、支持部材１の表面に塗布された塗着剤４はろう材３への浸漬によって蒸発し、支持部材１の表面上に残留しない。

【００２６】なお、第１の方法において、配列板５の代わりに、ワイヤで編まれたメッシュを用いてもよい。すなわち、メッシュの各網目部分を配列板５でいう貫通穴６として使用し、該網目部分にダイヤモンド粒２を入れ込んで、支持部材１の表面に配列させる。

【００２７】第２の方法では、前記第１の方法のようにダイヤモンド粒２を支持部材１の表面に直接的に配列するのではなく、粘着シート等の保持部材にいったん配列させてから、支持部材１の表面に転写するようにしている。

【００２８】図６（ａ）に示すように、配列板７には、ダイヤモンド粒２を配列させるための凹部８が形成されている。すなわち、配列板７には、図２や図３に示す配列と同様に凹部８を配列させている。なお、凹部８の口径Ｘも、ダイヤモンド粒サイズＤに対して、１．０Ｄ＜Ｘ＜２．０Ｄとすることは、前記第１の方法で述べた貫通穴６と同じである。

【００２９】前記配列板７上にダイヤモンド粒２を散布する。このときも、前記第１の方法で説明したように、配列板７に適当な振動を加えるなどして、ダイヤモンド粒２が全ての凹部８に入り込むようにする。全ての凹部８にダイヤモンド粒２が入り込んだならば、配列板７上の余分なダイヤモンド粒２をはけ等を用いて取り除く。

【００３０】次に、配列板７の凹部８が開口する面に粘着シート１０を貼り付ける。そして、図６（ｂ）に示すように、配列板７の上下を逆にする等して、粘着シート１０を剥がすと、粘着シート１０にダイヤモンド粒２が配列された状態で保持されることになる。

【００３１】前記粘着シート１０のダイヤモンド粒２を保持する粘着面を、塗着剤４が塗布された支持部材１の表面に貼り合わせるようにする。したがって、図７に示すように、ダイヤモンド粒２は、一端が粘着シート１０側で、他端が支持部材１の表面側で支持された状態となる。その後、支持部材１の表面側にダイヤモンド粒２を残し、粘着シート１０だけを取り除けば、ダイヤモンド粒２を支持部材１の表面上に配列させることができる。

【００３２】粘着シート１０だけを取り除く手法としては、例えば、粘着シート１０の接着材の溶解性と、支持部材１側の接着剤の溶解性とに差を持たせておけばよい。この場合、図７に示す状態で粘着シート１０の接着剤が溶けるような溶媒にすれば、支持部材１側の接着剤

４は保持力を維持したまま、粘着シート１０の接着力だけを弱め、粘着シート１０だけを取り除くことができる。

【００３３】以上述べたようにして支持部材１の表面にダイヤモンド粒２を配列させたならば、準備、ろう付けを行い、ダイヤモンド粒２を固定する。このろう付けの際に、支持部材１の表面に塗布された接着剤４はろう材３への拡散によって消滅し、支持部材１の表面上に残留しない。

【００３４】なお、第２の方法では、配列板７に凹部８を形成するようにしたが、貫通穴としてよい。この場合、図４に示す支持部材１を粘着シート１０に変更すれば、粘着シート１０にダイヤモンド粒を配列させることができるので、それを支持部材１の表面に転写すればよい。

【００３５】以上述べたように本実施の形態によれば、ダイヤモンド粒２を規則的に配列させているので、ＣＭＰコンディショナー面での凸体積がなくなり、安定したＣＭＰコンディショナー特性を得ることができる。また、ダイヤモンド粒２を、支持部材１の中心から放射状に配列させることにより、支持部材１の内側から外側にかけて密度が小さくなるよう配列するようにし、また、ダイヤモンド粒２が存在しない領域を放射状に確保するようにしたので、研磨時にスラリーを支持部材１の外側に向けて逃すことができ、マイクロスクラッチが減少する。そして、スラリーを逃すための特別な加工を支持部材１に施す必要がなくなるので、加工の手間やコストを軽減させることができる。

【００３６】なお、本実施の形態では、本発明という硬質砥粒としてダイヤモンド粒２を用いたが、その他の材質、例えば立方晶窒化ホウ素、窒化ホウ素、炭化珪素又は酸化アルミニウム等からなるものであってもよい。

【００３７】また、ダイヤモンド粒２の支持部材１への固着方法としては、ろう付け以外の方法、例えばニッケル電着等により固着させてもよい。

【００３８】ここで、参考の一例として、ダイヤモンド粒をろう付けにより固着する方法について説明すると、ろう材として、チタン、クロム、又はジルコニウムより選ばれた１種以上を０．５～２０ｗｔ％含む融点６５０℃～１２００℃の合金を用いることにより、ダイヤモンド粒とろう付け合金との界面に当該金属の炭化物層が形成される。ろう材に含まれるチタン、クロム、又はジルコニウムより選ばれた１種以上を０．５～２０ｗｔ％とするのは、０．５ｗｔ％より少ない含有量ではダイヤモンド－ろう付け合金の界面に当該金属の炭化物層が形成されないためであり、２０ｗｔ％添加すれば十分な接合強度を示す炭化物層が形成されるためである。

【００３９】ろう付け合金を融点６５０℃～１２００℃の合金とするのは、６５０℃未満のろう付け温度では、接合強度が得られず、１２００℃超のろう付け温度では、ダイヤモンドの劣化が起こるので好ましくないからである。ろう付け合金としては、例えば、上記成分を含有するニッケル（Ｎｉ）基合金等が有効に用いることができる。

【００４０】ろう付け合金の厚さは、ダイヤモンド粒の０．２～１．５倍の厚さが適当である。薄すぎると、ダイヤモンドとろう付け合金との結合強度が低くなり、厚すぎると、ろう材と支持部材との剥離がおこりやすくなるためである。

【００４１】ダイヤモンド粒の径は、５０μｍ～３００μｍとすることが好ましい。５０μｍ未満の微細ダイヤモンド粒では、十分な研磨速度が得られず、また、凝集しやすい傾向があり、脱落しやすくなるためである。また、３００μｍ超の粗粒のダイヤモンド粒では、研磨時の応力集中が大きくなり、脱落しやすくなるためである。

【００４２】

【発明の効果】以上述べたように本発明によれば、ＣＭＰコンディショナー面での凸体積がなくなり、安定したＣＭＰコンディショナー特性を得ることができるので、安定した量産ＣＭＰプロセスを実現することが可能となる。また、研磨時にスラリーを逃すことができ、マイクロスクラッチを減らすことができ、しかも、そのための特別な加工を支持部材に施す必要がなくなるので、加工の手間やコストを軽減させることができる。

【図面の簡単な説明】

【図１】ＣＭＰコンディショナーについて説明するための図である。

【図２】ダイヤモンド粒２の配列の一例を示す図である。

【図３】ダイヤモンド粒２の配列の一例を示す図である。

【図４】第１の方法によるダイヤモンド粒２の配列方法を説明するための図である。

【図５】配列板５を説明するための図である。

【図６】第２の方法によるダイヤモンド粒２の配列方法を説明するための図である。

【図７】第２の方法によるダイヤモンド粒２の配列方法を説明するための図である。

【図８】ＣＭＰ工程を説明するための図である。

【図９】従来のＣＭＰコンディショナーについて説明するための図である。

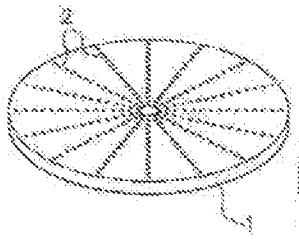
【図１０】逃し溝２０３を形成したＣＭＰコンディショナーを示す模式図である。

【符号の説明】

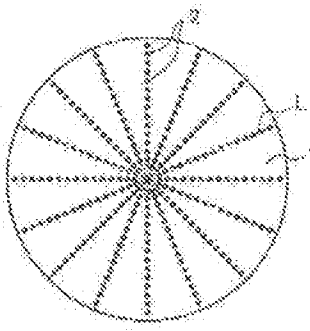
１　支持部材
２　ダイヤモンド粒
３　ろう材
４　接着剤
５　配列板

5a　飛散防止用壁
6　貫通穴
7　配列板
8　回転
9　ばね
10　発泡シート
101　化学スプリー
102　研磨布
103　半導体基板

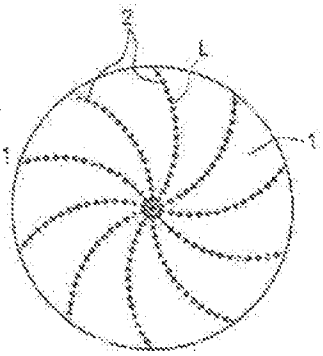
【図1】



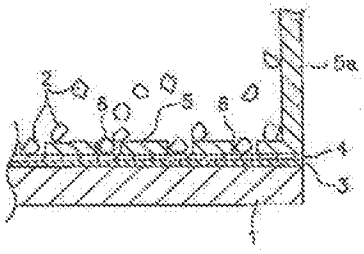
【図2】



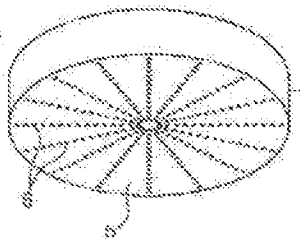
【図3】



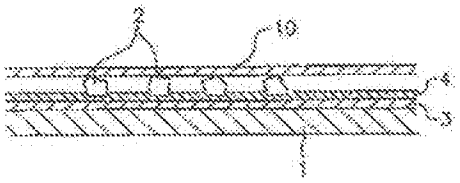
【図4】



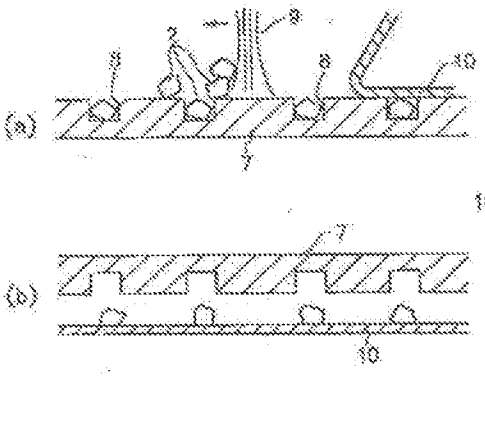
【図5】



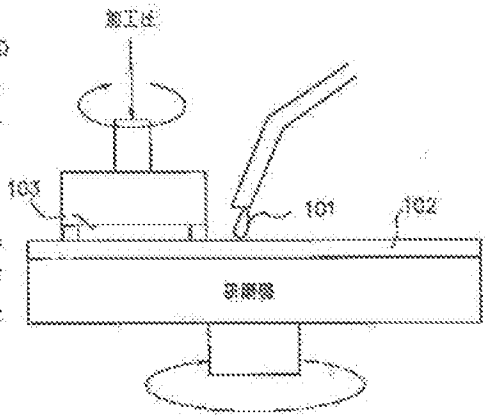
【図7】



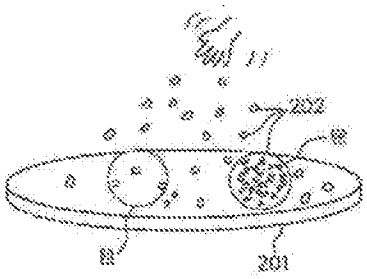
【図6】



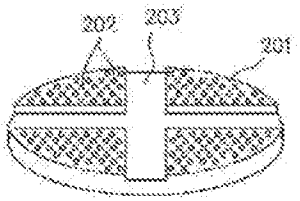
【図8】



【図９】



【図１０】



フロントページの続き

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３Ｑ０８３　ＡＡ１０　ＡＤ０５　ＢＢ０２　ＢＤ０２　ＢＫ０７　ＥＥ２０　ＦＦ２３

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Bibliographic data: JP2000167774 (A) — 2000-06-20

MANUFACTURE OF DIAMOND CUTTER AND DIAMOND CUTTER AND DIAMOND CUTTER MANUFACTURING JIG

Inventor(s): YOSHIDA TAKUMA; FUKADA NOBUO ± (YOSHIDA TAKUMA, ; FUKADA NOBUO)

Applicant(s): TOHO TITANIUM CO LTD; SANKYO DIAMOND KOGYO KK ± (TOHO TITANIUM CO LTD, ; SANKYO DIAMOND KOGYO KK)

Classification: **- international:** ***B24D3/00; B24D5/12;*** (IPC1-7): B24D3/00; B24D3/00; B24D5/12

- European:

Application number: JP19980301722 19981009

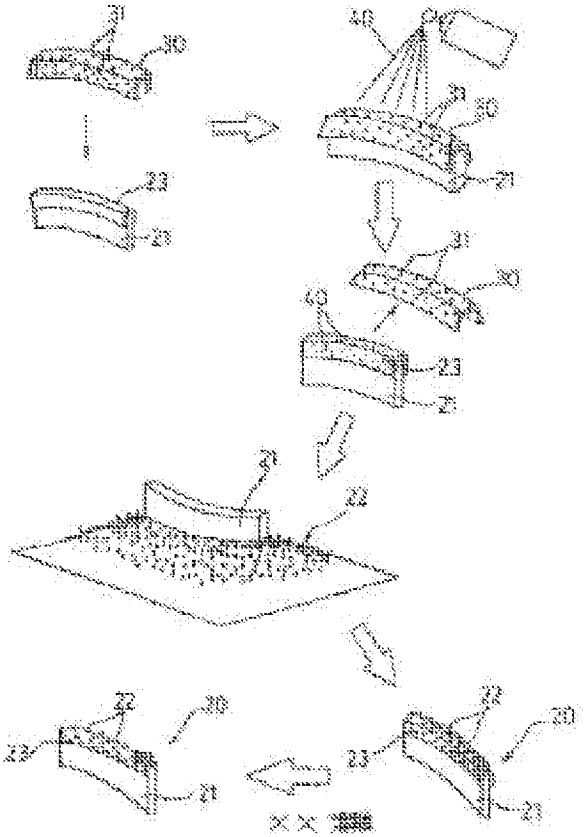
Priority number (s): JP19980301722 19981009

Also published as: JP4269018 (B2)

Abstract of JP2000167774 (A)

PROBLEM TO BE SOLVED: To increase fixation strength of diamond particles to prevent diamond particles from falling out by fixing diamond particles forming a diamond layer on the diamond layer at a predetermined interval mutually. SOLUTION: A manufacturing method of a diamond cutter includes a bond paste fixation process in which bond (registered trademark) paste in which binder and bond powders are blended is attached on a substrate, a screen covering process in which a screen 30 in which holes are drilled to a desired manner after the bond paste fixation process is covered in a bond paste fixation scope, an application process in which viscous substance 40 is applied from above the screen 30 on which the bond paste is covered, a screen removing process in which the screen 30 is removed after the application process,; and a diamond abrasive grain adhesion process in which diamond abrasive grains 22 come into contact with a bond paste face on which the viscous substance 40 adheres from a hole drilled part 31 of the screen 30 to form the substrate and the diamond layer integrally with each other by melting the bond after binder removing treatment.

Last updated: 04.12.2012 Worldwide
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特開2000－167774
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(43)公開日　平成12年６月20日(2000.6.20)

(51)Int.Cl.'		識別記号	F I		テーマコード'（参考）
Ｂ２４Ｄ	3/00	３４０	Ｂ２４Ｄ　3/00	３４０	３Ｃ０６３
		３２０		３２０Ｂ	
	5/12		5/12	Ｚ	

審査請求　未請求　請求項の数３　ＦＤ　（全　８　頁）

(21)出願番号　特願平10－301722

(22)出願日　平成10年10月９日(1998.10.9)

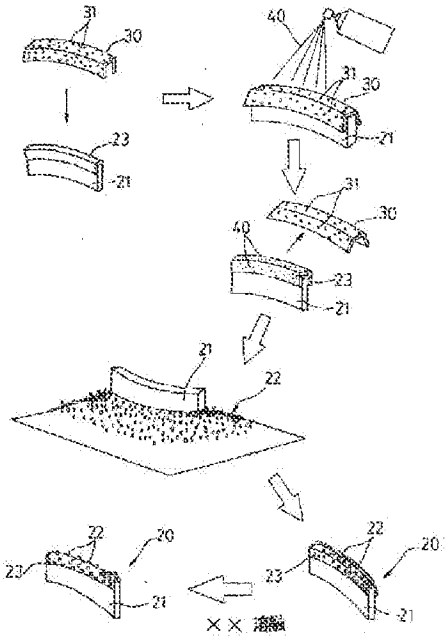
特許法第64条第２項ただし書の規定により図面第１図，４図，５図及び選択図の一部は不掲載とした。

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(54)【発明の名称】　ダイヤモンドカッターの製造方法及びダイヤモンドカッター並びにダイヤモンドカッター製造治具

(57)【要約】
【課題】　本発明の目的はダイヤモンド層を形成するダイヤモンド粒子が互いに所定間隔をもってダイヤモンド層に固着され、ダイヤモンド粒子の固着強度を高めてダイヤモンド粒子の脱落を防止できるダイヤモンドカッターの製造方法及びダイヤモンドカッター並びにダイヤモンドカッター製造治具を提供する。
【解決手段】　ダイヤモンドカッターＣの製造方法は、基板１０にバインダとボンド粉とを混練したボンド（登録商標）ペーストを取着させるボンドペースト取着工程と、ボンドペースト取着工程の後で所望に穿孔されたスクリーン３０をボンドペースト取着範囲に被覆するスクリーン被覆工程と、ボンドペーストを被覆したスクリーン３０上から粘性物質４０を塗布する塗布工程と、塗布工程の後でスクリーン３０を取り除くスクリーン除去工程と、スクリーン３０の穿孔部分３１から粘性物質４０が付着したボンドペースト面にダイヤモンド砥粒２２を接触させるダイヤモンド砥粒付着工程と、脱バインダ処理後ボンドを溶融させることによって基板１０とダイヤモンド層を一体に形成する。



【特許請求の範囲】

【請求項１】　取付部が形成された基板と、該基板の外周端面に取着されたダイヤモンド層と、を備えたダイヤモンドカッターの製造方法において、前記基板にバインダとボンド粉とを混練したボンドペーストを取着させるボンドペースト取着工程と、該ボンドペースト取着工程の後で所望に穿孔されたスクリーンを前記ボンドペースト取着範囲に被覆するスクリーン被覆工程と、前記ボンドペーストを被覆したスクリーン上から粘性物質を塗布する塗布工程と、該塗布工程の後で前記スクリーンを取り除くスクリーン除去工程と、前記スクリーンの穿孔部分から粘性物質が付着した前記ボンドペースト面にダイヤモンド砥粒を接触させるダイヤモンド砥粒付着工程と、脱バインダ処理後前記ボンドを溶融させることによって基板とダイヤモンド層を一体に形成してなることを特徴とするダイヤモンドカッターの製造方法。

【請求項２】　取付部が形成された基板と、該基板の外周端面に取着されたダイヤモンド層と、を備えたダイヤモンドカッターであって、前記ダイヤモンド層はボンド材料により所定間隔でダイヤモンド砥粒が配列されて前記基板に固定されていることを特徴とするダイヤモンドカッター。

【請求項３】　取付部が形成された基板と、該基板の外周端面に取着されたダイヤモンド層と、を備えたダイヤモンドカッターの製造過程で用いられるダイヤモンドカッター製造治具であって、ダイヤモンド層となる部分を被覆するカバー部材と、該カバー部材に所望間隔で形成された多数の小孔とから形成されていることを特徴とするダイヤモンドカッター製造治具。

【発明の詳細な説明】

【０００１】

【発明の属する技術分野】本発明はダイヤモンドカッターの製造方法及びダイヤモンドカッター並びにダイヤモンドカッター製造治具に係り、特に石材、コンクリートなどの切断に用いるのに好適なダイヤモンドカッターの製造方法及びダイヤモンドカッター並びにダイヤモンドカッター製造治具に関するものである。

【０００２】

【従来の技術】従来、取り付け部を有する基板の外周にダイヤモンド層が形成されたダイヤモンドカッターは公知である。そして、このようなダイヤモンドカッターとしては、例えば特公昭５５－２２１９４号公報で示されているように、「一時的な結合剤を使用して金属基材表面にダイヤモンド結晶とろう付け用合金の粒子を附着させて組立てたものを造る工程と、該組合せを還元性雰囲気または真空中で約１３００℃以下の温度迄に加熱してろう付け用合金を熔融する工程、該組合せを冷却して該ろう付け用合金を固化させダイヤモンドを基材に結合する工程よりなり、ここに該ろう付け用合金はニッケルおよびコバルトからなる群から選ばれた１種または２種の金属の少なくとも５０重量％、クロムの２～２６重量％、および硼素、珪素、燐より成る群より選ばれた１種または２種以上の元素の合計１２重量％以下よりなる、金属基材にダイヤモンド結晶を結合する」技術が知られている。

【０００３】また特開平９－２７２０６０号公報で示されているように、「化学的または物理的に表面処理された前記砥石刃取付部に、有機糊剤と結合材として自溶合金粉末の混合物を塗布し、その後乾燥させ、さらに有機糊剤を塗布し、砥粒を散布し、乾燥させ次いで、非酸化性雰囲気中において９００～１１５０°Ｃの加熱温度に加熱し、前記自溶合金の融着により前記砥石刃取付部に前記砥粒を固定し、前記加熱温度から徐々に冷却する」技術が知られている。

【０００４】上記従来技術では、基板等に取着させたボンドペーストにダイヤモンド砥粒を附着させるときに篩を利用したり、直接散布することにより、ダイヤモンド層を形成している。

【０００５】

【発明が解決しようとする課題】上述のように、ダイヤモンド砥粒を篩や散布することによっているため、ダイヤモンド層を形成するボンドペーストにダイヤモンド粒子が、一部分に団塊となって附着したり、ダイヤモンド粒子同士が隣接して結合することになり、ダイヤモンド粒子の固着が弱くなるという不都合があった。すなわち、このようにダイヤモンド粒子が団塊になったり、ダイヤモンド粒子が隣接した状態は、ダイヤモンド粒子を固定するためのボンドペーストが、ダイヤモンド粒子の周りに少ない状態となり、ダイヤモンド粒子の固着力の低下を意味するものである。

【０００６】本発明の目的は、ダイヤモンド層を形成するダイヤモンド粒子が互いに所定間隔をもってダイヤモンド層に固着され、ダイヤモンド粒子の固着強度を高めてダイヤモンド粒子の脱落を防止できるダイヤモンドカッターの製造方法及びダイヤモンドカッター並びにダイヤモンドカッター製造治具を提供することにある。

【０００７】

【課題を解決するための手段】上記課題は、請求項１のダイヤモンドカッターの製造方法によれば、取付部が形成された基板と、該基板の外周端面に取着されたダイヤモンド層と、を備えたダイヤモンドカッターの製造方法において、前記基板にバインダとボンド粉とを混練したボンドペーストを取着させるボンドペースト取着工程と、該ボンドペースト取着工程の後で所望に穿孔されたスクリーンを前記ボンドペースト取着範囲に被覆するスクリーン被覆工程と、前記ボンドペーストを被覆したスクリーン上から粘性物質を塗布する塗布工程と、該塗布工程の後で前記スクリーンを取り除くスクリーン除去工程と、前記スクリーンの穿孔部分から粘性物質が付着した前記ボンドペースト面にダイヤモンド砥粒を接触させ

るダイヤモンド砥粒付着工程と、脱バインダ処理後前記ボンドを溶融させることによって基板とダイヤモンド層を一体に形成してなる構成とすることにより、解決される。

【０００８】上記のように、ボンドペースト取着工程の後で所望に穿孔されたスクリーンを前記ボンドペースト取着範囲に被覆するスクリーン被覆工程と、前記ボンドペーストを被覆したスクリーン上から粘性物質を塗布する塗布工程を行なうことにより、ボンドペーストに塗布される粘性物質は、スクリーンを除去すると、スクリーンの小孔が形成された範囲だけとなる。したがって、スクリーンを除去した後でダイヤモンド砥粒を接触させると、ダイヤモンド粒子は、粘性物質が塗布された個所、すなわちスクリーンに形成された小孔の位置に付着することになる。このようにしてスクリーンに形成された小孔にしたがって、ダイヤモンド粒子を所定間隔で付着させたダイヤモンド層を形成することができる。

【０００９】また、上記課題は、請求項２に係るダイヤモンドカッターによれば、取付部が形成された基板と、該基板の外周端面に取着されたダイヤモンド層と、を備えたダイヤモンドカッターであって、前記ダイヤモンド層はボンドペーストにより所定間隔でダイヤモンド砥粒が配列されて前記基板に固定されている構成とすることにより、解決される。

【００１０】このようなダイヤモンド層はボンドペーストにより所定間隔でダイヤモンド砥粒が配列されて前記基板に固定されているダイヤモンドカッターによれば、ダイヤモンド粒子が所定間隔で配置されているために、ダイヤモンド粒子の固着強度が高まる。そして、ダイヤモンド層に均一にダイヤモンド粒子を配置して固着されているために、被切削材に対して均一な切り口となり、切削がスムーズに行なわれることになる。

【００１１】さらに、上記課題は、請求項３に係るダイヤモンドカッター製造治具によれば、取付部が形成された基板と、該基板の外周端面に取着されたダイヤモンド層と、を備えたダイヤモンドカッターの製造過程で用いられるダイヤモンドカッター製造治具であって、ダイヤモンド層となる部分を被覆するスクリーン部材と、該スクリーン部材に所望間隔で形成された多数の小孔とから形成されている構成とすることにより、解決される。

【００１２】このようなスクリーン部材によれば、スクリーン部材に形成する小孔の間隔や数を調整することにより、ダイヤモンド粒子を一定間隔で所望数配置することが可能となり、ダイヤモンドカッターにおけるダイヤモンド粒子の配置調整が可能となる。またダイヤモンド粒子が団塊になったり、隣接して配置されることがないために、各ダイヤモンド粒子が溶融ボンドによって確実に基板に固定できることになる。

【００１３】

【発明の実施の形態】ここでは、ダイヤモンドカッターとしてダイヤモンドチップを用いた例を示すが、これに限定されるものではなく、基板に直接ダイヤモンド層を形成してもよい。本発明のダイヤモンドカッターＣは、基板１０にダイヤモンドチップ２０を取着しており、このダイヤモンドチップは、基台にバインダペーストとボンド粉とを混練したボンドペーストを取着させ、ボンドペースト取着範囲に所望に穿孔されたスクリーンを被覆し、このスクリーン上から粘性物質を塗布し、塗布工程の後でスクリーンを取り除き、スクリーンの穿孔部分から粘性物質が付着したボンドペースト面にダイヤモンド砥粒を接触させ、脱バインダ処理後ボンドを溶融させることによって基板とダイヤモンド層を一体に形成することにより製造することが可能となる。

【００１４】以上のように、ダイヤモンド砥粒のばらつきがなく、均等に配置することが可能となり、ダイヤモンド層の全ての部分で切削が均等に行うことが可能となる。

【００１５】

【実施例】以下、本発明の一実施形態について、図を参照して説明する。なお、以下に説明する部材、配置等は、本発明を限定するものではなく、本発明の趣旨に沿って各種改変することができることは勿論である。

【００１６】図１乃至図６は本発明に係る実施例を示すものであり、図１はダイヤモンドカッターの一例としてダイヤモンドチップとして適用したときの製造工程を示す説明図、図２乃至図４は製造工程の拡大説明図、図５は製造工程のブロック図、図６はダイヤモンドカッターの一例を示す正面図である。また図７はダイヤモンドカッター製造治具としてのスクリーンの例を示す説明図である。

【００１７】本例のダイヤモンドカッターＣは、図６で示すように、基板１０と、ダイヤモンドチップ２０と、ダイヤモンド粒子（砥粒）２２と、を主要構成要素としており、ダイヤモンドチップ２０は、基台２１とダイヤモンド粒子（砥粒）２２とを固定するボンドペースト層２３から構成されている。そして上記円形基板１０の外周に、ダイヤモンド粒子（砥粒）２２が取着されたダイヤモンドチップ２０を焼結等により固着している。

【００１８】本例の基板１０は、鋼製であり、この鋼製基板１０は中心に取付孔１１が形成され、円形形状として形成されている。本例の基板１０は、冷間プレスで成形されている。また、本例の基板１０は、外周端面が同一円の面に形成され、所定間隔で冷却用溝等が形成されているものを用いている。このとき、基板１０の外周端面に若干の凹凸等が形成されている場合には、基台２１を基板外周端面と整合するような形状に成形する。しかし、外周端面に若干の凹凸等が形成されたり、溝を設けていないものを用いても良い。

【００１９】次に本発明に係るダイヤモンドカッターＣの製造方法について、図１乃至５を参照して説明する。

本発明のダイヤモンドカッターCは、基板１０にダイヤモンドチップ２０を取着しており、このダイヤモンドチップ２０は、基台２１にバインダペーストとボンド粉とを混練したボンドペースト２３を取着させ、ボンドペースト取着範囲に所望に穿孔されたスクリーン３０を被覆し、このスクリーン３０上から粘性物質４０を塗布し、塗布工程の後でスクリーン３０を取り除き、スクリーン３０の穿孔部分から粘性物質４０が付着したボンドペースト面にダイヤモンド砥粒２２を接触させ、少なくともボンドペースト２３中のバインダを除去した後、ダイヤモンド砥粒２２の周辺部分のボンドを溶融させて基板１０とダイヤモンド層を一体に形成することにより製造する。これをさらに詳細に説明する。

【００２０】はじめにペースト用バインダとボンド粉とを混練してボンドペースト２３を作成する。ボンドペースト２３を構成するペースト用バインダとボンド粉は、公知の材料を使用するものであり、本例で使用するボンドペースト２３としては、ダイヤとの化学的親和力の高いＴｉ、Ｃｒ等を含むボンドであり、ダイヤに対するボンドの濡れが良く、ダイヤ保持力がかなり強化されている。基台２１（基板１０）との接着力は、ボンドと基台２１（基板１０）が化学的に結合され、溶融に伴う拡散もあるので、接着力は比較的強固となる。具体的にはＣｕ－Ｓｎ－Ｔｉ系や、Ｎｉロウ系のボンドと、各種のバインダ用ペーストを用いてボンドペーストを形成している。

【００２１】本例で用いる粘性物質４０としては、例えば、市販の水溶性メチルセルロースを約３．３倍量の水で希釈した有機糊剤、その他糊材等の粘着のある物質を使用する。

【００２２】なお溶融のボンド合金化は９４０℃～９８０℃が適正温度範囲であり、ボンドとダイヤの濡れ具合が良好となる。また適正脱脂条件について説明すると、脱脂の昇温速度が速すぎると急激なバインダの分解ガスでボンドとダイヤの飛散が生じる。そこで、真空脱脂はバインダ熱分解温度まで５℃／ｍｉｎで昇温し２０ｍｉｎ保持の条件が有効である。使用金属粉としては、ブロンズ粉が溶融温度が低めで溶融状態が安定している。またＣｕＰの添加をすることにより、濡れ性を改善することができる。

【００２３】次に図１に示すように、上記混練されたペースト用バインダとボンド粉からなるボンドペースト２３を、ディスペンサなどの射出機や刷毛を使用して、ダイヤモンドチップ２０を構成する基台２１に塗布等により取着する。そして、次に述べるスクリーン３０を固着させないように、若干の所定時間を経過させて、乾燥させる。

【００２４】次いで、基台２１のボンドペースト２３の取着範囲にスクリーン３０を被覆する。本例のスクリーン３０は、断面コ字状の枠体であり、ボンドペースト２３が取着された基台端部に嵌合可能に形成されている。スクリーン３０はボンドペースト２３の取り付けを考慮して、基台２１より若干大き目で基台２１と略同形状に形成されている。そして、基台２１にスクリーン３０を被覆するように嵌合させる。

【００２５】なおスクリーン３０は基台端部を連続して被覆するように形成しても良く、或いは所定長さに形成し、この所定長さに形成された複数のスクリーン３０を用いて基台端部を被覆するように構成しても良い。スクリーン３０には、所望箇所に多数の小孔３１が穿孔されており、この小孔３１を介して、粘性物質４０が基台２１に塗布されるように構成されている。

【００２６】本例のスクリーン３０は、アルミ等の金属から形成されている。このようにスクリーン３０を金属製とすることにより、表面や小孔３１に付着した粘性物質４０を取り除いて再使用することが可能となり、低コスト化を実現するとともに、廃棄物を減少させて環境への影響を抑えることができる。またスクリーン３０を単一金属で形成することにより、リサイクルが可能となり資源の有効利用を図ることが可能となる。

【００２７】上記スクリーン３０の形状は、上記形状に限定されるものではなく、基台２１（基板１０）の端部形状に合わせて、各種形成するものである。例えば図７（ａ）に示すスクリーン３０は、端部側が薄く中心部に向けて厚みが増した形状の基台２１（基板１０）に用いられるものであり、図７（ｂ）に示すスクリーン３０は、端部がアール形状の基台２１（基板１０）に用いられるものである。さらに図７（ｃ）に示すスクリーン３０は端部側が薄く、且つ端部がアール形状の基台２１（基板１０）に用いられるものである。

【００２８】また、スクリーン３０は上記金属製に限らず、紙製或いは樹脂フィルム製とすることもできる。紙製や樹脂フィルム製のスクリーン３０とすれば、製造時において、各基台２１（基板１０）に常に新しいスクリーン３０を使用することが可能となり、生産効率の向上を図ることができる。

【００２９】そして基台２１に配設されたスクリーン３０に、粘性物質４０をディスペンサなどの射出機や刷毛を使用して塗布する。塗布された粘性物質４０は、スクリーン３０の小孔３１を介して基台２１に付着する。

【００３０】粘性物質４０を塗布後、基台２１からスクリーン３０を取り外す。スクリーン３０が取り外された基台２１表面には、図３に示すように、スクリーン３０の小孔３１と同様のパターンで粘性物質４０が付着している。

【００３１】次に基台２１にダイヤモンド砥粒２２を付着させる。ダイヤモンド砥粒２２としては通常４０～５０メッシュのダイヤモンド粒子を使用する。ダイヤモンド砥粒２２は、図１及び図３に示すように、予め作業台等の上に所定量が載せられており、このダイヤモンド砥

粒２２中に前記基台２１の粘性物質４０が付着した側を接触させ、基台２１にダイヤモンド砥粒２２を付着させる。

【００３２】次に基台２１にダイヤモンド砥粒２２を一体に形成するため、加熱炉の炉内にセットし、排気を行う。処理は、乾燥、昇温、減圧脱脂、溶融、炉冷（真空冷却）、取り出しの順で行なう。なお製造における加熱条件としては、温度１２０℃で乾燥し、５℃／ｍｉｎで昇温し、脱脂は、１８０℃２０ｍｉｎ保持～３８０℃２０ｍｉｎ保持で行なうと好適である。次に溶融について昇温を３０℃／ｍｉｎで行い、９００℃～１１００℃で１５ｍｉｎ保持する。次に、炉冷（真空冷却）を行なうが、約7℃／ｍｉｎで行なう。さらに外熱炉体移動を６００℃で行ない、その後取り出す。なお上記では、溶融接合時の炉内真空度１０－５～１０－６Ｔｏｒｒとしている。

【００３３】上記製造工程により、基台２１にダイヤモンド粒子を単一層に保持したダイヤモンドチップ２０を得ることができる。そしてこのダイヤモンドチップ２０を基板１０に取着してダイヤモンドカッターＣが形成される。なお上記実施例では複数の基台２１にダイヤモンド砥粒２２を取着させ、基台２１を基板１０に貼着した構成例を示したが、基板１０に直接ダイヤモンド砥粒２２を取着させた構成としても良い。またダイヤモンドカッターＣとしては、円形基板の外周にダイヤモンド層が形成されたダイヤモンドホイール、筒状の一方端面側にダイヤモンドチップを取りつけたコアビット、皿状のダイヤモンドカッター等各種形態のものとすることが可能である。

【００３４】上記各種形態のダイヤモンドカッターを製造するときも、スクリーンとしては、上記各ダイヤモンド層を形成する構成に合わせて、各種形成するものである。スクリーンの形状をダイヤモンドチップの形状に合わせて構成することにより、所望形状のダイヤモンド砥粒層を取着することができる。

【００３５】

【発明の効果】以上のように本発明に係るダイヤモンドカッターの製造方法によれば、ボンドペースト取着工程の後で所望に穿孔されたスクリーンを前記ボンドペースト取着範囲に被覆するスクリーン被覆工程と、前記ボンドペーストを被覆したスクリーン上から粘性物質を塗布する塗布工程を行なうことにより、ボンドペーストに塗布される粘性物質は、スクリーンを除去すると、スクリーンの小孔が形成された範囲だけとなる。したがって、スクリーンを除去した後でダイヤモンド砥粒を接触させると、ダイヤモンド粒子は、粘性物質が塗布された箇所、すなわちスクリーンに形成された小孔の位置に付着することになる。このようにしてスクリーンに形成された小孔にしたがって、ダイヤモンド粒子を所定間隔で付着させたダイヤモンド層を形成することができる。

【００３６】また、本発明のダイヤモンドカッターによれば、ダイヤモンド粒子が所定間隔で配置されているために、ダイヤモンド粒子の固着強度が高まる。そして、ダイヤモンド層に均一にダイヤモンド粒子を配置して固着されているために、被切削材に対して均一な切り口となり、切削がスムーズに行なわれることになる。

【００３７】さらに、本発明のダイヤモンドカッター製造治具としてのスクリーン部材によれば、スクリーン部材に形成する小孔の間隔や数を調整することにより、ダイヤモンド粒子を一定間隔で所望数配置することが可能となり、ダイヤモンドカッターにおけるダイヤモンド粒子の配置調整が可能となる。またダイヤモンド粒子が団塊になったり、隣接して配置されることがないために、各ダイヤモンド粒子が溶融ボンドによって確実に基板に固定できることになる。

【図面の簡単な説明】

【図１】本発明をダイヤモンドカッターの一例としてダイヤモンドチップとして適用したときの製造工程を示す説明図である。

【図２】図１の製造工程の拡大説明図である。

【図３】図１の製造工程の拡大説明図である。

【図４】図１の製造工程の拡大説明図である。

【図５】製造工程のブロック図である。

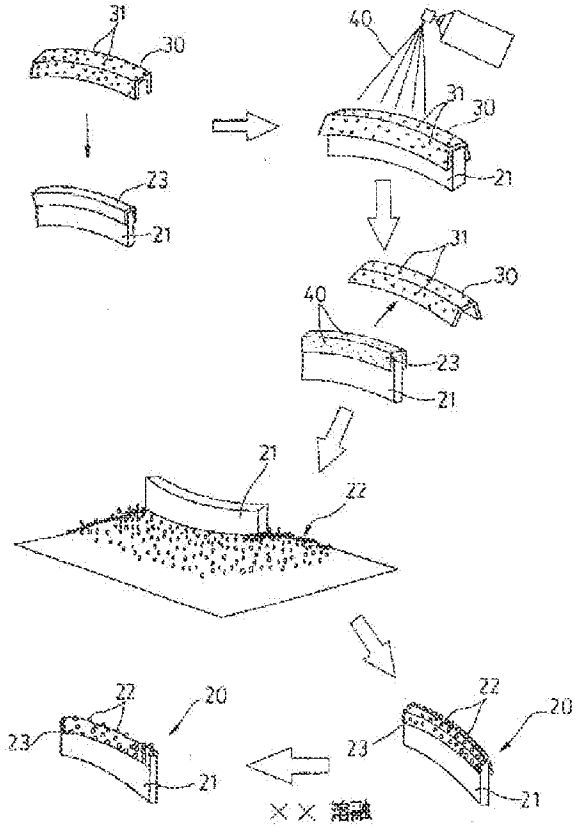
【図６】ダイヤモンドカッターの一例を示す正面図である。

【図７】ダイヤモンドカッター製造治具としてのスクリーンの例を示す説明図である。

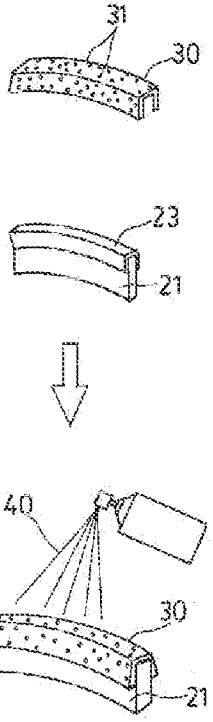
【符号の説明】

１０　基板
１１　取付孔
２０　ダイヤモンドチップ
２１　基台
２２　ダイヤモンド砥粒
２３　ボンドペースト
３０　スクリーン
３１　小孔
４０　粘性物質
Ｃ　ダイヤモンドカッター

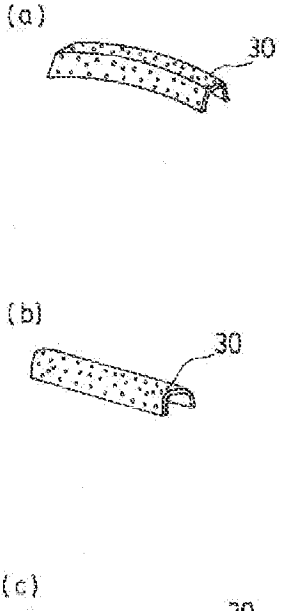
【図1】


31
30
40
23
21
22
20
××溶融

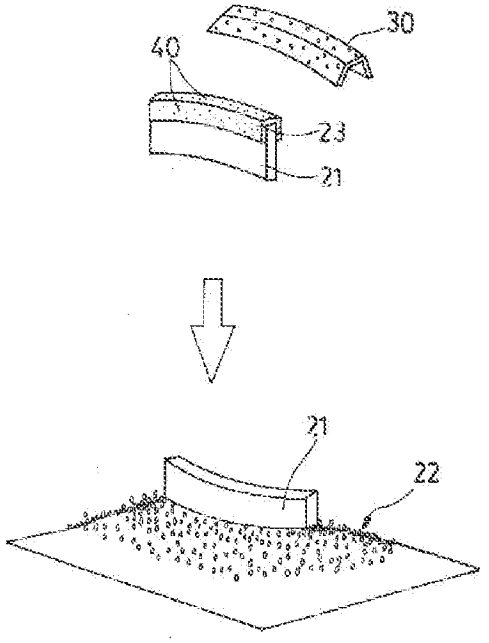
【図2】


31
30
23
21
40

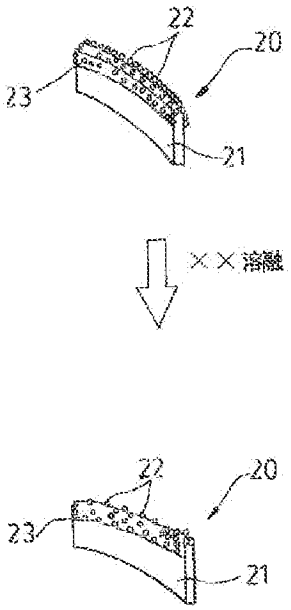
【図7】


(a)
30
(b)
30
(c)
30

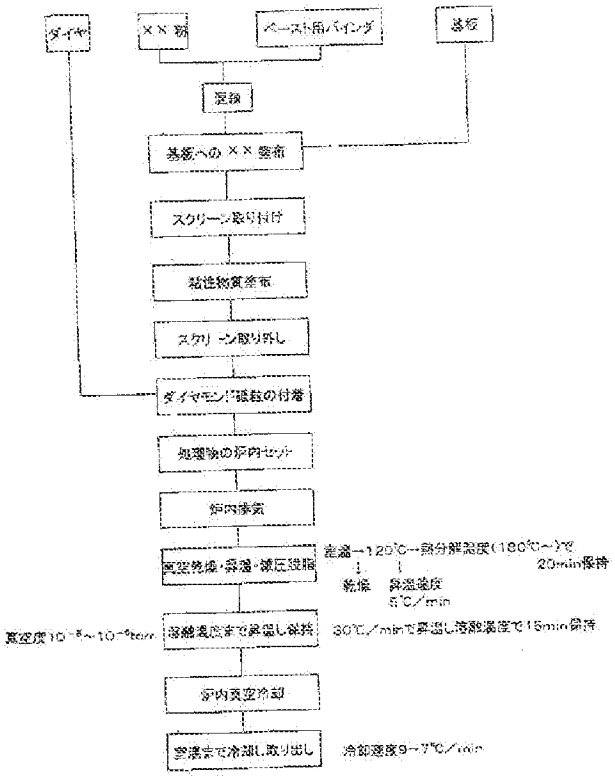
【図3】


30
40
23
21
22

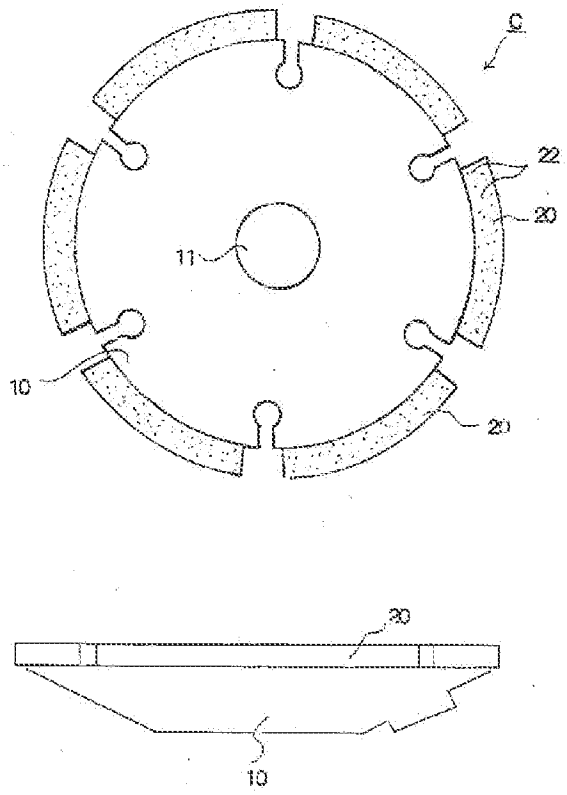
【図4】


22
20
23
21
××溶融

【図5】


ダイヤ
××粉
ペースト用バインダ
基板
混練
基板への××塗布
スクリーン取り付け
粘性物質塗布
スクリーン取り外し
ダイヤモンド砥粒の付着
処理後の炉内セット
炉内排気
真空発熱・昇温・減圧処理
室温→120℃→脱分解温度(180℃～)℃
20min保持
乾燥
昇温速度
5℃/min
真空度10⁻⁵～10⁻⁶torr
溶融温度まで昇温し保持
30℃/minで昇温し溶融温度で15min保持
炉内真空冷却
室温まで冷却し取り出し
冷却速度9～7℃/min

【図6】



フロントページの続き

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Fターム(参考)　3C063 AA02 AB03 BA03 BA12 BB02
BH02 EE31

No documents available for this priority number.

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Bibliographic data: JP2007044823 (A) — 2007-02-22

CMP PAD CONDITIONER IN SEMICONDUCTOR PLANARIZATION CMP PROCESS (CHEMICAL-MECHANICAL POLISHING)

Inventor(s): TANI KAZUNORI; IWASAKI HIDEKAZU ± (TANI KAZUNORI, ; IWASAKI HIDEKAZU)

Applicant(s): SOKEN KK ± (SOKEN:KK)

Classification: **- international:** ***B24B37/00; B24B53/12; B24D3/00; H01L21/304***
- cooperative:

Application number: JP20050232524 20050810

Priority number (s): JP20050232524 20050810

Abstract of JP2007044823 (A)

PROBLEM TO BE SOLVED: To reduce the time and trouble for replacing a CMP pad conditioner, and the down time and loss time of a CMP device by preventing abrasion, wearing and alteration of abrasive grains and fallout and defective of the abrasive grains to achieve long-time conditioning of the polishing pad. ; SOLUTION: This pad conditioner includes abrasive grains for pad polishing and grinding used in a semiconductor planarization CMP process, a bond layer holding the abrasive grains; and a bed to which the bond layer is fixed, wherein the abrasive grains or coating material coating the abrasive grains is a material having durability against CMP slurry. The material having durability is silicon carbide (SiC), diamond-like carbon, sapphire, alumina, TiO<SB>2</SB>, stabilized zirconia, ZrN, ZrC, WC, WC-Co, TiC, TiAlN, Tin-Ni, LiNbO<SB>3</SB>, NbC, LiTaO<SB>3</SB>, TaC or HfC. ; COPYRIGHT: (C) 2007,JPO&INPIT

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H01L 21/304	(2006.01)	H01L	21/304	622M	3C058
B24B 37/00	(2006.01)	B24B	37/00	A	3C063
B24D 3/00	(2006.01)	B24D	3/00	320A	
		B24D	3/00	330D	

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(22)出願日　平成17年8月10日 (2005.8.10)

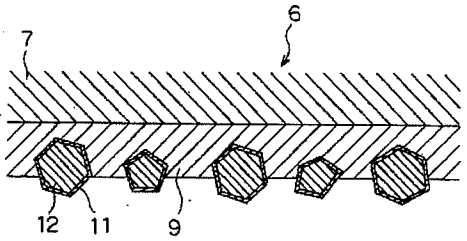
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Fターム(参考)　3C047 AA12　AA34　EE18　EE19　FF08

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(54)【発明の名称】半導体平坦化ＣＭＰプロセス（化学機械的研磨）におけるＣＭＰパッドコンディショナー

(57)【要約】　（修正有）
【課題】砥粒の磨耗、磨滅、変質や砥粒の脱落、欠損を防止できるので、長時間研磨パッドのコンディショニングを行うことができ、ＣＭＰパッドコンディショナーの交換の手間やＣＭＰ装置のダウンタイムならびにロス時間を減らすこと。
【解決手段】パッドコンディショナーは、半導体平坦化ＣＭＰプロセスに用いるパッド研磨研削用の砥粒と、この砥粒を保持するボンド層と、このボンド層を固着した台とよりなり、砥粒あるいは砥粒を被覆する被膜物質がＣＭＰスラリーに対して耐久性を持つ物質である。耐久性を持つ物質は、炭化珪素（ＳｉＣ）、ダイヤモンドライクカーボン、サファイア、アルミナ、TiO_2、安定化ジルコニア、ＺｒＮ、ＺｒＣ、ＷＣ、ＷＣ－Ｃｏ、ＴｉＣ、ＴｉＡｌＮ、Ｔｉｎ－Ｎｉ、$LiNbO_3$、ＮｂＣ、$LiTaO_3$、ＴａＣあるいはＨｆＣである。
【選択図】　図２



【特許請求の範囲】
【請求項1】
半導体平坦化ＣＭＰプロセスに用いるＣＭＰパッド研磨研削用の砥粒と、この砥粒を保持するボンド層と、このボンド層を固着した台とよりなり、上記砥粒はダイヤモンドよりＣＭＰスラリーに対して耐久性を持つ物質であることを特徴とする半導体平坦化ＣＭＰプロセス（化学機械的研磨）におけるＣＭＰパッドコンディショナー。
【請求項2】
半導体平坦化ＣＭＰプロセスに用いるＣＭＰパッド研磨研削用の砥粒と、この砥粒を保持するボンド層と、このボンド層を固着した台とよりなり、上記砥粒の表面にＣＭＰスラリーに対して耐久性を持つ物質が被覆されていることを特徴とする半導体平坦化ＣＭＰプロセス（化学機械的研磨）におけるＣＭＰパッドコンディショナー。
【請求項3】
半導体平坦化ＣＭＰプロセスに用いるＣＭＰパッド研磨研削用の砥粒と、この砥粒を保持するボンド層と、このボンド層を固着した台とよりなり、ＣＭＰスラリーに対して耐久性を持つ物質を上記砥粒表面及び上記ボンド層表面に被覆せしめたことを特徴とする半導体平坦化ＣＭＰプロセス（化学機械的研磨）におけるＣＭＰパッドコンディショナー。
【請求項4】
上記被覆した物質の膜厚が１０ｎｍ～９００μｍであることを特徴とする請求項２又は３記載の半導体平坦化ＣＭＰプロセス（化学機械的研磨）におけるＣＭＰパッドコンディショナー。
【請求項5】
上記各砥粒は夫々離間しており、上記砥粒のうち上記台からの高さが一番低い砥粒の高さと、上記台からの高さが一番高い砥粒の高さとの差が１００μｍ以内であることを特徴とする請求項１、２、３又は４記載の半導体平坦化ＣＭＰプロセス（化学機械的研磨）におけるＣＭＰパッドコンディショナー。
【請求項6】
上記各砥粒は夫々離間しており、上記被覆した砥粒のうち上記台からの高さが一番低い砥粒の高さと、上記台からの高さが一番高い砥粒の高さとの差が１００μｍ以内であることを特徴とする請求項２、３又は４記載の半導体平坦化ＣＭＰプロセス（化学機械的研磨）におけるＣＭＰパッドコンディショナー。
【請求項7】
上記ダイヤモンドよりＣＭＰスラリーに対して耐久性を持つ物質が、炭化珪素（ＳｉＣ）、ダイヤモンドライクカーボン、サファイア、アルミナ、安定化ジルコニアであることを特徴とする請求項１又は５記載の半導体平坦化ＣＭＰプロセス（化学機械的研磨）におけるＣＭＰパッドコンディショナー。
【請求項8】
上記ＣＭＰスラリーに対して耐久性を持つ物質が、炭化珪素（ＳｉＣ）、ダイヤモンドライクカーボン、サファイア、アルミナ、TiO_2、安定化ジルコニア、ＺｒＮ、ＺｒＣ、ＷＣ、ＷＣ－Ｃｏ、ＴｉＣ、ＴｉＡｌＮ、Ｔｉｎ－Ｎｉ、$LiNbO_3$、ＮｂＣ、$LiTaO_3$、ＴａＣあるいはＨｆＣであることを特徴とする請求項２、３、４、５又は６記載の半導体平坦化ＣＭＰプロセス（化学機械的研磨）におけるＣＭＰパッドコンディショナー。

【発明の詳細な説明】
【技術分野】
【0001】
本発明は半導体平坦化ＣＭＰプロセス（化学機械的研磨）におけるＣＭＰパッドコンディショナー、特に、化学機械的研磨（ＣＭＰ）装置に用いるＣＭＰパッドのコンディショナーに関するものである。
【背景技術】
【0002】

コンピュータのＣＰＵ能力向上の為には回路の集積度を上げる必要があり、これを実現する為に配線の多層化が行なわれているが、この時、シリコン基板上の各層間絶縁膜を平坦化するＣＭＰ（Chemical Mechanical Polishing）加工技術がキーテクノロジーとなる。半導体デバイスの多層配線化プロセスにおいては、半導体ウェハ表面の凹凸を極力平坦化することが重要であり、この半導体ウェハ表面の平坦化装置として化学的エッチングと研磨剤による機械的研磨とを組み合わせた研磨加工を行うＣＭＰ装置がある。

【0003】

図４は従来のＣＭＰ装置のコンセプトの一例を示す。１は回転自在な直径３００ｍｍの円板状の定盤、２は上記定盤１の略全面に設けたＣＭＰ研磨パッド、３は上記定盤１の回転中心軸に対して偏心した位置に設けた円板状の半導体ウェハ、４は上記半導体ウェハ３を保持し、上記半導体ウェハ３を上記研磨パッド２に押圧せしめる回転自在の半導体ウェハ押圧具、５は上記研磨パッド２上にコロイダルシリカ系、ヒュームドシリカ系、セリア系またはアルミナ系などのＣＭＰスラリーを供給するＣＭＰスラリー供給部、６は上記研磨パッド２の表面を再研磨または再研削（コンディショニング）するため上記半導体ウェハ３とは異なる位置で上記研磨パッド２上に載置した回転自在のＣＭＰパッドコンディショナーを示す。

【0004】

上記研磨パッド２は一般に大別して発泡ウレタンを基材としたパッドと無発泡のウレタンを基材としたパッドが使用されている。前者の発泡ウレタンを基材とする研磨パッドは弾力性があり後者の無発泡のウレタンを基材とするものに比べ平坦性が劣るという報告もある。発泡性ウレタン研磨パッドのＣＭＰパッドコンディショナーにはブラシ状の繊維を埋めこんだもの、あるいはダイヤモンド砥粒をメタルボンドした砥石状のＣＭＰパッドコンディショナーが使用される。一方、無発泡のウレタンの基材を用いたＣＭＰパッドコンディショナーにはダイヤモンド砥粒をメタルボンドした砥石状のＣＭＰパッドコンディショナーが用いられる。

【0005】

上記砥石状のＣＭＰパッドコンディショナー６は図４及び図５に示すように、円板状の金属製台金７と、この台金７の下面に設けた粒径５０μｍ～２００μｍのダイヤモンド砥粒８と、上記ダイヤモンド砥粒８を保持するための上記金属製台金７の下面あるいは台金表面全体を被覆するＮｉメッキボンド層等のボンド層９とよりなる。

【0006】

上記のような従来のＣＭＰ装置においては、上記ＣＭＰ研磨パッド２上にＣＭＰスラリーを供給し、上記定盤１と上記半導体ウェハ押圧具４とを相対的に回転せしめて、上記半導体ウェハ押圧具４により上記半導体ウェハ３を上記ＣＭＰ研磨パッド２上に押圧せしめれば、上記半導体ウェハ３の表面が研磨される。また、上記ＣＭＰパッドコンディショナー６を常時又は定期的に回転せしめれば、上記研磨パッド２の平坦度を維持、修復し、また目詰まりを防止せしめ、また、ＣＭＰ研磨パッド表面の面粗さを維持することによりＣＭＰスラリーの研磨特性を維持、回復せしめることができる。

【0007】

また、上記ＣＭＰパッドコンディショナー６におけるボンド層９により上記台金７を被覆しているので、上記ＣＭＰスラリーにより台金が溶出することがなく、ダイヤモンド砥粒８の脱落を防止できる。このようなＣＭＰ装置は特許文献１に記載されている。

【特許文献1】特開２００１－２３９４４９号

【発明の開示】

【発明が解決しようとする課題】

【0008】

しかしながら、上記従来のＣＭＰ装置においては、ある程度ＣＭＰ研磨パッド２のコンディショニングを続ければ、ＣＭＰパッドコンディショナー上の砥粒の切刃が研磨、磨滅、平坦化され、切れ味が鈍ることにより上記ＣＭＰ研磨パッド２の平坦度の修復が遅くなる。またダイヤモンド砥粒８等がＣＭＰパッドコンディショナー６のボンド層９から脱落

、欠損し、脱落したダイヤモンド砥粒８等がＣＭＰ研磨パッド２の表面に落下、固定され、あるいは転動してウェハ表面に傷をつけてしまうという欠点があった。

【0009】

本発明者は種々実験研究の結果、ダイヤモンド砥粒はコロイダルシリカ系、ヒュームドシリカ系、セリア系またはアルミナ系などのＣＭＰスラリーにより磨耗、磨滅、変質すること、また、この磨耗等により砥粒にかかる力が増大しダイヤモンド砥粒が脱落、欠損することを見出した。また、ＣＭＰスラリーに対して耐久性のある物質、例えば炭化珪素（ＳｉＣ）、ダイヤモンドライクカーボン、サファイア、アルミナ、TiO_2、安定化ジルコニア、ＺｒＮ、ＺｒＣ、ＷＣ、ＷＣ－Ｃｏ、ＴｉＣ、ＴｉＡｌＮ、Ｔｉｎ－Ｎｉ、$LiNbO_3$、ＮｂＣ、$LiTaO_3$、ＴａＣあるいはＨｆＣで砥粒あるいはＣＭＰコンディショナーの加工部表面を被覆すれば砥粒の磨耗等を少なくでき、研磨パッドの平坦度ならびに面粗さを維持し、砥粒の脱落等を防止できることを見出した。また、安価な砥粒にＣＭＰスラリーに対して耐久性のある物質を被覆すれば、安価で高品質のＣＭＰコンディショナーを提供できることを見出した。本発明はかかる知見をもとになされたものである。

【課題を解決するための手段】

【0010】

本発明の半導体平坦化ＣＭＰプロセス（化学機械的研磨）におけるＣＭＰパッドコンディショナーは、半導体平坦化ＣＭＰプロセスに用いるＣＭＰパッド研磨研削用の砥粒と、この砥粒を保持するボンド層と、このボンド層を固着した台とよりなり、上記砥粒はダイヤモンドよりＣＭＰスラリーに対して耐久性を持つ物質であることを特徴とする。

【0011】

本発明の半導体平坦化ＣＭＰプロセス（化学機械的研磨）におけるＣＭＰパッドコンディショナーは、半導体平坦化ＣＭＰプロセスに用いるＣＭＰパッド研磨研削用の砥粒と、この砥粒を保持するボンド層と、このボンド層を固着した台とよりなり、上記砥粒の表面にＣＭＰスラリーに対して耐久性を持つ物質が被覆されていることを特徴とする。

【0012】

本発明の半導体平坦化ＣＭＰプロセス（化学機械的研磨）におけるＣＭＰパッドコンディショナーは、半導体平坦化ＣＭＰプロセスに用いるＣＭＰパッド研磨研削用の砥粒と、この砥粒を保持するボンド層と、このボンド層を固着した台とよりなり、ＣＭＰスラリーに対して耐久性を持つ物質を上記砥粒表面及び上記ボンド層表面に被覆せしめたことを特徴とする。

【0013】

上記被覆した物質の膜厚は１０ｎｍ～９００μｍであることを特徴とする。

【0014】

上記各砥粒は夫々離間しており、上記砥粒のうち上記台からの高さが一番低い砥粒の高さと、上記台からの高さが一番高い砥粒の高さとの差が１００μｍ以内であることを特徴とする。

【0015】

上記各砥粒は夫々離間しており、上記被覆した砥粒のうち上記台からの高さが一番低い砥粒の高さと、上記台からの高さが一番高い砥粒の高さとの差が１００μｍ以内であることを特徴とする。

【0016】

上記ダイヤモンドよりＣＭＰスラリーに対して耐久性を持つ物質は、炭化珪素（ＳｉＣ）、ダイヤモンドライクカーボン、サファイア、アルミナ、安定化ジルコニアであることを特徴とする。

【0017】

上記ＣＭＰスラリーに対して耐久性を持つ物質は、炭化珪素（ＳｉＣ）、ダイヤモンドライクカーボン、サファイア、アルミナ、TiO_2、安定化ジルコニア、ＺｒＮ、ＺｒＣ、ＷＣ、ＷＣ－Ｃｏ、ＴｉＣ、ＴｉＡｌＮ、Ｔｉｎ－Ｎｉ、$LiNbO_3$、ＮｂＣ、$LiTaO_3$、ＴａＣあるいはＨｆＣであることを特徴とする。

【発明の効果】
【0018】
本発明の半導体平坦化ＣＭＰプロセス（化学機械的研磨）におけるＣＭＰパッドコンディショナーによれば、砥粒の磨耗、磨滅、変質や砥粒の脱落、欠損を防止できるので、長時間研磨パッドのコンディショニングを行うことができ、ＣＭＰパッドコンディショナーの交換の手間やＣＭＰ装置のダウンタイムならびにロス時間を減らすことができ、また、半導体ウェハ表面に傷が付くのを防止することができるという大きな利益がある。また、安価で高品質のＣＭＰコンディショナーを提供できる。
【発明を実施するための最良の形態】
【0019】
以下図面によって本発明の実施例を説明する。
【実施例】
【0020】
本発明のＣＭＰ装置用のＣＭＰパッドコンディショナー６においては、図１に示すように、ＣＭＰ研磨パッドのＣＭＰコンディショニング用砥粒としてダイヤモンド砥粒８を用いる代わりに、例えば粒径１００μｍ±４０μｍの、ダイヤモンドよりＣＭＰスラリーに対して耐磨耗性、耐磨滅性、耐変質性等の耐久性を持つ化学的に安定した結晶構造を持った例えば炭化珪素（ＳｉＣ）、ダイヤモンドライクカーボン、サファイア、アルミナ、安定化ジルコニアなどの物質からなる砥粒などの物質からなる砥粒１０を用いる。パッドコンディショナー用の台金７に形成せしめたＮｉメッキボンド層等のボンド層９の厚さは、上記砥粒１０の粒径のおよそ三分の一を突出せしめて固着せしめることができる程度が好適である。なお、各砥粒間隔は例えば約１０μｍ～１０ｍｍとし、ＣＭＰコンディショナーにかかる加圧力と相関してひとつの砥粒にかかる加圧力でＣＭＰコンディショナー全体の砥粒の数を決定することが望ましい。
【0021】
本発明によれば、上記砥粒１０はＣＭＰスラリー対して耐久性がダイヤモンドより大きいので、磨耗、磨滅、変質しづらく、砥粒の脱落、欠損を防ぐ事ができ、長期間ＣＭＰ研磨パッドのコンディショニングを続けることができるという大きな利益がある。
【0022】
なお、ＣＭＰスラリーをポリシャーとしての被研磨特性を試行したところ、ダイヤモンドは１時間程度で磨滅したが、サファイアを同じ量削るには２～３時間かかり、また、ＳｉＣを同量研磨するには１０～２０時間かかった。従って、ＣＭＰスラリーに対して耐久性のある物質としては特にＳｉＣが好ましい。
【0023】
また、上記各砥粒を夫々離間せしめ、また、上記砥粒のうち、台金からの先端高さが一番低い砥石の先端高さと、台金からの先端高さが一番高い砥粒の先端高さとの差を１００μｍ以内、例えば３μｍ以内としたＣＭＰコンディショナーを用いた場合、ＣＭＰパッドの面粗さを均一化することができ、ＣＭＰ加工品質を向上させ、半導体ウェハの品質を高めることができる。
【0024】
また、上記台金７は金属系の他に樹脂系、粉末冶金系であってもよい。
【実施例】
【0025】
本発明の第２の実施例においては、図２に示すように研磨パッドのコンディショニング用の砥粒として、その表面にスパッタリング、イオンプレーティング、真空蒸着法又は化学蒸着（ＣＶＤ）法等により、ＣＭＰスラリーに対して耐久性のある、例えば炭化珪素（ＳｉＣ）、ダイヤモンドライクカーボン、サファイア、アルミナ、TiO_2、安定化ジルコニア、ＺｒＮ、ＺｒＣ、ＷＣ、ＷＣ－Ｃｏ、ＴｉＣ、ＴｉＡｌＮ、Ｔｉｎ－Ｎｉ、$LiNbO_3$、ＮｂＣ、$LiTaO_3$、ＴａＣあるいはＨｆＣなどの物質１１を例えば約１０ｎｍ～９００μｍ被覆したダイヤモンド砥粒、ＳｉＣ砥粒、アルミナ砥粒あるいは立方晶窒

化ホウ素（ＣＢＮ）砥粒などの砥粒１２を用い、パッドコンディショナー用の台金７に形成せしめたＮｉメッキボンド層等のボンド層９に、上記砥粒１２の全部あるいは一部を上記ボンド層９で固着せしめる。

【0026】

この第２の実施例においては、上記物質１１はＣＭＰスラリーに対して耐久性があるので、磨耗等しづらく、砥粒の脱落等を防ぐ事ができ、長期間ＣＭＰ研磨パッドのコンディショニングを続けることができる。また、安価な砥粒にＣＭＰスラリーに対して耐久性のある物質を被覆すれば、安価で高品質のＣＭＰコンディショナーを提供できる。

【0027】

なお、上記物質１１を被覆する部分は、上記砥粒１２の切刃となる部分の表面だけであってもよい。

【0028】

また、各砥粒を夫々離間せしめ、また、上記被覆した砥粒のうち、台金からの先端高さが一番低い砥石の先端高さと、台金からの先端高さが一番高い砥粒の先端高さとの差を１００μｍ以内、例えば３μｍ以内としたＣＭＰコンディショナーを用いた場合、ＣＭＰパッドの面粗さを均一化することができ、ＣＭＰ加工品質を向上させ、半導体ウェハの品質を高めることができる。

【0029】

また、上記台金は金属系の他に樹脂系、粉末冶金系であってもよい。

【0030】

また、上記ＣＭＰスラリーに対して耐久性のある物質は炭化珪素（ＳｉＣ）、ダイヤモンドライクカーボン、サファイア、アルミナ、安定化ジルコニアなどのダイヤモンドよりＣＭＰスラリーに対して耐久性のある物質であることが好ましい。

【実施例】

【0031】

本発明の第３の実施例においては、図３に示すようにＣＭＰ研磨パッドのコンディショニング用の砥粒としてダイヤモンド砥粒、ＳｉＣ砥粒、アルミナ砥粒あるいは立方晶窒化ホウ素（ＣＢＮ）砥粒などの砥粒１２を用い、パッドコンディショナー用の台金７に形成せしめたＮｉメッキボンド層等のボンド層９に、上記砥粒１２の全部あるいは一部を上記ボンド層９の表面から突出せしめて固着せしめる。また加えて上記ボンド層９の表面から突出した砥粒１２の表面及び上記ボンド層９の表面にスパッタリング又はＣＶＤ法等により、ＣＭＰスラリーに対する耐久性のある、例えば炭化珪素（ＳｉＣ）、ダイヤモンドライクカーボン、サファイア、アルミナ、TiO_2、安定化ジルコニア、ＺｒＮ、ＺｒＣ、ＷＣ、ＷＣ－Ｃｏ、ＴｉＣ、ＴｉＡｌＮ、Ｔｉｎ－Ｎｉ、$LiNbO_3$、ＮｂＣ、$LiTaO_3$、ＴａＣあるいはＨｆＣなどの物質１１を例えば厚さ約１０ｎｍ～９００μｍで被覆せしめる。

【0032】

この第３の実施例においても、上記物質１１はＣＭＰスラリーに対して耐久性があるので、磨耗等しづらく、砥粒の脱落等を防ぐ事ができ、長期間ＣＭＰ研磨パッドのコンディショニングを続けることができる。また、安価な砥粒にＣＭＰスラリーに対して耐久性のある物質を被覆すれば、安価で高品質のＣＭＰコンディショナーを提供できる。

【0033】

なお、上記砥粒１２を被覆する部分は、上記砥粒１２の切刃となる部分の表面だけであってもよい。

【0034】

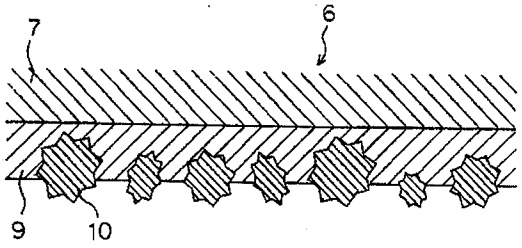
また、上記ＣＭＰスラリーに対して耐久性のある物質は炭化珪素（ＳｉＣ）、ダイヤモンドライクカーボン、サファイア、アルミナ、安定化ジルコニアなどのダイヤモンドよりＣＭＰスラリーに対して耐久性のある物質であることが好ましい。

【図面の簡単な説明】

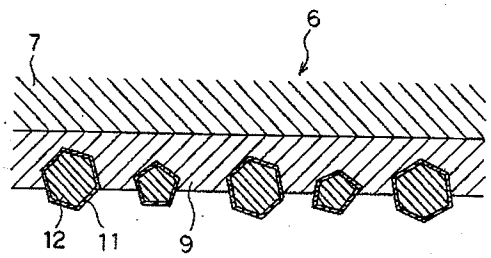
【0035】

【図1】本発明の半導体平坦化ＣＭＰプロセス（化学機械的研磨）におけるＣＭＰパッドコンディショナーの第１の実施例の要部を拡大した縦断側面図である。
【図2】本発明の半導体平坦化ＣＭＰプロセス（化学機械的研磨）におけるＣＭＰパッドコンディショナーの第２の実施例の要部を拡大した縦断側面図である。
【図3】本発明の半導体平坦化ＣＭＰプロセス（化学機械的研磨）におけるＣＭＰパッドコンディショナーの第３の実施例の要部を拡大した縦断側面図である。
【図4】従来の半導体平坦化ＣＭＰプロセス（化学機械的研磨）におけるＣＭＰ装置の斜視図である。
【図5】従来の半導体平坦化ＣＭＰプロセス（化学機械的研磨）におけるＣＭＰパッドコンディショナーの要部を拡大した縦断側面図である。
【符号の説明】
【0036】
１　定盤
２　ＣＭＰ研磨パッド
３　半導体ウェハ
４　半導体ウェハ押圧具
５　ＣＭＰスラリー供給部
６　ＣＭＰパッドコンディショナー
７　金属製台金
８　砥粒
９　ボンド層
１０　砥粒
１１　被覆物質
１２　砥粒

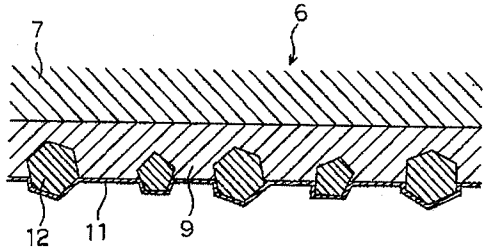
【図1】



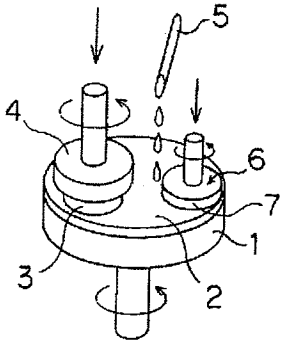
【図2】



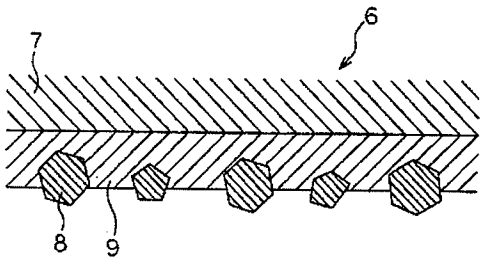
【図3】



【図4】



【図5】



(51)Int.Cl.	F I	テーマコード（参考）
	Ｂ２４Ｄ　3/00　３１０Ｄ	

Ｆターム(参考)　3C058 AA07 AA19 CB04 CB05 DA12 DA17
3C063 AB05 BA37 BB02 BB03 BB04 BB15 BC02 EE26 FF08 FF22
FF23

Espacenet

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

DIAMOND GRID-CHEMICAL MECHANICAL POLISH PAD DRESSER

No documents available for this priority number.

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

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

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

Application number: KR20000066159 20001108

Priority number(s): KR20000066159 20001108

Abstract of KR20020036138 (A)

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

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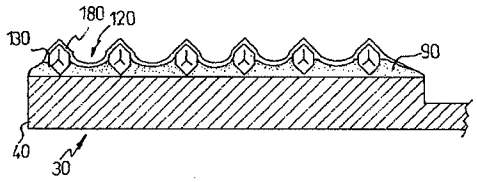
전체 청구항 수 : 총 48 항

(54) 다이아몬드 그리드 화학 기계적 연마 패드 드레서

(57) 요 약

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

대 표 도 - 도 3



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청구의 범위

청구항 1

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

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

청구항 2

제 1 항에 있어서,

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

청구항 3

제 1 항에 있어서,

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

청구항 4

제 1 항에 있어서,

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

청구항 5

제 1 항에 있어서,

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

청구항 6

제 5 항에 있어서,

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

청구항 7

제 5 항에 있어서,

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

청구항 8

제 1 항에 있어서,

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

청구항 9

제 1 항에 있어서,

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

청구항 10

제 1 항에 있어서,

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

청구항 11

제 1 항에 있어서,

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

청구항 12

제 1 항에 있어서,

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

청구항 13

제 1 항에 있어서,

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

청구항 14

제 12 항에 있어서,

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

청구항 15

제 1 항에 있어서,

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

청구항 16

제 14 항에 있어서,

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

청구항 17

제 14 항에 있어서,

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

청구항 18

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

청구항 19

제 18 항에 있어서,

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

청구항 20

제 18 항에 있어서,

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

청구항 21

제 18 항에 있어서,

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

청구항 22

제 18 항에 있어서,

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

청구항 23

제 18 항에 있어서,

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

청구항 24

a) 기판 부재를 제공하는 단계,

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

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

청구항 25

제 24 항에 있어서,

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

청구항 26

제 24 항에 있어서,

단계 b) 및 c)는 추가로,

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

b) 연마 입자들로 상기 형판의 구멍을 채우는 단계,

c) 형판 구멍내에 있지않은 연마 입자들을 제거하는 단계,

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

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

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

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

청구항 27

제 26 항에 있어서,

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

청구항 28

제 27 항에 있어서,

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

청구항 29

제 24 항에 있어서,

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

청구항 30

제 24 항에 있어서,

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

청구항 31

제 26 항에 있어서,

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

청구항 32

제 31 항에 있어서,

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

청구항 33

제 26 항에 있어서,

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

청구항 34

제 24 항에 있어서,

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

청구항 35

제 24 항에 있어서,

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

청구항 36

제 24 항에 있어서,

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

청구항 37

제 24 항에 있어서,

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

청구항 38

제 24 항에 있어서,

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

청구항 39

제 38 항에 있어서,

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

청구항 40

제 26 항에 있어서,

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

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

청구항 41

제 40 항에 있어서,

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

청구항 42

제 26 항에 있어서,

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

청구항 43

제 26 항에 있어서,

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

청구항 44

제 24 항에 있어서,

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

청구항 45

제 44 항에 있어서,

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

청구항 46

제 44 항에 있어서,

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

청구항 47

제 44 항에 있어서,

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

청구항 48

제 47 항에 있어서,

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

명세서

발명의 명칭

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

도면의 간단한 설명

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

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

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

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

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

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

발명의 상세한 설명

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

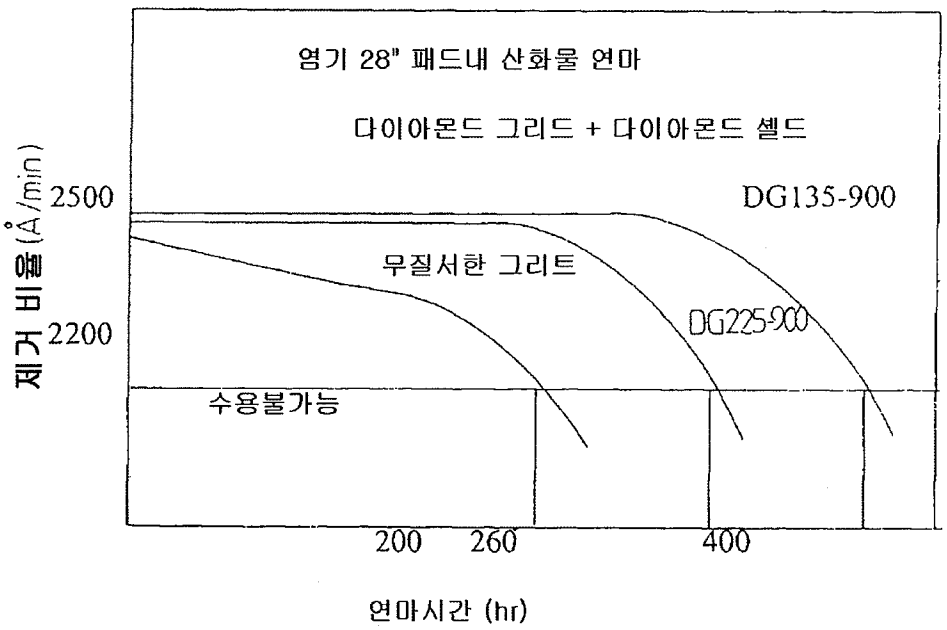
[0061] 실시예 1

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

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

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

[0065]



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

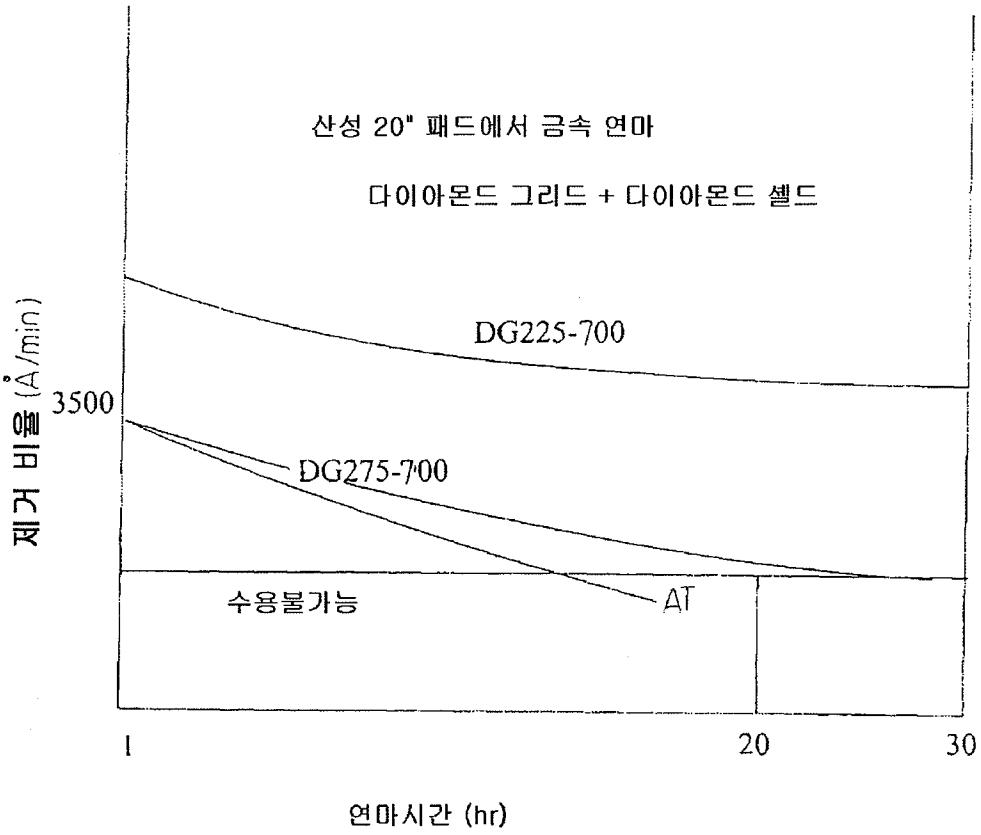
[0067] 실시예 2

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

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

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

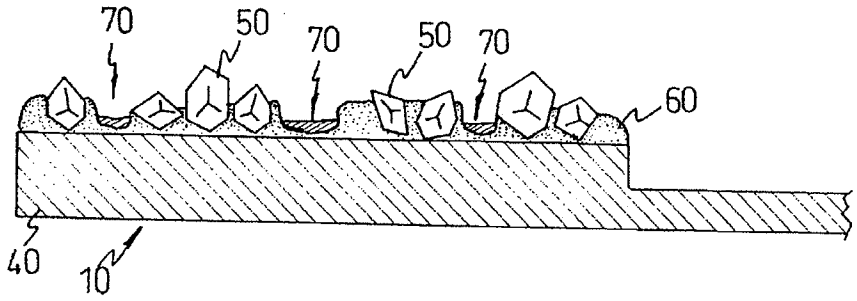
[0070]

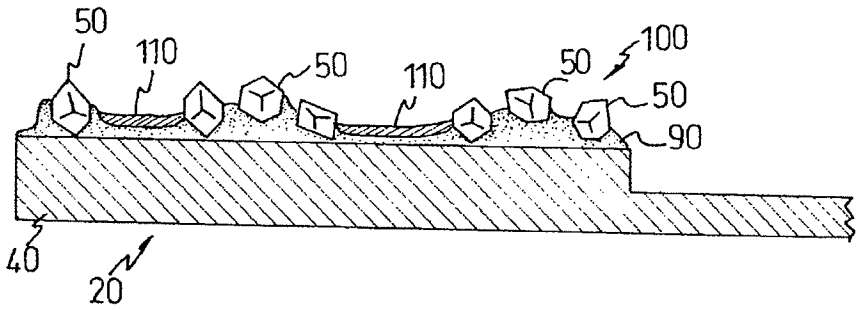


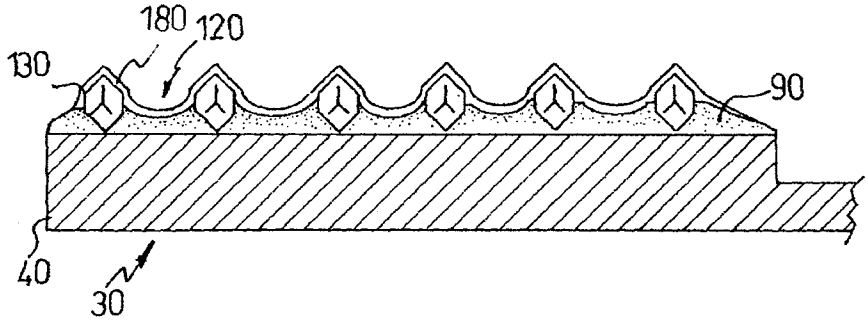
[0071] 표 2로부터 볼 수 있는 것 처럼, 종래 제조된 다이아몬드 디스크는 연마된 웨이퍼의 제거 비율을 유지할 수 없다. 또한, 금속 결합은 단지 1.5 시간동안 연마 슬러리의 산성 분위기에서 유지된다. 그 후, 다이아몬드는 떨어지기 시작하고 및 고가의 웨이퍼상에 많은 스크래치를 발생시킨다. 그러나, 본 발명의 디스크들은 산성 분위기에서 30 시간이상동안 유지되었다. 그러한 수명의 범위는 더 나은 CMP 패드 드레싱 결과를 얻을 수 있고, 및 종래 기술보다 상당히 개선된 웨이퍼 제조의 전체 비용 및 생산량을 구성한다. 물론, 상기는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

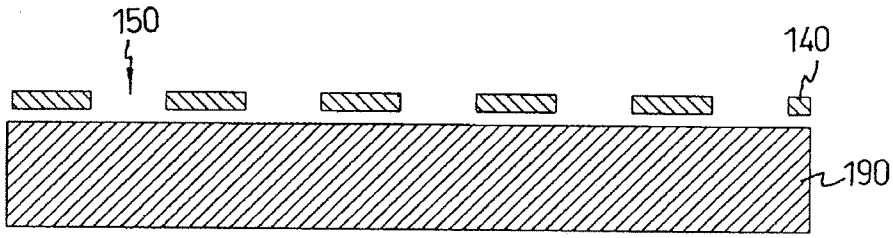
[0072] 많은 변경 및 다른 장치들이 본 발명의 범위 및 범주로부터 벗어남 없이 본 기술내에서 기술된 것들에 의해 발명되어졌고 및 첨부된 청구범위는 그러한 변경 및 장치들을 보호하기 위해 의도되어졌다. 따라서, 본 발명이 본 발명의 가장 실질적이고 및 바람직한 실시예로 간주되는 것과 관련하여 특별하고 및 상세하게 상기에 기술되어졌지만, 상기는 크기의 다양함, 재료, 형상, 형태, 기능, 작동 방법, 조립체, 및 용도에 제한을 두지 않고 포함된 많은 변경이 여기에서 설명된 개념 및 원리로부터 벗어남 없이 만들어질 수 있는 기술내에서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

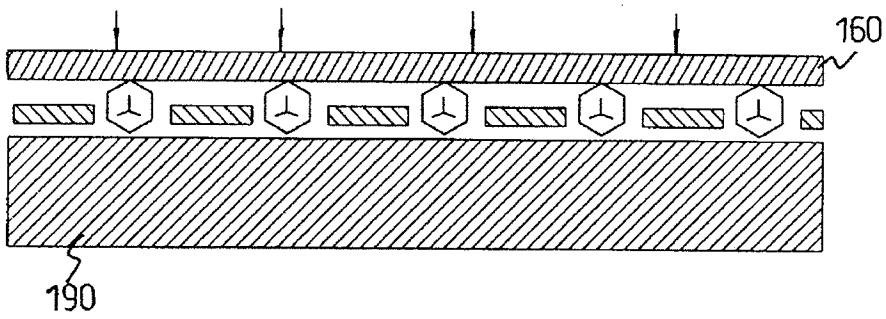
[0073] 상기한 바와 같이, 본 발명은 경납 합금 시트내에 연마 입자를 포함하기 위한 방법의 결과로써, 고른 공간이 만들어지고, 연마 그리트들은 CMP 패드에 적용될 때를 의미하는 기판 위의 거리 또는 균일한 높이로 연장하고, 그들은 패드 섬유내부 균일한 깊이로 돌출함에 따라, 상기 고른 공간 및 균일한 돌출로 CMP가 고르게 드레싱되고 손질되어 CMP 패드의 연마 효율성을 증가키고 및 그의 사용 수명을 연장시킨다.

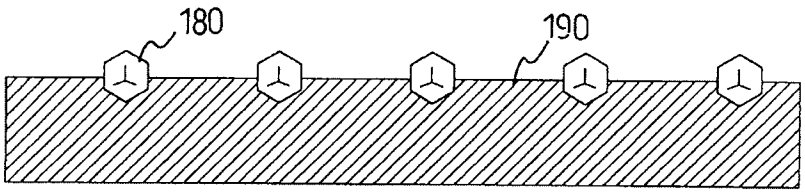

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요약

본 고안은 화학적-기계적-연마(CMP)용 폴리싱 패드의 평탄화를 향상시킴과 동시에, 이 패드의 컨디셔닝을 효과적으로 행하는 기능을 가지고, 더구나 컨디셔닝 성능이 우수하고, 수명이 길며, 컨디셔너의 제조가 용이하여 제조시의 품질을 현저히 감소시킬 수 있는 CMP 장치용 컨디셔너를 제공함에 그 목적이 있다.

이러한 목적을 달성하기 위한 본 고안은, 샹크(3)의 표면에 구형 형상의 비마스킹부(1)를 가지는 마스킹 시트(2)를 접착하고, 하나의 비마스킹부(1)에 하나의 다이아몬드 입자(4) 만을 예비 고정한 후에 마스킹 시트(2)를 박리시키고, 이어서 도금액 안에 넣어서 다이아몬드 입자 직경의 40~60% 정도의 두께를 가지도록 Ni 도금층(6)을 석출시키며, 다음에 1개 구형 형상의 비마스킹부(1)의 면적이 다이아몬드 입자(4)의 투영 면적의 110~160%에로 하고, 상기한 마스킹 시트(2)의 비마스킹부(1)의 전면적이 베스킹 시트(2)의 전면적의 0.04~0.2배의 범위로 하는 것이 특징으로 된 화학적-기계적-연마 패드용 다이아몬드 전착 컨디셔너를 제공한다.

본 고안에 의해 지수 형입도가 엄격한 제한 형상을 제현성 높게 용이하게 형성시킬 수 있고, 마스킹 시트(2)의 샹크(3)에 대한 밀착력이 높으며, 또한 샹크(3)에 전착되는 60~120메쉬의 다이아몬드 입자(4)가, 구형형의 비마스킹부(1)에 한 개씩 분산된다. 따라서 CMP용 폴리싱 패드의 평탄화를 향상시킴과 동시에, 이 패드의 컨디셔닝을 효과적으로 행하는 기능을 가지고, 더구나 컨디셔닝 성능이 우수하고, 수명이 길며, 컨디셔너의 제조가 용이하기 때문에 제조시의 불량률을 현저히 감소시킬 수 있다.

대표도

도 1

색인어

EX1057, Page 493

화학적-기계적-연마, 패드, 컨디셔너, 폴리싱, 다이아몬드

명세서

도면의 간단한 설명

도 1은 마스킹 시트를 정사한 상태의 부분 평면도

도 2는 다이아몬드 입자가 예비 고정된 상태의 부분 평면도

도 3은 본 고안 컨디셔너의 연삭면의 부분 단면도

도 4는 본 고안에 의한 일실시예인 컨디셔너의 작용면의 평면도

도 5는 본 고안에 의한 다른 실시예인 컨디셔너의 작용면의 평면도

<도면의 주요부분에 대한 부호의 설명>

1 : 구멍형상의 비마스킹부 2 : 마스킹 시트

3 : 상표 4 : 다이아몬드 입자

5 : 예비 도금층 6 : Ni 도금층

7 : 컨디셔너 8 : 비전착 영역

고안의 상세한 설명

고안의 목적

고안이 속하는 기술 및 그 분야의 종래기술

본 고안은 반도체의 웨이퍼 가공에 사용되는 CMP(Chemical mechanical planarization) 장치의 패드 컨디셔너에 관한 것이다. 보다 상세하게는, 본 고안은 CMP용 폴리싱 패드의 평탄화를 향상시킴과 동시에, 이 패드의 컨디셔닝을 효과적으로 행하는 기능을 가지고, 더구나 컨디셔닝 성능이 우수하고, 수명이 긴 CMP 장치용 컨디셔너에 관한 것이다.

반도체 웨이퍼의 연마를 반복하기 위하여, 연마 입과나 연마칩 등이 폴리싱 패드의 미세한 구멍에 들어가서 눌려붙어 있어단다는지, 연마 입자와 웨이퍼와의 화학 반응열에 의해 폴리싱 패드의 표면이 경화하여 연마 속도를 저하시키게 된다. 이 때문에 폴리싱 패드의 표면을 재생시켜 연마 속도를 회복시키는 컨디셔너라고 불리는 공구가 사용된다. 다이아몬드 입자는 우수한 컨디셔닝 재료이며, 다이아몬드 입자를 이용한 컨디셔닝이 실용화되어 있다. 이와 같은 CMP용 폴리싱 패드의 컨디셔너에 대해서는, 폴리싱 패드의 이물을 가급적 억제할 수 있으며, 더욱이 패드의 표면 상태를 항상 소정의 연마속도가 얻어지도록 일정하게 유지되는 것과, 다이아몬드 입자의 탈락이 없고, 패드면에 눌러붙을 일으키지 않는 것이 요구된다.

다이아몬드 입자를 컨디셔너 본체의 작용면에 금속 결합재를 사용하여 고착하는 방법에는 여러 가지가 있으나, 전착법은 비교적 용이하고 더욱이 다이아몬드 입자를 컨디셔너 본체의 작용면에 확실하게 고착시킬 수 있으므로 많이 사용되고 있다. 전착법에 의해 다이아몬드 입자를 고착하는 경우는, 컨디셔너 본체의 작용면에 다이아몬드 입자의 고정면을 남겨 두고서, 절연성의 마스킹 재료로 피복한다. 컨디셔너의 본체를 도금액에 담그고 입자 고정면에 입자를 놓고서 컨디셔너의 본체에 음극을 접속하고, 도금액에 양극을 접속하여 전기 도금을 행하여 다이아몬드 입자를 예비 고정한다. 다이아몬드 입자를 예비 고정한 후에 대부분의 입자는 그 일 부가 본체의 입자 고정면에 접한 상태에서 예비 고정되지만, 본체의 입자 고정면에 접하지 않은 상태에 부착하여, 부유하는 입자가 존재하는 경우도 있다. 컨디셔너의 본체를 다시 도금액에 넣어서, 본체에 음극을 접속하고 도금액에 양극을 접속하여 다이아몬드 입자 고정면의 도금을 행한다.

그러나 종래의 연마 슬urry의 제조에 있어서, 연마시너 용제의 적용인해 다이아몬드 입자의 고정력을 낮기 무고서, 철 연성의 마스킹 재료로 피복하는 경우에 비교적 크기의 작업한 지수 정밀도로 피복하는 것이 곤란하여 마스킹 되어 있지 않은 곳에 두 개 이상의 다이아몬드 입자가 동시에 들어가는 문제가 발생하였고, 마스킹 위치가 어긋나서 공업적 형상으로 제현하는 것이 곤란하였고, 피복층이 정밀하게 되지않고 요철(凹凸)이 상태였으며, 피복층의 일부분이 마찰에 의후에 공정에서 벗겨지는 경우가 있었기 때문에, CMP 패드를 컨디셔닝 하는데 있어서 불량률이 매우 높았다. 또한 종래의 컨디셔너는, 패드의 표면 상태를 항상 소정의 연마속도와 일어지도록 일정하게 유지시키는 것이 곤란하였으며, 다이아몬드 입자의 탈락이 많고, 패드면에 눈메움을 일으키는 경우가 많았으며, 종래의 컨디셔너를 사용하여 패드를 컨디셔닝하면, 웨이퍼의 표면거칠기 값 및 스크래치 개수가 높은 것이 큰 문제로 되어왔다.

고안이 이루고자 하는 기술적 과제

본 고안은 CMP용 폴리싱 패드의 평탄화를 향상시킴과 동시에, 이 패드의 컨디셔닝을 효과적으로 행하는 기능을 가지고, 더구나 컨디셔닝 성능이 우수하고, 수명이 길며, 컨디셔너의 제조가 용이하여 제조시의 불량률을 현저히 감소시킬 수 있는 CMP 장치용 컨디셔너를 제공함에 그 목적이 있다.

고안의 구성 및 작용

본 고안자들은, 상기의 과제를 해결하기 위하여 실험적인 연구를 거듭한 결과, 연속 컨디셔너의 연삭면에 있어서, 하나의 비마스킹부에 한 개씩의 다이아몬드 입자를 분산시키는 것에 의해, 연삭 작용을 감소시키고 컨디셔너의 수명이 증가되며, 연마속도의 컨디셔닝 속도가 높아지며, 웨이퍼의 표면거칠기 값이 낮아지며, 웨이퍼의 스크래치 개수가 감소됨을 확인하였다. 즉, 본 고안은, 상크(3)의 표면에 구형 형상의 비마스킹부(1)를 가지는 마스킹 시트(2)를 접착하고, 하나의 비마스킹부(1)에 하나의 다이아몬드 입자(4) 만을 에비 고정한 후에 마스킹 시트(2)를 박리시키고, 이어서 도금액 안에 넣어서 다이아몬드 입자 직경의 40~60% 정도의 두께를 가지도록 Ni 도금층을 석출시키며, 따옥이 1개의 구형 형상의 비마스킹부(1)의 면적이 다이아몬드 입자(4)의 투영 면적의 110~160%배로 하고, 상기한 마스킹 시트(2)의 비마스킹부(1)의 전면적이 마스킹 시트(2)의 전면적의 0.04~0.2배의 범위로 하는 것이 특징으로 된 화학적-기계적 연마 패드 컨디셔너를 제공하는 것이다.

아래에, 도면을 참고하여 본 고안을 상세히 설명한다. 도면 1은 마스킹 시트(2)를 접착한 상크의 부분 평면도이다. 도면 1에서 점으로 표시되는 부분이 비마스킹부(1)로서, 한 개의 비마스킹부(1)에 1개의 다이아몬드 입자(4)가 고착된다. 1개의 비마스킹부(1)에 고착되는 다이아몬드 입자(4)가 2개를 넘으면, 패드에 의한 연마에서 웨이퍼의 표면거칠기 값이 낮아지며, 웨이퍼의 스크래치 개수가 감소되기 쉽다. 도면 1에서 비마스킹부(1)의 전면적이 다이아몬드 입자(4)를 고착한 연삭면 적의 0.04~0.2배이다. 비마스킹부(1)의 전면적이 다이아몬드 입자(4)를 고착한 연삭면의 전면적의 0.04배 미만이면, 컨디셔너(7)로서의 연삭력이 부족한 문제가 있으며, 컨디셔너(7)의 수명이 짧아지게 된다. 또한 비마스킹부(1)의 전면적이 다이아몬드 입자(4)를 고착한 연삭면의 전면적의 0.3배 이상이면 패드에 의한 연마에서 웨이퍼의 표면거칠기 값이 낮아지며, 웨이퍼의 스크래치 개수가 감소되기 쉽다. 본 고안의 컨디셔너의 상크(3) 재료로는, 스테인레스 강재인 STS 304를 사용하는 것이 바람직하나, 경우에 따라서는 에폭 등의 합성수지 등이 사용될 수 있다.

본 고안의 컨디셔너를 제조하기 위해서는, 우선 상크(3)의 일지를 행하는 것이 바람직하다. 일지를 종료한 상크면에, 구형 형상의 비마스킹부(1)를 가지는 마스킹 시트(2)를 접착한다. 도면 1에서, 원형의 구형 형상의 비마스킹부(1)를 가지는 마스킹 시트(2)가 상크(3)에 접착되고, 상크(3)의 일부가 비마스킹부(1)의 형상으로 노출되어 있다. 본 고안에서는, 마스킹 시트(2)를 접착한 상태에서, 컨디셔너의 연삭면으로 되는 상크면에 에비 도금층(5)을 형성시키는 것이 바람직하다. 에비 도금을 실시하는 것에 의해, 다이아몬드 입자(4)와 상크(3)가 직접 접촉하는 것이 아니고, 다이아몬드 입자(4)의 상크(3)에 대한 고착강이 개량된다. 에비 도금하는 금속으로는 니켈 및 니켈 합금을 사용할 수가 있다. 본 고안에서는, 에비 도금을 실시한 상크를, 마스킹 시트(2)를 접착한 상태에서 도금 용기에 넣고, 도금 용기 중에서 상크의 마스킹 시트(2)를 접착한 면의 부분에 다이아몬드 입자(4)를 충전한다. 이어서, 상크에 음극을 접속하고, 도금액에 양극을 접속하여, 에비 도금층(5)의 형성을 위한 전기 도금을 실시한다. 에비 도금을 위한 금속은 다이아몬드 입자(4)를 상크(3)에 에비 고정할 수 있는 것이라면 제한이 없으나, 니켈 및 니켈 합금을 사용할 수가 있다. 각 다이아몬드 입자(4)의 일부분이 비마스킹부(1)에서 에비 도금층(5)을 사이에 두고 에비고정되며, 상크 표면에서 탈락하지 않는 상태로 되면, 에비 고정을 위한 도금을 종료하고, 상크를 도금 용기로부터 꺼낸다. 도면 2는, 다이아몬드 입자가 에비 고정된 상크의 부분 단면도이다. 본 고안에서, 상크(3)에 마스킹 시트(2)가 접착되고, 마스킹 시트의 구멍 형상의 비마스킹부(1)에 다이아몬드 입자(4)가 에비 도금층(5)에 의해 에비 고정되어 있다.

이어서 상크(3)의 표면으로부터 마스킹 시트(2)를 박리시키고, 더욱이 마스킹부의 일지와 활성화 처리를 행한 후에, 다시 상크(3)를 도금 용기에 넣고서 음극을 접속하고, 도금액에 양극을 접속하여 도금을 계속하여, 다이아몬드 입자(4)를 상크(3)에 견고하게 연삭면을 형성하고, 컨디셔너를 완성하게 된다. 도금에 사용하는 금속은, 에비 고정의 경우

와 마찬가지로 다이아몬드 입자(4)를 상크(3)에 고착할 수 있는 것이면 제한이 없고, 예를 들면 니켈 및 니켈 합금을 사용할 수 있다. 그림 3은 본 고안의 컨디셔너의 연삭면의 부분 단면도이다. 본 고안에서 다이아몬드 입자(2)는 상크(3)에 예비 도금층(5)에 의해 예비 고정되며, 더욱이 도금액에 의해 형성된 Ni 도금층(6)에 의해 완전히 고착되어 있다. 본 고안에서 이용하는 마스킹 시트(2)는, 도금 조건에 견딜 수 있는 것이면 제한은 없으며, 예를 들면 폴리 폴리 염화비닐 등의 수지 시트의 일면에 아크릴계 또는 비닐 에테르계 등의 점착제를 도포한 것에 박리시트를 적층시키고, 또한 구멍 형상의 비마스킹부(1)의 가공을 실시한 것을 용이하게 사용할 수가 있다. 마스킹 시트(2)의 두께란, 수지 시트 및 점착 제 층의 두께의 합이 되며 이러한 마스킹 시트의 두께는 다이아몬드 입자 직경의 80%~140%의 범위로 하는 것이 바람직하다. 또한 더욱이 1개의 구멍 형상의 비마스킹부(1)의 면적이 다이아몬드 입자의 투영 면적의 110~160%배의 범위로 되어야 한다. 마스킹 시트의 두께를 다이아몬드 입자 직경의 80% 미만으로 한다든지, 또는 한 개의 비마스킹부의 면적이 다이아몬드 입자의 투영 면적의 160%를 초과하면, 한 개의 비마스킹부에 2개 이상의 다이아몬드 입자가 고착되어서 균등한 분산이 이루어지지 않는다. 또한 비마스킹부의 면적이 다이아몬드 입자의 투영 면적의 110% 미만에서는, 다이아몬드 입자의 상크에 대한 부착이 곤란하게 된다. 따라서 마스킹의 두께를 전착할 다이아몬드 입자 경의 80~140%의 범위로 하고, 마스킹하지 않는 구멍의 직경을 전착할 입자경의 110~160%의 범위로 하는 것에 의해, 컨디셔너 금속에 전착되는 다이아몬드 입자(4)가, 비마스킹부(1)에 한 개씩 분산되도록 한다. 다이아몬드 입자의 평균 투영 면적은, 다이아몬드 입자가 분산된 상태에서 확대 사진을 촬영하는 것에 의해 구할 수 있다. 본 고안에서는, 상기한 마스킹 시트의 비마스킹부의 전면적이 마스킹 시트의 전면적의 0.04~0.2배의 범위이다. 마스킹 시트의 비마스킹부의 전면적은, 본 고안의 컨디셔너의 연삭면에서 다이아몬드 입자가 고착된 부분의 전면적에 대응한다. 마스킹 시트의 비마스킹부의 전면적이 마스킹 시트의 전면적의 0.04배 미만이면, 구멍 형상의 비마스킹부의 전면적이 다이아몬드 입자를 고착한 연삭면의 전면적의 0.04배 미만으로 되어, 컨디셔너로서의 연삭력이 부족할 염려가 있다. 마스킹 시트의 비마스킹부의 전면적이 마스킹 시트의 전면적의 0.2배를 초과하면, 구멍 형상의 비마스킹부의 전면적이 다이아몬드 입자를 고착 한 연삭면의 전면적의 0.2배를 초과하여, 연삭시에 눈메움이 발생할 염려가 있다.

《실시예》

컨디셔너 상크(3)의 연삭면으로 되는 작용면을 탈지하고, 마스킹 시트를 접착한 후에, 니켈에 의한 예비 도금층(5)을 형성하였다. 마스킹 시트(2)의 두께를 전착할 #120 메쉬 다이아몬드 입자경의 80%로 하였으며, 더욱이 다이아몬드 입자(4)를 전착시키기 위해 비마스킹부(1)의 직경을 전착할 다이아몬드 입자경의 140%로 하였다. 구멍 형상의 비마스킹부(1)의 전면적을 마스킹 시트(2)의 전면적의 0.1배가 되도록 하였다. 그 후에 컨디셔너의 본체를 도금액에 담그고 입자 고정면에 다이아몬드 입자(4)를 놓고서 컨디셔너의 본체에 음극을 접속하고, 도금액에 양극을 접속하여 전기 도금을 행하여 다이아몬드 입자(4)를 예비 고정하였다. 상크를 도금액으로부터 빼내고, 마스킹 시트(2)를 박리시키며, 탈지를 행한 후에 컨디셔너의 본체를 다시 도금액에 담그며, 본체에 음극을 접속하고 도금액에 양극을 접속하여 다이아몬드 입자 고정면의 최종 Ni 도금을 행함에 의해 다이아몬드 입자(4)를 상크의 연삭면에 고착하였다.

컨디셔너의 형상은 실시예 1에서는 도 4와 같은 다이아몬드 입자의 분포를 가지도록 하였으며, 실시예 2에서는 도 5와 같이 전착부가 스파이럴 형상이 되도록 하였고, 실시예 2에는 비전착 영역(8)이 형성되도록 하였다. 실시예 1 및 실시예 2는 컨디셔너의 형상을 제외하고는 다른 모든 제조 조건은 동일하였다. 비교예는 실시예 1 및 실시예 2의 컨디셔너의 성능과 비교를 행하기 위하여 양사에서 종래의 제조방법으로 제조되어 시중에서 판매되는 것으로, 비교예 1은 실시예 1과 동일 형 상이며, 비교예 2는 실시예 2와 동일 형상의 컨디셔너를 사용하였다. 실시예 1, 실시예 2, 비교예 1 및 비교예 2의 컨디셔너를 사용하여, 반도체의 웨이퍼 가공에 사용되는 화학-기계-연마 장치의 패드 컨디셔닝을 행한 결과를 표 1에 나타냈다.

[표 1]

	실시예 1	실시예 2	비교예 1	비교예 2
연마속도비	1.38	1.35	1	0.9
컨디셔닝속도(㎛/h)	35	34	12	11
컨디셔너수명(hr)	600	550	100	135
웨이퍼표면거칠기(nm)	0.33	0.34	0.43	0.44
웨이퍼의 스크래치 개수	1	1	7	8
패드의 표면상태	연삭흔 거의 없음	연삭흔 거의 없음	연삭흔 많음	연삭흔 많음

실시예 1 및 실시예 2의 패드 연마속도비, 컨디셔닝 속도 및 컨디셔너 수명이 비교예에 비해 현저히 높으며, 웨이퍼의 표면거칠기 및 스크래치 개수의 경우에서도 실시예 1과 실시예 2가 비교예에 비해 현저히 낮은 값을 보이며, 실시예 1 및 실시예 2의 컨디셔너를 사용한 경우 패드의 연삭흔이 거의 나타나지 않았으나, 비교예의 컨디셔너를 사용한 경

우는 패드의 표면에 연삭흔이 많이 나타났다.

고안의 효과

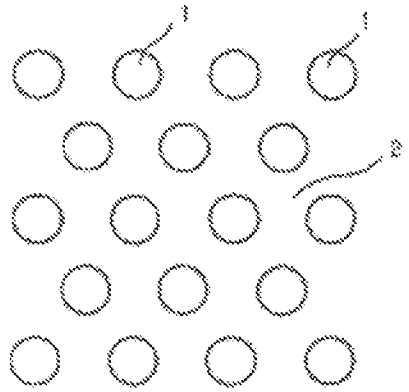
이상에서 서술한 바와 같이 본 고안은, 마스킹 시트(2)의 점착에 의해 마스킹을 행하므로 저수 정밀도가 엄격한 패턴 형상을 재현성 높게 용이하게 형성시킬 수 있으며, 마스킹 시트(2)의 컨디셔너 금속에 대한 밀착력이 높으며, 또한 컨디셔너 금속에 전착되는 80~120메쉬의 다이아몬드 입자(4)가, 비마스킹부(1)에 한 개 씩 분산된다. 따라서 CMP용 폴리싱 패드의 평탄화를 향상시킴과 동시에, 이 패드의 컨디셔닝을 효과적으로 행하는 기능을 가지고, 더구나 컨디셔닝 성능이 우수하고, 수명이 길며, 컨디셔너의 제조가 용이하기 때문에 제조시의 불량률을 현저히 감소시킬 수 있다.

(57) 청구의 범위

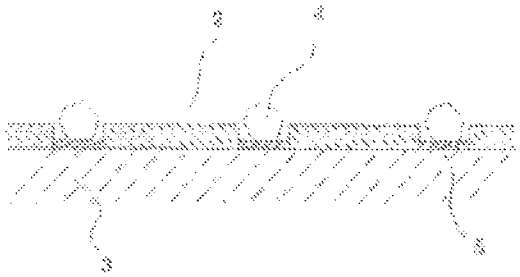
청구항 1.
Ni 도금층이 다이아몬드 입자 직경의 40~60% 정도의 두께를 가지며, 상크의 표면에 있어서, 1개의 구형 형상의 비마스킹부의 면적이 다이아몬드 입자의 투영 면적의 110~160%에로 하고, 마스킹 시트의 비마스킹부의 전면적이 마스킹 시트의 전면적의 0.04~0.2배의 범위로 하는 것이 특징으로 된 화학적-기계적-연마 패드 컨디셔너.

도면

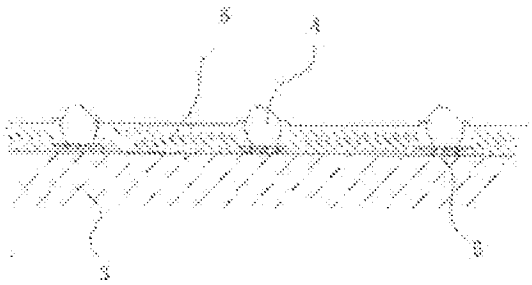
도면1



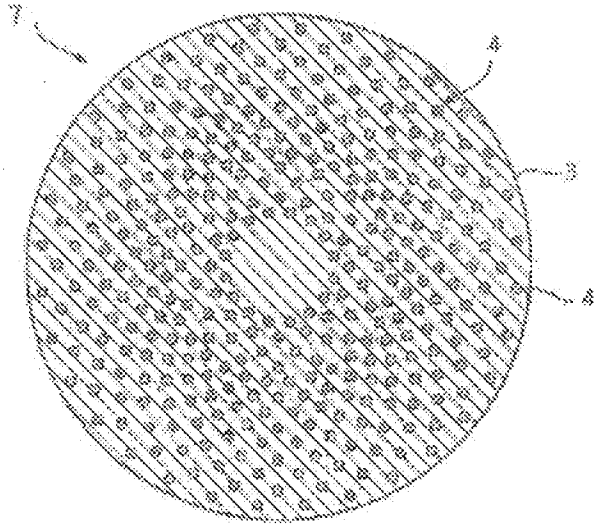
도면2



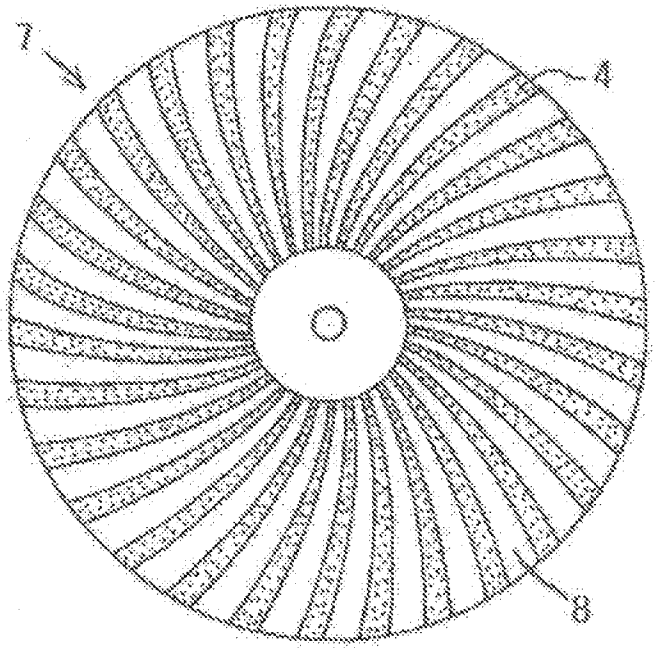
도면3



도면4



도면6



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CONTOURED CMP PAD DRESSER AND ASSOCIATED METHODS

No documents available for this priority number.

Inventor(s): SUNG CHIEN MIN [US] ± (SUNG CHIEN MIN)

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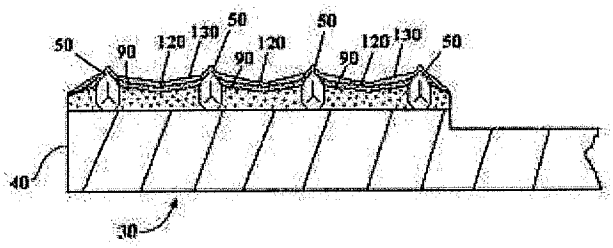
Application number: KR20077009784 20070430

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Abstract of KR20070063569 (A)

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



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(54) 윤곽지어진 CMP 패드 드레서 및 관련 방법들

(57) 요약

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

대표도

도 10

특허청구의 범위

청구항 1.

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

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

청구항 2.

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

청구항 3.

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

청구항 4.

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

청구항 5.

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

청구항 6.

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

청구항 7.

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

청구항 8.

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

청구항 9.

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

청구항 10.

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

청구항 11.

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

청구항 12.

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

청구항 13.

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

청구항 14.

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

청구항 15.

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

청구항 16.

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

청구항 17.

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

청구항 18.

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

청구항 19.

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

청구항 20.

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

청구항 21.

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

청구항 22.

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

청구항 23.

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

청구항 24.

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

청구항 25.

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

청구항 26.

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

청구항 27.

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

청구항 28.

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

청구항 29.

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

청구항 30.

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

청구항 31.

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

청구항 32.

다음을 포함하는 CMP 패드 드레서:

기판; 및

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

청구항 33.

다음을 포함하는 CMP 패드 드레서:

기판; 및

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

청구항 34.

다음을 포함하는 CMP 패드 드레서:

기판; 및

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

청구항 35.

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

청구항 36.

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

청구항 37.

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

청구항 38.

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

청구항 39.

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

청구항 40.

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

청구항 41.

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

청구항 42.

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

청구항 43.

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

청구항 44.

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

청구항 45.

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

청구항 46.

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

청구항 47.

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

청구항 48.

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

청구항 49.

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

청구항 50.

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

청구항 51.

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

청구항 52.

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

청구항 53.

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

청구항 54.

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

청구항 55.

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

청구항 56.

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

청구항 57.

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

청구항 58.

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

청구항 59.

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

청구항 60.

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

청구항 61.

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

청구항 62.

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

기판을 제공하는 단계; 및

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

명세서

기술분야

발명의 분야

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

배경기술

발명의 배경

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

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

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

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

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

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

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

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

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

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

발명의 요약

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

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

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

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

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

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

발명의 상세한 설명

발명의 상세한 설명

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

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

정의

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

발명

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

산업상 이용 가능성

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

도면의 간단한 설명

도면의 간단한 설명

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

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

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

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

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

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

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

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

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

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

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

도면

도면1

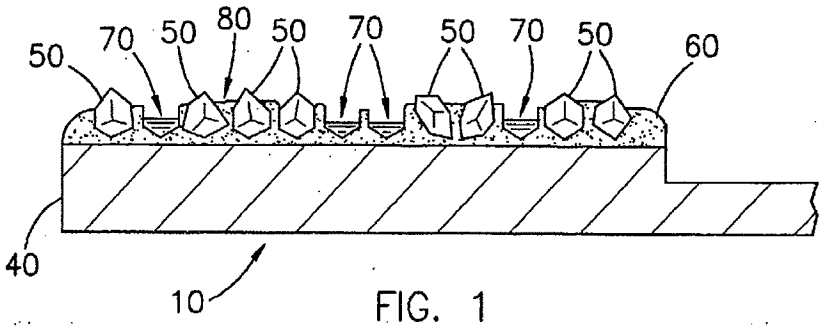


FIG. 1
(PRIOR ART)

도면2

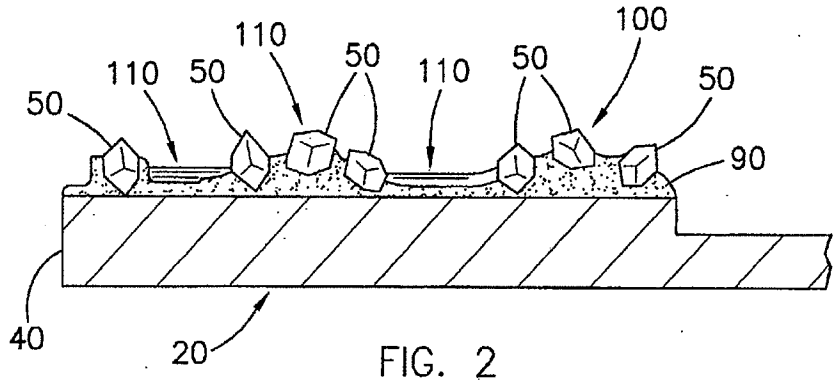


FIG. 2
(PRIOR ART)

도면3

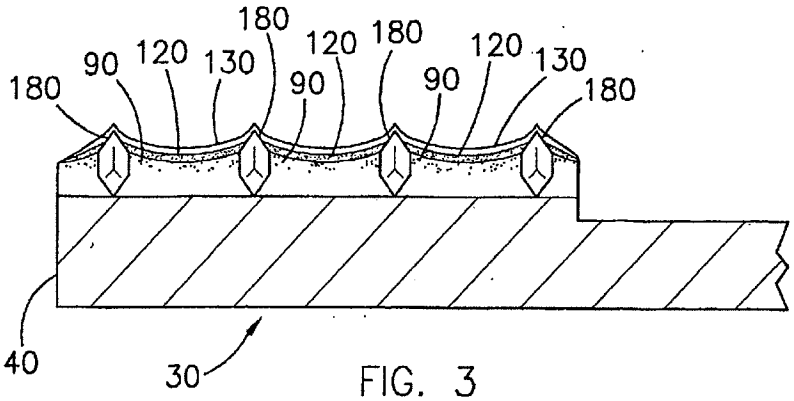
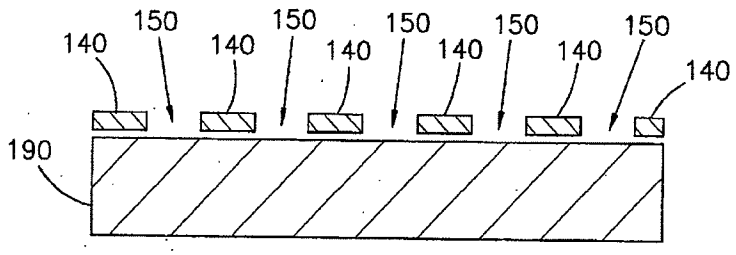
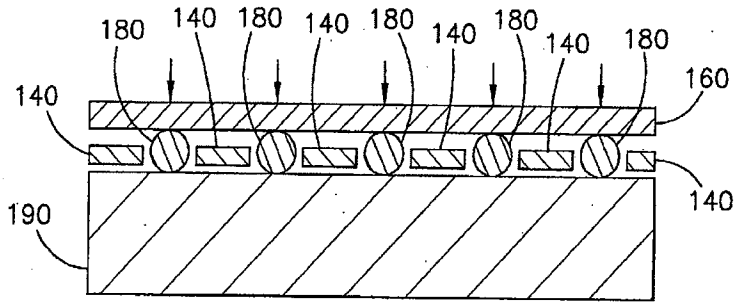


FIG. 3

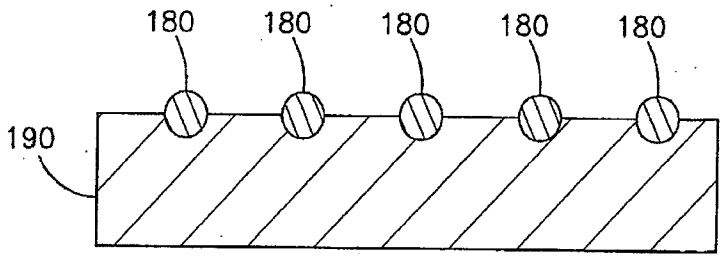
도면4



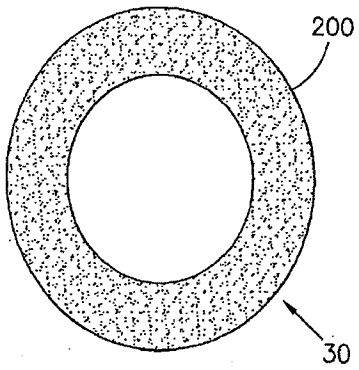
도면5



도면6



도면7



도면8

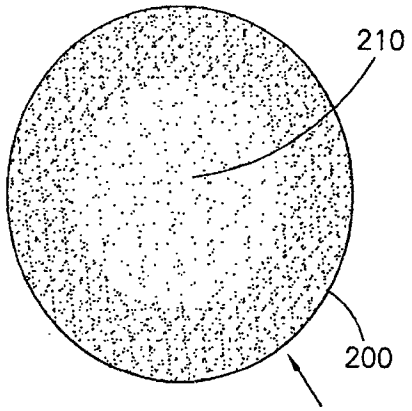


FIG. 8

도면9

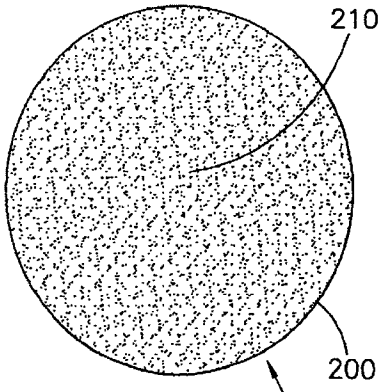


FIG. 9

도면10

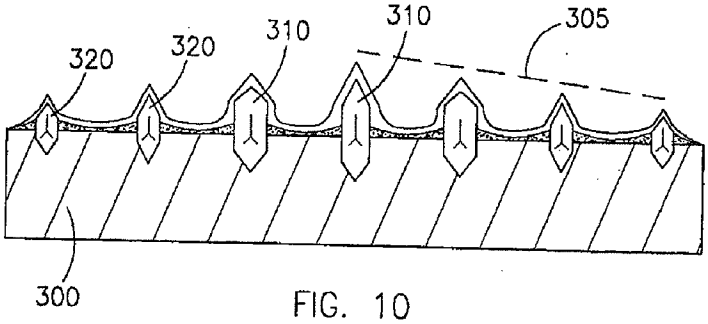
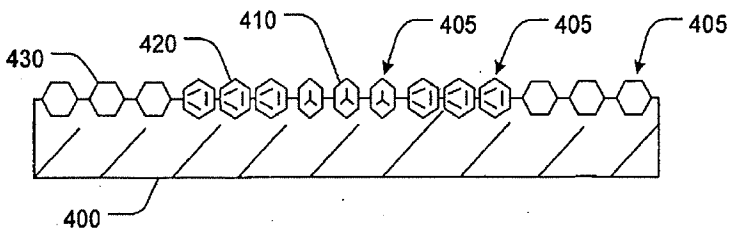


FIG. 10

도면11



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Publication Title:

SURFACE MOUNT AND FLIP CHIP TECHNOLOGY

Abstract:

Abstract of WO9531006

An integrated circuit chip has full trench dielectric isolation of each portion of the chip. A heat sink cap (100) is attached to a diamond passivation layer (96) on the substrate front side surface. The passivation layer is a CVD diamond film which provides both electrical insulation and thermal conductivity. In a flip chip version, frontside electrical contacts (174a, 174b) extend through the frontside passivation layer to the heat sink cap. In a surface mount version, vias are etched through the substrate, with surface mount posts (90a, 90g) formed on the vias, to contact the frontside electrical contacts and provide all electrical contacts on the substrate backside surface. The wafer is then scribed into die in both versions without need for further packaging.

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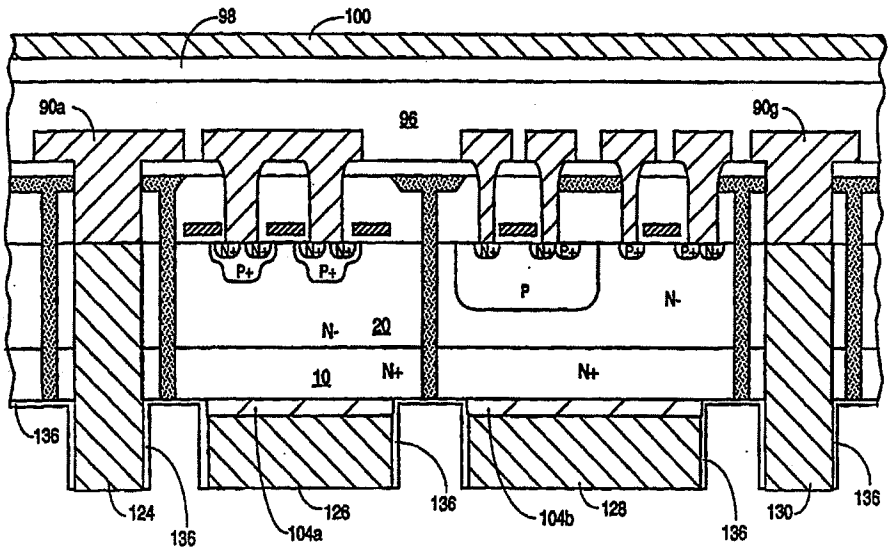
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(57) Abstract

An integrated circuit chip has full trench dielectric isolation of each portion of the chip. A heat sink cap (100) is attached to a diamond passivation layer (96) on the substrate front side surface. The passivation layer is a CVD diamond film which provides both electrical insulation and thermal conductivity. In a flip chip version, frontside electrical contacts (174a, 174b) extend through the frontside passivation layer to the heat sink cap. In a surface mount version, vias are etched through the substrate, with surface mount posts (90a, 90g) formed on the vias, to contact the frontside electrical contacts and provide all electrical contacts on the substrate backside surface. The wafer is then scribed into die in both versions without need for further packaging.

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SURFACE MOUNT AND FLIP CHIP TECHNOLOGY

BACKGROUND OF THE INVENTION

Field of the Invention

This invention relates to integrated circuit electrical isolation, and more specifically to integrated circuit total isolation using both surface mount and flip chip fabrication technologies with a diamond film passivation layer.

Description of the Prior Art

Dielectric isolation for integrated circuits is well known. A trench or groove is formed in the semiconductor chip substrate and is lined or filled with an insulative material, to electrically isolate various portions of the chip. For instance, a particular portion of the chip such as power (high voltage) transistors is surrounded by an insulated trench, thereby electrically isolating the power transistors from logic type (low voltage) transistors.

This prior art trench isolation has the significant disadvantage that the bottom of each trench is an area of substantial mechanical stress in the silicon substrate, thereby being detrimental to chip functionality. Such trenches thus reduce yield (increase cost). This deficiency is sometimes overcome by using "round hole" techniques to form the bottom of

each trench (providing a U-shaped rather than a rectangular cross-section) which however, also tends to increase cost.

Another method for achieving isolation uses two wafers, with an oxide layer formed on a surface of one wafer which is then bonded to the second wafer; the oxide layer provides the isolation. After bonding the two wafers, the backside of one of the wafers is polished away down to the oxide layer to isolate a portion of the remaining substrate. However, this is very expensive because there can be no defects in the bond between the two wafers. Defects are often induced by particles in the bond between the two wafers, causing low production yield and hence high cost. Also, oxidation induced stacking faults (OISF) due to processing temperature cycles are undesirably more pronounced in such a bond than in bulk silicon.

Therefore, the prior art is deficient in not providing practical and inexpensive dielectric isolation, especially for use with power transistors but also for other types of integrated circuits.

SUMMARY OF THE INVENTION

An integrated circuit structure and a fabrication method use trench dielectric isolation. This trench dielectric isolation is part of a chip fabrication process which allows the packaging for each chip to be formed on the wafer, thereby eliminating the prior art separate step of packaging a semiconductor die after the wafer is scribed into multiple die. Therefore the disclosed process is the equivalent of assembly or packaging the integrated circuit die at "waferscale", since each die is already packaged before the wafer is scribed.

In accordance with the invention, transistors (or other semiconductor devices) are conventionally

fabricated on a semiconductor substrate. Some of these devices may be power transistors (operating at high voltage) and others may be lower voltage logic-type transistors formed in another portion of the substrate. In one embodiment, the devices are field effect transistors having gate electrodes formed in an insulated layer overlying the principal surface of the semiconductor substrate. Alternatively, the transistors are IGBT's (insulated gate bipolar transistors), or bipolar transistors, or other semiconductor devices. The fabrication process technology may be a CMOS, NMOS, PMOS, DMOS, BiCMOS, or bipolar technology, for example. Then an interconnect pattern is conventionally formed overlying and interconnecting the gate electrodes (or other transistor elements), and for contacting the semiconductor regions (e.g. source and drain) formed in the substrate.

A plurality of insulated trenches are then formed penetrating the principal surface of the substrate into the depth thereof, and surrounding (in a top view) various portions of the substrate. The trenches are conventionally insulated by filling or lining them with an insulative material followed by a planarization. This step may be prior to metallization. Then a portion of the backside of the substrate is removed by conventional machining or chemical processes, exposing the bottom portion of each of the trenches, and thereby achieving full (total) electrical isolation of the various substrate portions surrounded by each trench.

Prior to the step of removing the backside portion of the substrate, the frontside surface is bonded to a heat sink cap or plate (made for instance of silicon) using epoxy or similar bonding materials. This heat sink cap also provides the needed mechanical support for the substrate, which is otherwise quite thin

following the substrate removal step.

Electrical contact is achieved in two different embodiments. In a surface mount embodiment, vias penetrate from the backside surface of the substrate to its principal (frontside) surface, allowing electrically conductive material formed in the vias to contact an interconnect structure already formed on the substrate frontside surface. Extensions of the vias on the substrate backside define posts for surface mount electrical contacts. Other similar posts formed on the backside surface of the substrate make contact to the bulk substrate. The heat sink cap attached to the frontside substrate surface has a mechanical and heat dissipation function, with no electrical connections provided thereby.

In a flip chip type embodiment, the heat sink cap also carries on it an electrical interconnect, making electrical contact to the conventional interconnect on the substrate frontside surface. This heat sink cap may be attached to a conventional flip chip backplate. In this embodiment the electrical contacts formed on the substrate backside surface are the conventional drain or collector contacts for e.g. power integrated circuits.

In both embodiments, the individual integrated circuits are packaged while still being part of a single wafer, by the application of the heat sink cap which is attached (by epoxy or other adhesive) to the wafer frontside surface. This cap replaces the conventional plastic or ceramic packaging which in the prior art is applied only after the wafer is scribed into individual die. In the present case the full trench isolation between the die allows the die to be scribed adjacent to the isolation trenches, together with attached front end mechanical support plate (cap), so no further packaging is needed.

Typically, the thickness of the substrate after the substrate removal step is less than about 50 micrometers. This very thin substrate is free of heat induced mechanical stresses, due to its mechanical flexibility. Advantageously, integrated circuit chips in accordance with the invention are inexpensively fabricated using essentially conventional steps which provide a high yield, thus reducing cost.

The dielectric isolation trenches may conventionally be lined, for instance with silicon dioxide (formed from the silicon trench walls), or may be completely filled with silicon dioxide, or may be lined with silicon dioxide and then filled with another material such as CVD (chemical vapor deposition) nitride, polysilicon or oxide. In another embodiment, the trenches are filled with conventional spun on glass. Other insulating materials may be used for the trench dielectric.

The heat sink cap (plate) is typically silicon, silicon carbide, silicon nitride, aluminum nitride, molybdenum, or other material which provides sufficient heat transfer and is conveniently fabricated. The removal of the backside of the substrate advantageously eliminates mechanical stresses normally present at the bottom of a trench formed in silicon. Thus the principal problem with prior art trench isolation is overcome, resulting in a thin, flexible substrate.

Also, the prior art requirement for a semiconductor buried layer in the substrate is eliminated, due to the very thin substrate and formation of a layer of conductive material on selective areas of the substrate backside surface. Thus the processing problems associated in the prior art with establishing an effective buried layer are avoided, and the parasitic bipolar transistor effects typically present in prior art integrated circuits

(especially for power integrated circuits) are eliminated by a backside contact which is a metal plate or metallization layer rather than a buried semiconductor layer.

The metallized drain (or collector) contact on the backside surface, which is in relatively close proximity to the active transistors (due to the thinness of the substrate), increases current carrying capability over that of the prior art chips which require either the buried layer or current being transmitted through the relatively thick bulk of the semiconductor substrate. Thus advantageously the actual thickness of the final substrate itself is the minimum necessary to support (mechanically and electrically) the active transistor regions. This thickness may be as little as 10 microns, in contrast to prior art integrated circuits typically having a 400 micron die thickness needed for mechanical support for the integrated circuit.

Additionally, the step of removing the backside portion of the substrate after trench formation means that the initial trench depth need not be precisely determined. In the prior art, formation of trenches of precise depth is a significant processing limitation. In the present case, since the bottom portion of each trench is removed, exact trench depth is not critical.

Also, the fabrication process in accordance with the invention advantageously allows for an isolated backside contact structure which is not a single contact but a plurality of individual backside contacts, each contact associated with a particular portion of the die. This allows for instance a first backside contact to a power transistor portion of the integrated circuit, and a second backside contact associated with a low voltage transistor portion of the integrated circuit. Thus the backside contacts are

selectable for a particular portion of the integrated circuit, and various portions of the chip need not share a common collector or common drain.

In another embodiment, the passivation layer which serves as an isolation layer and an attachment layer to bond the IC wafer (substrate) to the heat sink cap or plate is a CVD (Chemical Vapor Deposition) diamond film. Such material advantageously has high thermal conductivity and high electric resistivity. The diamond film is deposited on top of the substrate after the final front side surface metal patterning. A thermally conductive adhesive (such as silver epoxy) is applied to the exposed surface of the deposited diamond film to attach the substrate to the plate. This use of diamond film is compatible both with the surface mount and flip chip embodiments described above. In accordance with the invention the diamond layer may be patterned so that particular portions of the metal contact or bonding pads are connected to and through the support plate to external leads from the front side of the wafer, in the flip chip embodiment.

BRIEF DESCRIPTION OF THE DRAWINGS

Figs. 1 to 12 illustrate exemplary fabrication steps of the surface mount embodiment of the invention.

Figs. 13 to 17 illustrate exemplary fabrication steps of the flip chip type embodiment of the invention.

DETAILED DESCRIPTION OF THE INVENTION

Surface Mount Embodiment

Figs. 1 to 12 illustrate steps to fabricate a surface mount integrated circuit chip in accordance with the invention. It is understood that these are conventionally drawn cross sections of an integrated

circuit showing only a small portion of the circuit, i.e. a small portion of a wafer. No topside views are provided, since the top side geometry of the integrated circuit is conventional, as will be understood by one of ordinary skill in the art from the following description. Also, this is only an illustration of the surface mount approach, which is not limited to semiconductor devices of the type shown.

Fig. 1 shows initial steps in fabrication of this embodiment. Fig. 1 is conventional both in structure and fabrication as typically used for integrated circuits which include both power transistors and low voltage transistors on one chip. See U.S. Patent Application No. 07/948,276, filed 9/21/92, Hamza Yilmaz et al., entitled "BiCDMOS Process Technology and Structure", incorporated by reference, for an example of such a fabrication process. Shown in Fig. 1 is silicon (or other material) substrate 10 which is conventionally N+ doped, having formed on an upper portion thereof in one embodiment an epitaxial (silicon) layer 20 which is conventionally N- doped. The epitaxial layer is an option but is not a necessary part of the invention; the active portions of the transistors may be formed in the substrate. (It is to be understood that materials, dimensions, transistor structure, conductivity types, and other elements disclosed herein are illustrative only and not limiting.)

Substrate 10 is approximately 500 micrometers thick, and silicon layer 20 is 1 to 50 micrometers thick. Conventionally formed (in this illustrative embodiment) in silicon layer 20 are two "deep body" conventionally doped P+ regions 22 and 24. Formed respectively in body regions 22 and 24 are N+ source regions 26, 28, 30 and 32 each of which is conventionally doped. It is to be appreciated that in

this embodiment P+ body regions 22 and 24 and the associated other doped regions are a part of a power portion of the integrated circuit, i.e. power transistors.

Formed adjacent to regions 22 and 24 is P tub 38 which is conventionally doped. Formed in P tub 38 are N+ doped regions 40 and 42. Also formed in silicon layer 20 are P+ regions 46 and 48 which are similar in their doping level and structure to region 44, and N+ region 50 which is similar to regions 40 and 42. Formed immediately overlying the upper surface of silicon layer 20 is a conventional gate oxide layer 60 of e.g. silicon dioxide.

Formed conventionally overlying oxide gate layer 60 is a doped polysilicon gate electrode layer 54 including (as a result of a masking step) portions 54a, 54b, 54c and 54d and 54e which in this embodiment form the gate electrodes of various transistors.

Overlying gate electrode layer 54 is a second overlying (upper) insulative layer 62 formed of silicon dioxide and also covering the respective side edges of the gate electrodes 54a, ..., 54e.

Overlying upper oxide layer 62 is a BPSG layer 64 (borophosphorsilicate glass) formed to a thickness of 0.5 to 1.5 micrometers and then conventionally reflowed to completely cover gate electrodes 54a, ..., 54e.

Fig. 2 shows subsequent processing with a conventional masking layer 70 formed overlying BPSG layer 64. Masking layer 70 is conventionally patterned and the underlying structure etched, with the etchant penetrating through silicon layer 20 and into substrate 10 to define trenches 72a, 72b, 72c, 72d, and 72e. These trenches are 0.5 to 5 micrometers in width and 5 to 50 micrometers in depth. (The depth is relatively arbitrary for reasons discussed later and the width dimension is also not critical.) The trench etch is a

conventional anisotropic process. It is to be understood that trenches 72a, ..., 72e as shown here in cross-section, would in a topside view interconnect so as to surround various portions of the integrated circuit, i.e. so as to surround the power transistor portion 22, 24 thereby separating this portion from the portion including P tub 38.

Then (not shown) the remaining portions of masking layer 70 are conventionally stripped off.

Next in Fig. 3, a glass layer 76 is formed conventionally such as by being spun on in each of trenches 72a, ..., 72e thereby filling each of these trenches, and also overlying the upper surface of BPSG layer 64, to a thickness of 1 to 2 micrometers. Alternatives to SOG for layer 76 are CVD formed materials or plasma enhanced CVD formed materials. After formation of spun on glass layer 76, this material is cured. Spun on glass 76 here is the trench dielectric insulating material. In other embodiments, the trenches e.g. are lined with a layer of grown silicon dioxide and then filled with another material such as CVD oxide, or nitride, or polysilicon. (These are conventional processes for forming dielectric layers.)

Next in Fig. 4, first an etch back (planarization) step using for instance CMP of the spun on glass layer 76 effectively removes those parts of layer 76 overlying the gate electrodes 54a, ..., 54e. An alternative to CMP is to apply a layer of photoresist then etch back using a dry etch.

Following the etch back step, an LTO (low temperature oxide) layer 80 is deposited over the upper surface of the structure to a thickness of 0.5 to 2.0 micrometers. Following the LTO layer 80 deposition, an electrical contact mask layer (not shown) is applied overlying LTO layer 80. The contact mask layer is then

conventionally patterned and the underlying layers etched, with the etch extending through LTO layer 80, through the remaining portions of spun on glass layer 76, and down to the principal surface of the silicon layer 20, thus exposing portions of the principal surface of the silicon layer 20 and defining contact openings 84a, ..., 84g.

Next in Fig. 5, a layer of conductive material 90 (such as aluminum) is conventionally deposited over the surface of the substrate, filling the various contact openings 84a, ..., 84g and making contact with the underlying semiconductor regions formed in silicon layer 20. Then conductive layer 90 is conventionally masked, the masking layer is patterned, and layer 90 is etched to define electrical contacts 90a, ..., 90g.

Then in Fig. 6 a conventional passivation layer of e.g. plasma nitride material is formed overlying electrical contacts 90a, ..., 90g. Passivation layer 96 is 0.5 to 2.5 micrometers thick.

Overlying passivation layer 96, an epoxy material layer 98 is conventionally deposited 25 to 250 micrometers thick. This epoxy material 98 is e.g. a high performance epoxy resin composition that is thermally conductive (such as silver epoxy).

Next a "cap" (plate) 100 is affixed to epoxy layer 98 which is then cured, bonding cap 100 to passivation layer 96. Cap 100 is about 500 micrometers thick and is e.g. undoped silicon. A single cap 100 covers the entire wafer which includes substrate 10 and silicon layer 20. Other bonding materials and processes other than epoxy may be used to affix cap 100. Cap 100 achieves thermal conductivity through epoxy layer 98 and also provides sufficient mechanical support for the substrate 10, silicon layer 20, and the active transistor regions and interconnect formed thereon.

Next in Fig. 7, the backside surface of substrate

10 is partially removed by grinding, etching, or CMP (chemical-mechanical polishing) so that the total thickness of substrate 10 and/or silicon layer 20 is in the range of 5 to 100 microns. This is in contrast to the original total thickness of structures 10 and 20 of about 500 microns. (Again, these dimensions are illustrative and not limiting.) The goal is to reduce the thickness of substrate 10 and silicon layer 20 to a minimum necessary for electrical functioning of the transistors formed therein. The removing step reduces the thickness of substrate 10 so that the bottom portion of each of trenches 72a, ..., 72e is exposed, and thereby the insulative material formed in each of these trenches is exposed at the backside surface of substrate 10. This achieves full electrical isolation of each portion of substrate 10. It is to be understood that in this case the attachment of heat sink cap 100 is done prior to the removing step to ensure that the substrate, after the removing step, remains in one integrated piece. Conventional masking, implantation, and annealing steps may be added to this process, as desired to form N+ or P+ contacts on the backside surface of substrate 10 for subsequent metal layer 104.

Next in Fig. 8, the backside surface of substrate 10 is metallized by a conventional sputtering or evaporative process to form a metal layer 104 (aluminum) thereon having a thickness of e.g. 0.15 to 1.5 micrometers. Then metal layer 104 is conventionally covered with a mask layer, the mask layer is patterned, and metal layer 104 then etched to define backside contact pads 104a, 104b on the backside surface of substrate 10. Each of contact pads 104a, 104b is associated with a selected portion of the integrated circuit die as defined by the trenches 72a, ..., 72e. Of course each portion of the die need not

have a contact pad; contact pads 104a, 104b are only provided to those portions of the die which require same. (It is to be understood that conventionally some types of transistors do not require backside contacts.)

Next in Fig. 9, a protective/passivation layer 110 is deposited on the backside surface of substrate 10 overlying contact pads 104a and 104b. Then layer 110 is conventionally patterned to define openings 112a and 112b. Each opening 112a and 112b is associated with a particular electrical contact 90a, 90g on the upper side surface of silicon layer 20, as shown.

Next in Fig. 10, a deep silicon etch is undertaken through openings 112a and 112b to define vias 116a, 116b extending through substrate 10 and/or silicon layer 20 to expose the underside portions of electrical contacts 90a, 90g. This deep silicon etching step uses a wet or wet/dry or dry etching technique.

Vias 116a, 116b need only be wide enough to establish good electrical paths to contacts 90a, 90g and otherwise their dimensions in terms of width are not critical; neither is their cross-sectional shape critical. Then as shown in Fig. 10 the remaining portions of layer 110 may be conventionally stripped off or kept as a passivation layer.

Next in Fig. 11 by a plating step (electrodeposition), vias 116a and 116b are filled with a solderable metal (e.g. nickel, gold, copper) to define posts 124, 130 extending therefrom. In the same electrodeposition step, metal posts 126, 128 are deposited on metal contacts 104a, 104b.

Alternatively, posts 126, 128 are formed by screen printing the desired type of metal on respectively the backside 104, 104b interconnection leads to establish the desired thickness for the surface mount posts. The diameter of the surface mount posts may vary, depending on the particular application.

Next in Fig. 12, an (optional) passivation layer 136 is deposited over all exposed surfaces of the surface mount posts 124, 126, 128 and 130 to a thickness of 0.8 to 1.2 micrometers. Passivation layer 136 is e.g. polyamide or low temperature oxide.

Then portions of passivation layer 136 are removed (by masking and etching or other means, for instance CMP) from the bottom surfaces of each surface mount post 124, 126, 128, 130, so as to leave the post bottom surfaces exposed for soldering, such as to a printed circuit board. The height of the surface mount posts extending from the lower backside surface of substrate 10 is conventionally 10 to 300 micrometers, or any other height as needed depending on the mounting application.

In Fig. 12 all of the electrical interconnections to outside of the chip are provided on the backside surface of substrate 10 by the surface mount posts 124, 126, 128 and 130.

Subsequently (not shown) the wafer is scribed into individual integrated circuit die along predescribed scribe lines which are for instance adjacent to dielectrically filled trenches. Thus as a result of the scribing step, the various integrated circuit die are already packaged by the top side cap 100 and the bottom side passivation layer 136; hence no further packaging is needed.

Flip Chip Embodiment

In the flip chip embodiment, the initial processing steps are identical to those shown in Figs. 1 to 5 for the surface mount embodiment, resulting in the structure of Fig. 13. Again, the flip chip embodiment is not limited to semiconductor devices of the type shown. The structure of Fig. 13 is identical to that of Fig. 5, except that in Fig. 13 the next step is that passivation layer 160 (which is similar in

material and thickness to passivation layer 96 in Fig. 6) is masked using a mask layer (not shown) which is patterned, with subsequent etching to define contact openings 164a, 164b, therethrough. Contact openings 164a, 164b, expose a portion of the underlying electrical contacts 90a, 90g. The goal in this embodiment is to define both substrate frontside and backside electrical contacts; openings 164a, 164b define the frontside contact openings.

Next in Fig. 14, silicon (or other suitable material) heat sink cap 172 is provided, having formed thereon (prior to assembly to the substrate 10) metal (aluminum or a solderable material) interconnect structures 176a, 176b. Interconnect structures 176a, 176b include interconnect lines on the surface of cap 172 which faces contacts 90a, 90b; these interconnect lines have a thickness of e.g. 10 to 50 micrometers. Cap 172 is electrically insulated on all surfaces by e.g. oxide dielectric layer 175. Interconnect structures 176a, 176b are then conventionally soldered or sintered to underlying contacts 90a, 90g. This electrical contact can be provided by any other conventional method.

Cap 172 is attached to the underlying structure by a high performance thermally conductive (and electrically insulative) epoxy resin layer 168 (or other high performance adhesive) formed on passivation layer 160 to a thickness of 25 to 250 micrometers. Adhesive layer 168 is prevented from fouling the electrical interconnections (between elements 176a, 176b and 90a, 90b) by masking and etching the adhesive layer 168 or by polishing it away from the interconnections. For instance, adhesive layer 168 is initially formed in a thick layer covering structures 176a, 176b, then etched back to expose the ends of structures 176a, 176b using a wet or dry etch.