

[54] METHOD OF COATING DIAMOND PARTICLES WITH METAL

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3,276,852 10/1966 Lemelson .....51/295
3,356,473 12/1967 Hull et al. ....51/309
3,293,012 12/1966 Smiley et al. ....51/293
3,178,273 4/1965 Libal.....51/293

Primary Examiner—Donald J. Arnold
Attorney—Rowland V. Patrick

[52] U.S. Cl. ....51/295, 51/298, 51/308, 51/309
[51] Int. Cl. ....B24b 1/00
[58] Field of Search.....51/295, 298, 293, 309, 308; 117/100

[57] ABSTRACT

Titanium (or zirconium) coated single diamonds have high cohesion bonding between coating and diamond and have external surfaces adhesion-receptive to subsequently applied less oxidizable metal, resin, and ceramic coatings which permit the double coated particles to be mounted at elevated temperatures in or on extraneous supports with or without application of pressure, without loosening the cohesion bonding and without resort to nonoxidizing atmospheres; or permitting them to be directly mounted in nonoxidizing atmospheres, in either case providing long life support for single or multiple diamond arrays as in cutting and abrading implements.

[56] References Cited

UNITED STATES PATENTS

2,319,331 5/1943 Kurtz.....51/295
2,411,867 12/1946 Brenner.....51/309

7 Claims, 6 Drawing Figures

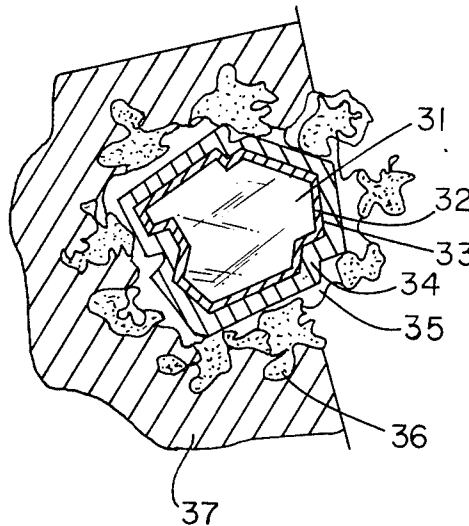


FIG. 1

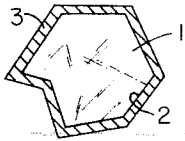


FIG. 2

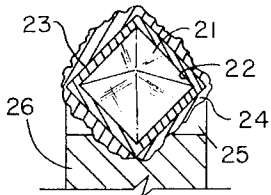
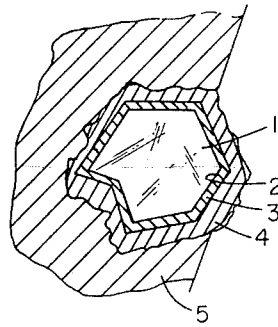


FIG. 3

FIG. 4

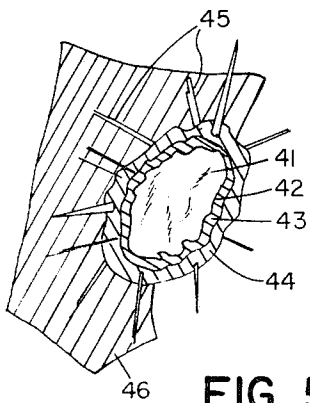
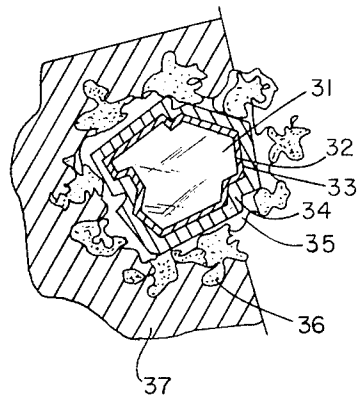


FIG. 5

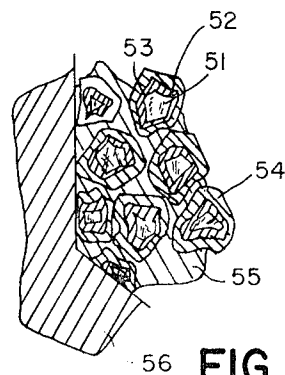


FIG. 6

## A METHOD OF COATING DIAMOND PARTICLES WITH METAL

This invention relates to diamond bonding and provides single diamond particles bearing over their entire surfaces metallic coatings which render the diamonds bondable to each other and to other surfaces with or without resorting to vacuum or inert gas environments and having very high energies of adhesion at the diamond-coating interface.

Currently, diamond particles are supported for cutting, abrading, and other uses by attaching them, singly or multiply, to the surface of a support or by embedding them, in single or multiple layers or other conglomerate configuration, partially or wholly in metallic, ceramic or organic matrices.

In these attaching or embedding operations, the diamond surface interface bond is usually created either in the act of attaching the diamond to the support or in the act of hardening or sintering the matrix. When it was recognized that conventional binder metals, for example, copper and silver alloys provide little if anything more than mechanical bonds to diamonds due to the failure of such alloys to wet the diamond surface and to the absence of any chemical bonds providing anything more than very low energies of adhesion, the art, looking for improved bonds, began to carry out the brazing or embedding operations in the presence of transition metals, particularly titanium or zirconium, either as such or as hydrides.

Thus, U.S. Pat. No. 2,570,248 suggested use of an admixture of titanium hydride and copper powders applied to the junction of two bodies to be brazed; and Hall U.S. Pat. No. 2,728,651 suggested applying a slurry of titanium hydride in an organic liquid to a diamond and then sprinkling these titanium hydride coated diamonds onto a solder alloy layer and heating and dissociating the hydride and melting the solder and dissolving the titanium.

Both of these patentees, however, recognized that the attachment heating operation, where titanium was present, had to be carried out in a vacuum or reducing atmosphere, thus requiring expensive equipment to provide vacuum chambers large enough to accommodate the tool holder or matrix-forming press.

A molding operation utilizing diamond powder together with a powdered hydride and metallic binder mixture in alcohol plus a molten metallic infiltrant is described in U.S. Pat. No. 3,293,012, also carried out in a nonoxidizing atmosphere with pressure.

In the course of making the present invention, it has been made evident that industry has erred in attempting to create a strong diamond interface bond during the act of attaching the diamond to or embedding it in an extraneous support; and that it has failed to appreciate that detrimental consequences ensue when titanium or zirconium components are subjected, in admixture with or in close proximity to non-diamond reactive alloys, simultaneously to heat treatment thereby forming an indiscriminate single bond layer from melted alloy and dissolved titanium, or that these consequences are avoidable.

In accordance with this invention, diamond bonds of great strength are provided by creating the diamond surface interface bond prior to any operation of permanently attaching it to an ultimate extraneous support or seating it in a matrix, so that only diamond-reactive materials need be present when creating the diamond surface bond. These materials, forming the diamond surface interface bond, because they are oxidizable, are then covered with a less oxidizable protective layer or layers so that subsequent attaching or embedding operations with non-oxidizable brazing alloys can be carried out in the air without oxidizing the titanium. Operation in a nonoxidizing atmosphere is thus required only during the creation of the diamond surface interface and is not thereafter needed.

Essentially, this invention provides single diamond particles with bonded coatings of those transition metals which are most likely to react with diamond or carbon, namely, titanium or zirconium or mixtures thereof. The coatings are in the form of thin continuous layers of substantially uniform thickness

which are bonded throughout the entire interface between the film and the diamond exterior surface with high energies of adhesion commensurate with those created by chemical bonds such as are present in titanium or zirconium carbide structure.

Instead then of merely mechanically depositing these metals from a slurry or other dispersion thereof on the diamond for subsequent dissolution in a molten metallic binder or solder, it has been found that when a dry mechanical deposit on a diamond surface, of these metals, partly at least in the hydride form, in the absence of other binder, is heated to a suitable temperature in a vacuum, a bond having surprisingly large energy of adhesion is created displaying at the interface some sort of reaction or electron interaction of a chemical or physico-chemical nature involving or equivalent to the formation of carbide. Apparently because the titanium or zirconium is not dissolved in any non-wetting binder and is alloy-free and otherwise uncontaminated, its attraction and attachment to the diamond surface is of an entirely different order of magnitude.

After the film or coating of the transition metal is bonded over the entire diamond particle external surface, a further layer of a different less oxidizable metal, ceramic or resin can be applied thereover to permit subsequent attaching operations to take place without resort to nonoxidizing atmospheres.

One of the beauties of the present invention is that a whole batch of diamond particles having mechanical deposits contacting each other can have the deposits converted into heat bonded coatings all at one time without fusing adjacent particles together, since the heat treatment is at a far less temperature than that which would melt the titanium or zirconium deposits. When one attempts to coat diamond particles with molten brazing materials or binder alloys, fusion takes place between the coated diamonds which therefore can no longer be recovered in individual, discrete, flowable condition but rather fuse into a solid mass. After the above conversion operation in accordance with this invention, one possesses discrete, unitary, free flowing coated diamond particles which can be further covered with one or more layers of different materials which provide surfaces that are readily attachable metallurgically in air to extraneous metal, organic or ceramic substrata or other supports by conventional means.

Because of the small amount of non-diamond binding or matrix material needed, bodies having very high concentration of diamonds can be achieved running as high as 200-260 carats per cubic inch where the body is hot pressed. Minimizing non-diamond material reduces friction and heat when the body is used for cutting and abrading purposes. The strength of the bond permits great protrusion of diamonds from their supporting surface, giving better clearance without danger of pullout and greatly prolonging life.

### EXAMPLE 1 (FIG. 1)

Diamond chips weighing an aggregate of 200 carats, 200-250 mesh size, and cleaned by conventional solvents to rid their surfaces of organic contaminants are placed in a mechanical tumbler along with approximately 6 grams of titanium or zirconium powder (at least some of which, 10 percent or more, is in the hydride form) having a mesh size of 600 and tumbled for a length of time under room temperature and atmospheric pressure conditions until each diamond particle bears over its entire exterior surface a clinging deposit of the powder.

The diamonds with the deposits thereon are then placed together in a graphite crucible and brought down to a vacuum of about  $10^{-5}$  mm. of mercury and to a minimum of about  $10^{-4}$  and subjected to a temperature of 850°-900° C. for a period of at least 10-15 minutes. The particles in the crucible are allowed to cool in the vacuum and then are removed from the vacuum chamber.

Each particle is discrete and unattached to other particles except by occasional frangible attachment, which can be

readily broken without damage to the surface. Under the microscope, each particle is seen to have acquired a very thin smooth even external metallic film of substantially uniform thickness which may be for example about 5 percent by weight of the combined weight of the film and the enclosed diamond. In the accompanying FIG. 1 the diamond core is shown at 1, the diamond surface interface with the surrounding titanium film (shown in cross section) is at 2, and 3 is the titanium film.

The surprisingly great magnitude of the heat and vacuum induced interface bond leads to the conclusion that, because of the high electrochemical activity of titanium and its diamond wetting capability in the presence of its hydride, it has reacted with the diamond to form carbide. Nevertheless, it appears that, because of the time limitation, any carbide formation has occurred primarily only at the interface with the diamond surface, leaving the external surface of the smooth coating uncarbided. This is evidenced by the fact that, in subsequent brazing operations, more than adequate metallurgical adhesion of the coated particle is secured, which would not be the case were the external surface carbided, as conventional brazing materials do not adhere well to titanium carbide surfaces. Similarly, when the coated particles are submitted to subsequent hot pressing operations, the coating is not loosened as is the case with nickel-clad diamonds. The bonds of this invention do withstand de-bonding forces imparted as a result of difference in the coefficients of expansion of the diamond and the coating.

The initial layer formed on the diamond particles is thus viewed as being a non-homogeneous layer progressing outwardly from titanium carbide content at and near the interface towards all or entirely all uncarbided titanium content on and near the external surface rendering the coated particle adhesion-receptive to metallic as well as nonmetallic bonding materials.

In any event, the coated particle has the high cohesion characteristics of metal-carbide structure at the interface, yet permits further application to the external surface of the coated particle of protective layers by conventional techniques with the resulting outer adhesions being greater than would be provided were the external surface of the initial layer constituted of titanium carbide. This dual function characteristic of the single applied titanium layer is achieved by omitting from the coating the metal alloys or brazing materials which were heretofore used but produced only low diamond adhesions, not far removed from those of solely mechanical bonds, and by limiting the depth of carbide formation. Of course the uncarbided surface may be subsequently carburized as by heating in a pack of carbon or methane where such carburizing is desired, as to reduce oxidation or create a harder surface and is not incompatible with good adhesion to subsequent bonding materials.

While the temperature range above given is preferred, a minimum temperature is about 350° C., i.e., sufficient to dissociate the hydride and the maximum is that temperature at which graphitization of the diamond under the prevailing conditions would occur, say 1,250° C. No advantage, however, has been found in elevating the temperature over 1,000° C. and the time, temperature conditions should be always such as to minimize formation of any significant amounts of carbide on the external surface of the coated particles. Because of this dual function of the single applied layer, one does not include, as has been done previously in initial titanium coatings, any other alloy or brazing material, producing far lower adhesions more commensurate with solely mechanical bonds.

#### EXAMPLE 1a

The same procedure is followed except the deposit is an admixture of 1 part of titanium and 1 part zirconium by weight (again each partially in the form of hydrides).

#### EXAMPLE 2 (FIG. 2)

A further layer of copper or nickel is plated onto the entire FIG. 1 particle to a thickness of 0.002 - 0.005 inch. Instead of electroplating or electroless plating, conventional vacuum deposit techniques, including sputtering, may be utilized.

The coated particles are then embedded in a metallic matrix by the following procedure to produce the product shown (again greatly magnified) in FIG. 2 wherein 1 is the diamond particle; 2 is the interface; 3 is the titanium layer; 4 is the copper or nickel layer; and 5 is a fragment of the metallic matrix:

The particles are mixed with copper powder and subjected to a hot press in a graphite mold or a cold press and subsequently sintered or infiltrated with brazing material by usual techniques.

Alternatively, a resin powder with or without metallic or other filler may be used.

Where the matrix is ceramic, vitrifying techniques may be used.

The copper or nickel layer 4 may be constituted of successive layers of copper and nickel.

If desired, the copper coated, the nickel coated, or the double copper, nickel coated particle may be sintered or fused in situ at the sintering or fusing temperatures of the less-oxidizable metal or metals in a nonoxidizing atmosphere producing metallurgical cohesion bonding between the transition metal and the overlying less-oxidizable metal.

It is to be understood that a protective layer or layers such as 4 is essential only in those cases where further operations are to be done without the benefit of nonoxidizing atmospheres. However, the addition of a metal layer or layers is helpful wherever press techniques are to be utilized since the softer metal will not damage the surface of the mold.

Rather than mixing the coated particles with other metal powders, the coated diamond particles 1, 2, 3, 4 can be used themselves as powdered metals with conventional powdered metallurgical techniques such as cold pressing, sintering, or infiltration for manufacturing diamond impregnated bodies.

#### EXAMPLE 3 (FIG. 3)

A coated product prepared as in Example 2 can be mounted on a tool by applying a conventional brazing material between the coated particle and a clean surface of a steel shank and the brazing material then heated with a gas torch in air to melt the brazing material.

The resulting product is shown in FIG. 3 wherein 21 is the diamond; 23 is the titanium layer; 24 is the copper or nickel layer; 25 is the brazing material and 26 is a portion of the steel shank.

#### EXAMPLE 4 (FIG. 4)

Coated particles prepared in accordance with Example 2 are covered with a layer of sponge iron by the following procedure:

Brazing material is applied to the coated particles which are then mixed with sponge iron powder and placed in vacuum or hydrogen furnace to braze the iron particles to the surface of the coated diamond.

A batch of said products are then embedded in a resin matrix by conventional procedure.

This produces the product shown in FIG. 4 wherein 31 is the diamond; 32 is the interface; 33 is the titanium layer; 34 is a metallic layer; 35 is a brazing material; 36 are sponge iron particles; and 37 is a portion of a resin matrix.

#### EXAMPLE 5 (FIG. 5)

A coated product prepared in accordance with Example 1 is plated with a coating of copper or nickel to a thickness of about 0.002 - 0.005 inch. During this operation, minute whiskers, fibers or filaments, of ceramic, glass or aluminum oxide are placed in the metal plating solution and allowed to

settle on the surface of the diamond during the course of approximately 25 minutes. Subsequent agitation of the diamonds in the plating solution results in further deposit of whiskers on the coated diamond particle surfaces during the plating operation.

The whisker-covered particles are then embedded in a resin matrix by conventional techniques to provide a grinding wheel section as shown in FIG. 5, wherein 41 is a natural or synthetic diamond particle; 42 is the interface; 43 is the titanium layer; 44 is a copper or nickel coating; 45 are whiskers; and 46 is a portion of the resin matrix.

EXAMPLE 6 (FIG. 6)

FIG. 6 shows a fragment of a wheel dressing tool having an unusually high diamond concentration which produces more cutting points and eliminates undercutting action by dressed abrasive particles.

A low carbon steel shank is provided having a cavity about 0.150 inch in diameter by 0.250 inch deep. Diamond particles bearing the titanium coatings of this invention overlaid with a nickel or copper layer (or both) are hot pressed into the cavity with copper and silver brazing alloy powders at 750° C. in air.

This produces the product shown in FIG. 6 wherein 51 are diamond particles; 52 is the interface; 53 is the titanium layer; 54 is the plated metal coating; 55 is the brazing alloy; and 56 is a portion of the cavitied rigid steel shank.

Instead of over-coating the titanium layer with metal, as in Example 2, the titanium may be over-coated directly with a ceramic or resin material as by applying them in powdered form and then vitrifying or hot pressing the applied powder in a conventional manner.

As can be seen from the variety of products shown in the accompanying drawings and hereinbefore described, the coated diamond particles of this invention may be used in making a great variety of cutting or abrading products including dressing tools, dressing wheels, saw blades, grinding wheels, grinding wheel segments either single or multiple layer, saw blade segments or inserts, drilling bits, routing, boring, turning, countersinking, and single or multiple point tools, chamfering and edging tools and wire and other saws and all other kinds of honing, lapping, cutting and abrading devices; or may be used to manufacture optical lens generators or button inserts.

The protective metallic layer may be of any metal bonding material which can be electroplated, electroless plated, vacuum deposited or sputtered, for example, nickel, copper, iron, zinc, tin, silver, gold, cadmium or cobalt or their alloys or admixtures thereof.

Useful brazing or soldering bonding materials are copper, bronze, copper-silver alloy, copper-beryllium alloy, copper-zinc alloy or any other metallurgical brazing material or solders which have adhesion to the protective layer or to the transition metal coating if the metallic protective layer is omitted.

What is claimed is:

1. A method of coating diamond particles comprising mixing a batch of individual discrete diamond particles with transition metal powder consisting essentially of powders selected from the class consisting of powders of titanium, zirconium, and their hydrides, and mixtures thereof, to form a dry deposit of said powder over the external surfaces of said diamond particles and then subjecting the particles with their deposits thereon in a single container to a temperature in the range of a minimum of about 350° and a maximum of 1,000° C. in a nonoxidizing atmosphere to coat the entire surface of each particle with a thin continuous layer consisting essentially of said transition metal, cooling the particles and removing them from said atmosphere in the form of individual discrete, nonadherent, flowable transition metal coated particles, each containing a single diamond particle.

2. A method as claimed in claim 1 wherein the deposit consists essentially of a transition metal and its hydride.

3. A method as claimed in claim 1 wherein the deposits consist essentially of titanium and titanium hydride.

4. A method as claimed in claim 1 wherein the deposits consist essentially of titanium and titanium hydride and the particles and deposits are subjected to a temperature of 350°-1,000° C. at a minimum of 10<sup>-4</sup> mm. of Hg for a minimum period of 10 minutes.

5. A method as claimed in claim 4 wherein during the temperature treatment titanium carbide structure is formed at the interfaces between the metal coatings and the diamonds.

6. A method as claimed in claim 4 wherein the temperature is 850°-900° C. for a period of from 10 to 15 minutes.

7. A method as claimed in claim 4 wherein the temperature is 850°-900° C. for a period of from 10 to 15 minutes.

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