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(54) Title: POLYCRYSTALLINE DIAMOND MATERIAL

(57) Abstract: A polycrystalline diamond material comprising a mass of diamond particles or grains exhibiting inter-granular bonding and a binder material comprises a non-metallic catalyst material for diamond, the non-metallic catalyst material for diamond comprising at least one nitrogen compound derived from an ammonium compound and/or at least one halide compound.

POLYCRYSTALLINE DIAMOND MATERIAL

Field

This disclosure relates to polycrystalline diamond (PCD) material, and to a method of making such material.

Background

Cutter inserts for machine and other tools may comprise a layer of polycrystalline diamond (PCD) bonded to a cemented carbide substrate. PCD is an example of a superhard material, also called superabrasive material, which has a hardness value substantially greater than that of cemented tungsten carbide.

Components comprising PCD are used in a wide variety of tools for cutting, machining, drilling or degrading hard or abrasive materials such as rock, metal, ceramics, composites and wood-containing materials. PCD comprises a mass of substantially inter-grown diamond grains forming a skeletal mass, which defines interstices between the diamond grains. PCD material comprises at least about 80 volume % of diamond and may be made by subjecting an aggregated mass of diamond grains to an ultra-high pressure of greater than about 5 GPa and temperature of at least about 1,200 degrees centigrade in the presence of a sintering aid, also referred to as a catalyst material for diamond. Catalyst material for diamond is understood to be material that is capable of promoting direct inter-growth of diamond grains at a pressure and temperature condition at which diamond is thermodynamically more stable than graphite. Some catalyst materials for diamond may promote the conversion of diamond to graphite at ambient pressure, particularly at elevated temperatures. Examples of catalyst materials for diamond are

cobalt, iron, nickel and certain alloys including any of these. PCD may be formed on a cobalt-cemented tungsten carbide substrate, which may provide a source of cobalt catalyst material for the PCD. The interstices within PCD material may at least partly be filled with the catalyst material.

A well-known problem experienced with this type of PCD material, however, is that the residual presence of the catalyst material for diamond, in particular a metallic catalyst material for diamond, for example Co, Ni or Fe, in the interstices has a detrimental effect on the performance of the PCD material at high temperatures. During application, the PCD material heats up and thermally degrades, largely due to the presence of the metallic catalyst material that catalyses graphitisation of the diamond and also causes stresses in the PCD material due to the large difference in thermal expansion between the metallic catalyst material and the diamond microstructure.

One approach to addressing this problem is to remove, typically by leaching, the catalyst material, also referred to as a catalyst/solvent in the art, from the PCD material.

US 3,745,623 and US 4,636,253 teach the use of heated acid mixtures in the leaching process in which mixtures of HF, HCl, and HNO_3 and HNO_3 and HF, respectively, are used.

US 4,288,248 and US 4,224,380 describe removal of the catalyst/solvent by leaching the PCD tables in a hot medium comprising HNO_3-HF (nitric acid and hydrofluoric acid), alone or in combination with a second hot medium comprising HCl-HNO_3 (hydrochloric acid and nitric acid).

US 2007/0169419 describes a method of leaching a portion or all of the catalyst/solvent from a PCD table by shielding the portion of the PCD table not to be leached and immersing the shielded PCD table in corrosive solution to dissolve the catalyst/solvent in water and aqua regia. The leaching process is

accelerated by the use of sonic energy, which agitates the interface between the PCD table and the corrosive solution to accelerate the dissolution rate of the catalyst/solvent.

US 4,572,722 discloses a leaching process that is accelerated by forming a hole in the PCD table by laser cutting or spark emission prior to or during the leaching process. The PCD table is then leached by using conventional acid leaching techniques, electrolytic leaching and liquid zinc extraction.

An alternative approach to addressing the problem is to use a non-metallic catalyst material for diamond that produces a more thermally stable PCD material.

JP2795738 (B2) describes sintering a mixture of diamond powder and metal carbonates at pressures of 6-12 GPa and temperatures of 1700-2500°C to give sintered polycrystalline material consisting of 0.1-15 vol% non-metallic binder in a sintered diamond layer.

JP4114966 describes the use of carbon powder added as a sintering aid to diamond powder and an alkali earth carbonate, in order to improve the sinterability of the non-metallic system.

JP2003226578 also addresses the problem of poor sinterability, which describes the use of oxalic acid dihydrate as a sintering aid in a carbonate-based non-metallic solvent/catalyst system.

JP2002187775 describes the addition of other organic compounds to achieve a sintered carbonate-based non-metallic PCD, and similarly the addition of metal carbides is described in JP6009271.

Summary

In general terms, this disclosure relates to comprises a polycrystalline diamond material having a non-metallic catalyst material for diamond.

Viewed from a first aspect there is provided a polycrystalline diamond material comprising a mass of diamond particles or grains exhibiting inter-granular bonding and a binder material comprising a non-metallic catalyst material for diamond, the non-metallic catalyst material for diamond comprising at least one nitrogen compound derived from an ammonium compound and/or at least one halide compound.

The ammonium compound may comprise an anion selected from the group comprising the carbonates, phosphates, hydroxides, oxides, sulphates, borates, titanates, silicates, halides, and combinations thereof.

The halide compound may comprise a cation selected from the group comprising the alkali metals, alkali earth metals, transition metals, ammonium, and combinations thereof.

In some embodiments, the non-metallic catalyst material for diamond may comprise one or more of lithium chloride, sodium chloride, potassium chloride, rubidium chloride, caesium chloride, magnesium chloride, calcium chloride, strontium chloride, barium chloride, yttrium chloride, zirconium chloride, zinc chloride, niobium chloride, all oxidation states thereof, and mixtures thereof.

In some embodiments, the average particle size of the diamond particles or grains may be from about 5 nanometres to about 50 microns, or from about 20 nanometres to about 20 microns, or from about 50 nanometres to about 10 microns.

In some embodiments, the diamond content of the polycrystalline diamond material may be at least about 80 percent, at least about 88 percent, at least

about 90 percent, at least about 92 percent or even at least about 96 percent of the volume of the polycrystalline diamond material. In one or more embodiments, the diamond content of the polycrystalline diamond material may be at most about 98 percent of the volume of the polycrystalline diamond material.

The content of the non-metallic catalyst material for diamond may, for example, be at most about 20 volume percent, at most about 10 volume percent, at most about 8 volume percent, or even at most about 4 volume percent of the PCD material.

Viewed from a further aspect there is provided a method for making polycrystalline diamond material, the method including providing a mass of diamond particles or grains, contacting the diamond particles or grains with a binder material comprising a non-metallic catalyst material for diamond, the non-metallic catalyst material for diamond comprising at least one ammonium compound and/or at least one halide compound, consolidating the diamond particles or grains and binder material to form a green body, and subjecting the green body to a temperature and pressure at which diamond is thermodynamically stable, sintering and forming polycrystalline diamond material.

In some embodiments, the salts may be combined with the diamond particles or grains via infiltration, mixing, milling, chemical vapour deposition, colloidal (sol-gel) deposition, atomic layer deposition, physical vapour deposition, and the like.

In some embodiments, the diamond particles or grains and the binder material may be mixed in powder form with appropriate binding aids.

The diamond particles or grains may be suspended in a liquid medium, the non-metallic catalyst material for diamond precipitating *in situ* onto the

surfaces of respective diamond particles or grains in the liquid medium in order to coat the diamond particles or grains.

In some embodiments, the diamond particles or grains prior to contact with the binder material may have an average particle or grain size of from about 5 nanometres to about 50 microns, or from about 20 nanometres to about 20 microns, or from about 50 nanometres to about 10 microns.

In some embodiments, a multimodal mixture of diamond particles or grains of varying average particle or grain size may be provided.

The polycrystalline diamond material may be a stand-alone compact or may be attached to a substrate, such as a metal carbide substrate, for example.

Sintering may be carried out at pressures of 4 GPa or more, or 7 GPa or more, and temperatures of 1000°C or more, or 1700°C or more, for sintering times of 10 minutes or longer, or sintering times of 30 seconds or longer, or one minute or longer.

In some embodiments, sintering may be carried out at pressures of 7 GPa or less and temperatures of 1800°C or less.

According to another aspect, there is provided a wear element comprising a polycrystalline diamond material as defined above.

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Enhanced thermal stability of the polycrystalline diamond material over conventional metal catalysed polycrystalline material and lower sintering temperatures and pressures than for other non-metallic catalyst materials for diamond may be obtained through one or more embodiments.

Detailed description of embodiments

As used herein, "polycrystalline diamond" (PCD) material comprises a mass of diamond grains, a substantial portion of which are directly inter-bonded with each other and in which the content of diamond is at least about 80 volume percent of the material. In one embodiment of PCD material, interstices between the diamond grains may at least partly be filled with a binder material comprising a non-metallic catalyst for diamond.

As used herein, "non-metallic catalyst material for diamond" is a material that is capable of catalysing intergrowth of polycrystalline diamond particles or grains under conditions of temperature and pressure at which diamond is more thermodynamically stable than graphite.

As used herein, "interstices" or "interstitial regions" are regions between the diamond grains of PCD material.

A multi-modal size distribution of a mass of grains is understood to mean that the grains have a size distribution with more than one peak, each peak corresponding to a respective "mode". Multimodal polycrystalline bodies are typically made by providing more than one source of a plurality of grains, each source comprising grains having a substantially different average size, and blending together the grains or particles from the sources. Measurement of the size distribution of the blended grains typically reveals distinct peaks corresponding to distinct modes. When the grains are sintered together to form the polycrystalline body, their size distribution is further altered as the grains are compacted against one another and fractured, resulting in the overall decrease in the sizes of the grains. Nevertheless, the multimodality of the grains is usually still clearly evident from image analysis of the sintered article.

As used herein, a green body is an article that is intended to be sintered or which has been partially sintered, but which has not yet been fully sintered to form an end product. It may generally be self-supporting and may have the general form of the intended finished article.

As used herein, a superhard wear element is an element comprising a superhard material and is for use in a wear application, such as degrading, boring into, cutting or machining a workpiece or body comprising a hard or abrasive material.

A polycrystalline diamond material according to some embodiments comprises diamond having increased thermal stability over conventional solvent/catalyst sintered diamond composite materials. In some embodiments, the polycrystalline diamond material includes a binder comprising a non-metallic catalyst material for diamond. The non-metallic catalyst material for diamond comprises at least one nitrogen compound derived from an ammonium compound and/or at least one halide containing compound.

A method for making polycrystalline diamond material, in some embodiments, includes contacting a mass of diamond particles or grains with a binder material comprising a non-metallic catalyst material for diamond. The non-metallic catalyst material for diamond is at least one ammonium compound and/or at least one halide compound.

The salts may be combined with diamond by, for example, infiltration, mixing, milling, chemical vapour deposition, colloidal (sol-gel) deposition, atomic layer deposition, physical vapour deposition and other similar processes that would be appreciated by those skilled in the art.

The non-metallic binder material may be combined with the diamond particles or grains in powder form. It can be mixed in a conventional mixing process

such as, for example, a planetary ball milling process, typically in the presence of a milling aid such as methanol, for example. Milling balls, such as Co-WC milling balls, may be used to mill the binder and diamond powders together. The binder and diamond mixture may then typically be dried at a temperature of 50 to 100 °C to remove the methanol and other volatile residues and then consolidated into a green body ready for sintering.

In an alternative embodiment, the non-metallic binder material may be combined with the diamond particles or grains in a sol-gel process. Diamond powder is suspended in a liquid under vigorous stirring to form a diamond suspension. The liquid is typically water although the person skilled in the art will appreciate that any appropriate liquid medium can be used. A first salt of the desired ammonium cation and/or halide anion may be chosen such that it is soluble in a solvent, but forms an insoluble salt with a chosen anion/cation, as the case may be, in the diamond suspension. A second salt of the desired anion/cation may be chosen such that it is soluble in a solvent, but the anion/cation forms an insoluble salt respectively with the ammonium cation and/or halide anion of the first salt.

The two salt containing solutions are added concomitantly drop wise to the diamond suspension such that an insoluble precipitate consisting of the non-metallic catalyst material for diamond forms on the surface of the respective diamond particles or grains.

The liquid containing the suspended diamond particles or grains is stirred during the drop wise addition. This stirring may be accomplished by a heater-stirrer and magnetic stirrer, or by an overhead stirrer, or by ultrasonication, or any other suitable method that is able effectively to disperse the diamond particles in the liquid.

The diamond powder with precipitated salt may be removed from suspension and dried at a temperature suitable for removing any residual suspension

medium or solvents that may be present. The drying temperature may typically be around 50 to 100°C, or a temperature that does not volatise the ammonium or halide compound. Alternatively, the diamond with precipitated salt may be dried under vacuum at a moderate temperature or room temperature. The diamond with precipitated salt may be stationary during drying, or may be agitated, tossed or moved in a way that increases the efficiency or rate of drying. The diamond particles and binder material are consolidated to form a green body.

Prior to contact with the binder material, the diamond particles may have an average particle size ranging from about 5 nanometres to about 50 microns.

The green body, once formed, is placed in a suitable container and introduced into a high pressure and high temperature press. Pressure and heat are applied in order to sinter the diamond particles together, typically at pressures of around 4 to 7 GPa or more and temperatures of around 1000 to 1700°C or more.

In some embodiments, the grain boundaries of the diamond particles or grains may contain reduced levels of contaminants that originate from residues of the starting salts, thereby enabling stronger diamond-diamond bonding and improved material properties. In the case of ammonium cations, the lower concentration of contaminants is expected because the ammonium cations will dissociate under sintering conditions to form hydrogen and nitrogen, which are liberated as gases.

In some embodiments, the sintered PCD may contain an amount of dissolved nitrogen or hydrogen gas. The hydrogen gas liberated during HPHT is expected to have the beneficial effect of helping to reduce the carbon monoxide or carbon dioxide intermediate to diamond, thereby enabling the use of lower pressures and temperatures.

In other embodiments, for example where halides are used, lower pressures and temperatures may also be used to sinter the PCD. For example, 7 GPa or less and 1800°C or less, as opposed to the more conventional 8 GPa or more and 2300°C or more for more conventional non-metallic catalyst systems, may be used. Although wishing not to be bound by theory, it is believed that the disruption of the C-O bonds by the chloride ion reduces the temperature at which the potassium carbonate becomes catalytically active.

In some embodiments where, for example, compounds containing an ammonium cation are used, the anion may be any one or more of the following: carbonates, phosphates, hydroxides, oxides, sulphates, borates, titanates, silicates, halides and the like.

In some embodiments where, for example, compounds containing halide anions are used, the cation may be any one or more of the following: alkali metals, alkali earth metals, and transition metals. Examples of such compounds may include lithium chloride, sodium chloride, potassium chloride, rubidium chloride, caesium chloride, magnesium chloride, calcium chloride, strontium chloride, barium chloride, yttrium chloride, zirconium chloride, zinc chloride, niobium chloride, all oxidation states thereof, and mixtures thereof.

In some embodiments, mixtures of ammonium and halide compounds may be used.

The diamond grain sizes in the sintered PCD may range from about 5 nanometres to about 50 microns, or from about 20 nanometres to about 20 microns, or from about 50 nanometres to about 10 microns. The diamond size distributions may be monomodal or multimodal.

The non-metallic PCD may be monolithic, or may be attached to a suitable substrate, for example a Co-WC substrate. The interface between the PCD and the substrate may be planar or non-planar.

The non-metallic PCD may be leached partly or fully, using any appropriate leaching process that would be understood by a person skilled in the art.

Examples

A number of embodiments are described in more detail with reference to the examples below, which are not intended to be limiting.

Example 1:

An approximate eutectic mixture of $CaCO_3$ and $Ca(OH)_2$ was mixed with NH_4Cl in the ratio of 0.4 moles $CaCO_3$ with 0.4 moles $Ca(OH)_2$ and 0.2 moles NH_4Cl. This binder mixture was mixed with diamond in a ratio of 4.5 g diamond to 0.5 g binder mixture. This combined mixture was densely packed into an air tight metal container suitable for HPHT processing. This container was then subjected to HPHT processing to temperatures above 1500ºC and pressures above 6.8 GPa and held for times ranging from 10 minutes to 60 minutes. It was expected that there would be an intergrown diamond compact after HPHT processing.

Example 2:

An equimolar mixture of $MgCO_3$ and $Mg(OH)_2$ (in the absence of phase diagrams in the available literature for this system, it was assumed that an equimolar mixture would be sufficiently close to an eutectic composition) was mixed with NH_4Cl in the ratio of 0.4 moles $MgCO_3$ with 0.4 moles $Mg(OH)_2$ and 0.2 moles NH_4Cl. This binder mixture was mixed with diamond in a ratio of 4.5 g diamond to 0.5 g binder mixture. This combined mixture was densely packed into an air tight metal container suitable for HPHT processing. This container was then subjected to HPHT processing to temperatures at 1500ºC and pressures above 6.8 GPa and held for times ranging from 10 minutes to

60 minutes. It was expected that there would be an intergrown diamond compact after HPHT processing.

Example 3

An approximate eutectic mixture of $CaCO_3$ and $Ca(OH)_2$ was mixed with NH_4Cl in the ratio of 0.4 moles $CaCO_3$ with 0.4 moles $Ca(OH)_2$ and 0.2 moles NH_4Cl. This binder mixture was mixed with diamond in a ratio of 9 g diamond to 1 g binder mixture. This combined mixture was densely packed into an air tight metal container suitable for HPHT processing. This container was then subjected to HPHT processing to the following temperatures: 1600ºC, 1800ºC, and 2000ºC and a pressure of 8 GPa and held for a time of 10 minutes. It was expected that there would be an intergrown diamond compact after HPHT processing under all of these conditions.

Example 4:

An equimolar mixture of $MgCO_3$ and NH_4 Oxalate was mixed as a binder with diamond in a ratio of 4.5 g diamond to 0.5 g binder mixture. This combined mixture was densely packed into an air tight metal container suitable for HPHT processing. This container was then subjected to HPHT processing to temperatures above 1500ºC and pressures above 6.8 GPa and held for times ranging from 10 minutes to 60 minutes. It was expected that there would be an intergrown diamond compact after HPHT processing.

Example 5:

NH_4 Oxalate was mixed as a binder with diamond in a ratio of 4.5 g diamond to 0.5 g binder mixture. This combined mixture was densely packed into an air tight metal container suitable for HPHT processing. This container was then subjected to HPHT processing to temperatures above 1500ºC and pressures above 6.8 GPa and held for times ranging from 10 minutes to 60 minutes. It

was expected that there would be an intergrown diamond compact after HPHT processing.

Example 6:

K_2CO_3 and KCl were dried at 50°C for 24 hours, then were planetary ball milled separately for 45 minutes at 90 rpm, then combined in a molar ratio of 70:30. This mix was combined with diamond powder of average particle size 10 micron in an amount of 5vol% mix to 95vol% diamond. Being very hygroscopic, the salt mix was dried between steps as well as stored when necessary in a vacuum oven. Practical difficulties with pressure generation were experienced, so that no sintering was achieved in the experiments. However, it is expected that sintering at greater than 1000°C and greater than 7 GPa for more than 5 minutes will cause sintering, with 1260°C, 7.7 GPa and 1 hour expected to result in well sintered non-metallic PCD with very good thermal stability and wear behaviour. These temperatures are unusually low for sintering PCD, and this benefit is thought to be due to the presence of the chloride ions which may destabilise the carbonate anions and increase their reactivity as a catalyst material for diamond.

Claims

1. A polycrystalline diamond material comprising a mass of diamond particles or grains exhibiting inter-granular bonding and a binder material comprising a non-metallic catalyst material for diamond, the non-metallic catalyst material for diamond comprising at least one nitrogen compound derived from an ammonium compound and/or at least one halide compound.

2. A polycrystalline diamond material according to claim 1, wherein the ammonium compound comprises an anion selected from the group comprising the carbonates, phosphates, hydroxides, oxides, sulphates, borates, titanates, silicates, halides, and combinations thereof.

3. A polycrystalline diamond material according to claim 1, wherein the halide compound comprises a cation selected from the group comprising the alkali metals, alkali earth metals, transition metals, ammonium, and combinations thereof.

4. A polycrystalline diamond material according to claim 3, wherein the non-metallic catalyst material for diamond comprises one or more of lithium chloride, sodium chloride, potassium chloride, rubidium chloride, caesium chloride, magnesium chloride, calcium chloride, strontium chloride, barium chloride, yttrium chloride, zirconium chloride, zinc chloride, niobium chloride, oxidation states thereof, and/or mixtures thereof.

5. A polycrystalline diamond material according to any one of the preceding claims, wherein the diamond particles or grains have an average particle or grain size of from about 5 nanometres to about 50 microns.

6. A polycrystalline diamond material according to any one of the preceding claims, wherein the diamond content of the polycrystalline diamond material is at least 80 percent and at most 98 percent of the volume of the polycrystalline diamond material.

7. A polycrystalline diamond material according to any one of the preceding claims, wherein the polycrystalline diamond material comprises at most 20 volume percent of the non-metallic catalyst material for diamond.

8. A method for making polycrystalline diamond material, the method including providing a mass of diamond particles or grains, contacting the diamond particles or grains with a binder material comprising a non-metallic catalyst material for diamond, the non-metallic catalyst material for diamond comprising at least one ammonium compound and/or at least one halide compound, consolidating the diamond particles or grains and binder material to form a green body, and subjecting the green body to a temperature and pressure at which diamond is thermodynamically stable, sintering and forming polycrystalline diamond material.

9. A method according to claim 8, wherein the ammonium compound comprises an anion selected from the group comprising the carbonates, phosphates, hydroxides, oxides, sulphates, borates, titanates, silicates, halides, and combinations thereof.

10. A method according to claim 8, wherein the halide compound comprises a cation selected from the group comprising the alkali metals, alkali earth metals, transition metals, ammonium, and combinations thereof.

11. A method according to claim 10, wherein the non-metallic catalyst material for diamond comprises any one or more of lithium chloride, sodium chloride, potassium chloride, rubidium chloride, caesium chloride, magnesium chloride, calcium chloride, strontium chloride,

barium chloride, yttrium chloride, zirconium chloride, zinc chloride, niobium chloride, all oxidation states thereof, and/or mixtures thereof.

12. A method according to any one of claims 8 to 11, wherein the method includes subjecting the green body in the presence of the non-metallic catalyst material for diamond to a pressure and temperature at which diamond is more thermodynamically stable than graphite.

13. A method according to claim 12, wherein the pressure is at least about 4 GPa and the temperature is at least about 1000°C.

14. A method according to claim 12, wherein the pressure is at most about 8 GPa and the temperature is at most about 2300°C.

15. A wear element comprising a polycrystalline diamond material according to any one of claims 1 to 7.