



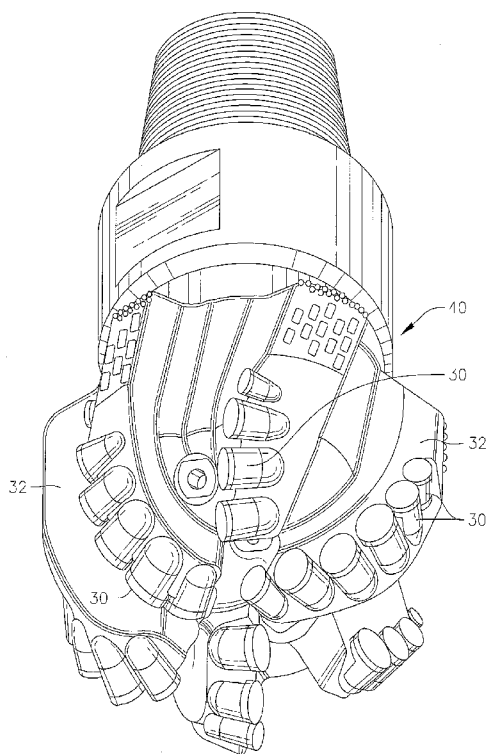
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[Continued on nextpage]

(54) **Title:** THERMALLY STABLE MATERIALS, CUTTER ELEMENTS WITH SUCH THERMALLY STABLE MATERIALS, AND METHODS OF FORMING THE SAME



(57) **Abstract:** A thermally stable ultra-hard material, a cutting element incorporating such thermally stable ultra-hard material, and methods for forming the same. A thermally stable ultra-hard diamond element is combined with a second ultra-hard material volume forming an assembly. One or more surfaces of the thermally stable diamond element that face the second diamond volume are coated with a coating prior to combining the thermally stable diamond element with the second diamond volume. The assembly is sintered at high pressure and high temperature to form PCD from the second diamond volume.

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THERMALLY STABLE MATERIALS, CUTTER ELEMENTS WITH SUCH
THERMALLY STABLE MATERIALS, AND METHODS OF FORMING THE SAME

BACKGROUND

5 [0001] The present invention relates to thermally stable ultra-hard materials, and more particularly to a cutting element incorporating a thermally stable ultra-hard material, and methods for forming the same. Ultra-hard materials are often used in cutting tools and rock drilling tools. Polycrystalline diamond material is one such ultra-hard material, and is known for its good wear resistance and hardness. To form polycrystalline diamond, diamond particles are sintered at high pressure and high temperature (HPHT sintering) to produce an ultra-hard polycrystalline structure. A catalyst material is added to the diamond particle mixture prior to sintering and/or infiltrates the diamond particle mixture during sintering in order to promote the intergrowth of the diamond crystals during HPHT sintering, to form the polycrystalline diamond (PCD) structure. Metals conventionally employed as the catalyst are selected from the group of solvent metal catalysts selected from Group VIII of the Periodic table, including cobalt, iron, and nickel, and combinations and alloys thereof. After HPHT sintering, the resulting PCD structure includes a network of interconnected diamond crystals or grains bonded to each other, with the catalyst material occupying the interstitial spaces or pores between the bonded diamond crystals. The diamond particle mixture may be HPHT sintered in the presence of a substrate, to form a PCD compact bonded to the substrate. The substrate may also act as a source of the metal catalyst that infiltrates into the diamond particle mixture during sintering.

15 [0002] The amount of catalyst material used to form the PCD body represents a compromise between desired properties of strength, toughness, and impact resistance versus hardness, wear resistance, and thermal stability. While a higher metal catalyst content increases the strength, toughness, and impact resistance of the resulting PCD body, this higher metal catalyst content also decreases the hardness and wear resistance as well as the thermal stability of the PCD body. This trade-off makes it difficult to provide PCD having desired levels of hardness, wear resistance, thermal stability, strength, impact resistance, and toughness to meet the service demands of particular applications, such as in cutting and/or wear elements used in subterranean drilling devices.

25 [0003] Thermal stability is desired during wear or cutting operations. Conventional PCD bodies may be vulnerable to thermal degradation when exposed to elevated temperatures during cutting and/or wear applications. This vulnerability results from the differential that exists between the thermal expansion characteristics of the metal catalyst disposed interstitially within the PCD body and the thermal expansion characteristics of the intercrystalline bonded diamond. This differential thermal expansion is known to start at temperatures as low as 400 °C, and can induce thermal stresses that are detrimental to the

1 intercrystalline bonding of diamond and that eventually result in the formation of cracks that
can make the PCD structure vulnerable to failure. Accordingly, such behavior is not
desirable.

5 [0004] Another form of thermal degradation known to exist with conventional PCD
materials is one that is also related to the presence of the metal catalyst in the interstitial
regions of the PCD body and the adherence of the metal catalyst to the diamond crystals.
Specifically, the metal catalyst is known to cause an undesired catalyzed phase
transformation in diamond (converting it to carbon monoxide, carbon dioxide, or graphite)
with increasing temperature, thereby limiting the temperatures at which the PCD body may
10 be used.

15 [0005] To improve the thermal stability of the PCD material, the catalyst material may be
removed from the PCD body after sintering, to form thermally stable PCD. This thermally
stable PCD material (referred to as TSP) is formed by first HPHT sintering diamond particles
in the presence of a metal catalyst, forming a PCD body with the catalyst occupying the
interstitial regions between the diamond crystals. Then, the catalyst material is removed from
the PCD body, leaving a network of empty interstitial spaces between the diamond crystals.
For example, one known approach is to remove a substantial portion of the catalyst material
from at least a portion of the sintered PCD by subjecting the sintered PCD construction to an
acid leaching process, such as that disclosed for example in U.S. Patent No, 4,224,380.
20 Applying the leaching process to the PCD results in a thermally stable material portion
substantially free of the catalyst material. If a substrate was used during the HPHT sintering,
it is generally removed from the PCD body prior to leaching.

25 [0006] As another way to improve the thermal stability of the PCD material, a carbonate
catalyst has been used to form the PCD. The carbonate catalyst is mixed with the diamond
powder prior to sintering, and promotes the growth of diamond grains during sintering.
When a carbonate catalyst is used, the diamond remains stable in polycrystalline diamond
form with increasing temperature, rather than being converted to carbon dioxide, carbon
monoxide, or graphite. Thus the carbonate PCD is more thermally stable than PCD formed
with a metal catalyst.

30 [0007] As another way to provide a more thermally stable ultra-hard diamond, diamond
bodies with high diamond content have been provided, by reducing the amount of catalyst
material. Additionally, binderless polycrystalline diamond has been formed, without the use
of a catalyst material. The resulting diamond material has a uniform intercrystalline diamond
microstructure, without catalyst material interspersed between the diamond crystals. As a
35 result, the binderless diamond body does not suffer from differential thermal expansion
between diamond and catalyst.

 [0008] However, while thermally stable diamond with a reduced amount of catalyst has
high hardness and wear-resistance at elevated temperature, it is difficult to incorporate into a

1 cutting element for use in a drilling or cutting tool. For example, binderless diamond
material may be formed in small quantities, due to the specialized processes used in its
formation. Bonding these small pieces of thermally stable diamond to other components of a
cutting element can cause cracking, which can lead to early failure of the cutting element.

5 SUMMARY

[0009] This summary is provided to introduce a selection of concepts that are further
described below in the detailed description. This summary is not intended to identify key or
essential features of the claimed subject matter, nor is it intended to be used as an aid in
10 limiting the scope of the claimed subject matter.

[0010] The present disclosure relates to thermally stable ultra-hard materials, and more
particularly to a cutting element incorporating a thermally stable ultra-hard material, and
methods for forming the same. In one embodiment, a thermally stable ultra-hard diamond
element is provided, and is combined with a second ultra-hard material volume, such as a
15 volume of diamond powder or an unsintered or pre-sintered diamond part. At least a surface
of the thermally stable diamond element that faces the second diamond volume are coated
with a coating prior to combining the thermally stable diamond element with the second
diamond volume. The assembly is then sintered at high pressure and high temperature to
form PCD from the second diamond volume. During sintering, the coating at the interface
20 between the thermally stable element and the second diamond volume melts. The melted
coating acts as a slip plane between the two elements, allowing relative movement at the
interface. This effect enables the second ultra-hard material volume to densify during
sintering, shrinking relative to the more fully dense thermally stable ultra-hard element. As a
result, the coating reduces the stresses along the interface and reduces cracking in the cutting
25 element.

[0011] In one embodiment, a method for making an ultra-hard construction includes
applying a coating on a surface of a thermally stable ultra-hard element. The thermally stable
element has a theoretical density of at least 98% and a diamond volume content of at least
90%. The method also includes combining the thermally stable ultra-hard element with a
30 second ultra-hard material volume to form an assembly. The coated surface of the thermally
stable element faces the second ultra-hard material volume. The method also includes
sintering the assembly at high pressure and high temperature. The thermally stable ultra-hard
element is bonded to the sintered ultra-hard element to form an ultra-hard body having a
working surface and/or a cutting edge. The thermally stable ultra-hard element forms at least
35 a portion of the working surface or the cutting edge of the ultra-hard body. In one
embodiment the thermally stable element has a theoretical density of at least 99%. In one
embodiment the thermally stable element has a diamond volume content of at least 98%, and
in another embodiment at least 99%.

BRIEF DESCRIPTION OF THE DRAWINGS

[0012] Embodiments of **THERMALLY** STABLE MATERIALS, CUTTER ELEMENTS WITH SUCH THERMALLY STABLE MATERIALS, AND METHODS OF **FORMING** THE SAME are described with reference to the following figures.

[0013] Figure 1A illustrates a schematic view of a material microstructure of a thermally stable diamond material according to an embodiment (with dimensions exaggerated and not to scale, for clarity).

[0014] Figure 1B illustrates a schematic view of a material microstructure of a thermally stable diamond material according to an embodiment (with dimensions exaggerated and not to scale, for clarity).

[0015] Figure 1C illustrates a schematic view of a material microstructure of a **thermally** stable diamond material according to an embodiment (with dimensions exaggerated and not to scale, for clarity).

[0016] Figure 2A illustrates a perspective view of a diamond construction incorporating a thermally stable diamond element, according to an embodiment.

[0017] Figure 2B illustrates a perspective view of a diamond construction incorporating a thermally stable diamond element, according to an embodiment.

[0018] Figure 2C illustrates a cross-sectional view of a diamond construction incorporating one or more thermally stable diamond elements, according to an embodiment.

[0019] Figure 3A illustrates a side view of an assembly including a thermally stable diamond element and a coating, prior to sintering of the assembly.

[0020] Figure 3B illustrates a side view of the assembly of Figure 3A after sintering.

[0021] Figure 4 illustrates a flowchart illustrating a method of forming a diamond construction incorporating a thermally stable diamond element, according to an embodiment.

[0022] Figure 5 illustrates a perspective view of a drag bit incorporating the cutting element of Figure 3B.

DETAILED DESCRIPTION

[0023] The present disclosure relates to thermally stable ultra-hard materials, and more particularly to a cutting element incorporating a thermally stable ultra-hard material, and methods for forming the same. In one embodiment, a thermally stable ultra-hard diamond element is provided, and is combined with a second ultra-hard material volume, such as a volume of diamond powder or an unsintered or pre-sintered diamond part. One or more surfaces of the thermally stable diamond element that face the second diamond volume are coated with a coating prior to combining the thermally stable diamond element with the second diamond volume. The assembly is then sintered at high pressure and high temperature to form PCD from the second diamond volume. During sintering, the coating at the interface between the thermally stable element and the second diamond volume melts.

1 The melted coating acts as a slip plane between the two elements, allowing relative movement at the interface. This effect enables the second ultra-hard material volume to densify during sintering, shrinking relative to the more fully dense thermally stable ultra-hard element. As a result, the coating reduces the stresses along the interface and reduces cracking
5 in the cutting element.

[0024] For clarity, as used herein, the term "PCD" refers to conventional polycrystalline diamond that has been formed with the use of a metal catalyst during an HPHT sintering process, forming a microstructure of bonded diamond crystals with the catalyst material occupying the interstitial spaces or pores between the bonded diamond crystals. "Thermally
10 stable PCD" (or "TSP") refers to PCD material which has been subsequently treated to substantially remove the catalyst material from at least a portion of the PCD body, such as by leaching the entire or part of the PCD body after HPHT sintering, to remove the catalyst material from the interstitial regions of the PCD body. "Binderless" diamond refers to a polycrystalline diamond matrix that is formed without the use of a metal catalyst, such as by
15 converting graphite directly to diamond at ultra-high pressure and temperatures. This material is not referred to as PCD, in order to clarify that it does not utilize a metal catalyst, and to distinguish it from thermally stable PCD (TSP). This binderless diamond material may have a diamond volume content of at least 99%. Finally, "carbonate PCD" refers to PCD formed in the presence of a carbonate catalyst.

[0025] Embodiments of the present disclosure are directed to thermally stable ultra-hard materials with a theoretical density of at least 98% and a diamond volume content of at least 90%. This includes both TSP that has a diamond volume content of at least 98%, carbonate PCD with a diamond volume content of 90-95%, as well as binderless diamond material. These materials are referred to herein as "thermally stable diamond materials" or a "thermally
25 stable diamond element."

[0026] As described below, embodiments herein provide a thermally stable diamond material with a theoretical density of at least 98% and a diamond volume content of at least 90%. In one example, this thermally stable diamond material is a binderless diamond, formed through a process that does not utilize a metal catalyst material. A region of a
30 binderless thermally stable diamond material 10A is schematically illustrated in Figure 1A, according to an embodiment of the present disclosure. The thermally stable, binderless diamond material 10A has a polycrystalline microstructure including multiple diamond grains or crystals 12 bonded to each other. As shown in Figure 1A, this material microstructure is substantially devoid of gaps or interstitial spaces between the diamond crystals 12. The
35 diamond crystals 12 are bonded directly to each other. The binderless diamond material 10A is essentially pure carbon, with a diamond volume fraction of substantially 100%. There is substantially no binder phase or catalyst material between the diamond crystals 12.

1 [0027] This material is described as "substantially" devoid of gaps and interstitial spaces,
and "substantially" 100% diamond, in order to allow for the possibility of small imperfections
and deviations within the binderless diamond material 10A which may leave small gaps or
spaces between some of the diamond crystals. In one embodiment the material
5 microstructure of the binderless thermally stable diamond material has a diamond volume
content of at least 98%, and in another embodiment at least or about 99%, and in another
embodiment at least or about 99.5%, and in another embodiment at least or about 99.8%, and
in another embodiment at least or about 99.9%. In one embodiment the thermally stable
diamond material 10A has a fine diamond grain size, such as an average diamond grain size
10 less than 1 micron, such as about 50 nm or less. In other embodiments the thermally stable
diamond material has an average grain size of about 1-10 microns.

[0028] To form a binderless diamond material such as the material 10A shown in Figure
1A, carbon, in the form of graphite, buckeyballs, or other carbon structures, is subjected to an
ultra-high HPHT sintering process without a catalyst material. In one embodiment, this
15 process includes HPHT sintering at ultra-high temperature and pressure, above that applied
during conventional HPHT sintering to form PCD. In one embodiment, the pressure is
between about 100-160 kbar, such as about 150 kbar, and the temperature is about 1600-2500
°C. For example, when sintering graphite, the pressure may be about 150 kbar, or about 150-
160 kbar. When sintering other types of carbon, such as buckeyballs or other complex
20 carbon structures, the pressure may be about 110-120 kbar. For reference, conventional
HPHT sintering to form PCD may be performed at about 50-60 kbar. In another
embodiment, the temperature is about 2200-2300 °C.

[0029] The method phase transforms the graphite (or other form of carbon) into
polycrystalline diamond. That is, during the HPHT sintering process, the graphite converts
25 into polycrystalline diamond, without the assistance of a catalyst material. Once the HPHT
sintering is complete, the result is a thermally stable polycrystalline diamond matrix
including bonded together diamond crystals substantially devoid of interstitial spaces, as
discussed above.

[0030] In another embodiment, the thermally stable binderless diamond material 10A is
30 formed by depositing layers in a chemical vapor deposition process, to form a thermally
stable diamond element with substantially 100% diamond volume content.

[0031] The binderless diamond material 10A is inherently thermally stable, due to its
uniform diamond content. The binderless diamond material 10A is one phase, and thus there
is no differential thermal expansion between different phases of the material. As a result,
35 diamond bodies formed from this thermally stable diamond material 10A can exhibit very-
high strength even at elevated temperatures, where conventional PCD suffers from thermal
degradation due to the differential expansion of the diamond and catalyst phases.

[0032] In another embodiment, a TSP material is provided as the thermally stable diamond material with a theoretical density of at least 98%. The TSP is made up of diamond and substantially empty interstitial spaces, providing a diamond volume content of at least 98%, and in another embodiment at least 99%. A region of a TSP material 10B having a diamond volume content of at least 98% is schematically illustrated in Figure 1B. The TSP material 10B has a polycrystalline microstructure including multiple diamond grains or crystals 14 bonded to each other, with interstitial spaces or pores 16 between the diamond crystals. This polycrystalline microstructure is formed by first forming PCD and then removing the catalyst material from the interstitial spaces 16. That is, diamond powder is subjected at an HPHT sintering process in the presence of a metal catalyst. In one embodiment, the HPHT sintering process includes applying a pressure of about 50 kbar or greater, and a temperature of about 1,350 to 1,550 °C. At this temperature and pressure, the catalyst material melts and infiltrates into the diamond powder mixture. The catalyst promotes the growth of diamond crystals during the HPHT sintering process, forming PCD. The result is a PCD material with the catalyst material occupying the interstitial spaces 16 between the diamond crystals 14. In one embodiment, the diamond grains in the PCD material are about 1-20 microns in size.

[0033] Subsequently, this catalyst material is removed, such as by subjecting the PCD material to a chemical treatment, such as acid leaching, aqua regia bath, electrochemical treatment, or liquid metal infiltration, or combinations of these. After removing the catalyst, the result is a TSP material 10B with interstitial spaces 16 that are substantially empty, as shown in Figure 1B. This TSP material 10B has a diamond volume fraction of at least 98%, and in another embodiment at least 99%. In another embodiment, a TSP material with a diamond volume fraction of at least 97% may be used.

[0034] The term "removed", as used with reference to the catalyst material removed from TSP, is understood to mean that a substantial portion of the catalyst material no longer resides within the treated region of the diamond body. However, it is to be understood that some small amount of catalyst material may still remain in the part, e.g., within the interstitial regions and/or adhered to the surface of the diamond crystals. Additionally, the term "substantially free", as used herein to refer to the catalyst material in the treated region of the diamond body, is understood to mean that there may still be some small/trace amount of catalyst material remaining within the treated diamond body as noted above.

[0035] In another embodiment, a carbonate PCD material is provided as the thermally stable diamond material with a theoretical density of at least 98% and a diamond volume content of at least 90%. In one embodiment the carbonate PCD is made up of about 90-95% diamond (by volume), as well as carbonate catalyst material, providing a total theoretical density of at least 98%, and in another embodiment at least 99%. A region of a carbonate PCD material 10C having a theoretical density of at least 98% is schematically illustrated in

Figure 1C. The carbonate PCD material IOC has a polycrystalline microstructure including multiple diamond grains or crystals 18 bonded to each other, with interstitial spaces or pores between the diamond crystals. This polycrystalline microstructure is formed by subjecting a diamond powder to an HPHT sintering process in the presence of a carbonate catalyst. In one embodiment, the HPHT sintering process includes applying a pressure of about 65 kbar or greater, and a temperature of about 2000 to 2500 °C. At this temperature and pressure, the carbonate catalyst material melts and infiltrates into the diamond powder mixture. The catalyst promotes the growth of diamond crystals during the HPHT sintering process, forming carbonate PCD. The result is a carbonate PCD material with the carbonate catalyst material 19 occupying the interstitial spaces between the diamond crystals 18. In one embodiment, the diamond grains 18 in the carbonate PCD material are about 1-20 microns in size, and provide a diamond volume content of at least 90%. In another embodiment the carbonate PCD has a diamond volume content of at least 95%. In one embodiment, the carbonate catalyst material is magnesium carbonate (MgCO_3)

[0036] The diamond materials 10A, 10B, and IOC are thermally stable diamond materials having a theoretical density of at least 98%, or higher, and a diamond volume content of at least 90%, or in another embodiment at least 95%. These materials show improved thermal stability as compared to conventional PCD and other forms of diamond with lower diamond volume content or lower theoretical density. In one embodiment, the thermally stable diamond element is expected to be thermally stable above 700 °C, and in another embodiment above 750 °C, and maintains high strength at elevated temperatures (for example, a strength of about 3.0 GPa above 1000 °C). In one embodiment, the thermally stable diamond matrix is expected to exhibit a hardness of about 4000 HV or greater.

[0037] In certain embodiments, to form the diamond materials 10A, 10B, or IOC, high temperatures and pressure are applied. Due to the high temperature and pressure used to form these thermally stable diamond materials, the materials may be formed in small quantities. The amount of space within a central pressure cell of a high-pressure press is limited, and in some cases just a small volume of material can be HPHT sintered in the press to form the thermally stable diamond material. It is then desirable to incorporate the small volume of thermally stable diamond material into a larger ultra-hard construction, such as a diamond construction, for use in a cutting tool.

[0038] According to an embodiment of the present disclosure, a method for forming an ultra-hard construction incorporating a thermally stable ultra-hard element is provided. In one embodiment, the method includes forming a diamond construction incorporating a thermally stable diamond element. In this method, the thermally stable diamond material (binderless diamond, carbonate PCD, or TSP, with a theoretical density of at least 98% and a diamond volume content of at least 90%) is formed, cut, or machined into a desired shape, forming a thermally stable diamond element. The thermally stable diamond element is then

1 combined with a second ultra-hard material volume, such as a diamond volume, into a
combined ultra-hard assembly. This combined assembly is subjected to a second HPHT
sintering process to form a sintered ultra-hard body that incorporates the thermally stable
ultra-hard element. Optionally, the combined assembly also includes a substrate, such that
5 the ultra-hard body is bonded to the substrate during sintering. In one embodiment, the
combined assembly includes a diamond body incorporating a thermally stable diamond
element.

[0039] The thermally stable diamond element is combined with the second diamond
volume in order to incorporate the thermally stable diamond element into a sintered diamond
10 body that can be utilized in a cutting tool.

[0040] According to an embodiment of the disclosure, an ultra-hard assembly 20 prior to
sintering is shown in Figure 3A. The ultra-hard assembly 20 includes a thermally stable
ultra-hard element 22, a second ultra-hard material volume 24, and a substrate 26. In this
embodiment, each of these components is cylindrical in shape, stacked together with the
15 second ultra-hard material volume 24 interposed between the thermally stable ultra-hard
element 22 and the substrate 26. In one embodiment, the ultra-hard assembly 20 is a
diamond assembly, including a thermally stable diamond element 22, a second diamond
volume 24, and a substrate 26.

[0041] In an embodiment, the thermally stable diamond element 22 is a TSP material,
20 carbonate PCD material, or binderless diamond material with a theoretical density of at least
98%, as described above. The second diamond volume 24 is a volume of diamond that is not
fully densified. In one embodiment, the second diamond volume is a volume of diamond
powder. Optionally, the diamond powder may include a catalyst material mixed with the
diamond powder, to promote the formation of PCD from the diamond powder during
25 sintering. In other embodiments, the second diamond volume may be an unsintered green-
state part or volume, or a pre-sintered diamond body that is not fully densified. Prior to
sintering, the second diamond volume includes diamond that has not yet been formed into a
polycrystalline diamond structure. During sintering, this second diamond volume is
converted into PCD.

30 [0042] The substrate 26 is useful for attaching the diamond body to a cutting tool. The
substrate may also provide a source of a solvent metal catalyst for HPHT sintering. The
substrate can be selected from the group including metallic materials, ceramic materials,
cermet materials, and combinations thereof. Examples include carbides such as WC, W₂C,
TiC, VC, and SiC. In one embodiment, the substrate is formed of cemented tungsten carbide.

35 [0043] As discussed above, the thermally stable element may be formed during a first
sintering process. The second diamond volume 24 and the comparatively dense thermally
stable element 22 are both sintered together during a second sintering process, to incorporate
them together into one sintered diamond body. However, the second diamond volume 24 and

1 the thermally stable diamond element 22 undergo different amounts of densification during
this second HPHT sintering process. The thermally stable diamond element is already
substantially fully dense, having already undergone an HPHT sintering process to provide a
theoretical density of 98% or higher. This element is substantially fully dense and does not
5 undergo substantial additional densification during the second HPHT sintering process. By
contrast, the second diamond volume 24 forming the remainder of the diamond body, such as
a loose diamond powder, is not fully dense, and it undergoes major densification during the
second HPHT sintering process. As a result, the second diamond volume 24 experiences
shrinkage relative to the thermally stable element 22.

10 [0044] During sintering, the surface of the thermally stable element may constrain the
shrinkage of the second diamond volume, making it difficult to achieve full densification of
the second diamond volume at the interface with the thermally stable element. This can
introduce stresses in the resulting diamond body at the interface between the thermally stable
diamond element 22 and the PCD element formed from the second diamond volume 24.
15 These stresses in the material can later cause cracks to form, leading to early failure of the
diamond body.

[0045] According to an embodiment of the present disclosure, a coating 28 is provided
between the thermally stable ultra-hard element 22 (such as the thermally stable diamond
element 22) and the second ultra-hard material volume 24 (such as the second diamond
20 volume 24) prior to the second HPHT sintering process. As the heat and pressure are applied
during the second HPHT sintering process, the coating 28 melts into a liquid form. The
liquid coating forms a slip plane between the thermally stable diamond element 22 and the
second diamond volume 24, allowing the materials to shift relative to each other along the
liquid plane. The materials can move relative to each other to accommodate shrinkage of the
25 second diamond volume relative to the thermally stable element as the second diamond
volume densities. This freedom of relative movement during sintering reduces the stresses in
the final diamond body. The assembly is shown after sintering as the sintered diamond
construction 50 in Figure 3B. After sintering, the second diamond volume 24' shifted along
the slip plane created by the coating, and exhibits shrinkage with respect to the thermally
30 stable diamond element 22. The second diamond volume 24' is indicated with a prime (')
indication in Figure 3B to show that it is now post-sintering. After sintering, the second
diamond volume has been formed into a PCD element 24'. The PCD element 24' is bonded
to both the thermally stable diamond element 22 and the substrate 26. In one embodiment,
the diamond grains in the PCD element 24' are about 1-40 microns in size.

35 [0046] Referring again to Figure 3A, the coating 28 is formed on the surface of the
thermally stable diamond element 22 that faces the second diamond volume 24. During the
sintering process, the coating 28 melts and liquefies. The melted, liquid coating spreads
between the two diamond volumes and wets the two facing surfaces at the interface between

1 them (see interface 66A in Figure 2A, below). The coating is chosen from a material that has good wetting properties for wetting diamond, such as, for example, cobalt.

5 [0047] In this liquid form, a portion of the coating may move into the second diamond volume 24, as the second diamond volume is not yet fully dense. As a result, the material for the coating is chosen to be a material that is compatible with the formation of PCD in the second diamond volume 24. In one embodiment, the coating is cobalt, and in one embodiment, pure cobalt, without any additives or mixtures. The coating may be any solvent metal catalyst selected from Group VIII of the Periodic table, including cobalt, iron, nickel, combinations, and alloys thereof. In one embodiment the coating is chosen from the group of
10 cobalt, nickel, and copper. In one embodiment, the coating is devoid of refractory metals (such as niobium or molybdenum), in order to minimize the formation of carbide in the PCD body 24'.

15 [0048] The coating 28 is separate from a catalyst that may also be provided to assist in sintering the second diamond volume 24. In one embodiment, a metal catalyst infiltrates into the second diamond volume 24 during sintering, such as from the substrate, and/or is provided and mixed within the second diamond volume 24 prior to sintering. This catalyst material melts and moves through the second diamond volume 24 during sintering, reaching the top of the second diamond volume at the interface with the coating 28. This catalyst material promotes the formation of PCD in the second diamond volume. In an embodiment,
20 the coating 28 is chosen to have a melting temperature that is higher than the melting temperature of the catalyst. Thus, as heat is applied, the catalyst melts first and moves through the second diamond volume. The second diamond volume is then occupied by the melted catalyst, which prevents the coating from moving down into the second diamond volume when the coating melts. The prior-melted catalyst displaces the melted coating 28 and prevents the melted coating 28 from moving through the second diamond volume. In this
25 case, the melted, liquid coating remains substantially in place between the two diamond volumes, which is beneficial for accomplishing its slip plane function.

30 [0049] In one embodiment, the coating 28 has a melting point that is higher or slightly-higher than the melting point of the catalyst material that is used to promote the formation of PCD in the second diamond volume 24. As a result, the catalyst melts and begins to move through the second diamond volume before the coating melts. This difference in melting temperature can be obtained even when the coating and the catalyst are both cobalt, as the catalyst material is not pure cobalt. For example, when the catalyst is cobalt infiltrated from a tungsten carbide cobalt (WC-Co) substrate, the catalyst is a eutectic mix of cobalt, tungsten, and carbon, which has a melting point of about 1300 °C. This melting point is lower than the
35 melting point of pure cobalt, which is about 1500 °C. Thus, the catalyst melts before the coating melts. It is noted that during sintering, the cobalt coating will actually melt at a

1 temperature lower than 1500 °C, because the coating will become mixed with the catalyst
once the catalyst melts and infiltrates. Thus, sintering may still take place below 1500 °C.

[0050] In one embodiment, the melted coating 28 may partially move into the second
diamond volume 24 during sintering, but does not infiltrate the thermally stable element 22,
5 As discussed above, the thermally stable diamond element 22 is already fully dense, with few
or no interstitial spaces between the diamond crystals, leaving little or no room for the
coating to flow into the thermally stable element. The coating material diffuses in
substantially one direction, away from the thermally stable diamond element and into the
second diamond volume, or stays substantially in place between the two diamond volumes,
10 This behavior helps to maintain the thermal stability of the thermally stable element, as the
coating material does not infiltrate into the thermally stable element and does not create a
two-phase structure which could suffer from differential thermal expansion.

[0051] During sintering, the coating melts and wets the surface of the thermally stable
diamond element, spreading out along the surfaces between the two facing diamond volumes,
15 After the sintering process, when the diamond construction is cooled, the coating cools and
provides a strong bond between the two diamond volumes along their facing surfaces. This
bond is formed by the melting and cooling of the coating material, and does not rely on
infiltration of the coating material into either diamond volume. In one embodiment, the
coating remains substantially in place along the surfaces between the two diamond volumes,
20 and does not infiltrate or penetrate substantially into either diamond volume. In one
embodiment, the coating penetrates slightly into the second diamond volume. Additionally,
in one embodiment, use of a solvent catalyst material such as cobalt, nickel, iron, or mixtures
thereof as the coating may also promote some diamond bonding between the first and second
diamond volumes, depending on sintering conditions and the degree of penetration of the
25 coating into the second diamond volume.

[0052] When the sintering is complete, the sintered diamond body includes a region
formed from the second diamond volume and a region formed from the thermally stable
diamond element. The region formed from the second diamond volume has a substantially
higher metal catalyst content than the region formed from the thermally stable element.
30 Optionally, if a small or trace amount of the coating material has infiltrated into the thermally
stable element, it may be removed by leaching.

[0053] The coating 28 may be applied by a sputter deposition process onto the surface or
surfaces of the thermally stable diamond element 22 facing the second diamond volume 24.
In one embodiment, the coating 28 is approximately 50-100 microns thick, prior to the
35 sintering process. In one embodiment, rather than coating the thermally stable element, a
separate disc of material may be inserted between the thermally stable element and the
second diamond volume. In the case where the thermally stable element is a small piece fully
submerged into the second diamond volume, the thermally stable element may be coated in

1 three dimensions on each outside surface of the thermally stable diamond element facing the second diamond volume. In another embodiment, the coating may be applied to the second diamond volume, such as when the second diamond volume is provided in a partially compacted disc or tape.

5 [0054] In one embodiment, the coating 28 is selected to have a melting point above the melting point of the solvent catalyst alloy, but below the maximum temperature reached during sintering. In one embodiment, the coating is not highly reactive (an example of a highly reactive material is an alkaline earth metal). In one embodiment, the coating is not a strong carbide former. In addition to cobalt and nickel, other materials expected to be
10 suitable as a coating include palladium and the lanthanide series of rare earth elements (such as gadolinium, terbium, dysprosium, holmium, erbium, lutecium, and thulium, although the maximum sintering temperature may be needed to be raised for several of these elements). Coating may be applied with any number of processes such as but not limited to electrochemical plating, electroless coating, sputtering, physical vapor deposition, or
15 chemical vapor deposition. The melting points of these materials vary from 1300-1600 °C.

[0055] A separate coating or an intermediate layer or disc may be included between the second diamond volume 24 and the substrate. 26. Such a layer is optional, and is separate from the coating 28. The coating 28 is applied between the two diamond volumes that form the sintered diamond body.

20 [0056] Figure 4 shows a flowchart illustrating a method of forming a diamond construction incorporating a thermally stable diamond element, according to an embodiment. In one embodiment, the method includes providing a thermally stable diamond element having a theoretical density of at least 98% and a diamond volume content of at least 90% (block S01), and coating a surface of the thermally stable diamond element (block 102). The
25 method then includes combining the thermally stable diamond element with a second diamond volume to form an assembly (block 103). The coated surface of the thermally stable element faces the second diamond volume. The method then includes sintering the assembly at high pressure and high temperature to form PCD (block 104). The PCD is a polycrystalline diamond element formed from the second diamond volume. In one
30 embodiment, sintering includes applying a pressure of about 50-60 kbar and a temperature of about 1,350 to 1,550 °C. After sintering, the thermally stable diamond element is bonded to the polycrystalline diamond element to form a diamond body. The thermally stable diamond element forms at least a portion of the working surface and/or the cutting edge of the diamond body.

35 [0057] The above method forms a diamond construction incorporating a thermally stable diamond element. A diamond construction 50A according to an embodiment of the disclosure is shown in Figure 2A. The diamond construction 50A includes a diamond body 52A bonded to a substrate 54A. The diamond body includes a working surface 56A opposite

1 the substrate, and a cutting edge 58A at an edge of the working surface. The diamond body 52A meets the substrate 54A at an interface 60A, where the two facing surfaces of the diamond body and the substrate meet.

[0058] According to embodiments of the present disclosure, the diamond body 52A
5 includes two regions —a thermally stable region (formed from the thermally stable diamond element) and a PCD region (formed from the second diamond volume). The thermally stable region includes a thermally stable diamond element 62A, and the PCD region includes a PCD element 64A. The two elements 62A, 64A meet each other at an interface 66A, defined by the facing surfaces of the two elements 62A, 64A. In one embodiment, the thermally stable
10 diamond element 62A forms at least part of the cutting edge 58A and/or the working surface 56A of the diamond body 52A.

[0059] In Figure 2A, the two elements 62A and 64A are shaped as discs or cylinders, with the thermally stable diamond element 62A stacked on top of the PCD element 64A. The thermally stable diamond element 62A substantially covers the entire top surface of the PCD
15 element 64A. The PCD element 64A is positioned between the thermally stable diamond element 62A and the substrate 54A, such that the thermally stable element 62A does not contact the substrate. However, in other embodiments, the thermally stable diamond element and PCD element may have other shapes and configurations.

[0060] For example, in Figure 2B, a diamond construction 5QB includes a diamond body
20 52B with a thermally stable diamond element 62B that is shaped as a wedge or pie-shaped piece, and a mating PCD element 64B. The two elements 62B, 64B meet at an interface 66B that is defined by the mating surfaces of the two elements. In this case, the side surfaces of the wedge-shaped piece 62B (along the interface 66B) face the PCD element 64B, and these surfaces are coated with the coating prior to the second sintering process. The coated wedge-
25 shaped piece 62B is then combined with diamond powder to form the desired shape of the body 52B, and the combined assembly is sintered (optionally, with the substrate 54B). The thermally stable diamond element 62B forms a portion of the cutting edge 58B of the diamond body 52B, along the portion of the circumference spanned by the angle α ,

[0061] Other configurations and shapes may be used in other embodiments. For
30 example, the thermally stable diamond element may be formed into pieces of desired size and shape such as individual granules, particles, segments or the like, or may be formed into pieces of random shapes and sizes. The pieces may then be incorporated into the diamond body and may be fully surrounded by the PCD body. Another example is shown in Figure 2C, showing a cross-sectional view of a diamond construction 50C according to an
35 embodiment. The diamond construction 50C includes a diamond body 52C bonded to a substrate 54C. The diamond body 52C includes one or more thermally stable diamond elements 62C embedded within a PCD element 64C. The thermally stable elements may be fully submerged within the PCD element, or may form part of the exposed working surface,

1 cutting edge, or side surface. In this embodiment, each of the surfaces of the thermally stable element that face the PCD element is coated as described herein,

[0062] The diamond constructions shown in Figures 2A-2C include a diamond body that incorporates a thermally stable diamond element to form at least a portion of the cutting edge
5 or working surface of the diamond body. The remainder of the diamond body is formed by a PCD element formed from the second diamond volume 24 (see Figures 3A-3B). The PCD element facilitates the incorporation of the thermally stable element into the diamond body and the bonding of the diamond body to the substrate (if a substrate is utilized). In one embodiment, the thermally stable element forms at least a part of the working surface and/or
10 the cutting edge of the diamond body, such as at least 5% of the cutting edge (as measured by the circumference of the diamond body).

[0063] In one embodiment, the thermally stable diamond element 62A, 62B is smaller than the PCD element 64A, 64B. That is, the thermally stable diamond element occupies less than half of the volume of the sintered diamond body. In one embodiment, the diamond
15 volume content of the thermally stable diamond element is greater than that of the PCD element. For example, the thermally stable diamond element may have a diamond volume content of at least 95%, and in another embodiment at least 98%. The PCD element may have a diamond volume content of less than 95%, for example, ranging from 85% up to but less than 95%.

[0064] After sintering, the second diamond volume forms a PCD body that incorporates the thermally stable diamond element. In one embodiment, when the coating is a metal catalyst, it melts during sintering and partially infiltrates into the second diamond volume. As a result, after sintering, the coating may be dissipated into the second diamond volume and difficult to detect. There may be a higher content of this catalyst metal in the region near
20 the coating than remote from the coating. When the coating is a different material than the catalyst used to form PCD, such as aluminum or copper, the sintered PCD body may show a residual amount of this material near the interface with the thermally stable element. Accordingly, in one embodiment the PCD element includes a higher level of the coating material near the interface with the thermally stable element than remote from the interface
25 (such as near the substrate).

[0065] In an embodiment, the coating remains substantially in place between the two diamond volumes during sintering. After sintering, this coating can be detected as a coating layer spread along the facing surfaces of the two diamond volumes,

[0066] The diamond constructions 50A, 50B shown in Figures 2A-2B are formed as
35 cutting elements for incorporation into a cutting tool. Figure 5 shows a drag bit 40 incorporating the cutting element of Figure 2A, 2B, or 3B, according to an embodiment of the disclosure. The drag bit 40 may include several cutting elements 30 that are each attached to blades 32 that extend along the drag bit. The drag bit may be used for high-temperature rock

1 drilling operations. In other embodiments, other types of drilling or cutting tools incorporate
cutting elements that have a thermally stable diamond element forming at least a portion of
the cutting edge of the cutting element, such as, for example, rotary or roller cone drilling
bits, or percussion or hammer drill bits. In one embodiment, the cutting element is a shear
5 cutter.

[0067] Diamond constructions manufactured according to the methods disclosed herein
are expected to display an improved yield with reduced cracking. The interface between the
PCD element and the thermally stable diamond element exhibits a reduction in cracks
propagating through the PCD, as such cracks have been noted in PCD formed in the presence
10 of a fully dense thermally stable ultra-hard element. In one embodiment, the polycrystalline
diamond element exhibits shrinkage at the interface with the thermally stable element and is
substantially devoid of cracks at the interface.

[0068] While the thermally stable element and the PCD body have been described above
as a diamond bonded body, it is to be understood that these elements may be formed from
15 ultra-hard materials other than diamond. As used herein, the term "ultra-hard" is understood
to refer to those materials known in the art to have a grain hardness of about 4,000 HV or
greater. Such ultra-hard materials may include those capable of demonstrating physical
stability at temperatures above about 750° C, and for certain applications above about 1,000°
C, that are formed from consolidated materials. Such ultra-hard materials may include but are
20 not limited to diamond, cubic boron nitride (cBN), diamond-like carbon, boron suboxide,
aluminum manganese boride, and other materials in the boron-nitrogen-carbon phase diagram
which have shown hardness values similar to cBN and other ceramic materials.

[0069] Although only a few example embodiments have been described in detail above,
those skilled in the art will readily appreciate that many modifications are possible in the
25 example embodiments without materially departing from this invention. Accordingly, all
such modifications are intended to be included within the scope of this disclosure as defined
in the following claims. It is the express intention of the application not to invoke 35 U.S.C.
§ 112, paragraph 6 for any limitations of any of the claims herein, except for those in which
the claim expressly uses the words 'means for' together with an associated function.

1 WHAT IS CLAIMED IS:

1. A method for making an ultra-hard construction comprising:
applying a coating on a surface of a thermally stable ultra-hard element having a
5 theoretical density of at least 98%;

combining the thermally stable ultra-hard element with a second ultra-hard material
volume to form an assembly, with the coated surface of the thermally stable element facing
the second ultra-hard material volume; and

sintering the assembly at high pressure and high temperature;

10 wherein the thermally stable ultra-hard element is bonded to the sintered ultra-hard
element to form an ultra-hard body having a working surface and/or a cutting edge, and
wherein the thermally stable ultra-hard element forms at least a portion of the working
surface and/or the cutting edge of the ultra-hard body.

15 2. The method of claim 1, wherein the thermally stable ultra-hard element is a
thermally stable diamond element having a diamond volume content of at least 90% and
wherein the coating comprises a solvent metal.

20 3. The method of claim 2, further comprising melting the coating during the
sintering.

4. The method of claim 3, further comprising forming a slip plane between the
thermally stable diamond element and the second diamond volume during the sintering.

25 5. The method of claim 4, further comprising shrinking the second diamond
volume relative to the thermally stable diamond element during the sintering.

30 6. The method of claim 2, wherein the thermally stable diamond element
comprises a material microstructure comprising bonded-together diamond crystals
substantially devoid of interstitial regions between the diamond crystals and has a diamond
volume content of at least 98%.

35 7. The method of claim 6, further comprising forming the thermally stable
diamond element by phase-transforming graphite into diamond during a first high pressure
high temperature sintering process at a temperature of about 1600-2500 °C and a pressure of
about 100-160 kbar, prior to sintering the assembly.

1 8. The method of claim 2, further comprising forming the thermally stable
diamond element by sintering a mixture of diamond particles in the presence of a carbonate
catalyst material to form a material microstructure comprising bonded-together diamond
crystals and interstitial spaces between the diamond crystals, with the carbonate catalyst
5 material residing in the interstitial spaces.

 9. The method of claim 2, wherein sintering the assembly to form the
polycrystalline diamond element comprises sintering in the presence of a catalyst material to
promote the formation of the polycrystalline diamond element, and wherein the coating
10 comprises a melting temperature that is higher than a melting temperature of the catalyst
material.

 10. The method of claim 2, further comprising infiltrating the coating in
substantially one direction into the diamond powder and away from the thermally stable
15 element during sintering.

 11. The method of claim 2, wherein the thermally stable diamond element has a
diamond volume content of at least 95%.

20 12. The method of claim 1, further comprising mounting the ultra-hard
construction on a bit body.

 13. A cutting element comprising:
a diamond body bonded to a substrate, the diamond body having a working surface;
25 wherein the diamond body comprises:
a polycrystalline diamond element; and
a thermally stable diamond element bonded to the polycrystalline diamond
element at an interface, the thermally stable diamond element forming at least a portion of the
working surface of the diamond body,
30 wherein the thermally stable diamond element comprises a material microstructure
comprising bonded-together diamond crystals having a theoretical density of at least 98%.

 14. The cutting element of claim 13, wherein the polycrystalline diamond element
35 exhibits shrinkage at the interface and is substantially devoid of cracks at the interface.

 15. The cutting element of claim 13, wherein the polycrystalline diamond element
comprises a first region proximate the interface and a second region remote from the

1 interface, and wherein the first region comprises an increased amount of a non-diamond residual material, wherein the non-diamond residual material comprises a solvent metal catalyst.

5 16, The cutting element of claim 15, wherein the polycrystalline diamond element exhibits shrinkage at the interface and is substantially devoid of cracks at the interface,

17. A bit comprising a bit body and the cutting element of claim 13, mounted on said bit body.

10 18, A method for making a diamond construction comprising:
applying a coating on a surface of a thermally stable diamond element having a theoretical density of at least 98% and a diamond volume content of at least 90%;

15 combining the thermally stable diamond element with a second diamond volume to form an assembly, with the coated surface of the thermally stable element facing the second diamond volume, wherein the second diamond volume comprises a diamond powder;

sintering the assembly at high pressure and high temperature in the presence of a catalyst material to form a polycrystalline diamond element from the diamond powder, wherein sintering comprises melting the catalyst material;

20 melting the coating during the sintering, wherein the coating melts after the catalyst material melts, and wherein melting the coating comprises forming a slip plane between the thermally stable diamond element and the second diamond volume during the sintering; and

25 cooling the coating to bond the polycrystalline diamond element to the thermally stable diamond element to form a diamond body having a working surface and/or a cutting edge, wherein the thermally stable diamond element forms at least a portion of the working surface or the cutting edge of the diamond body.

19, The method of claim 17, wherein applying a coating comprises applying a metal solvent coating.

30 20, The method of claim 17, further comprising mounting the diamond construction on a bit body.

FIG. 1A

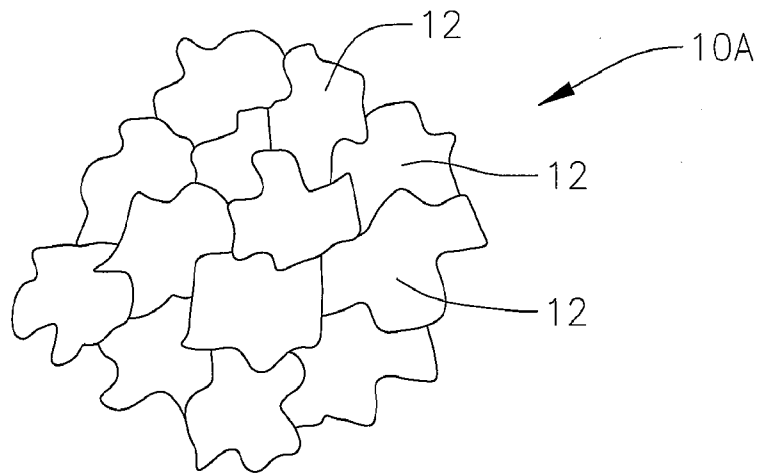


FIG. 1B

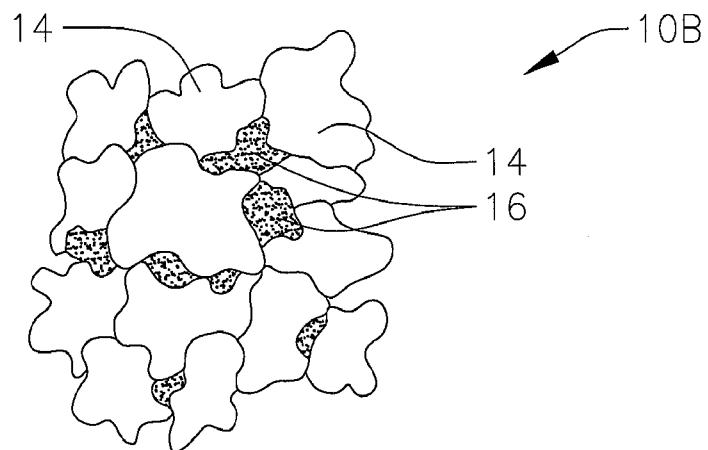


FIG. 1C

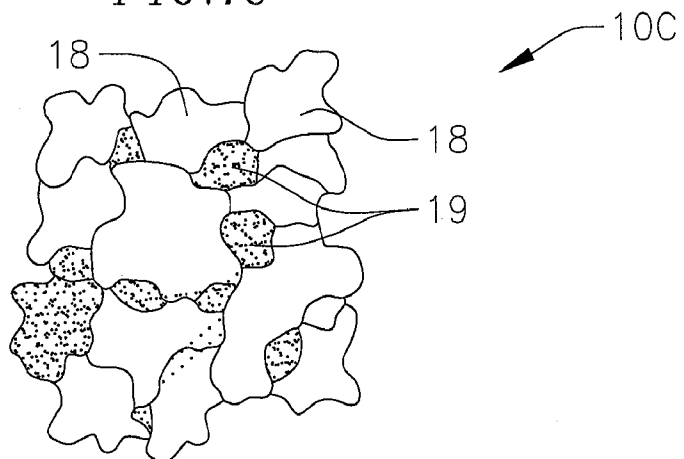


FIG. 2A

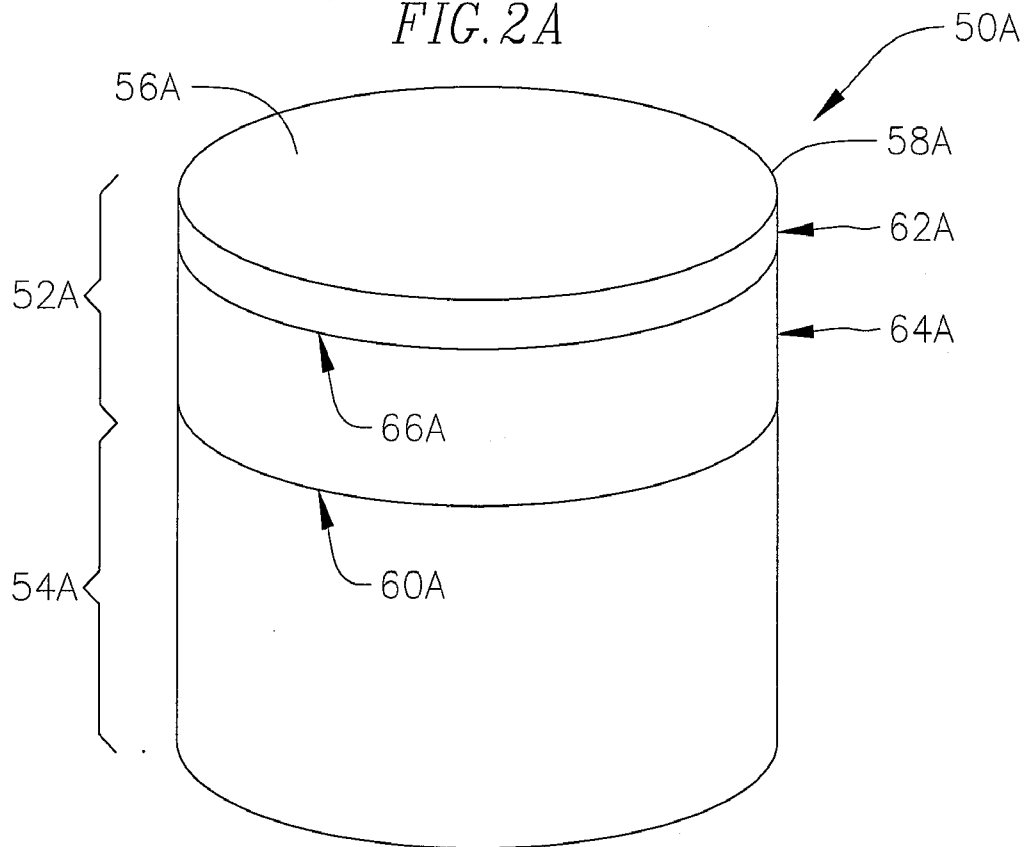
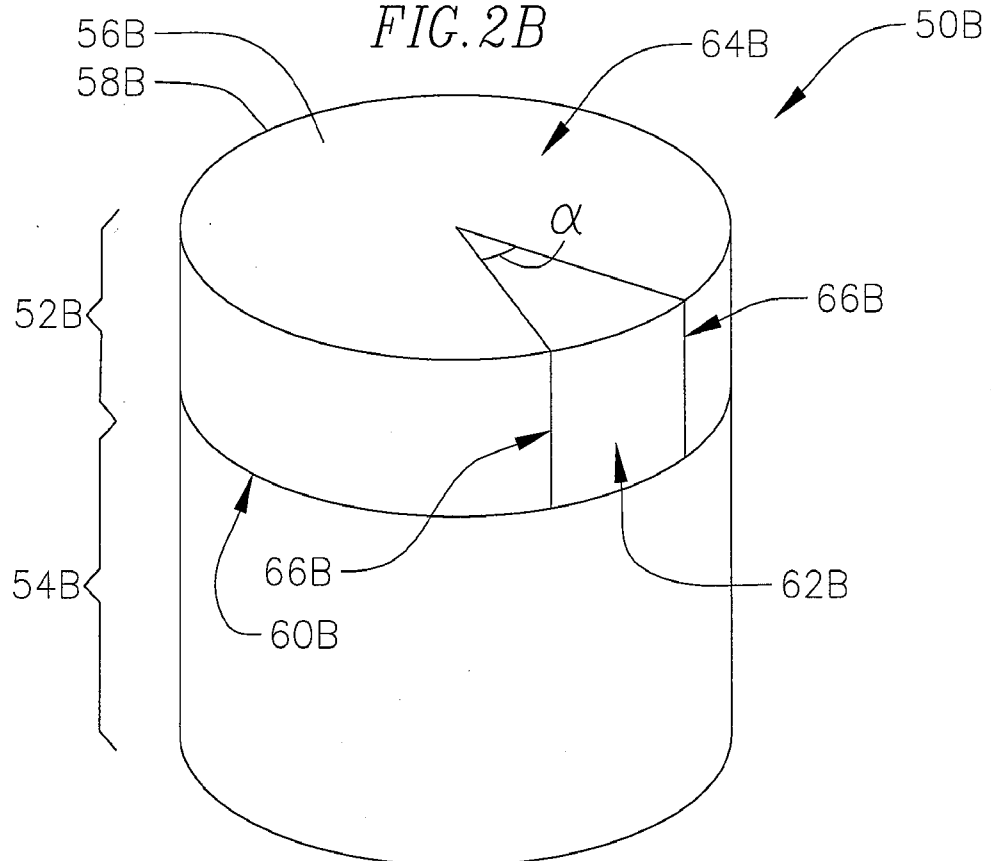
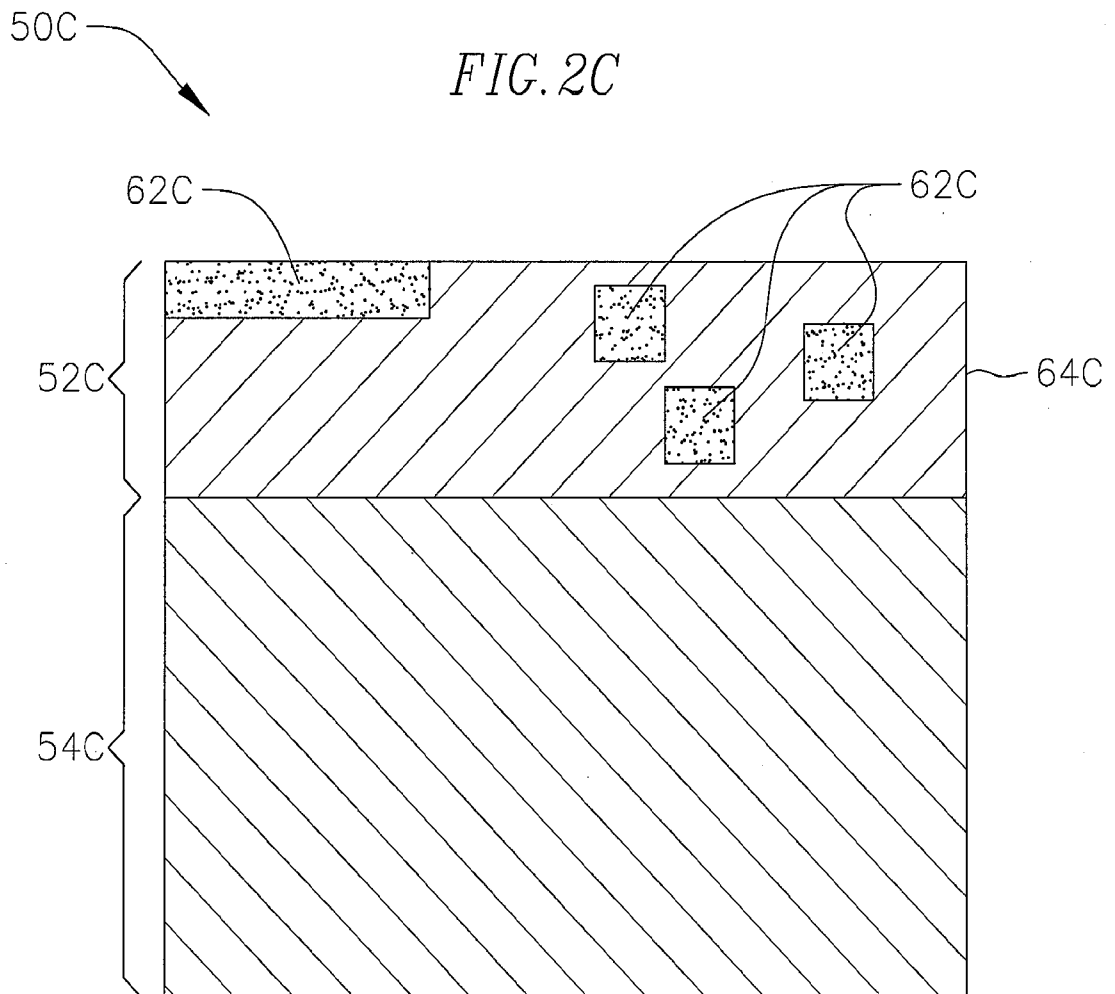


FIG. 2B





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FIG. 3A

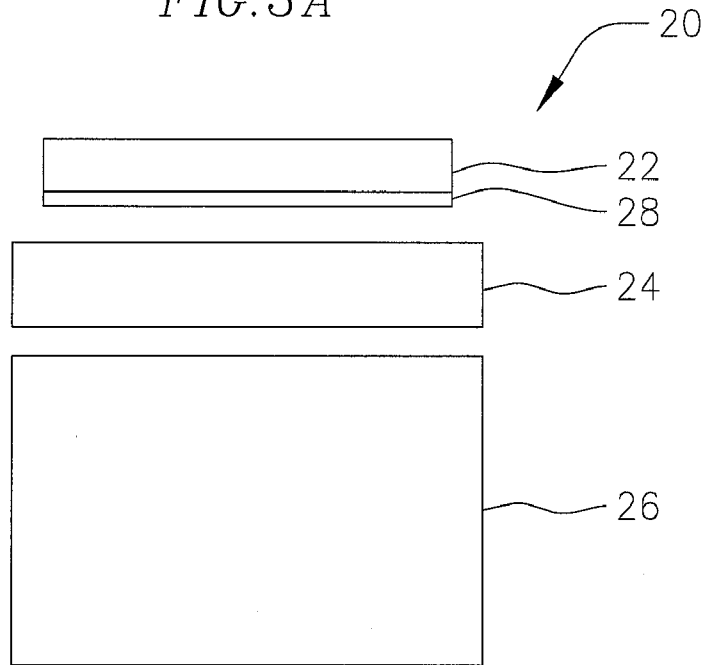


FIG. 3B

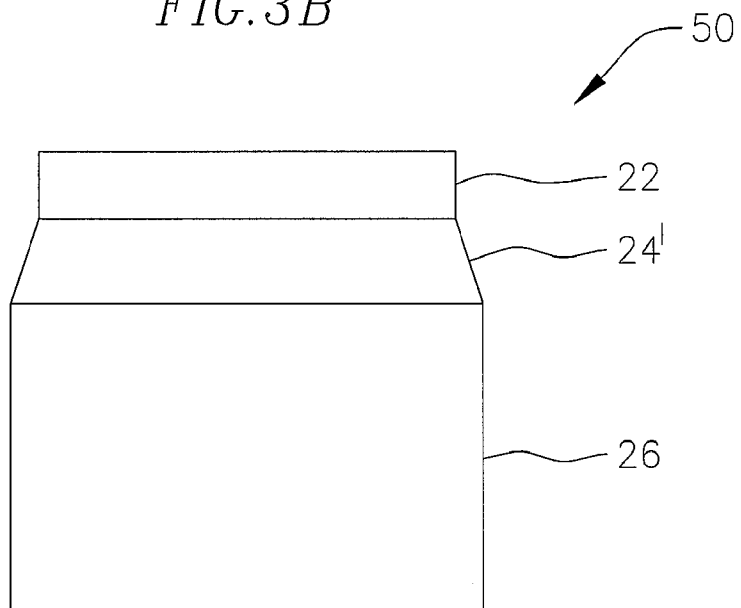


Figure 4

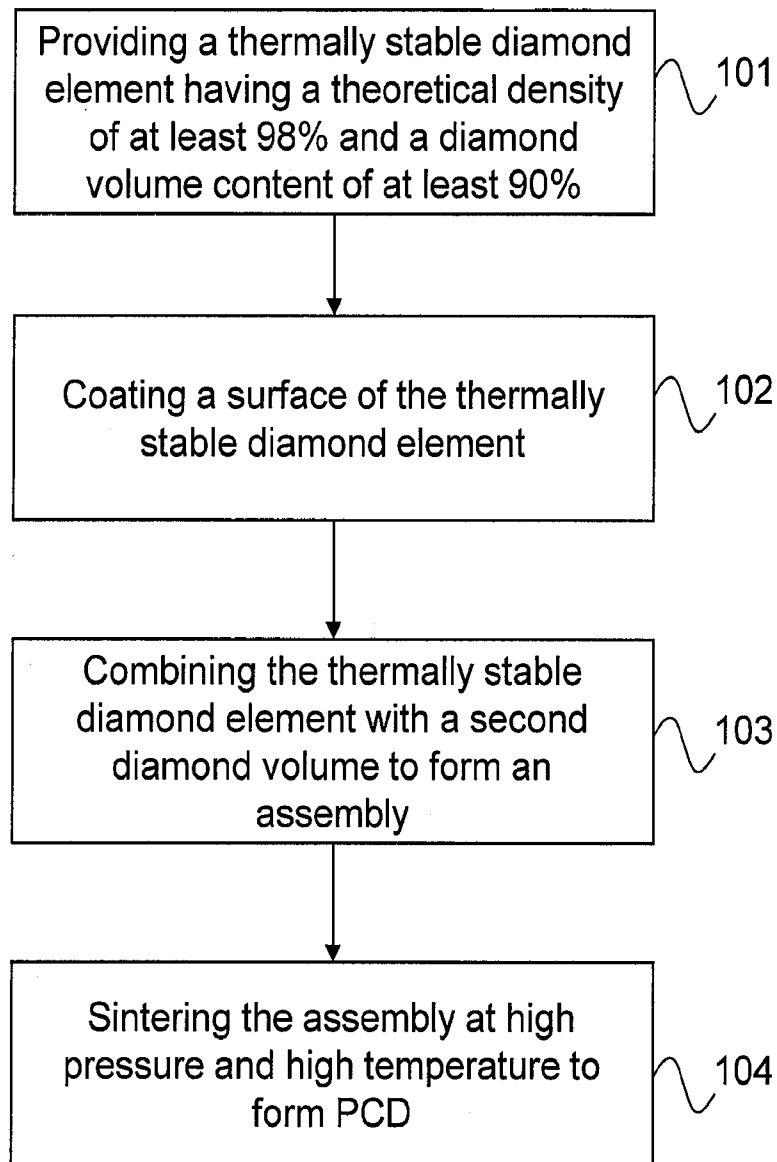
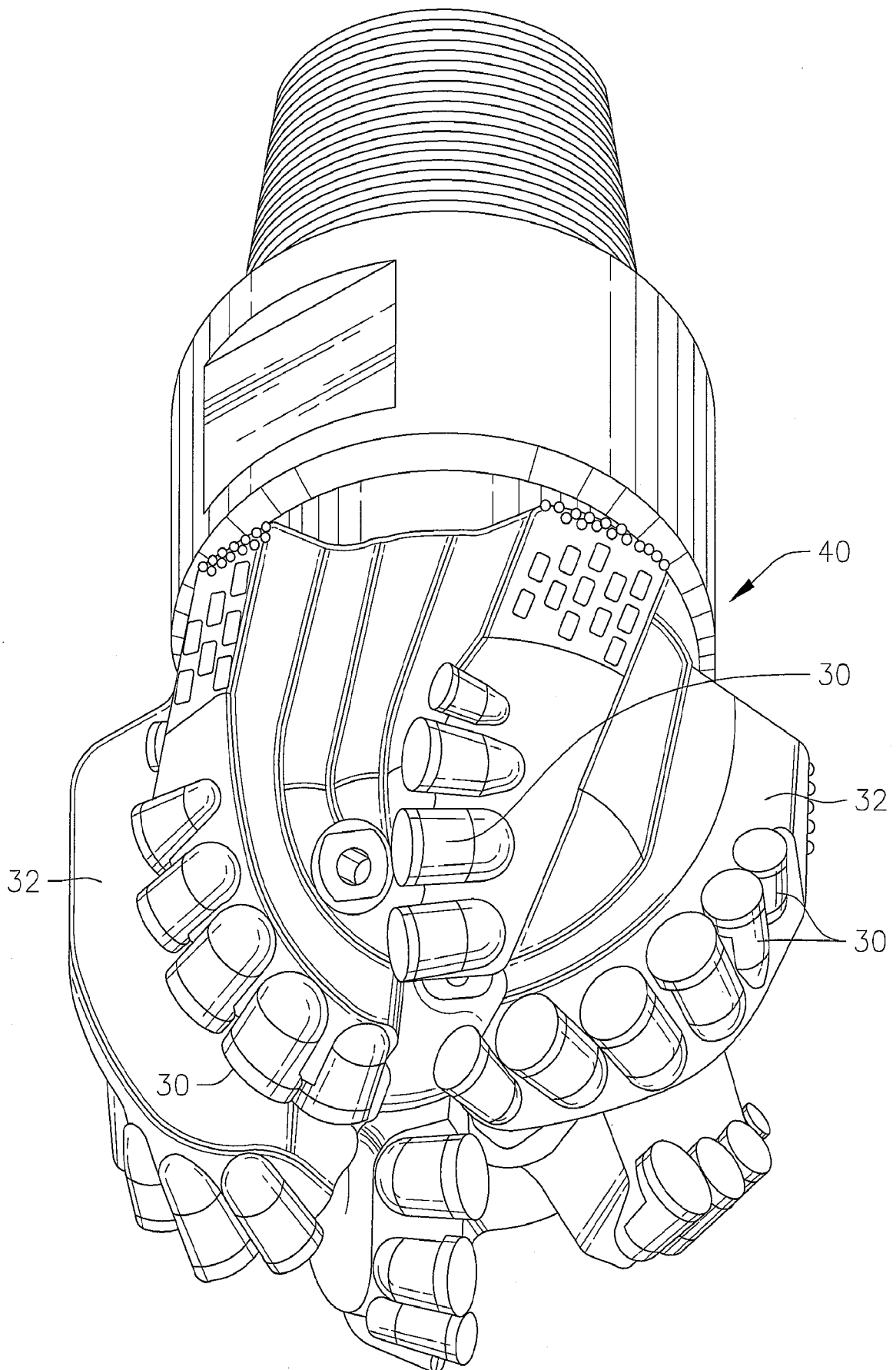


FIG. 5



INTERNATIONAL SEARCH REPORT

International application No.
PCT/US2012/070697**A. CLASSIFICATION OF SUBJECT MATTER****E21B 10/46(2006.01)i, B24D 3/10(2006.01)1**

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

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

E21B 10/46; E21B 10/36; B24D 3/02; C01B 31/06; E21B 10/42; B24D 11/00

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Korean utility models and applications for utility models

Japanese utility models and applications for utility models

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

eKOMPASS(KIPO internal) & Keywords: thermally, stable, diamond, polycrystalline, cutting element, and volume percent

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	US 2010-0084197 Al (VORONIN et al.) 08 April 2010 See abstract ; paragraphs [0040] , [0048] , [0061] , [0062] , [0064] , [0067] , [0070] , [0083] , [0092] , [0103] ; and figures 4, 8, 10 .	1-20
Y	US 2009-0095538 Al (MIDDLEMISS STEWART N.) 16 April 2009 See abstract ; paragraph [0037] , [0068] ; claim 8; and figure 2 .	1-20
A	US 2008-0073126 Al (SHEN et al.) 27 March 2008 See abstract ; paragraph [0074] ; claim 6; and figure 3 .	1-20
A	US 2006-0254830 Al (RADTKE, ROBERT P.) 16 November 2006 See abstract ; paragraphs [0028] , [0041] ; and figure 3 .	1-20
A	US 5011514 A (CHO et al.) 30 April 1991 See abstract ; and column 2, lines 12-23 .	1-20



Further documents are listed in the continuation of Box C.



See patent family annex.

* Special categories of cited documents:

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"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

"&" document member of the same patent family

Date of the actual completion of the international search

15 April 2013 (15.04.2013)

Date of mailing of the international search report

16 April 2013 (16.04.2013)

Name and mailing address of the ISA/KR

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Authorized officer

LEE, Jong Kyung

Telephone No. 82-42-481-3360



INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No.

PCT/US2012/070697

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