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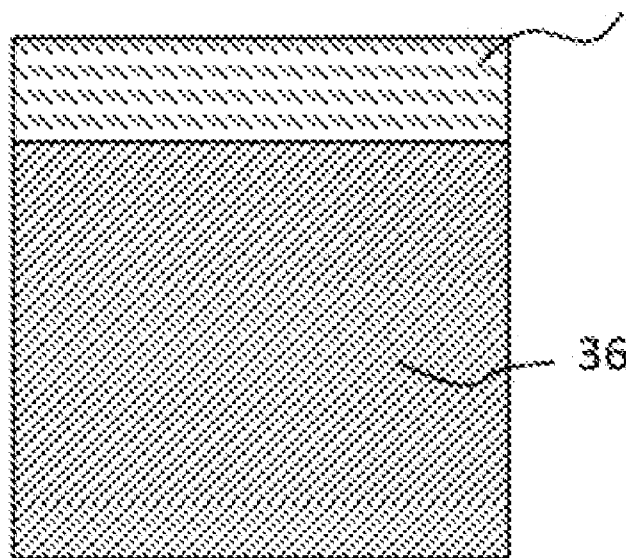
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(54) Title: ERUPTION CONTROL IN THERMALLY STABLE PCD PRODUCTS



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(57) Abstract: A method of making a polycrystalline diamond cutting element includes placing a body of polycrystalline diamond including a matrix phase of bonded together diamond grains and a plurality of empty interstitial spaces between the bonded together diamond grains adjacent a first substrate material to form an assembly and subjecting the assembly to high pressure/high temperature conditions that include an initial pressure ramping, a pressure hold, and a second pressure ramping.

ERUPTION CONTROL IN THERMALLY STABLE PCD PRODUCTS

BACKGROUND

[0001] Polycrystalline diamond (“PCD”) materials and PCD elements formed therefrom are well known in the art. Conventional PCD may be formed by subjecting diamond particles in the presence of a suitable solvent metal catalyst material to processing conditions of high pressure/high temperature (HPHT), where the solvent metal catalyst promotes desired intercrystalline diamond-to-diamond bonding between the particles, thereby forming a PCD structure. The resulting PCD structure produces enhanced properties of wear resistance and hardness, making such PCD materials extremely useful in aggressive wear and cutting applications where high levels of wear resistance and hardness are desired. FIG. 1 illustrates a microstructure of conventionally formed PCD material 10 including a plurality of diamond grains 12 that are bonded to one another to form an intercrystalline diamond matrix first phase. The catalyst/binder material 14, e.g., cobalt, used to facilitate the diamond-to-diamond bonding that develops during the sintering process is dispersed within the interstitial regions formed between the diamond matrix first phase. The term “particle” refers to the powder employed prior to sintering a superabrasive material, while the term “grain” refers to discernable superabrasive regions subsequent to sintering, as known and as determined in the art.

[0002] The catalyst/binder material used to facilitate diamond-to-diamond bonding can be provided generally in two ways. The catalyst/binder can be provided in the form of a raw material powder that is pre-mixed with the diamond grains or grit prior to sintering. In some cases, the catalyst/binder can be provided by infiltration into the diamond material (during high temperature/high pressure processing) from an underlying substrate material that the final PCD material is to be bonded to. After the catalyst/binder material has facilitated the diamond-to-diamond bonding, the catalyst/binder material is generally distributed throughout the diamond matrix within interstitial regions formed between the bonded diamond grains. Particularly, as shown in FIG. 1, the binder material 14 is not continuous throughout the microstructure in the

conventional PCD material 10. Rather, the microstructure of the conventional PCD material 10 may have a uniform distribution of binder among the PCD grains. Thus, crack propagation through conventional PCD material will often travel through the less ductile and brittle diamond grains, either transgranularly through diamond grain/binder interfaces 15, or intergranularly through the diamond grain/diamond grain interfaces 16.

[0003] Solvent catalyst materials may facilitate diamond intercrystalline bonding and bonding of PCD layers to each other and to an underlying substrate. Solvent catalyst materials used for forming conventional PCD include metals from Group VIII of the Periodic table, such as cobalt, iron, or nickel and/or mixtures or alloys thereof, with cobalt being the most common. Conventional PCD may include from 85 to 95% by volume diamond and a remaining amount of the solvent catalyst material. However, while higher metal content increases the toughness of the resulting PCD material, higher metal content also decreases the PCD material hardness, thus limiting the flexibility of being able to provide PCD layers having desired levels of both hardness and toughness. Additionally, when variables are selected to increase the hardness of the PCD material, brittleness also increases, thereby reducing the toughness of the PCD material.

[0004] PCD is commonly used in earthen drilling operations, for example in cutting elements used on various types of drill bits. Although PCD is extremely hard and wear resistant, PCD cutting elements may still fail during normal operation. Failure may occur in three common forms, namely wear, fatigue, and impact cracking. The wear mechanism occurs due to the relative sliding of the PCD relative to the earth formation, and its prominence as a failure mode is related to the abrasiveness of the formation, as well as other factors such as formation hardness or strength, and the amount of relative sliding involved during contact with the formation. Excessively high contact stresses and high temperatures, along with a very hostile downhole environment, also tend to cause severe wear to the diamond layer. The fatigue mechanism (including both thermal and/or mechanical fatigue) involves the progressive propagation of a surface crack, initiated on the PCD layer, into the material below the PCD layer until the crack length is sufficient for spalling or chipping. Lastly, the impact mechanism involves the

sudden propagation of a surface crack or internal flaw initiated on the PCD layer, into the material below the PCD layer until the crack length is sufficient for spalling, chipping, or catastrophic failure of the cutting element.

SUMMARY

[0005] 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 limiting the scope of the claimed subject matter.

[0006] In one aspect, embodiments disclosed herein relate to a method of making a polycrystalline diamond cutting element that includes placing a body of polycrystalline diamond including a matrix phase of bonded together diamond grains and a plurality of empty interstitial spaces between the bonded together diamond grains adjacent a first substrate material to form an assembly and subjecting the assembly to high pressure/high temperature conditions that include an initial pressure ramping, a pressure hold, and a second pressure ramping.

[0007] In another aspect, embodiments disclosed herein relate to a method of forming a polycrystalline ultra-hard material that includes placing a volume of ultra-hard material adjacent to a substrate material including a Group VIII-containing material to form an assembly, subjecting the assembly to a first high pressure/high temperature condition sufficient to cause the Group VIII-containing material to melt and partially infiltrate the volume of ultra-hard material, and subjecting the combination to a second high pressure/high temperature condition sufficient to cause the Group VIII-containing material to further infiltrate the volume of ultra-hard material, where the pressure of the second high pressure/high temperature condition is higher than that of the first high pressure/high temperature condition.

[0008] In yet another aspect, embodiments disclosed herein relate to a polycrystalline diamond compact that includes a polycrystalline diamond body including a matrix phase of bonded together diamond grains and a plurality of interstitial spaces between

the bonded together diamond grains, where the polycrystalline diamond body is substantially free of eruptions, and a substrate attached to the polycrystalline diamond body at an interface, where the polycrystalline diamond body has at least two regions, a first region adjacent the interface and a second region opposite the interface, where the first region of the polycrystalline diamond body has an infiltrant material disposed within the interstitial spaces and being substantially free of a catalyst material used to form the polycrystalline diamond body, and where the interstitial spaces in the second region of the polycrystalline diamond body are substantially free of the infiltrant material and the catalyst material used to form the polycrystalline diamond body.

[0009] Other aspects and advantages of the claimed subject matter will be apparent from the following description and the appended claims.

BRIEF DESCRIPTION OF DRAWINGS

[0010] Embodiments of the present disclosure are described with reference to the following figures. The same numbers are used throughout the figures to reference like features and components.

[0011] FIG. 1 shows the microstructure of conventionally formed polycrystalline diamond.

[0012] FIG. 2 is a picture of a reinfiltrated PCD diamond table after removing the metal matrix.

[0013] FIG. 3 is a picture of a reinfiltrated PCD diamond table, after removing the metal matrix, formed according to embodiments of the present disclosure.

[0014] FIGS. 4.1-4.3 shows a schematic of formation of a cutting element in accordance with the present disclosure.

[0015] FIG. 5 shows a fixed cutter drill bit.

[0016] FIG. 6 shows a hole opener.

[0017] FIG. 7 shows is a cross-sectional view of a conventionally formed reinfiltrated diamond compact.

DETAILED DESCRIPTION

[0018] In one aspect, embodiments disclosed herein relate to use of multiple pressure stages during the formation of a polycrystalline diamond compact cutter, and the polycrystalline diamond compact cutters formed by such processing.

[0019] Polycrystalline ultra-hard materials, and compacts formed therefrom, are specifically engineered having a polycrystalline ultra-hard material body having a material microstructure that is substantially free of substrate material eruptions, e.g., catalyst or infiltrant material eruptions, and thereby free of localized concentrations, regions or volumes of the catalyst or infiltrant material therein, and substantially free of any other substrate constituent material. As used herein, “eruptions” refer to precipitated regions of carbide grains and binder pools (catalyst or infiltrant material) formed from the substrate material, which may create inclusions that are substantially larger than the interstitial regions formed in a polycrystalline diamond body. Eruptions created by binder pools may leave voids within the material microstructure once the binder is removed. In other words, pores may not be present in the reinfiltrated polycrystalline diamond body until the binder pools are removed. Thus, “eruptions” may refer to inclusions or collections of precipitated material different from the remaining polycrystalline diamond microstructure, while “pores” refer to the voids left in the polycrystalline diamond microstructure after removal of the inclusions. As used herein, the eruptions may be at least an order of magnitude larger than conventional interstitial regions. Eruptions may occur during HPHT bonding methods of attaching a diamond body to a substrate without pressure control, where the eruptions precipitate from the substrate into the diamond body.

[0020] For example, FIG. 7 shows an example of a diamond compact 400 formed by HPHT bonding of a preformed diamond body 410 to a tungsten carbide substrate 420 without using the pressure stages described herein. During the HPHT step, eruptions 430 of tungsten carbide and cobalt precipitated from the substrate 420 into the diamond body 410 in a branched pattern. Eruptions 430 occurring from the HPHT bonding process of attaching a diamond body 410 to a tungsten carbide substrate 420 are distinct

from substrate material that may infiltrate into the diamond body 410 during the HPHT bonding process to form the metallic bonds. For example, as described above, eruptions 430 may be made of precipitated tungsten carbide and cobalt, which has a tree-like or dendrite form extending into the diamond body, and which may have a substantially larger size than the infiltrated materials residing in interstitial spaces between diamond grains bonded together. During the HPHT bonding process, substrate material may also infiltrate into the diamond body. Infiltration occurs when the temperature of the HPHT bonding process reaches the melting temperature of the substrate material. For example, when the HPHT bonding process temperature reaches the melting point of cobalt, the cobalt from the tungsten carbide substrate may melt and infiltrate into the diamond body, thereby filling at least a portion of the empty interstitial regions. Further, prior to attachment to the substrate, the diamond body 410 may include bonded together diamond grains and substantially empty interstitial regions between the bonded together diamond grains. After the HPHT bonding process, the diamond body 410 may have an amount of infiltrated cobalt disposed within the interstitial regions. Further, the present disclosure also relates to the use of a two-stage pressure profile during HPHT sintering and formation of polycrystalline diamond to result in a more uniform polycrystalline diamond microcrystalline structure.

[0021] Eruptions may have a distinct form and composition from the remaining diamond microstructure and may also produce a non-uniform substrate microstructure. Thus, in accordance with the present disclosure, the catalyst or infiltrant material in such polycrystalline ultra-hard material body is instead evenly dispersed throughout the material microstructure, or throughout at least a region of material microstructure for those embodiments where the catalyst or infiltrant material has been removed therefrom. In an example embodiment, such polycrystalline ultra-hard materials and compacts are formed by controlling the HPHT process used to sinter the polycrystalline ultra-hard material, to regulate the manner in which the catalyst or infiltrant material melts and is infiltrated into the adjacent ultra-hard material before and during the sintering process.

[0022] For example, FIG. 2 shows an example of a conventional pre-formed diamond table 200 that had been reinfiltrated with cobalt by HPHT processing without using a two-stage pressure profile. The diamond table 200 has a plurality of pores 210 formed therein. Particularly, eruptions of a metal matrix precipitated from a substrate into the diamond table 200 during processing, and when the metal matrix material was subsequently removed, the pores 210 were left in the diamond table 200. In other words, the pores 210 in the diamond table 200 are not formed until the metal matrix is removed, such as by leaching. In contrast, FIG. 3 shows a diamond table 300 formed according to methods of the present disclosure, where the preformed diamond table 300 had been attached to a substrate (not shown) by HPHT bonding using a two-stage pressure. As shown, the diamond table 300 has a substantially continuous microstructure, where substantially no eruptions have been formed by the reinfiltration of cobalt.

[0023] Further, eruptions may create a non-uniform microstructure in the polycrystalline diamond. Particularly, eruptions formed using conventional methods of attaching a tungsten carbide substrate to a diamond body may include precipitated tungsten carbide grains and cobalt pools extending into the diamond body in a tree-shaped or branched pattern. Thus, the attached polycrystalline diamond body may have a microstructure including a plurality of bonded together diamond grains, a plurality of interstitial regions disposed among the bonded together diamond grains, and extensions of precipitated tungsten carbide grains and cobalt pools extending from the interface between the tungsten carbide substrate and diamond body a distance into the diamond body and through the bonded together diamond grains and interstitial regions. However, constructions formed according to methods of the present disclosure may have an attached diamond body that is substantially free of eruptions. In other words, the attached diamond body may have a substantially uniform microstructure including a plurality of bonded together diamond grains and a plurality of interstitial regions disposed among the bonded together diamond grains.

[0024] Aspects of the present disclosure involve the use of an HPHT profile that is controlled to minimize or reduce the number of eruptions of the substrate materials into

the diamond body. The controlled HPHT process may involve a process used in the initial formation of a polycrystalline diamond body on a substrate or/and it may involve a subsequent HPHT process whereby a previously formed polycrystalline diamond (PCD) body is attached to a substrate.

[0025] As used herein, the term “PCD” refers to polycrystalline diamond that has been formed, at high pressure/high temperature (HPHT) conditions, through the use of a catalyst, such as solvent metal catalysts from Group VIII of the Periodic table, non-metallic catalysts including carbonates, as well as non-catalyst formed polycrystalline diamond formed with even higher temperatures and pressure than those used to form polycrystalline diamond with cobalt.

[0026] In accordance with particular embodiments of the present disclosure, the two-stage pressure profile of the HPHT process may be particularly applicable for attaching previously formed diamond bodies to a substrate in an HPHT process to reduce or avoid the formation of eruptions within the diamond body. In such embodiments, the diamond body attached to the substrate may be previously treated to be rendered thermally stable so that the diamond body is substantially free of the catalyst material used to form the diamond body. When the diamond body is then attached to a substrate in an HPHT process, the bond is formed by a continuous metal bond. However, other embodiments of the present disclosure also relate to the use of a two-stage pressure profile in the HPHT process used to form the polycrystalline diamond microstructure.

Forming Polycrystalline Diamond

[0027] A polycrystalline diamond body may be formed in a conventional manner, such as by a high pressure, high temperature sintering of “green” particles to create intercrystalline bonding between the particles. “Sintering” may involve a high pressure, high temperature (HPHT) process. Examples of high pressure, high temperature (HPHT) process can be found, for example, in U.S. Patent Nos. 4,694,918; 5,370,195; and 4,525,178. Briefly, to form the polycrystalline diamond object, an unsintered mass of diamond crystalline particles is placed within a metal enclosure of the reaction cell of a HPHT apparatus. A suitable HPHT apparatus for this process is described in U.S.

Patent Nos. 2,947,611; 2,941,241; 2,941,248; 3,609,818; 3,767,371; 4,289,503; 4,673,414; and 4,954,139. A metal catalyst, such as cobalt or other Group VIII metals, may be included with the unsintered mass of crystalline particles to promote intercrystalline diamond-to-diamond bonding. The catalyst material may be provided in the form of powder and mixed with the diamond grains, or may be infiltrated into the diamond grains during HPHT sintering. For example, a minimum temperature may be about 1200 °C (2192 °F) and a minimum pressure may be about 35 kilobars. In some embodiments, processing may be conducted at a pressure of at least 45 kbar and 1300 °C (2372 °F). Those of ordinary skill in the art will appreciate that a variety of temperatures and pressures may be used, and the scope of the present invention is not limited to specifically referenced temperatures and pressures. Further, it is also within the scope of the present disclosure that the two-step pressure profile disclosed below may be used in the polycrystalline diamond formation process.

[0028] Diamond grains useful for forming a polycrystalline diamond body may include any type of diamond particle, including natural or synthetic diamond powders having a wide range of grain sizes. For example, such diamond powders may have an average grain size in the range from submicrometer in size to 100 micrometers, and from 1 to 80 micrometers in other embodiments. Further, one skilled in the art would appreciate that the diamond powder may include grains having a mono- or multi-modal distribution.

[0029] Moreover, the diamond powder used to prepare the PCD body may be synthetic diamond powder or natural diamond powder. Synthetic diamond powder is known to include small amounts of solvent metal catalyst material and other materials entrained within the diamond crystals themselves. The diamond grain powder, whether synthetic or natural, may be combined with or already includes a desired amount of catalyst material to facilitate desired intercrystalline diamond bonding during HPHT processing. Suitable catalyst materials useful for forming the PCD body include those solvent metals selected from the Group VIII of the Periodic table, with cobalt (Co) being the most common, and mixtures or alloys of two or more of these materials. In a particular embodiment, the diamond grain powder and catalyst material mixture may include 85 to 95% by volume diamond grain powder and the remaining amount catalyst material. In

other embodiments, the diamond grain powder can be used without adding a solvent metal catalyst in applications where the solvent metal catalyst can be provided by infiltration during HPHT processing from the adjacent substrate or adjacent other body to be bonded to the PCD body.

[0030] The diamond powder may be combined with the desired catalyst material, and the reaction cell is then placed under processing conditions sufficient to cause the intercrystalline bonding between the diamond particles. In the event that the formation of a PCD compact including a substrate bonded to the PCD body is desired, a selected substrate is loaded into the container adjacent the diamond powder mixture prior to HPHT processing. Additionally, in the event that the PCD body is to be bonded to a substrate, and the substrate includes a metal solvent catalyst, the metal solvent catalyst used for catalyzing intercrystalline bonding of the diamond may be provided by infiltration, in which case, a metal solvent catalyst does not have to be mixed with the diamond powder prior to HPHT processing.

[0031] In an example embodiment, the device is controlled so that the container is subjected to a HPHT process including a pressure in the range of from 40 to 70 kilobars and a temperature in the range of from about 1320 to 1600 °C, for a sufficient period of time. During this HPHT process, the catalyst material in the mixture melts and infiltrates the diamond grain powder to facilitate intercrystalline diamond bonding. During the formation of such intercrystalline diamond bonding, the catalyst material may migrate into the interstitial regions within the microstructure of the so-formed PCD body that exists between the diamond bonded grains. It should be noted that if too much additional non-diamond material is present in the powdered mass of crystalline particles, appreciable intercrystalline bonding is prevented during the sintering process. Such a sintered material where appreciable intercrystalline bonding has not occurred is not within the definition of PCD. Following such formation of intercrystalline bonding, a polycrystalline diamond body may be formed that has, in one embodiment, at least about 80 percent by volume diamond, with the remaining balance of the interstitial regions between the diamond grains occupied by the catalyst material. In other embodiments, such diamond content may include at least 85 percent by volume of the

formed diamond body, and at least 90 percent by volume in yet another embodiment. However, one skilled in the art would appreciate that other diamond densities (or gradients of diamond densities) may be used in alternative embodiments. In particular embodiments, the polycrystalline diamond bodies being leached in accordance with the present disclosure include what is frequently referred to in the art as “high density” polycrystalline diamond, which refers to a diamond body having a diamond content of at least 90 percent by volume. However, in other embodiments, the high density polycrystalline diamond used in the method of the present disclosure may have a density of at least 92 percent by volume up to 97 percent by volume. One skilled in the art would appreciate that conventionally, as diamond density increases, the leaching time (and potential inability to effectively leach) similarly increases.

[0032] Further, one skilled in the art would appreciate that, frequently, a diamond layer is sintered to a carbide substrate by placing the diamond particles on a preformed substrate in the reaction cell and sintering. However the present disclosure is not so limited. Rather, the polycrystalline diamond bodies treated in accordance with the present disclosure may or may not be attached to a substrate.

[0033] In a particular embodiment, the polycrystalline diamond body is formed using solvent catalyst material provided as an infiltrant from a substrate, for example, a WC-Co substrate, during the HPHT process. In such embodiments where the polycrystalline diamond body is formed with a substrate, it may be desirable to remove the polycrystalline diamond portion from the substrate prior to leaching so that leaching agents may contact the diamond body in an unshielded manner, i.e., from each side of the diamond body without substantial restriction.

[0034] In various embodiments, a formed PCD body having a catalyst material in the interstitial spaces between bonded diamond grains is subjected to a leaching process (before or after attachment to a substrate), whereby the catalyst material is removed from the PCD body. As used herein, the term “removed” refers to the reduced presence of catalyst material in the PCD body, and is understood to mean that a substantial portion of the catalyst material no longer resides in the PCD body. However, one

skilled in the art would appreciate that the leaching process is limited in that trace amounts of catalyst material may still remain in the microstructure of the PCD body within the interstitial regions and/or adhered to the surface of the diamond grains. Such trace amounts may result from limited access of leaching agents during the leaching process, and because of this limited access, alternative methods may be used to reduce the thermal coefficient differential between the remaining catalyst material and diamond.

[0035] Rather than actually removing the catalyst material remaining in the interstitial spaces and/or adhered to the surface of the diamond grains from the PCD body or compact, the selected region of the PCD body or compact can be rendered thermally stable by treating the catalyst material in a manner that reduces or prevents the potential for the catalyst material to adversely impact the intercrystalline bonded diamond at elevated temperatures due to the thermal mismatch between the diamond and the remaining catalyst material as well as potential back conversion or graphitization. For example, the catalyst material can be combined chemically with another material or transformed into another material, thus causing it to no longer act as a catalyst material. Accordingly, as used herein, the terms “removing substantially all” or “substantially free” as used in reference to the catalyst material is intended to cover the different methods in which the catalyst material can be treated to no longer adversely impact the intercrystalline diamond in the PCD body or compact with increasing temperature.

[0036] The quantity of the catalyst material remaining in the material PCD microstructure after the PCD body has been subjected to a leaching treatment may vary, for example, on factors such as the treatment conditions, including treatment time, as well as whether the PCD body is attached to the substrate body before or after leaching. Further, one skilled in the art would appreciate that it may be desired in certain applications to allow a small amount of catalyst material to remain in the PCD body. In a particular embodiment, the PCD body may include up to 1-2 percent by weight of the catalyst material. However, one skilled in the art would appreciate that the amount of residual catalyst present in a leached PCD body may depend on the diamond density of the material and body thickness.

[0037] A conventional leaching process involves the exposure of an object to be leached with a leaching agent, such as described in U.S. Patent No. 4,224,380. In select embodiments, the leaching agent may be a weak, strong, or mixtures of acids. In other embodiments, the leaching agent may be a caustic material such as NaOH or KOH. Suitable acids may include, for example, nitric acid, hydrofluoric acid, hydrochloric acid, sulfuric acid, phosphoric acid, or perchloric acid, or combinations of these acids. In addition, caustics, such as sodium hydroxide and potassium hydroxide, have been used to the carbide industry to digest metallic elements from carbide composites. In addition, other acidic and basic leaching agents may be used as desired. Those having ordinary skill in the art will appreciate that the molarity of the leaching agent may be adjusted depending on the time desired to leach, concerns about hazards, etc.

[0038] Once the leaching step is completed and the PCD body is removed from the leaching agent, the resulting material microstructure of the leached portion of the diamond body may include a first matrix phase of the bonded-together diamond grains and a second phase of a plurality of empty interstitial regions dispersed within the matrix phase. In other words, at the end of the leaching process, the treated interstitial regions may be substantially empty so that the second phase may be described as a plurality of voids or empty regions dispersed throughout the diamond-bonded matrix phase. Thus, the leached portion of the diamond body may be substantially free of the catalyst material used to initially form or sinter the diamond body, and may be referred to as thermally stable polycrystalline diamond.

Reattachment

[0039] Preformed diamond bodies may be attached (or reattached) to a substrate subjecting the polycrystalline diamond body and substrate to the HPHT conditions disclosed herein, to facilitate attachment to a bit, cutting tool, or other end use application or device. Furthermore, methods of attaching (or reattaching) a diamond layer to a substrate may result in the migration of an infiltrant material, the source of which may be the substrate and/or an optional intermediate material. In a particular

embodiment, the source of infiltrant material may be a substrate that is attached to preformed diamond body during the HPHT process.

[0040] As used herein, the term “infiltrant material” is understood to refer to materials that are other than the catalyst material used to initially form the diamond body, and may include materials identified in Group VIII of the periodic table that have subsequently been introduced into the already formed diamond body. The type of infiltrant material is not a limitation on the scope of the present disclosure. Additionally, the term “infiltrant material” is not intended to be limiting on the particular method or technique used to introduce such material into the already formed diamond body. The infiltrant material may be selected from the group of materials including metals, ceramics, cermets, or combinations thereof. In an example embodiment, the infiltrant material is a metal or metal alloy selected from Group VIII of the Periodic Table, such as cobalt, nickel, iron or combinations thereof. It is to be understood that the choice of material or materials used as the infiltrant material can and will vary depending on such factors including but not limited to the end-use application, and the type and density of the diamond grains used to form the polycrystalline diamond matrix first phase, and the mechanical properties and/or thermal characteristics desired for the polycrystalline diamond construction.

[0041] After forming a diamond body, the catalyst material residing in the interstitial regions may be removed so that an infiltrant material may take its place in the interstitial regions. That is, once the catalyst material used to initially form the diamond body is removed from the diamond body, the remaining microstructure includes a polycrystalline matrix phase with a plurality of interstitial voids forming what is essentially a porous material microstructure.

[0042] The voids or pores in the polycrystalline diamond body may be filled with the infiltrant material using a number of different techniques. Further, voids throughout the diamond body or voids throughout a portion of the diamond body may be filled with the replacement material. In an example embodiment, the infiltrant material may be introduced into the diamond body by liquid-phase sintering under HPHT conditions. In

such example embodiment, the infiltrant material may be provided in the form of a sintered part or a green-state part that contains the infiltrant material and that is positioned adjacent one or more surfaces of the diamond body. The assembly is placed into a container that is subjected to HPHT conditions sufficient to melt the infiltrant material within the sintered part or green-state part and cause it to infiltrate into the diamond body. In an example embodiment, the source of the infiltrant material may be a substrate that will be used to form a compact from the polycrystalline diamond construction by attachment to the diamond body during the HPHT process.

[0043] The term “filled”, as used herein to refer to the presence of the infiltrant material in the voids or pores of the diamond body that resulted from removing the catalyst material used to form the diamond body therefrom, is understood to mean that a substantial volume of such voids or pores contain the infiltrant material. However, it is to be understood that there may also be a volume of voids or pores within the same region of the diamond body that do not contain the infiltrant material, and that the extent to which the infiltrant material effectively displaces the empty voids or pores will depend on such factors as the particular microstructure of the diamond body, the effectiveness of the process used for introducing the infiltrant material, and the desired mechanical and/or thermal properties of the resulting polycrystalline diamond construction. In some embodiments, when introduced into the diamond body, the infiltrant fills substantially all of the voids or pores throughout the diamond body. In some embodiments, complete migration of the infiltrant material through the diamond body is not realized, in which case a region of the diamond body may not include the infiltrant material. This region devoid of the infiltrant material from such incomplete migration may extend from the region including the infiltrant to a surface portion of the diamond body, such as a cutting surface of the diamond body.

[0044] In an example embodiment, a substrate is used as the source of the infiltrant material and to form the polycrystalline construction. Substrates useful in this regard may include substrates that are used to form conventional PCD, e.g., those formed from metals, ceramics, and/or cermet materials that contain a desired infiltrant. In an example embodiment, the substrate is formed from WC-Co, and is positioned adjacent

the diamond body after the metal catalyst material used to initially form the same been removed, and the assembly is subjected to HPHT conditions sufficient to cause the cobalt in the substrate to melt and infiltrate into and fill the voids or pores in the polycrystalline diamond matrix.

[0045] Once the diamond body has been filled with the infiltrant material, it may then be treated to remove a portion of the infiltrant material therefrom. In some embodiments, where the infiltrant material did not migrate completely through the diamond body, a subsequent infiltrant removal step may not be conducted, or may useful as a clean up process to ensure a uniform infiltrant removal depth. Treating the compact to remove such infiltrant material may render the polycrystalline diamond body or compact thermally stable by treating the infiltrant material in a manner that reduces or prevents the potential for the infiltrant material to adversely impact the intercrystalline bonded diamond at elevated temperatures. Generally, infiltrant materials are problematic when heat is generated at the cutter impact point of the compact. Specifically, heat generated at the exposed part of the polycrystalline diamond body, caused by friction between the polycrystalline diamond and the work material, may result in thermal damage to the polycrystalline diamond in the form of cracks (due to differences in thermal expansion coefficients) which may lead to spalling of the polycrystalline diamond layer, delamination between the polycrystalline diamond and the substrate, and back conversion of diamond to graphite causing rapid abrasive wear. Thus, increased thermal stability may be achieved by treating the compact to remove such infiltrant material using such methods as leaching or other methods known in the art.

[0046] In an example embodiment, the infiltrant material is removed from the diamond body a depth of less than about 0.7 mm from the desired surface or surfaces, and in some embodiments, in the range of from about 0.05 to 0.5 mm. Ultimately, the specific depth of the region formed in the diamond body by removing the infiltrant material will vary depending on the particular end-use application.

[0047] In some embodiments, a detached polycrystalline diamond layer may be treated to first remove the catalyst material initially used to form the polycrystalline bonds in the

polycrystalline diamond layer. The resulting thermally stable polycrystalline diamond body may then be attached to a substrate using an HPHT process for a period of time and at a temperature sufficient to meet the melting point of an infiltrant material present in the substrate such that the infiltrant material migrates to the polycrystalline diamond body. The resulting polycrystalline diamond compact may then be treated to remove a portion of the infiltrant material therefrom. Techniques useful for removing a portion of the infiltrant material from the diamond compact include the same techniques described above for removing the catalyst material used to initially form the diamond compact from the polycrystalline diamond body, *e.g.*, such as by leaching or the like. Depending on the application, it may be desired that the process of removing the infiltrant material be controlled so that the infiltrant material be removed from a targeted region of the diamond compact extending a determined depth from one or more diamond compact surfaces. These surfaces may include working and/or nonworking surfaces of the diamond compact.

[0048] Referring now to FIGS. 4.1-4.3 collectively, an embodiment of the process steps of the present disclosure is shown. As shown in FIG. 4.1, a polycrystalline diamond body 30 having a catalyzing material found in the interstitial regions between the diamond grains (as described above) may be formed attached to a carbide substrate 34. The polycrystalline diamond body 30 may be detached (shown in FIG. 4.2) from the substrate 34 simultaneous with or prior to removal of the catalyzing material from the interstitial spaces. Further, as shown in FIG. 4.3, the diamond body 30 may then be attached (or reattached) to a substrate 36 through sintering, and in particular using multiple stages of elevated pressure.

Two Stage Pressure Profile

[0049] In accordance with embodiments of the present disclosure, the HPHT process in which the diamond material (either preformed body or mass of diamond particles) is attached to a substrate and infiltrated with a catalyst or infiltrant material, may include a two-stage pressure profile. The first stage may include elevated temperatures (to temperatures sufficient to cause infiltration of the catalyst or infiltrant material), and a

first stage of pressure ramping. However, after a short hold at the first stage of elevated pressure, the pressure is further ramped to a predetermined second stage pressure and held for the duration of the HPHT process. In one or more embodiments, the second pressure hold may be longer than the first pressure hold. Further, in one or more embodiments, the first pressure ramping is at a greater ramp rate than the second pressure ramping. Suitable ramp rates for the first pressure ramping (of the internal cell pressure increase) may include rates of 0.8 to 15 kbar/sec, and at least about 1, 2, 3, or 5 kbar/sec in one or more embodiments, and no more than about 14, 13, 10, or 8 kbar/sec in one or more embodiments, where any lower limit may be used in combination with any upper limit. Suitable ramp rates for the second pressure ramping may include rates of may include rates of 0.8 to 15 kbar/sec, and at least about 1, 1.2, 1.5, or 2 kbar/sec in one or more embodiments, and no more than about 6, 5, 4, or 3 kbar in one or more embodiments, where any lower limit may be used in combination with any upper limit. As mentioned above, these pressure ramp rates are the ramp rates for the internal cell pressure experienced by the diamond body, etc., not the external application of hydraulic pressure applied that will trigger internal cell pressure increases. The correlation between the hydraulic pressure applied and the internal cell pressure will vary for each cell configuration, as understood by those of ordinary skill in the art, and thus, because the internal cell pressure is what effects the infiltration process as it is the pressure experienced by the cell contents, it is the internal cell pressure that is discussed herein.

[0050] Further, suitable hold periods for the first pressure hold may range from about 20 seconds to up to 240 seconds, and may be less than 120 seconds or 60 seconds in particular embodiments, and suitable hold periods for the second pressure hold may range from about 40 seconds up to 320 seconds, but may be less than 240 seconds or 180 seconds in particular embodiments. It is also within the scope of the present disclosure that subsequent pressure stages may be reached in further step-wise fashion.

[0051] The temperature reached during the two-stage HPHT process may range, for example, from 1300 to 1600 °C, but also may depend on the melting temperature of the infiltrant material selected. The total pressure range for the two-stage HPHT process

may generally range from at least 40 kbar to 85 kbar, where the first pressure stage may range from 50 kbar to 65 kbar in one or more embodiments, (e.g., 57 kbar), and the second pressure stage may range from 60 kbar to 82 kbar in one or more embodiments. In one or more embodiments, the first pressure stage may have a lower limit of any of 40, 45, 50, 55, or 60 kbar, and an upper limit of any of 50, 55, 60, 65, or 68 kbar, where any lower limit can be used in combination with any upper limit. Further, in one or more embodiments, the second pressure stage may have a lower limit of any of 55, 60, 65, or 70 kbar, and an upper limit of any of 60, 65, 70, 75, 80, or 82 kbar, where any lower limit can be used in combination with any upper limit.

[0052] Further, like the ramp-hold-ramp pattern in the pressure increase, the HPHT conditions may also include an initial temperature ramping, a temperature hold, and a second temperature ramping, in one embodiment. In another embodiment, the high pressure/high temperature conditions may include a monotonic increase in temperature through the initial pressure ramping, the pressure hold, and the second pressure ramping. In yet another embodiment, the high pressure/high temperature conditions may include an initial temperature ramping during the initial pressure ramping and a temperature hold through the pressure hold and second pressure ramping. In one or more embodiments, a substantial majority of the temperature increase may occur during the first pressure ramping, where the second stage observes a temperature increase of less than 100 degrees, less than 75 degrees, less than 50 degrees, less than 25 degrees, or less than 20 degrees Celsius.

[0053] The cutting elements of the present disclosure may be incorporated in various types of cutting tools, including for example, as cutters in fixed cutter bits or on borehole enlargement tools such as reamers. Thus, the structure on which the cutting elements of the present disclosure may be installed may be referred to as a cutting element support structure, *i.e.*, a blade for fixed cutter bit or a reamer.

[0054] Referring now to FIG. 5, an embodiment of a fixed cutter drill bit 100 is shown. As shown in FIG. 5, drill bit 100 includes a bit body 110 having a threaded upper pin end 111 and a cutter face 112. The cutter face 112 may include a plurality of ribs or

blades 120 arranged about the rotational axis L of the drill bit and extending radially outward from the bit body 110. Cutting elements, or cutters, 150 are embedded in the blades 120 at predetermined angular orientations and radial locations relative to a working surface and with a desired back rake angle and side rake angle against a formation to be drilled. Cutters 150 are conventionally attached to a drill bit or other downhole tool by a brazing process so that the ultra hard cutting table faces into the direction of rotation of the bit. In the brazing process, a braze material is positioned between the cutter substrate and the cutter pocket. The material is melted and, upon subsequent solidification, bonds (attaches) the cutter in the cutter pocket.

[0055] A plurality of orifices 116 are positioned on the bit body 110 in the areas between the blades 120, which may be referred to as “gaps” or “fluid courses.” The orifices 160 are commonly adapted to accept nozzles. The orifices 160 allow drilling fluid to be discharged through the bit in selected directions and at selected rates of flow between the blades 120 for lubricating and cooling the drill bit 100, the blades 120 and the cutters 150. The drilling fluid also cleans and removes the cuttings as the drill bit 100 rotates and penetrates the geological formation. Without proper flow characteristics, insufficient cooling of the cutters 150 may result in cutter failure during drilling operations. The fluid courses are positioned to provide additional flow channels for drilling fluid and to provide a passage for formation cuttings to travel past the drill bit 100 toward the surface of a wellbore (not shown).

[0056] FIG. 6 shows a general configuration of a hole opener 830 that includes one or more cutting elements of the present disclosure. The hole opener 830 includes a tool body 832 and a plurality of blades 838 disposed at selected azimuthal locations about a circumference thereof. The hole opener 830 generally includes connections 834, 836 (e.g., threaded connections) so that the hole opener 830 may be coupled to adjacent drilling tools that include, for example, a drillstring and/or bottom hole assembly (BHA) (not shown). The tool body 832 generally includes a bore therethrough so that drilling fluid may flow through the hole opener 830 as it is pumped from the surface (e.g., from surface mud pumps (not shown)) to a bottom of the wellbore (not shown). The tool body 832 may be formed from steel or from other materials known in the art. For

example, the tool body 832 may also be formed from a matrix material infiltrated with a binder alloy.

[0057] The blades 838 shown in FIG. 6 are spiral blades and are generally positioned at substantially equal angular intervals about the perimeter of the tool body so that the hole opener 830. This arrangement is not a limitation on the scope of the invention, but rather is used merely to illustrative purposes. Those having ordinary skill in the art will recognize that any downhole cutting tool may be used.

[0058] 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 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. In the claims, means-plus-function clauses are intended to cover the structures described herein as performing the recited function and not only structural equivalents, but also equivalent structures. Thus, although a nail and a screw may not be structural equivalents in that a nail employs a cylindrical surface to secure wooden parts together, whereas a screw employs a helical surface, in the environment of fastening wooden parts, a nail and a screw may be equivalent structures. It is the express intention of the applicant 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.

CLAIMS

What is claimed:

1. A method of making a polycrystalline diamond cutting element, comprising:
placing a body of polycrystalline diamond comprising a matrix phase of bonded together diamond grains and a plurality of empty interstitial spaces between the bonded together diamond grains adjacent a first substrate material to form an assembly;
and
subjecting the assembly to high pressure/high temperature conditions that include an initial pressure ramping, a pressure hold, and a second pressure ramping.
2. The method of claim 1, further comprising:
placing a plurality of diamond particles adjacent a second substrate material comprising a catalyst material;
subjecting the plurality of diamond particles and the second substrate material to second high temperature/high pressure conditions to form a polycrystalline diamond body comprising a matrix phase of bonded together diamond grains and a plurality of interstitial spaces between the bonded together diamond grains occupied by the catalyst material bonded to the second substrate material;
removing the second substrate material from the polycrystalline diamond body; and
removing substantially all of the catalyst material from the interstitial spaces of the polycrystalline diamond body.
3. The method of claim 1, wherein the high pressure/high temperature conditions include an initial temperature ramping, a temperature hold, and a second temperature ramping.
4. The method of claim 1, wherein the high pressure/high temperature conditions include a monotonic increase in temperature through the initial pressure ramping, the pressure hold, and the second pressure ramping.

5. The method of claim 1, wherein the high pressure/high temperature conditions include an initial temperature ramping during the initial pressure ramping and a temperature hold through the pressure hold and second pressure ramping.
6. The method of claim 1, wherein the high pressure/high temperature conditions further include a second pressure hold following the second pressure ramping.
7. The method of claim 6, wherein the second pressure hold is longer than the first pressure hold.
8. The method of claim 1, wherein the first pressure ramping is at a greater ramp rate than the second pressure ramping.
9. The method of claim 1, wherein the first pressure ranges from about 50 to 70 kbar.
10. The method of claim 1, wherein the second pressure ranges from 70 to 82 kbar.
11. The method of claim 1, wherein the first substrate material comprises a plurality of carbide particles bonded together by a infiltrant material.
12. The method of claim 11, wherein during the subjecting step, the infiltrant material infiltrates into at least some of the empty interstitial spaces.
13. The method of claim 12, further comprising: removing at least a portion of the infiltrant materials from the interstitial spaces.
14. A method of forming a polycrystalline ultra-hard material, comprising:
 - placing a volume of ultra-hard material adjacent to a substrate material comprising a Group VIII-containing material to form an assembly;
 - subjecting the assembly to a first high pressure/high temperature condition sufficient to cause the Group VIII-containing material to melt and partially infiltrate the volume of ultra-hard material; and

subjecting the combination to a second high pressure/high temperature condition sufficient to cause the Group VIII-containing material to further infiltrate the volume of ultra-hard material,
the pressure of the second high pressure/high temperature condition is higher than that of the first high pressure/high temperature condition.

15. The method of claim 14, wherein the volume of ultra-hard material comprises a body of polycrystalline diamond comprising a matrix phase of bonded together diamond grains and a plurality of empty interstitial spaces between the bonded together diamond grains.
16. The method of claim 14, wherein the first high pressure/high temperature condition has a temperature maximum that is substantially the same as the second high pressure/ high temperature condition.
17. The method of claim 14, further comprising: holding the first high pressure/high temperature condition for a period of time prior to ramping to the second high pressure/high temperature condition.
18. The method of claim 14, wherein the volume of ultra-hard material comprises a mass of diamond particles.
19. The method of claim 14, further comprising:
placing a plurality of ultra-hard particles adjacent a second substrate material comprising a catalyst material;
subjecting the plurality of ultra-hard particles and the second substrate material to second high temperature/high pressure conditions to form the ultrahard material comprising a matrix phase of bonded together ultra-hard particle grains and a plurality of interstitial spaces between the bonded together ultra-hard particle grains occupied by the catalyst material bonded to the second substrate material;
removing the second substrate material from the polycrystalline diamond body; and
removing substantially all of the catalyst material from the interstitial spaces of the ultra-hard material.

20. A polycrystalline diamond compact, comprising:
- a polycrystalline diamond body comprising a matrix phase of bonded together diamond grains and a plurality of interstitial spaces between the bonded together diamond grains, the polycrystalline diamond body being substantially free of eruptions; and
 - a substrate attached to the polycrystalline diamond body at an interface,
- the polycrystalline diamond body comprising at least two regions, a first region adjacent the interface and a second region opposite the interface, the first region of the polycrystalline diamond body comprising an infiltrant material disposed within the interstitial spaces and being substantially free of a catalyst material used to form the polycrystalline diamond body, and the interstitial spaces in the second region of the polycrystalline diamond body being substantially free of the infiltrant material and the catalyst material used to form the polycrystalline diamond body.
21. A cutting tool, comprising: at least one polycrystalline diamond compact of claim 20 disposed thereon.

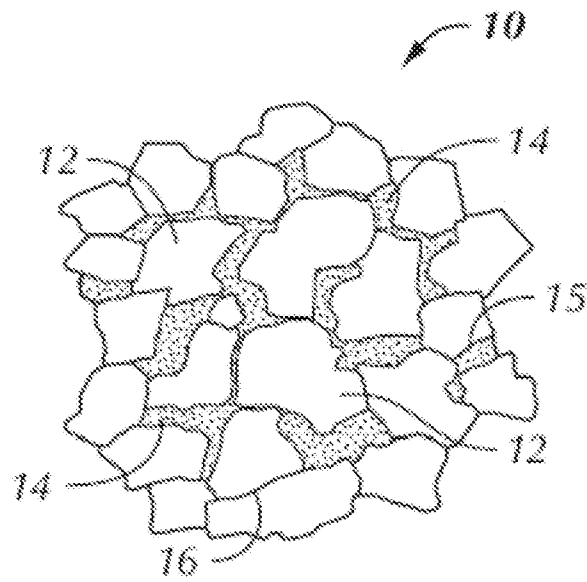


FIG. 1
(Prior Art)

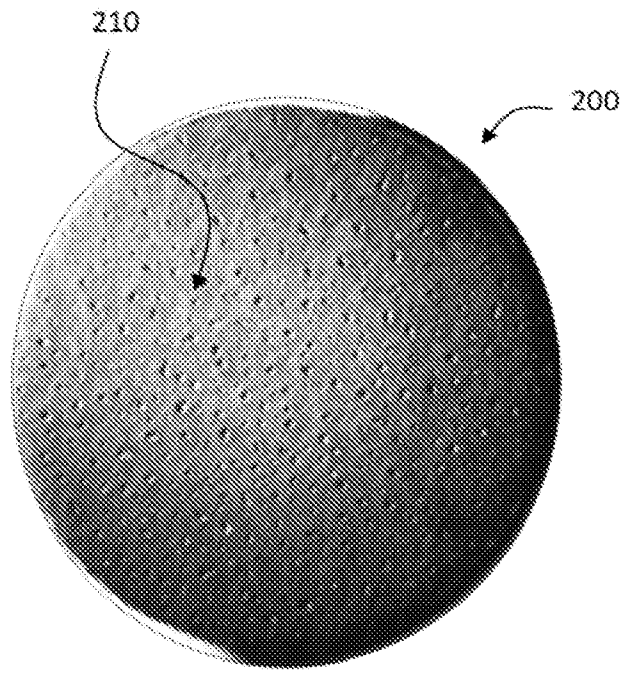


FIG. 2

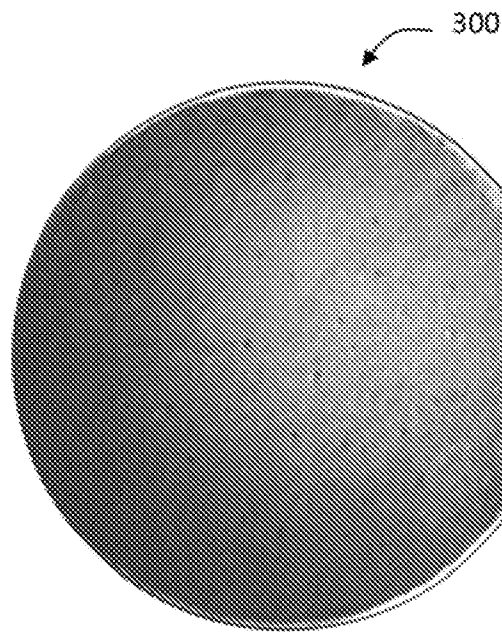


FIG. 3

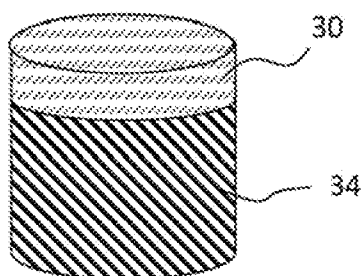


FIG. 4.1

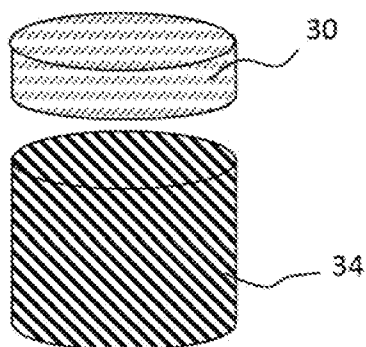


FIG. 4.2

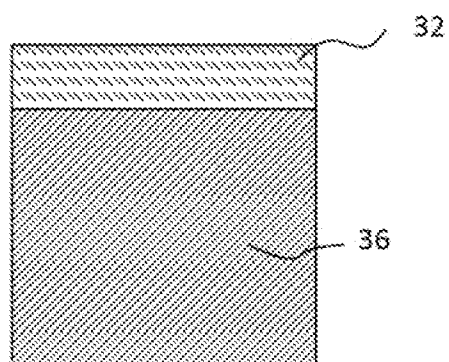


FIG. 4.3

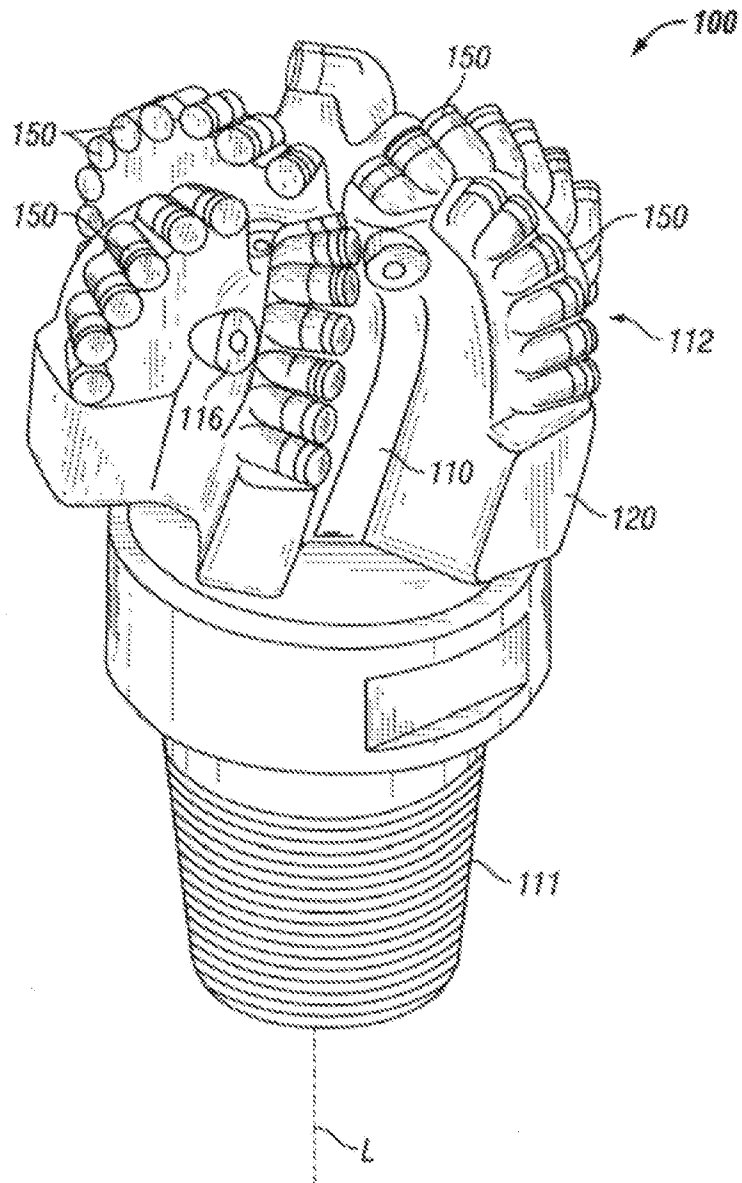


FIG. 5

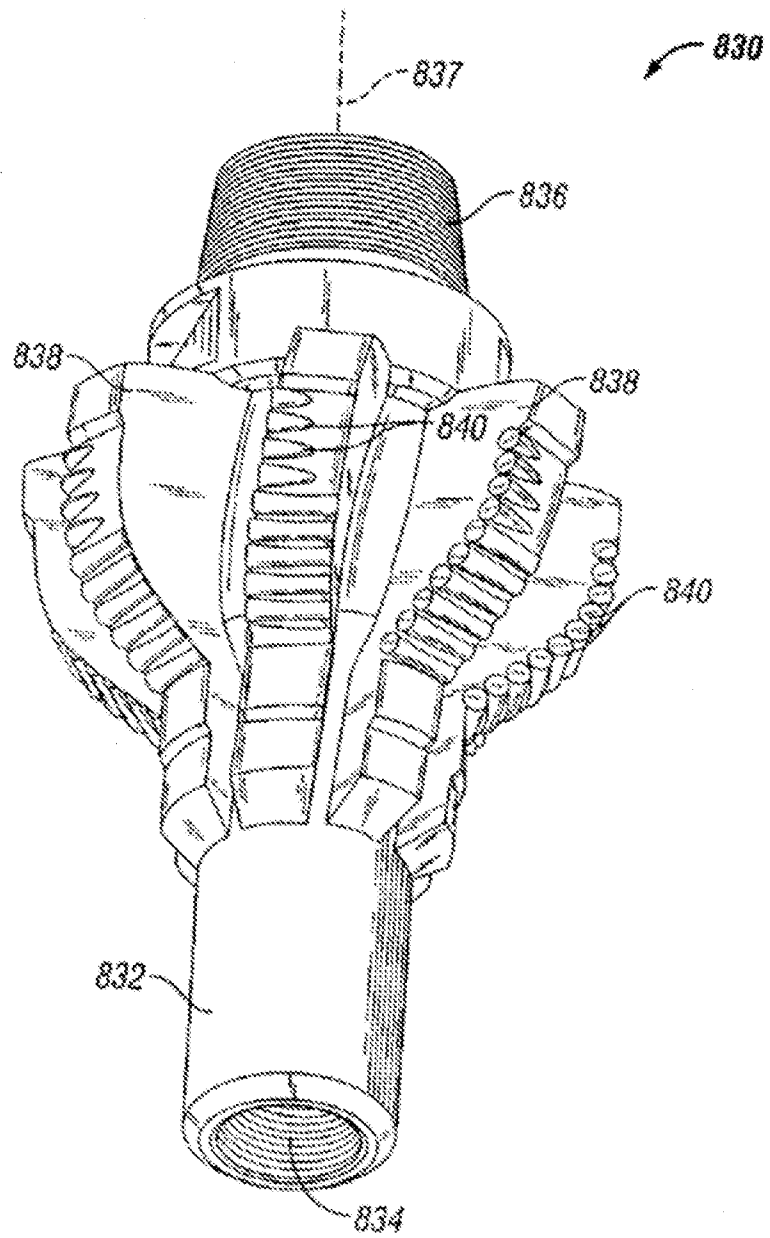


FIG. 6

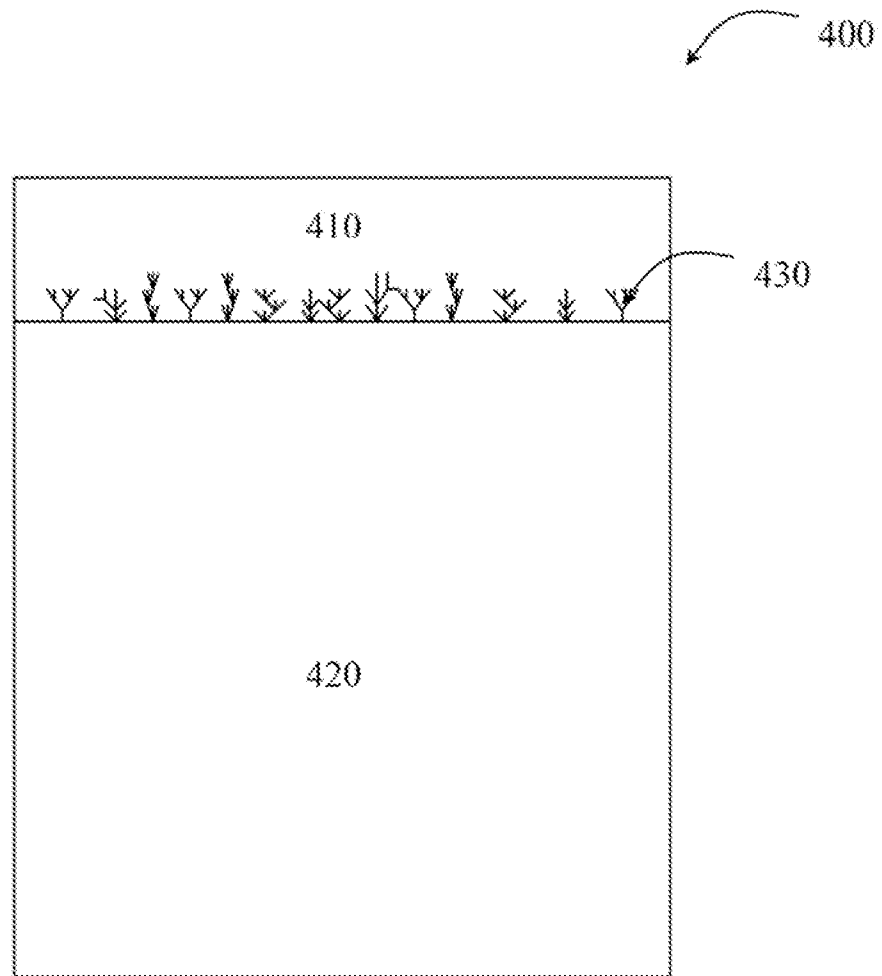


FIG. 7
(Prior Art)

INTERNATIONAL SEARCH REPORT

International application No.
PCT/US2013/071894**A. CLASSIFICATION OF SUBJECT MATTER****E21B 10/46(2006.01)i, B24D 3/10(2006.01)i**

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; B24D 3/10; E21B 10/36; B22F 7/02; B22F 7/04; B24D 7/14; B01J 3/06

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: PCD cutting element, ultra-hard material, high pressure/high temperature, pressure ramping, and substrate

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 2007-0144790 A1 (FANG et al.) 28 June 2007 See paragraphs [0007], [0028]-[0029], [0041]-[0057], [0069], [0078] and figures 2A, 6.	14-18, 20-21
Y		1, 3-13
A		2, 19
Y	US 6521174 B1 (BUTCHER et al.) 18 February 2003 See column 6, line 57 - column 7, line 6 and figure 5.	1, 3-13
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A	US 5510193 A (CERUTTI et al.) 23 April 1996 See column 5, line 60 - column 6, line 47 and figures 1-2.	1-21



Further documents are listed in the continuation of Box C.



See patent family annex.

* Special categories of cited documents:

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"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

13 March 2014 (13.03.2014)

Date of mailing of the international search report

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Information on patent family members

International application No.

PCT/US2013/071894

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