



(51) International Patent Classification:
C22C 26/00 (2006.01)

(21) International Application Number:
PCT/IB2010/050864

(22) International Filing Date:
1 March 2010 (01.03.2010)

(25) Filing Language: English

(26) Publication Language: English

(30) Priority Data:
0903344.0 27 February 2009 (27.02.2009) GB

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(81) Designated States (unless otherwise indicated, for every kind of national protection available): AE, AG, AL, AM, AO, AT, AU, AZ, BA, BB, BG, BH, BR, BW, BY, BZ, CA, CH, CL, CN, CO, CR, CU, CZ, DE, DK, DM, DO, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, GT,

HN, HR, HU, ID, IL, IN, IS, JP, KE, KG, KM, KN, KP, KR, KZ, LA, LC, LK, LR, LS, LT, LU, LY, MA, MD, ME, MG, MK, MN, MW, MX, MY, MZ, NA, NG, NI, NO, NZ, OM, PE, PG, PH, PL, PT, RO, RS, RU, SC, SD, SE, SG, SK, SL, SM, ST, SV, SY, TH, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, ZA, ZM, ZW.

(84) Designated States (unless otherwise indicated, for every kind of regional protection available): ARIPO (BW, GH, GM, KE, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM, ZW), Eurasian (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European (AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HR, HU, IE, IS, IT, LT, LU, LV, MC, MK, MT, NL, NO, PL, PT, RO, SE, SI, SK, SM, TR), OAPI (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG).

Declarations under Rule 4.17:

— as to applicant's entitlement to apply for and be granted a patent (Rule 4.17(ii))

Published:

— with international search report (Art. 21(3))
— before the expiration of the time limit for amending the claims and to be republished in the event of receipt of amendments (Rule 48.2(h))

(54) Title: POLYCRYSTALLINE DIAMOND

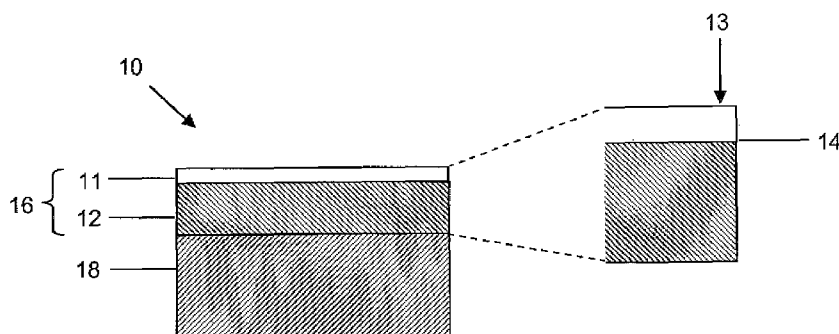


FIG 1

(57) Abstract: The invention relates to a polycrystalline diamond (PCD) body comprising PCD material having a graphitisation onset temperature, the PCD body having a working surface and comprising a first region remote from the working surface, the first region containing a catalysing material; and a second region extending a depth from the working surface into the PCD body, the second region being substantially free of catalysing material; the depth having a thermal gradient characteristic that when the temperature at the working surface is 900 degrees centigrade, the temperature at the depth is in the range from 780 degrees centigrade to 850 degrees centigrade and to inserts, machine tools and drill bits comprising the PCD body.

POLYCRYSTALLINE DIAMOND

5 Field

The invention relates to polycrystalline diamond (PCD) bodies, particularly but not exclusively for use in boring into the earth, high wear applications, machine tools, mining or other degradation of hard materials. More particularly, the PCD
10 bodies are for use in applications in which resistance to thermal degradation of the PCD bodies is valued.

Background

15 Polycrystalline diamond (PCD) is an example of a superhard, also called superabrasive, material comprising a mass of substantially inter-grown diamond grains, forming a skeletal mass defining interstices between the diamond grains. PCD material typically comprises at least about 80 volume % of diamond and may be made by subjecting an aggregated mass of diamond grains to an ultra-
20 high pressure of greater than about 5 GPa and temperature of at least about 1,200°C in the presence of a sintering aid. Suitable sintering aids for PCD may also be referred to as a solvent / catalyst material for diamond. Solvent / catalyst material for diamond is understood be material that is capable of promoting direct inter-growth of diamond grains at a pressure and temperature
25 condition at which diamond is thermodynamically more stable than graphite, and of promoting the conversion of diamond to graphite at ambient pressure. Examples of solvent / catalyst materials for diamond are cobalt, iron, nickel and certain alloys including any of these. PCD may be formed on a cobalt-cemented tungsten carbide substrate, which may provide a source of cobalt solvent /
30 catalyst for the PCD. The interstices within PCD material may be wholly or partially filled with binder or filler material, which may be solvent / catalyst material.

Components comprising PCD are used in a wide variety of tools for cutting, machining, drilling or degrading hard or abrasive materials such as rock, metal, ceramics, composites and wood-containing materials. For example, PCD
5 bodies are commonly used as cutter inserts on drill bits used for boring into the earth in the oil and gas drilling industry. PCD bodies are also used for machining and milling metal-containing bodies, such as may be used in the auto manufacturing industry. In many of these applications the temperature of the PCD material becomes elevated as it engages a rock formation, workpiece or
10 body with high energy. Examples of PCD composite abrasive compacts are described in US patents numbers 3,745,623; 3,767,371 and 3,743,489.

PCD is extremely hard and abrasion resistant, which is the reason it is the preferred tool material in some of the most extreme machining and drilling
15 conditions, and where high productivity is required. A disadvantage of PCD containing a solvent / catalyst material for diamond as a filler material may be its relatively poor thermal stability above about 400°C.

United States patent number 6,878,447 discusses that, due to the presence of
20 the binder-catalysing material, diamond is caused to graphitise as temperature increases, which typically limits its use to operation temperatures of up to about 750 degrees centigrade. The patent discloses a polycrystalline diamond element comprising a body with a working surface, wherein a first volume of the body remote from the working surface contains a catalysing material, a second
25 volume of the body adjacent to the working surface is substantially free of the catalysing material to a depth from the working surface, wherein a thermal gradient of the bonded diamonds causes a 950 degrees centigrade temperature at the working surface to be less than 750 degrees centigrade at the depth.

30 United States patent number 7,377,341 discloses a thermally stable ultra-hard compact construction comprising a body formed from an ultra-hard material, such as diamond, having a thermally stable region positioned adjacent a

working surface of the body, and a metallic substrate connected to the body. The thermally stable region may be formed from a material selected from the group consisting of consolidated materials that are thermally stable at temperatures greater than about 750 degrees centigrade, or even greater than
5 about 1,000 degrees centigrade and that are substantially free of a catalyst material. The body and metallic substrate are joined together by high pressure / high temperature process.

Summary

10

The purpose of the invention is to provide a thermally stable PCD body having enhanced impact toughness and reduced manufacturing cost. More particularly, the purpose is to provide a PCD body comprising a thermally stable stratum that is as thin as possible while maintaining key benefits of having a thermally stable
15 stratum.

20

As used herein, a "working surface" of a body is any part of the body which may in use contact a workpiece or other body being worked. It is understood that any portion of a working surface is also a working surface.

25

As used herein, the "graphitisation onset temperature" is the lowest temperature at which at least 10 percent by mass of the diamond within at least a portion of a given PCD body converts into a form of non-diamond carbon, such as graphitic carbon, after thirty minutes at the temperature.

30

As used herein, "catalysing material" means a material that is capable of promoting the sintering and inter-bonding of diamond grains to form polycrystalline diamond material at a temperature and pressure at which diamond is thermodynamically more stable than graphite. Examples of
catalysing material are cobalt, iron, nickel and manganese, and certain alloys including any of these.

As used herein, the phrases "substantially free" or "substantial absence" in relation to catalysing material within a PCD body means that a volume or the interstices within a volume are substantially but not necessarily completely devoid of catalysing material, any content of catalysing material that may be present being sufficiently low that it does not materially promote the degradation of a region of PCD in which it is present. Some of the diamond surfaces within the volume may still be in contact with catalyst material remaining within the proximate interstices.

- 5
- 10 A first aspect of the invention provides a polycrystalline diamond (PCD) body comprising PCD material having a graphitisation onset temperature, the PCD body having a working surface and comprising a first region remote from the working surface, the first region containing catalysing material, and a second region extending a depth from the working surface into the PCD body, the
- 15 second region being substantially free of catalysing material; the depth having a thermal gradient characteristic that when the temperature at the working surface is about 900 degrees centigrade, the temperature at the depth is in the range from about 780 degrees centigrade to about 850 degrees centigrade, or a temperature at the depth in the range from about 800 degrees centigrade to
- 20 about 820 degrees centigrade, or even a temperature at the depth within 10 degrees centigrade either side of the graphitisation onset temperature.

Embodiments of the invention have the advantage that the substantial absence of catalysing material within the PCD body volume defined by the depth (the

25 second region) thus determined results in significantly improved thermal stability of the PCD body (compact) proximate the working surface while avoiding excessive and unnecessary removal of catalysing material to a greater depth. Such excessive removal adds unnecessary cost to the manufacturing process and is believed to result in inferior impact strength.

30

In one embodiment, the depth is in the range from about 20 to about 90 microns from the working surface, or the depth is in the range from about 40 to about 70 microns from the working surface.

- 5 In one embodiment, bonded diamond grains of the PCD body have a size distribution in which more than one peak or mode is evident. In one embodiment, the bonded diamond grains have a size distribution characteristic that at least 20% of the grains have an average size greater than 10 microns, and at least 10% of the grains have an average size in the range from 10 to 20
10 microns. In one embodiment, the bonded diamond grains have an average size in the range from 5 microns to 20 microns.

Embodiments of the invention have the advantage of enhanced working life and cutting or penetration rate of the PCD body in rock drilling or earth boring
15 applications, and shear cutting rock drilling in particular.

A second aspect of the invention provides an insert for a machine tool or drill bit, the insert comprising an embodiment of a PCD body according to the present
20 invention.

In one embodiment, the insert comprises a PCD body joined to a cemented carbide substrate, and in one embodiment, the PCD body is part of a cutter insert for use in boring into the earth, particularly for drilling oil and gas wells. In another embodiment, the PCD body is part of an insert for use in pavement
25 degradation, mining, machining, including turning, milling, drilling or in certain other wear applications.

A third aspect of the invention provides a machine tool or drill bit comprising an embodiment of an insert according to the second aspect of the present
30 invention.

Embodiments of the PCD bodies according to the present invention have the advantage that they exhibit extended operating lives or cutting rates.

Drawings

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Non-limiting embodiments will now be described with reference to the figures, of which

FIG 1 shows a schematic longitudinal cross sectional view of an embodiment of a PCD body, as well as a magnified view of a part of the cross section.

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FIG 2 shows a schematic longitudinal cross sectional view of an embodiment of a PCD body, as well as a magnified view of a part of the cross section.

FIG 3 shows a frequency graph of number of diamond grains versus grain size for an embodiment of a PCD body.

15

The same reference numbers refer to the same features in all drawings.

Detailed description of embodiments

20

Where meanings of terms used herein are not sufficiently clear, a term or terms should be understood to have the same meaning as in US patent number 6,878,447.

25

With reference to FIG 1 and FIG 2, embodiments of PCD inserts 10 each comprise a PCD body 16 comprising PCD material, the bodies integrally bonded to respective cobalt-cemented tungsten carbide substrates 18, the PCD material having a respective graphitisation onset temperature, and the PCD body 16 having a working surface 13. A first region 12 of the PCD body 16 remote from the working surface 13 contains a catalysing material (not shown) and a second region 11 of the body 16 adjacent to the working surface 13 is substantially free

30

of catalysing material to a depth 14 from the working surface 13. The depth 14 is such that when a portion of the working surface 13 is heated to 900 degrees centigrade, the temperature at the depth 14 is within ten degrees centigrade of the graphitisation onset temperature of the PCD material and in the range from 5 780 to 850 degrees centigrade. In one embodiment, the depth is in the range from about 60 microns to about 80 microns.

With reference to FIG 3, an embodiment of a PCD body comprises PCD material in which the sintered diamond grains have a size distribution that can 10 be resolved into more than one distribution, each having a single different peak or mode. Such distributions may be referred to as multimodal distributions. In this embodiment, the mean size of the diamond grains is in the range from about 5 to about 20 microns and the size distribution can be resolved into at least three distinct peaks.

15

The size distribution of the diamond grains within PCD material is measured by means of image analysis carried out on a polished surface of a PCD body. A Saltykov correction may be applied to convert the size distribution obtained from the two-dimensional image data to a particle size distribution in three 20 dimensions, as is standard practice. As used herein, particle size is in terms of the equivalent circle diameter of the particle, determined as the diameter of a circle having an area equal to the area of a shape of a cross section of a particle.

25 The polycrystalline diamond element may be made using an ultra-high pressure sintering method as is well known in the art, in which an aggregate mass of diamond particles is subjected to a pressure of at least about 5.5GPa and a temperature of at least about 1,250 degrees centigrade in the presence of a catalysing material. During this step, catalysing material such as molten cobalt 30 is allowed to infiltrate into an aggregate mass of diamond grains, which may be formed into an unbonded, or at least a weakly bonded, porous pre-form comprising diamond grains. The catalysing material infiltrates and fills

- substantially all of the pores or interstitial regions within the aggregate mass, promoting the intergrowth of the diamond grains to form a sintered PCD body. After the sintering step, catalysing material is removed from a region adjacent a working surface of the PCD body by any of various means known in the art, for example by using an acid liquor (e.g. hydrofluoric acid, nitric acid or mixtures thereof) to leach out catalysing material. Other exemplary methods are disclosed in international patent publication number WO2007042920, and in South African patent publication number 2006/00378.
- 10 The graphitisation onset temperature is found by subjecting each of a set of samples of a type of PCD body (element) to heat treatment at various temperatures within the range from about 760 degrees centigrade to about 850 degrees centigrade in air for thirty minutes. It is important that the samples are substantially identical to each other in terms of the nature and size distribution of diamond grains, the type and content of solvent / catalyst material, sinter quality, and were manufactured using substantially the same process and process conditions. It is also important that the PCD body has at least a portion from which the solvent / catalyst has not been removed or depleted, since this is the portion that must be inspected and analysed for evidence of conversion of diamond to graphite. It is not necessary to deplete any portion of the samples of solvent / catalyst material when carrying out the process of determining the graphitisation onset temperature, and in general it is preferable not to do so. After each heat treatment, the treated sample is removed and subjected to micro-Raman analysis to determine whether there is evidence that diamond grains, or at least portions of diamond grains have converted to graphite or other non-diamond carbon. Micro-Raman spectroscopy is suitable for measuring the levels of diamond, graphite and other non-diamond carbon. Graphite appears as a distinct peak at about 1,580 inverse centimetres and other non-diamond carbon appears as a peak to the left of the diamond peak. In some cases, this will be evident from mere visual inspection. If more than about 10 volume percent of the diamond within conversion has occurred, this means that temperature used in the heat treatment of that sample exceeded the critical

degradation temperature (graphitisation onset temperature). It has been found that the temperature range over which a very substantial portion of the diamond converts to graphite is rather narrow, and was about 15 degrees centigrade in one example. The amount of diamond to graphite conversion over about 20
5 degrees centigrade may be so extensive that very little, if any diamond can be detected within a portion of the heat treated sample.

Once the graphitisation onset temperature has been determined for a given grade or type of PCD body, the depth from a working surface from which solvent
10 / catalyst material should be depleted must be determined. This may be done by means of a wear test known in the art. For example, a well-known test is to engage a cutting edge of an element with a workpiece of rock or other abrasive material moving at high speed relative to the element, and so to effect a cutting action on the workpiece. The time period of the engagement may be varied
15 depending on the type of workpiece material and its speed of relative movement, among other factors, as would be appreciated by the person of ordinary skill. Such tests are known to be carried out in various ways, such as "log turning", in which a generally cylindrical workpiece is caused to rotate about its cylindrical axis on a lathe, for example, or a "vertical borer" test, in which the
20 workpiece has a generally disc-like form and is caused to rotate about a substantially vertical axis. The action of cutting the workpiece results in the wear of the element proximate the cutting edge and the formation of a wear scar, the dimensions of which may readily be measured. The depth of the wear scar may be used as an indicator of the wear resistance and likely performance
25 of an element in application, all else being equal.

A wear test of the kind described above and which is known in the art may be used to determine whether a depth associated with a second volume according to the invention has been achieved within a polycrystalline diamond body
30 (element). For example, a (body) element having a second volume from which catalyst material has been substantially removed or depleted to a depth may be subjected to a wear test wherein the relative speed of the workpiece is

increased to the point where the temperature or proximate the cutting edge is 900 degrees centigrade. The achievement of such a temperature may be determined by observation of the colour of the light emitted from the cutting edge, which glows white-hot at this temperature. A more accurate
5 measurement of the temperature may be achieved by means of an optical pyrometer, which is a device that measures temperature by analysing the frequencies of light given off by glowing-hot bodies. After the wear test, once the cutting edge has been allowed to cool down, the element is removed from the test apparatus and the wear scar dimensions, its length and depth in
10 particular, are measured. It has been found that wear scar dimensions are substantially greater for elements having a second volume depth less than that according to the invention. This is believed to be because a first portion proximate the second volume at the depth is degraded by conversion of diamond to graphite or other non-diamond carbon at temperatures above about
15 815 degrees centigrade. Evidence of such degradation or conversion may be measured at or proximate the depth by means of Raman or micro-Raman spectroscopy, or may be evident from visual inspection under a microscope, and provides an indication that the depth of the tested sample is too shallow, since the temperature at the depth during the test was too high. In order to determine
20 a correct depth, the test should be repeated using elements having various different depths, using a trial-and-error proximation approach.

Unexpectedly, it has been found that there is not a single value for the graphitisation onset temperature corresponding to all PCD bodies, and that the
25 this temperature depends on factors such as the size distribution of the diamond grains, the type and content of the catalysing material and the quality of the sintering of the diamond grains. It believed that certain improved PCD bodies as have been developed in the recent past have higher graphitisation onset temperature than previous PCD bodies.

30

An embodiment of the invention is described in more detail with reference to the example below, which is not intended to limit the invention.

Example

Fifteen polycrystalline diamond (PCD) bodies (compacts) having multimodal diamond grain size distribution as shown in FIG 3 were manufactured in a conventional way using a ultra-high pressure, high temperature sintering process. The compacts were substantially cylindrical in shape, having a diameter of about 16mm. The compacts comprised a layer of PCD integrally bonded onto a cobalt-cemented tungsten carbide (WC) substrate, the PCD layers being 2.2mm thick. The diamond content of the PCD layer was about 92% by volume, the balance being cobalt and minor precipitated phases such as WC.

The input diamond powder was prepared by blending diamond powders having different average sizes, the size distribution of the grains within the resulting blended powder have the size distribution characteristic that 9.77 wt.% of the grains had average grain size less than 5 microns, 7.64 wt.% of the grains had average size in the range from 5 microns to 10 microns, and 82.58 wt.% of the grains had average grain size greater than 10 microns. The blended powder was formed into an aggregated mass, which was integrally bonded to a cobalt-cemented tungsten carbide (WC) substrate during the sintering step, as is conventional in the art. During this step, cobalt from the substrate infiltrated the aggregated mass of diamond grains, filling pores between them and promoting the intergrowth of the diamond grains, resulting in a polycrystalline diamond compact bonded to a substrate. The diamond grains within the PCD thus produced had a multimodal size distribution having the characteristic that 34.7 wt.% of the grains had average grain size less than 5 microns, 40.4 wt.% of the grains had average size in the range from 5 microns to 10 microns, and 24.9 wt.% of the grains had average grain size greater than 10 microns. The grain size distribution of the sintered PCD is different from that of the input grains due to mutual crushing of the grains at high pressure, in addition to the shift towards coarser grain sizes that normally occurs during the sintering process.

Polycrystalline diamond bodies (compacts) comprising diamond grains having a size distribution with more than one peak, each corresponding to a respective "mode", have been found to have advantageous properties. So-called "multimodal" PCD is disclosed in US patents 5,505,748 and 5,468,268. Multimodal polycrystalline bodies are typically made by providing more than one source of a plurality of grains, each source comprising grains having a substantially different average size, and blending together the grains or particles from the sources. Measurement of the size distribution of the blended grains typically reveals distinct peaks corresponding to distinct modes. The blended grains are then formed into an aggregate mass and subjected to a sintering step at high or ultra-high pressure and elevated temperature, typically in the presence of a sintering agent. The size distribution of the grains is further altered as the grains are compacted against one another and fractured, resulting in the overall decrease in the sizes of the grains. Nevertheless, the multimodality of the grains is usually still clearly evident from image analysis of the sintered article.

Nine of the compacts were used to determine the minimum graphitisation temperature. The PCD layer was detached from the carbide substrate of each of the nine compacts by means of electro-discharge machining (EDM), yielding nine un-backed PCD discs, each about 2.2mm thick. Each of the PCD discs was treated in air for thirty minutes at a different temperature in the range from 650 degrees centigrade to 850 degrees centigrade, specifically at 650, 700, 730, 750, 770, 800, 815, 825 and 850 degrees centigrade. After heat treatment, each sample was fractured to expose a cross-section, the exposed cross-section then being visually examined and subjected to micro-Raman analysis in order to detect the presence of diamond and / or graphite. An external surface of each sample was similarly examined to determine whether a different result would be observed for the fracture and external surfaces.

In the cases of all samples treated at temperatures in the range of 650 degrees centigrade to 800 degrees centigrade, no evidence of graphite was detected and the diamond had clearly not degraded to any significant degree. However, substantially all of the diamond had converted to graphite in the samples treated at temperatures in the range from 815 degrees centigrade to 850 degrees centigrade. This indicated that the minimum graphitisation temperature was in the range from 800 degrees centigrade to 815 degrees centigrade.

The PCD faces or end working surfaces (i.e. the flat end faces of the PCD, coterminous with the working end of the cylindrical compacts) of each of six of the remaining compacts were then polished and treated with acid to remove substantially all of the cobalt from different respective depths. This was done by masking the major portion each compact by suitable means as is known in the art, leaving only that portion exposed from which the cobalt was to be removed. The masked samples were bathed in hot hydrofluoric / nitric acid for about 3.5 hours at a temperature close to the boiling point of the acid under ambient pressure, which was sufficient to remove substantially all of the cobalt from the exposed portion. A different volume of PCD was exposed for each sample, the depth of the exposed portion being different in each case. This resulted in the cobalt in each sample being removed to a different depth from the flat PCD face, the depths being in the range from 40 microns to 140 microns in steps of about 20 microns. Six "leached" compacts were thus produced.

A further ten PCD compacts were used to establish suitable wear test conditions under which to determine which of the leached samples displayed the required thermal character proximate a leached working surface. The wear test involved causing a PCD compact to engage a rapidly rotating log of sandstone mounted onto a lathe, a working edge (surface) of the PCD cutting into the sandstone. As is typical in such tests, the engaged working edge glowed red hot initially. The speed of rotation of the sandstone log was increased to the point at which the working edge glowed white hot, indicating that 950 degrees centigrade had been achieved.

Once the required turning speed had been determined, each of the six leached PCD compacts was subjected to the wear test for three minutes using that speed. After each test, the PCD at the leached depth was visually inspected
5 and subjected to micro-Raman analysis in order to detect the presence of substantial graphite. Using this method, the sample with the optimum leach depth was identified, this depth being in the range from 40 to 60 microns.

Further compacts were the prepared having a leach depth of 50 microns from
10 the flat working surfaces and subjected to standard performance wear tests. Unfortunately it was not possible to compare directly the performance of these compacts with those having the leached depth characteristic disclosed in the prior art, since the prior art asserts that the temperature at the depth should be less than 750 degrees centigrade. Since the graphitisation temperature for the
15 PCD grade used in this example was found to be in the range from 800 degrees centigrade to 815 degrees centigrade, there was no marker or means of identifying a depth at which 750 degrees centigrade was achieved. Nevertheless, the performance wear test results of the optimum compacts made according to this example provided qualitative positive indication that they were
20 at least as effective as the best available in the art, while having the shallower leach depth than any compact known in the art.

Claims

1. A polycrystalline diamond (PCD) body comprising PCD material having a graphitisation onset temperature, the PCD body having a working surface and comprising a first region remote from the working surface, the first region containing catalysing material; and a second region extending a depth from the working surface into the PCD body, the second region being substantially free of catalysing material; the depth having a thermal gradient characteristic that when the temperature at the working surface is 900 degrees centigrade, the temperature at the depth is in the range from 780 degrees centigrade to 850 degrees centigrade.
2. A PCD body as claimed in claim 1, in which the temperature at the depth is in the range from 800 degrees centigrade to 820 degrees centigrade.
3. A PCD body as claimed in claim 1 or claim 2, in which the temperature at the depth is within 10 degrees centigrade either side of the graphitisation onset temperature.
4. A PCD body as claimed in any of the preceding claims, in which the depth is in the range from 20 to 90 microns from the working surface.
5. A PCD body as claimed in any of the preceding claims, in which the bonded diamond grains have a size distribution in which more than one peak or mode is evident.
6. A PCD body as claimed in any of the preceding claims, in which bonded diamond grains of PCD body have a size distribution characteristic that at least 20% of the grains have an average size greater than 10 microns, and at least 10% of the grains have an average size in the range from 10 to 20 microns.

7. A PCD body as claimed in any of the preceding claims, in which the bonded diamond grains have an average size in the range from 5 microns to 20 microns.

5 8. An insert for a machine tool or drill bit, comprising a PCD body as claimed in any of claims 1 to 7.

9. A machine tool or drill bit comprising an insert as claimed in claim 8.

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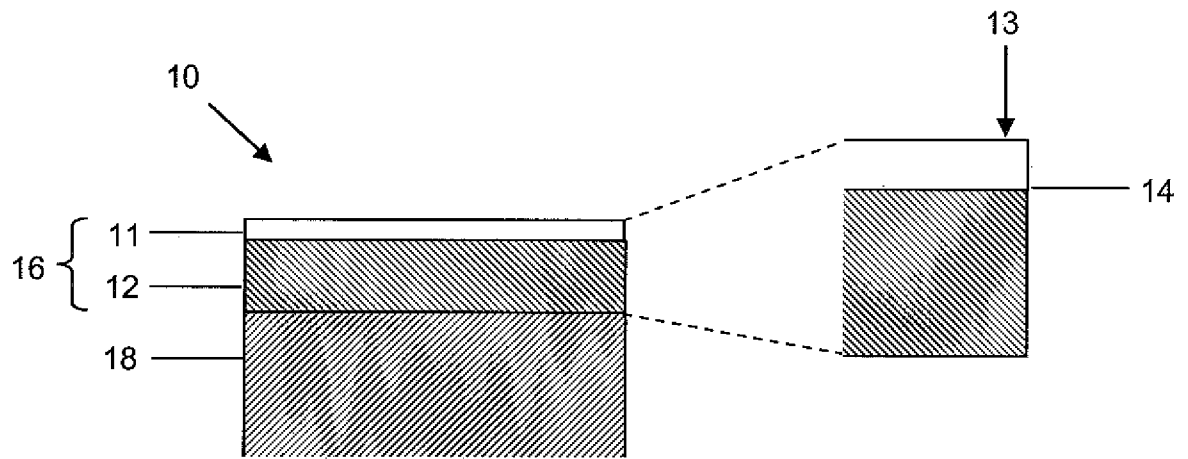


FIG 1

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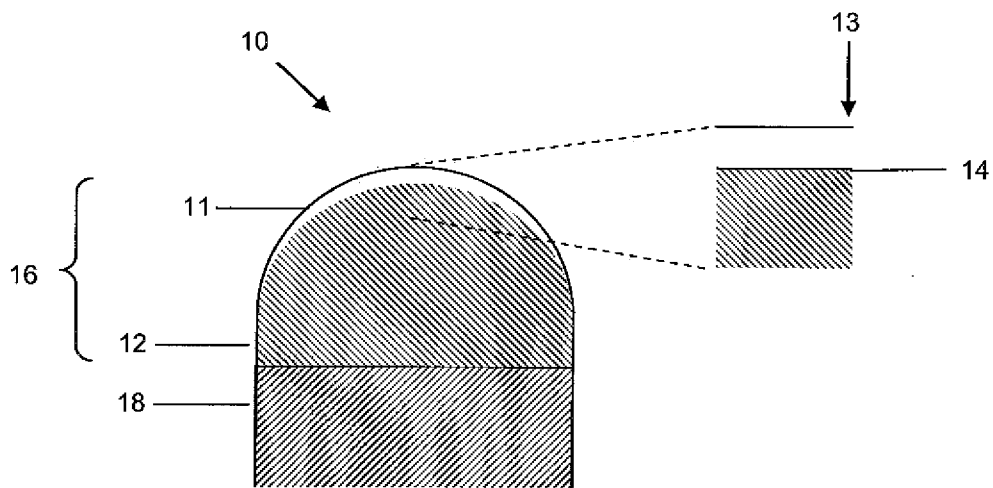


FIG 2

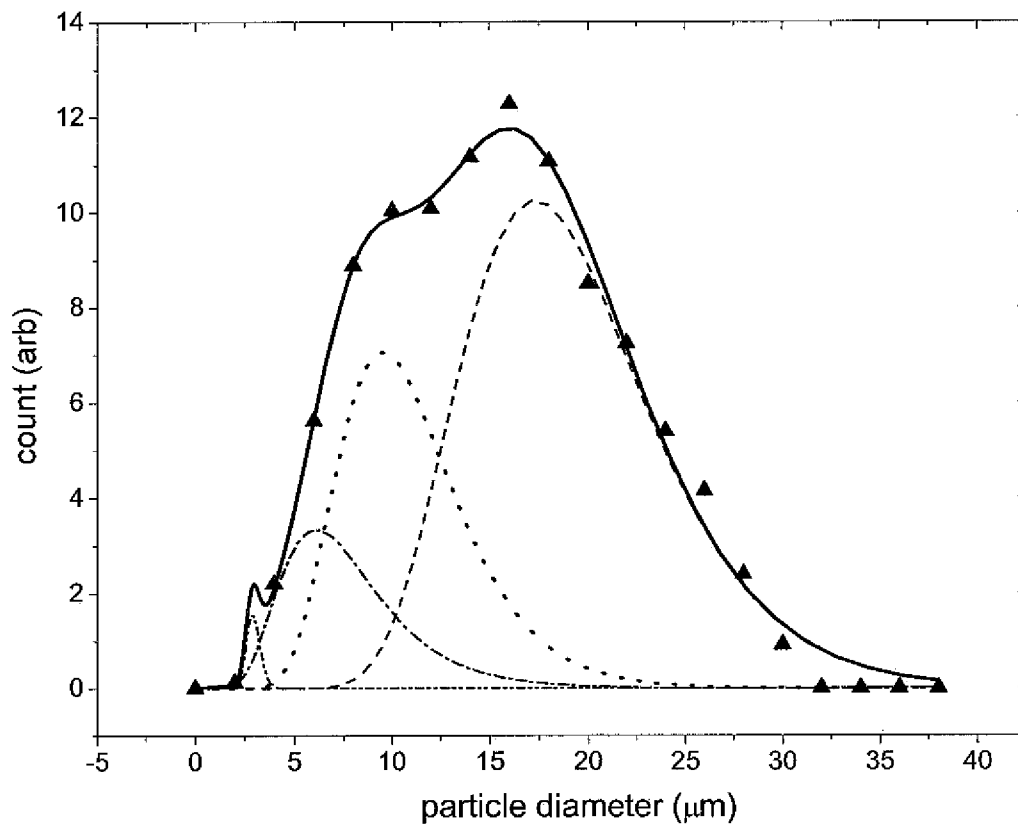


FIG 3

INTERNATIONAL SEARCH REPORT

International application No

PCT/IB2010/050864

A. CLASSIFICATION OF SUBJECT MATTER

INV. C22C26/00

ADD.

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)

C22C

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

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

EPO-Internal

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 2005/129950 A1 (GRIFFIN NIGEL D [GB] ET AL) 16 June 2005 (2005-06-16) page 4, paragraph 60 - page 6, paragraph 82; claims 1-48; figures 1-18 -----	1-9
X	US 2005/115744 A1 (GRIFFIN NIGEL D [GB] ET AL) 2 June 2005 (2005-06-02) page 5, paragraph 65 - page 7, paragraph 88; claims 1-31; figures 1-18 -----	1-9
X	US 2008/010905 A1 (EYRE RONALD K [US]) 17 January 2008 (2008-01-17) claims 1-25; figures 1-12; examples 1-2 -----	1-9
X	EP 1 190 791 A2 (CAMCO INT UK LTD [GB]) 27 March 2002 (2002-03-27) claims 1-50; figures 1-36 -----	1-9



Further documents are listed in the continuation of Box C.



See patent family annex.

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Date of the actual completion of the international search

15 June 2010

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INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No

PCT/IB2010/050864

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