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(54) **COMPOSITE MATERIAL**

(57) A composite material comprising a cemented carbide matrix embedded with diamond entities which are homogeneously distributed throughout; wherein the constituents of the cemented carbide include a metal carbide phase and a binder phase; wherein the grain size of the metal carbide is 0.6-8 microns; wherein the material comprises 5-65 vol% diamond entities; characterized in that: at least 20% of the diamond entities contains pores and / or cracks that are filled with constituent(s) of the cemented carbide.

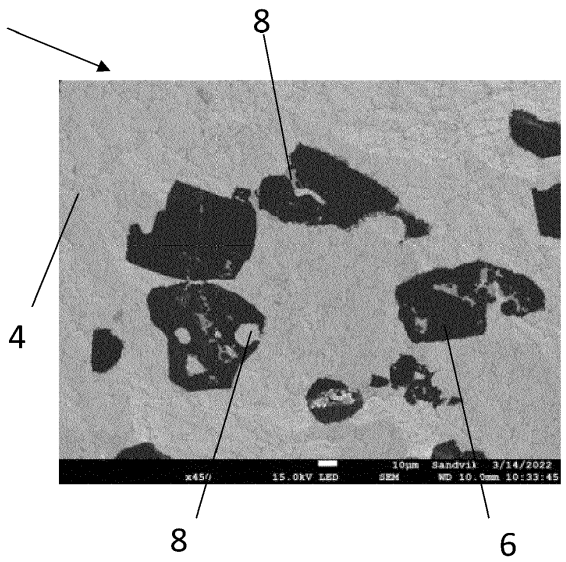


Fig 1b

EP 4 385 644 A1

Description

TECHNICAL FIELD

[0001] The present invention relates to a composite material comprising diamond entities in a cemented carbide matrix for use as inserts in mining or rock cutting applications or wear parts and a method of producing the same.

BACKGROUND

[0002] PCD (poly crystalline diamond) is well known for being highly wear resistant, making it a popular choice for making industrial applications. However, for toughness demanding mining applications, such as inserts for percussive and rotary drilling (with the exception of oil and gas) or mechanical cutting, PCD works less well due to its brittle behaviour, which limits the lifespan of the inserts. Cemented carbide has a unique combination of high elastic modulus, high hardness, high compressive strength, high wear and abrasion resistance together with a good level of toughness. Therefore, cemented carbide is commonly used in products such as mining and cutting inserts. Cemented carbide comprises a hard ceramic (carbide) phase and a binder phase.

[0003] It is desirable to combine the high wear resistance of the PCD with the toughness of the cemented carbide. Therefore, composite materials comprising diamond in a cemented carbide matrix have been developed, such as those disclosed in US7647992 and WO2009128034. However, known composite materials comprising diamond and cemented carbide are not so well balanced regarding the wear resistance to toughness relation making them prone to breaking and unreliable when for example used for mining inserts.

[0004] The best performing prior art PCD-inserts used for percussive drilling have layers with different diamond concentrations in the domed region of the insert, however the problem with this is that it demands a rather complex manufacture procedure which makes the material very expensive. The problem to be solved is how to provide a new material that is able to provide high performance without excessive manufacturing costs.

DEFINITIONS

[0005] By "cemented carbide" is herein meant a material that comprises at least 50 wt% tungsten carbide, possibly other hard constituents common in the art of making cemented carbides and a metallic binder phase preferably selected from one or more of Fe, Co and Ni.

[0006] By "HPHT" is herein meant a "High Pressure High Temperature" process with pressures about at or above the diamond stable region (>50 kBar) and with temperatures of at least about 1000 C.

[0007] By "SCCG" is herein meant sintered cemented carbide granules wherein each granule the cemented carbide is at least 90% dense, preferably fully dense.

[0008] By "diamond entity" is herein meant either a single diamond grain or two or more diamond grains bonded together, i.e. a diamond cluster.

[0009] By "homogenously distributed throughout" is herein meant that the diamond entities are evenly distributed throughout the composite material and that no distinguishable pattern in the distribution of the diamond entities can be seen. Examples of distinguishable patterns could be a gradient in either size, volume or number of the diamond entities or that the material contains satellite structures wherein there would be a plurality of the smaller diamond entities surrounding a larger diamond entity.

SUMMARY OF INVENTION

[0010] According to a first aspect of the present invention there is a composite material comprising a cemented carbide matrix embedded with diamond entities which are homogeneously distributed throughout; wherein the constituents of the cemented carbide include a metal carbide phase and a binder phase; wherein the average grain size of the metal carbide is 0.6- 8 microns; wherein the material comprises 5-65 vol% diamond entities; characterized in that: at least 20% of the diamond entities contains pores and / or cracks that are filled with constituent(s) of the cemented carbide.

[0011] Advantageously, this provides a composite material wherein the high wear resistance has been maintained with increased toughness, therefore providing a material that is more reliable and less prone to chipping and breaking. Furthermore, the properties of both the diamond parts and the surrounding cemented carbide matrix can be tailored to suit the application the material is being used for. The toughness of the composite material is further enhanced from the presence of binder in pores and / or cracks of the diamond entities. The wear resistance is maintained due to the reduction in diamond crystal size and from the formation of new diamond to diamond bonds. This material provides the same performance as the best performing prior art PCD-inserts that are currently used for percussive drilling which layers with different diamond concentrations in the domed region of the insert but at a lower cost due to that fact the diamond content

is lower and the manufacturing process is simpler.

[0012] According to another aspect of the present application there is an insert for a mining or rock cutting or wear part application comprising the material as described hereinbefore or hereinafter.

[0013] Advantageously, if the material of the present invention is used for inserts for mining, rock cutting or wear parts applications, the lifetime of the inserts will be increased due to the wear resistant nature of the material in combination with the increased toughness.

[0014] According to another aspect of the present invention there is a method for making a material as described hereinbefore or hereinafter comprising the steps of:

a) providing friable diamond grains;
b) providing sintered granules of cemented carbide;
c) blending the friable diamond grains with the sintered cemented carbide granules to form a homogenous powder blend;
d) placing the powder blend into preformed refractory metal cup;
e) providing a refractory metal lid, a pre-sintered or sintered cemented carbide base on top of the powder blend to close the cup;
f) pre-compacting the powder in the refractory metal cup;
g) surrounding the cup with a pressure media;
h) inserting the pressure media surrounded cup into a high pressure high temperature container;
i) placing the above container in a high pressure high temperature press and sintering at high pressure and high temperature to form a composite material.

[0015] Advantageously, this results in a more homogenous blend which provides uniform properties throughout the volume of the material, higher powder density, reduced and more controllable shrinkage during HPHT sintering cycle which means that there is greater control over the final shape of the product being produced. Further, it means that the properties of the cemented carbide matrix can be steered when the granules are made prior rather than during the HPHT sintering which ultimately results in greater control over the properties of the material. Further, it reduces the risk that the diamond entities dissolve and break which form satellites which result in nonuniform properties. The composition and the sintering temperature of the cemented carbide granules defines the final WC-Co structure in terms of grain size, binder content and properties of the cemented carbide matrix.

BRIEF DESCRIPTION OF DRAWINGS

[0016]

Figure 1a: SEM image of the composite material F at x140 magnification.

Figure 1b: SEM image of the composite material F at x450 magnification.

Figure 2: Schematic drawing of the insert

Figure 3: SEM image of benchmark material R at x1000 magnification.

DETAILED DESCRIPTION

[0017] Figures 1a and 1b are SEM images at x140 and x450 magnification respectively that show a composite material 2 comprising a cemented carbide matrix embedded 4 diamond entities 6 which are homogeneously distributed throughout; wherein the constituents of the cemented carbide include a tungsten carbide and a binder phase; wherein the grain size of the metal carbide is 0.6-8 microns; wherein the material comprises 5-65 vol% diamond entities and wherein at least 20% of the diamond entities contains pores and / or cracks 8 that are filled with constituent(s) of the cemented carbide. The diamond entities could have either a regular or irregular shape. Figure 1a clearly shows the homogeneous distribution of the diamond entities in the cemented carbide matrix. Figure 1b clearly shows the presence of the cracks and pores in the diamond entities that are filled with constituents of the cemented carbide.

[0018] In one embodiment the material comprises between 35-95 vol% cemented carbide, preferably between 40-90 vol% cemented carbide, more preferably between 45-85 vol% cemented carbide, even more preferable between 50 - 80 vol% cemented carbide, most preferable between 45-75 vol% cemented carbide.

[0019] Preferably the average grain size of the tungsten carbide is between 0.6 - 8μm, more preferably between 0.7 - 6 μm, even more preferably between 0.8 - 5 μm, most preferably between 0.9 -4 μm. The average WC grain size is

evaluated either using the Jeffries method described below from at least one different micrograph for each material, preferably two or more. If several micrographs are used an average value was then calculated from the mean grain size values obtained from the individual micrographs (for each material respectively). The procedure for the mean grain size evaluation using a modified Jeffries method was the following:

A rectangular frame of suitable size was selected within the SEM micrograph so as to contain a minimum of 150 WC grains. The grains inside the frame and those intersected by the frame are manually counted, and the mean grain size is obtained from equations (1-3):

$$M = \frac{L_{scale\,mm} \times 10^{-3}}{L_{scale\,micro} \times 10^{-6}} \quad (1)$$

$$vol\%WC = 100 \times \left(-1.308823529 \times \frac{\left(\frac{wt\%Co}{100} - 1\right)}{\left(\frac{wt\%Co}{100} + 1.308823529\right)} \right) \quad (2)$$

$$d = \frac{1500}{M} \times \sqrt{\frac{L_1 \times L_2 \times vol\%\,WC}{\left(n_1 + \frac{n_2}{2}\right) \times 100}} \quad (3)$$

Where:

d = WC grain size (μm)
L_1, L_2 = length of sides of the frame (mm)
M = magnification$L_{scale\,mm}$ = measured length of scale bar on micrograph in mm
$L_{scale\,micro}$ = actual length of scale bar with respect to magnification (μm)
n_1 = no. grains fully within the frame
n_2 = no. grains intersected by frame boundary
wt%Co = known cobalt content in weight %.

[0020] Equation (2) is used to estimate the WC fraction based on the known Co content in the material. Equation (3) then yields the mean WC grain size from the ratio of the total WC area in the frame to the number of grains contained in it. Equation (3) also contains a correction factor compensating for the fact that in a random 2D section, not all grains will be sectioned through their maximum diameter.

[0021] Optionally, or when the binder is not Co, the WC-grain size could also be determined using EBSD on a cross-section of an ion polished sintered sample. This is more precise, but more time consuming method also give information regarding grain size distribution. When comparing Jeffries and EBSD grain size the area d50 value from EBSD corresponds well with the Jeffries value. "D50" is the equivalent diameter Dn where the combined area of the grains smaller than Dn is equal 50% of the total grain area

[0022] Settings and method for EBSD analysis on WC grain size are:

Table 1. Settings for the EBSD analysis in Aztec 6.0.

Parameters	Typical settings	
	Ex 1. WC- Map	Ex 2. WC- Map
Binning mode	2x2	2x2
Speed of acquisition	64.73 Hz	64.81 Hz
Area	20x20	16x23
Step size	0.05 μm	0.05 μm

[0023] The post-processing was performed using AztecCrystal 2.2 software. For WC auto-cleaning was used with an addition of Pseudo-symmetry rotations removal of axis 0001 with and angle of 30 degrees (allowed deviating angle 5 degrees).

[0024] WC-WC boundaries were defined as having a misorientation angle larger than 3 degrees and boundaries being closed. Boarder grains were excluded. Smallest grain was defined as having size of 13 pixels in area.

[0025] Preferably the binder phase of the cemented carbide, and therefore also one of the constituents that fills the pores / and or cracks in the diamond entities, is selected from cobalt, nickel, iron or a mixture thereof, more preferably cobalt. The binder phase may also contain Cr, V, Ti additions to control the grain growth during sintering of the granules and during HPHT.

[0026] In one embodiment the distribution of the diamond entities in the cemented carbide matrix is a normal distribution. In other words, there is a single modal distribution of the diamond entities. In another embodiment, there may be a multi modal distribution of the diamond entities.

[0027] The diamond entities are homogenously distributed throughout the cemented carbide matrix in three dimensions in terms of distance between the neighbouring diamond entities throughout the material. The volume of the diamond entities throughout the material is homogenously distributed, meaning that the diamond entities are evenly distributed throughout the composite material and that no distinguishable pattern in the distribution of the diamond entities can be seen. Examples of distinguishable patterns could be a gradient in either size or number of the diamond entities or that the material contains satellite structures wherein there would be a plurality of the smaller diamond entities surrounding a larger diamond entity. This could be analysed by comparing SEM or LOM images from a near in the bulk of the material and an area near the surface of the material. The difference between the volume of the diamond entities in the area near the surface and the volume of diamond entities in the area in the bulk of the material (i.e. ((highest value- lowest value)/Highest value)*100) is less than 15%, preferably less than 10%. By an "area near the surface" is defined as $1/10^{th}$ of the distance from the surface (i.e. the cutting edge) and by an "area in the bulk of the material" is defined as being $1/10^{th}$ of the distance from the substrate if there is a substrate present or $1/10^{th}$ of the distance from the bottom of the insert if there is no substrate present.

[0028] In one embodiment, the binder content of the cemented carbide matrix 4 is between 5 - 20 weight percent (wt%). Advantageously, this provides a material with a cemented carbide matrix having an optimal balance between hardness and toughness. Preferably, the binder content in the cemented carbide part of the matrix is >3 wt%, more preferably >4wt%; most preferably >5 wt%, the binder content is <20 wt%, more preferably <15wt%, most preferable <14 wt%. This is measured by using energy dispersive spectroscopy (EDS) on cemented carbide areas of the sintered sample phase.

[0029] In one embodiment the cemented carbide matrix further comprises one or more elements selected from Cr, Ta, Ti, Nb, Mo, Zr and V present as elements or as carbides, nitrides or carbonitrides or a mixture thereof in contents from 100 ppm up to 15 wt% depending on the element added and the purpose of the addition. Advantageously, the addition of one or more of these elements is that they act as a grain growth inhibitor will control grain growth in granules. Further it lowers the melting point for HPHT synthesis which is beneficial as it reduces the fatigue on the cemented carbide dies in the press, thereby saving money and material. When the grain growth inhibitor is chromium it also provides the advantage of increasing the plastic deformation and corrosion resistance of the material.

[0030] In one embodiment the cemented carbide matrix further comprises a gamma phase selected from a carbide or nitride of niobium, tantalum, titanium or a mixture thereof. Advantageously, the presence of the gamma phase increases the wear resistance of cemented carbide matrix. When the gamma phase is tantalum or niobium the plastic deformation resistance at elevated temperatures is increased.

[0031] In one embodiment the D50 of the diamond entities is between 10 - 500 μm, preferably between 10-300 μm, more preferably between 12-250 μm, even more preferably between 12-200 μm, even more preferably between 15-200 μm, even more preferably between 20-200 μm, most preferable between 12-150 μm. This is measured on SEM image using the Fiji Image J software and the particle size function with the diamonds as the targeted phase. Advantageously this enables the formation of a homogenous blend between the diamond entities within the cemented carbide matrix. This size range also allows for a higher degree of fracture of the diamond particles during processing and densifying thus creating new cracks and cavities that also will be infiltrated by the constituents of the cemented carbide thus further increasing the toughness of the diamond entities.

[0032] In one embodiment the diamond entities include single crystal diamond. The average diameter or D50 of the diamond single crystals are between 6 -100 μm, preferable 6-80 μm, more preferable 8-60 μm, even more preferable 10-80 μm, most preferable 15- 80 μm or 12-60 μm. The diamond single crystal grain size is analysed using EBSD on a ground, lapped and ion polished samples where the diamonds and the CC-matrix is in the same height level. To be indexed as different diamond crystals the difference in orientation is = or >10 degrees.

[0033] In one embodiment the diamond entities are at least partially in the form of diamond clusters. Diamond clusters being defined as being two or more diamond grains bonded together.

[0034] According to the present invention the pores and / or cracks 8 in the diamond entities 6 are filled through

infiltration. The elements in the cemented carbide source are tougher compared to the diamond and hence the enhancement is provided by supplying the diamond with a tougher material(s). These element(s) may also improve the retention, i.e. a reduced risk of pull-out, of the diamond to the cemented carbide through increased contact area. By employing a diamond feed stock having existing imperfections such as cracks, cavities etc. or diamond feedstock that is capable of creating such imperfections during the manufacturing process, it allows for the pores and/or cracks to be filled with elements from the cemented carbide matrix. The definition of a diamond entity containing pores and/or cracks that are filled with constituent(s) from the cemented carbide matrix are defined as cemented carbide element(s) that are visible in less than or equal to 1kX inside or in the periphery of the diamond entity 6 using a SEM and back scatter electrons.

[0035] In one embodiment, at least one of the elements from the cemented carbide source is infiltrated into at least 20 % of the diamond entities 6, preferably at least 25 % and even more preferably at least 30 wt%.

[0036] In one embodiment of the present invention, at least 20 %, preferably at least 25 % and even more preferably at least 30 wt% of the diamond entities contains pores and/or cracks that are filled with at least one of the elements from the cemented carbide source.

[0037] In one embodiment at least 25% of the diamond entities comprise a plurality of crystals having a two or more different orientations wherein having different orientations is defined as two or more substantially neighbouring diamonds crystals having at least 10 degrees difference in orientation.

[0038] Figure 2 shows an insert 10 for a mining or rock cutting or wear part application comprising the material as described hereinbefore or hereinafter. The Inserts typically comprise a base portion 12; a working tip potion 14 and a core 16. It should however be understood that the insert could have a different form. The insert 10 could for example have a symmetrical or asymmetrical formation. In one embodiment, the insert 10 has a domed working tip portion 14 comprising the composite material 2 as described hereinbefore or hereinafter and a base portion 12 comprising cemented carbide.

[0039] The composition of the cemented carbide of the base portion 12, otherwise known as a substrate, comprises a cemented carbide having a composition within the ranges as described hereinbefore and hereinafter.

[0040] In one embodiment the cemented carbide base portion contains 4-15 wt% Co.

[0041] In one embodiment the cemented carbide base portion contains Cr.

[0042] In one embodiment the cemented carbide base portion has a room temperature hardness between 900 - 1650 Vickers.

[0043] In one embodiment the cemented carbide base portion has a fracture toughness K1C >10 mPa/m measured with Palmqvist method from 30 kg or 100 kg Vickers indents using Shetty's formula.

[0044] Preferably, for a percussive application as top hammer or DTH (Down the hole) drilling the binder concentration is between 4-12 wt%, more preferably between 4-10 wt% most preferable 5-8 wt%. Preferably, the average grain size of the hard metal is between 0.7-5 μm, more preferably between 1-4 μm with a room temperature hardness of 1200-1650 HV20.

[0045] Preferably, for rotary applications the binder concentration is between 8-20 wt%, more preferably between 8-15 wt%, most preferable 10-15 wt%. Preferably, the average grain size of the hard metal is between 2-10 μm, more preferable between 2- 8 μm most preferable between 2-6 μm with a room temperature hardness of between 1000-1300 HV20.

[0046] Preferably, for mechanical rock cutting the binder concentration is between 6-15 wt%, more preferably between 6-12 wt%. Preferably, the average grain size of the hard metal is between 6-18 μm, more preferably between 6-15 μm with a room temperature hardness of 800 - 1100 HV20.

[0047] Preferably, for a wear part applications for example but not limited to support inserts in drill bits the binder concentration is between 3-10 wt%, more preferably between 3-8 wt% most preferable 3-7 wt%. Preferably, the average grain size of the hard metal is between 0.6- 4 μm, more preferably between 0.6-3 μm with a room temperature hardness of 1300-2000 HV20.

[0048] Preferably, the diameter of the base portion 12 is between 5-40 mm, more preferable 7-30 mm, most preferable 7-24 mm.

[0049] Preferably, the thickness of the tip portion 14 is between 0.1-15 mm more preferable 0.2-10 mm, even more preferable 0.5 - 5 mm, most preferable 0.8 -4 mm when measured along the longitudinal axis.

[0050] Preferably, the volume of the tip portion 14 is between 2- 50 vol% of the total volume of the insert 10, more preferable 5-40 vol%, most preferable 8-30 vol%.

[0051] Alternatively, the insert 10 may be freestanding without a cemented carbide base.

[0052] The application further relates to a method for making a material as described hereinbefore or hereinafter comprising the steps of:

a) providing friable diamond grains;
b) providing sintered granules of cemented carbide;
c) blending the diamond grains with the sintered cemented carbide granules to form a homogenous powder blend;
d) placing the powder blend into a preformed refractory metal cup;

e) providing a refractory metal lid, or, a pre-sintered or sintered cemented carbide base on top of the powder blend to close the cup;
f) pre-compacting the powder in the refractory metal cup;
g) surrounding the cup with a pressure media;
h) inserting the pressure media surrounded cup into a high pressure high temperature container;
i) placing the above container in a high pressure high temperature press and sintering at high pressure and high temperature to form a composite material.

[0053] By friable it is herein meant the diamond grains comprise one or more of the following characteristics; the diamond grains have rough surfaces; cavities; pores; cracks; multi/polycrystalline structure; inclusions; crystallographic defects and / or an elongated, irregular, sharp or angular shape. Friable diamond grains have the tendency to break up into smaller fragments when under pressure.

[0054] Examples of diamond feedstock with more friable characteristics are grits designed for resin and or vitrified bond system grinding wheels. Another example of diamond feedstock with more friable characteristics are polycrystalline diamond powder, for example those designed for lapping or polishing, that comprise of many smaller crystals bound together to form a larger polycrystalline diamond grain.

[0055] In the art of sintering PCD, recommendations can be found to use a diamond feedstock with low crystal defects, hence tough diamonds. However, in this invention it has surprisingly been found suitable to use diamond feedstock with more friable characteristics which would be considered non-intuitive as friable diamond crystals are typically not as tough. The composite material however has unexpectedly improved toughness and wear resistance.

[0056] The friable diamond granules could for example be produced by, but not limited, freeze spray drying or spray drying and have a relative density of about 15-40% compared to the density of diamonds (3.52 g/cm^3). Examples of diamond feedstock with more friable characteristics are grits designed for resin and or vitrified bond system grinding wheels. Contribution to the friable characteristics of such grits may be shapes that are more elongated and irregular, rougher surfaces, multi/polycrystalline structure inside such grains, as well as inclusions. Another example of diamond feedstock with more friable characteristics are polycrystalline diamond powder, for example those designed for lapping or polishing, that comprise of many smaller crystals bound together to form a larger polycrystalline diamond grain. In the art of sintering PCD, recommendations can be found to use a diamond feedstock with low crystal defects, hence tough diamonds. However, in this invention it has surprisingly been found suitable to use diamond feedstock with more friable characteristics which would be considered non-intuitive as friable diamond crystals are typically not as tough.

[0057] In one embodiment the D50 or average diameter of cemented carbide granules is in the range of 5-60 microns.

[0058] The sintered cemented carbide granules can be manufactured in different ways. Spray dried granules are prepared using conventional means, i.e. preparing a slurry is prepared where powders with the desired composition and WC grain size are mixed with an organic binder, usually PEG and a liquid, usually a water/ethanol blend. The slurry is then spray dried to form granules.

[0059] The sintering temperature of the cemented carbide granules is used both to control the WC-grain size and the density and is preferable between 1250 - 1550 °C, more preferably between 1270-1500 °C, most preferably between 1300-1500 °C. Depending on the sintering temperature the sintered cemented carbide granules are preferably fully dense or at least 90% dense, depending on the composition and sintering temperature of the granules. The sintering can be performed in vacuum, or in N_2/Ar atmosphere, or, at least partly, in a carburizing atmosphere which can be provided by one or more carbon containing gases e.g. CO_2, CO and CH_4.

[0060] The sintering process are usually started with a de-binding step where the organic binder is removed. The de-binding step is usually performed at a temperature between 300 and 600°C.

[0061] In one embodiment the sintered granules of cemented carbide particles are substantially fully dense.

[0062] Using fully dense or near fully dense cemented carbide granules below a certain D50 or D90 is beneficial to controlling the homogeneity when blending diamond which is a lighter material with SCCG which is a heavier material. The cemented carbide granule size also defines the smallest distance to the next diamond entity.

[0063] In one embodiment the WC grain size within the sintered cemented carbide granules is between 0.6 - 8 μm. Advantageously, this grain size range provides the means to balance and optimize the hardness and toughness for mining applications. The grain is measured by image analysis on SEM images either from secondary or back-scatter electron images using Jeffries method giving an average grain size or from analysis of an EBSD-image on an ion polished surface using the area D50 value.

[0064] Preferably, the average WC grain size in the sintered cemented carbide granules is preferably between 0.6- 8 microns, more preferably between 0.7-5 microns, even more preferably between 0.8-3 microns, most preferably between 0.9-4 microns.

[0065] The binder phase content, preferable Co, in the SCCG prior to HPHT is between 6-20 wt%, more preferable 7-15 wt% Co, most preferable 8-13 wt% Co. In the composite after HPHT the binder content in the cemented carbide part can thus range from about 3 wt% to about 18 wt%, depending on the amounts of diamonds and the degree of binder

infiltration. The binder phase content in the cemented carbide part of the composite after HPHT can be analysed by EDS (energy dispersive spectroscopy) or more preferable WDS (wavelength dispersive spectroscopy) on a sufficient large ion polished area where only cemented carbide is present.

[0066] In one embodiment the D50 size of SCCG is between 5-60 microns. Advantageously, this range provides good flowability and high powder density and the mass of each SCCG is more equal to the mass of a diamond particle. The D50 size of the cemented carbide granules is preferably between 5-40 microns, even more preferably 5-30 microns. The particle size distribution was measured using laser diffraction fully compliant with ISO 13320 for the complete size range from 0.1 μm to 8750μm from Sympatec GmbH using a Helos BR instrument with Rodos M/Vibri dry sampling unit. The powder is analysed with a combination of R3 (0.9 to 175μm) and R5 (4.5 to 875μm) measuring ranges. For each measuring range the samples are analysed three times using 0.5g of powder. The results from the two measuring ranges were then combined in the Windox 5.7.2.2 software to cover the range 0.9 to 875 μm.

[0067] In one embodiment the D90 size of SCCG is <80 μm, preferably <70 μm, more preferably < 60 μm, most preferable <50 μm. Advantageously, this provides a smaller distance to the next diamond entity and is also important for the mass of the granule which should be as close to the mass of the diamonds in the feed as possible to reduce the risk of separation during blending and filling of the cup which will be of great importance for the homogeneity of the final material.

[0068] In one embodiment the (D90 - D10) range of cemented carbide granules is < 50 μm, preferably < 40 μm, more preferably < 30 μm. Advantageously, a narrow distribution of the sintered cemented carbide granules provides a more homogenous distribution of the diamond entities within the cemented carbide matrix and the distances between the diamond entities within the composite material will be easier to control and thus the properties of the material will be more even.

[0069] The D10, D50 and D90 are calculated using Windox software. D10, D50, or D90 is defined as the size value corresponding to cumulative size distribution at 10%, 50%, or 90% respectively, which represents the size of particles below which 10%, 50%, or 90% of the sample lies. Alternative notations are x10, x50 and x90, as used in Windox software.

[0070] In one embodiment the powder density of the SCCG powder is >35%, preferably >40%, more preferably >45% compared with the fully dense sintered bodies of such granules. The powder density (or apparent density) is measured by using a Hall flow meter and filling a known volume (Hall density cup) using a funnel placed above where the powder is added.

[0071] In one embodiment the SCCG powder has a tap density is preferable >40%, more preferably >50%, most preferably >55% relative to a full sintered body. The tap density is obtained when filling a known volume (Hall density cup or similar) with the powder granules and tap or "knock" to make them pack even tighter. Advantageously, a high granule density provides that the diamond grains are fixed in their position after filling the refractory metal cup. Moreover, it allows a lower shrinkage during HPHT which is beneficial for the shape and size control and also for avoiding sudden pressure drops during HPHT (so called blow-outs) which can result in catastrophic failures of the cemented carbide dies in the HPHT cell.

[0072] In one embodiment the cemented carbide granules have graphite or other sp^2-carbon on their surface prior to the HPHT step. Advantageously, this will lower the binder-melting point and ease the infiltration of the diamond grains and will convert into diamond since the HPHT process is carried out at or above the diamond stable region in presence of a catalytic metal (Co).

[0073] In one embodiment the interior of the cemented carbide granules has a majority of fcc-Co and the binder layer around the remaining granules have a majority of hcp-Co after HPHT.

[0074] For step c) the blending could be done by vibrating, turbola blending or shaking for example in a commercial paint shaker.

[0075] For step d) the refractory metal cup is preferably made from titanium but could also be made from niobium or tantalum or any other suitable refractory metal. The cup is shaped as required by the product being formed.

[0076] For step e) either a refractory metal lid, or, a pre-sintered or a sintered cemented carbide pre-shaped base is inserted on top of the powder blend inside the refractory metal cup in order to close the cup. The choice of the cemented carbide base in terms of grain size and composition is made depending on the target application. By "pre-sintered is herein meant that the cemented carbide base has not been sintered to full density prior to being placed in the cup. It will reach full density during the subsequent HPHT step.

[0077] For step f) either a refractory metal or a sintered hard metal pre-shaped substrate is inserted on top of the powder blend inside the refractory metal cup in order to close the cup. Advantageously if a hard metal substrate is added this enables a cemented carbide base portion to be formed and the shape of the cutting tip to be designed or adjusted to fit the application for example by allowing a higher amount of cemented carbide granule and diamond blend in one part or one side of the tip. The choice of the cemented carbide base portion in terms of grain size and binder content is made depending on the target application. The tip could have either a symmetric or asymmetric geometry.

[0078] For step g) the pressure media could for example is hBN or an NaCl mixture that becomes molten during the high temperature high pressure stage at or above the diamond stable region.

[0079] For step h) the high pressure container for example could be, but not limited to a natural and synthetically reconstituted pyrophyllite cube or cylinder.

[0080] For step i) a typical HPHT cycle comprises a fast ramp for 50-65 seconds to a max pressure of 52 kBar and a temperature of 1225°C and then a smooth transition into a lower ramp of 200-300 seconds at 52 kBar gradually climbing up to a sintering soak temperature. the typical soaking temperature is between 1350-1425°C for 100-200 seconds a sharp transition into a down ramp with maintained pressure of 52 kBar for 200-400 seconds; an instant cut of electrical power and a natural cooling ramp with cooling water jackets dissipating the heat for 40 seconds and a gradual release of the applied pressure. The temperature is controlled by W-Re thermocouples inside the cube. The full cycle is about 15-25 minutes. The sintering temperature used is typically 1300-1500°C, preferably 1320-1450°C, most preferable 1350-1420°C. The sintering pressure used is typically 50kBar to 60kBar, preferably 50kBar-55kBar, most preferably 52kBar.

[0081] The pressure and the contact with the cemented carbide granules will to a large extent break up the diamond and the metal binder, in the cemented carbide melts and infiltrates and fills the cavities in the diamond entities and or cementing the diamond crystals and or enabling the formation of new diamond entities.

[0082] Following HPHT the outer diamater of the part is then cleaned up using centerless grinding and when needed the cup on the dome is removed either using grinding or by blasting with SiC-grits. If the part is a mining insert it is then ground to the exact dimensions required. If required the inserts can then be subjected to shot blasting and / or tumbling, for example high energy tumbling. The inserts can then be shrink fit to be brazed into a cavity in a drill bit.

EXAMPLES

Example 1 - summary of samples

[0083] The inventive samples were produced by blending diamonds and sintered cemented carbide granules (SCCG) in the desired composition using a Caulk VARI-MIX II vibrating unit for 2.5 minutes. The amount of powder blend to be filled to the Ti-cups was calculated by the volume of the Ti-cup and the desired height of the cutting layer. The comparable samples were manufactured using only SCCG powder. Cylindrical cemented carbide base portions (substrates) were manufactured using conventional methods and then the desired dome geometry of the tip portion was formed on top.

[0084] Table 2 shows the summary of the powder blends and substrates used in the samples

Table 2: Summary of powder blends and substrates

Sample	Composition of the SCCG	Type of diamond source	Diamond feedstock sizes	Volume of the diamond (%)	Composition and hardness of cemented carbide substrate
A (inventive)	13 wt% Co, 0.56 wt% Cr, WC (SCCG1-)	Resin bond RVG810 from Hyperion	230/270 US mesh	30	12 wt% Co, 0.5 wt% TiC, 2.5 wt% (Ta,Nb)C, 90.2 HRA
B (inventive)	13 wt% Co, 0.56 wt% Cr, WC (SCCG-1)	Resin bond RVG810 from Hyperion	230/270 US mesh	50	12 wt% Co, 0.5 wt% TiC, 2.5 wt% (Ta,Nb)C, 90.2 HRA
C (comparative)	13 wt% Co, 0.56 wt% Cr, WC (SCCG-1)	Freeze dried diamond granules < 500 microns with MBM diamonds from Hyperion	Mixture of 20 wt% 4-8 micron & 80 wt% 20-30 microns	30	12 wt% Co, 0.5 wt% TiC, 2.5 wt% (Ta,Nb)C, 90.2 HRA
D (comparative)	13 wt% Co, 0.56 wt% Cr, WC (SCCG-1)	an	n/a	0	12 wt% Co, 0.5 wt% TiC, 2.5 wt% (Ta,Nb)C, 90.2 HRA
E (inventive)	12 wt% Co, WC (SCCG-2)	Resin bond RVG810 from Hyperion RVG810	325/400 US mesh	15	12 wt% Co, 0.5 wt% TiC, 2.5 wt% (Ta,Nb)C, 90.2 HRA

(continued)

Sample	Composition of the SCCG	Type of diamond source	Diamond feedstock sizes	Volume of the diamond (%)	Composition and hardness of cemented carbide substrate
F (inventive)	12 wt% Co, WC (SCCG-2)	Resin bond RVG810 from Hyperion RVG810	230/270 US mesh	30	6 wt% Co, 0.6 wt% Cr, WC, HV20 1420
G (inventive)	12 wt% Co, WC (SCCG-2)	Resin bond RVG810 from Hyperion	325/400 US mesh	30	6 wt% Co, 0.6 wt% Cr, WC, HV20 1420
H (comparative)	12 wt% Co, WC (SCCG-2)	n/a	n/a	0	6 wt% Co, 0.6 wt% Cr, WC, HV20 1420
I (comparative)	6 wt% Co, WC (SCCG-3)	Resin bond RVG810 from Hyperion	230/270 US mesh	10	6 wt% Co, WC, HV20 1450
J (comparative)	6 wt% Co, WC (SCCG-3)	Resin bond RVG810 from Hyperion	230/270 US mesh	30	6 wt% Co, WC, HV20 1450
K (inventive)	11 wt% Co, 1.11 wt% Cr, WC (SCCG-4)	Resin bond RVG810 from Hyperion	325/400 US mesh	30	6 wt% Co, 0.6 wt% Cr, WC, HV20 1420
L (inventive)	8 wt% Co, 0.8 wt% Cr, WC (SCCG-5)	Resin bond RVG810 from Hyperion	230/270 US mesh	30	6 wt% Co, 0.6 wt% Cr, HV20 1420
M (comparative)	8 wt% Co, 0.8 wt% Cr, WC (SCCG-5)	n/a	n/a	0	6 wt% Co, 0.6 wt% Cr, HV20 1420
O (comparative)	7.6 wt% Co 0.27 wt% Nb 1.15 wt% Ta, WC (SCCG-6)	Resin bond RVG810 from Hyperion	230/270 US mesh	30	6 wt% Co, 0.6 wt% Cr, HV20 1420
P (comparative)	11 wt% Co, 1.11 wt% Cr, WC (SCCG-4)	n/a	n/a	0	6 wt% Co, 0.6 wt% Cr, HV20 1420

[0085] Sample C is a comparative sample wherein soft freeze spray dried diamond containing granules was used in the powder mixture having a D100 diamond granule size if 500 μm, having a bi-modal distribution of the diamond grain sizes with maxima at 6 μm and 25μm, a powder density of the granules of 1.11 g/cm^3 and a relative density of the diamond in the diamond containing granules compared to pure diamond of 32%. The soft diamond granules also contained 10 wt% PEG-binder that was removed in a mixture of hydrogen and nitrogen gas up to 500 °C prior to the HPHT-step.

[0086] Table 3 shows the properties of the cemented carbide granules in the powder mixture:

Table 3: Properties of the cemented carbide granules in the powder mixture

SCCG source in samples	Sintering temp. (°C)	Average WC grain size in the sintered cemented carbide granules (μm)	Sintered density (g/cm3) [1]	D50 size of the cemented carbide granules (μm)	D10 size of the cemented carbide granules (μm)	D90 size of the cemented carbide granules (μm)	Powder density of sintered granules (g/cm^3)	Relative density of sintered cemented carbide granules (%)	Tap density (g/cm^3)
SCCG-1 used in samples A, B, C, D[2]	1360	0.75	14.16	16.7	9.1	28.5	7.9	56	9.0
SCCG-2 used in samples E, F, G, H[2]	1350	1.1[2] 1.2[3]	14.4	18.1	11.0	29.2	7.2	50	8.1
SCCG-3 used in samples I and J	1275	*1.3*[2]	14.95	97.5	47.5	162.1	6.30	42	7.18
SCCG-4 used in samples K and P	1275	*2.8*[2]	14.24	50.8	30.4	68.0	6.050	42	n/a
SCCG-5 used in samples L and M	1275	*1.1*[2]	14.50	52.7	35.2	68.4	6.704	46	n/a
SCCG-6 used in sample O	1275	*0.90*[2]	14.7	72.9	31.9	138.3	6.09	41	6.69

[1] The sintered density is obtained from the producer or can be calculated from the nominal composition. If calculated the carbide forming metals are included in the form of their carbides; TaC and NbC and Cr is regarded as fully dissolved in the binder phase and included as an element.
[2]By Jeffries

[0087] The sintered cemented carbide granules SCCG-3, SCCG-4, SCCG-5 and SCCG-6 have been manufactured using soft spray dried cemented carbide granules with a relative density of about 25% and average granule size around 80-100 microns and with a maximum size of 250 microns. The spray dried granules were placed on yttrium oxide coated graphite trays. 1.5 kg spray dried granules were loaded on each tray that have an inside diameter of 278 mm. The sintering consisted of a de-binding step to remove the PEG from the spray dried granules and then a solid-state sintering step at 1275°C for 60min under a partial pressure of 250 mbar. The partial pressure consisted of equal flow of argon and carbon monoxide. After sintering the granules were deagglomerated using approximately 2kg cylindrical cemented carbide milling bodies in a small ball mill for 20 minutes. The deagglomeration was ran under dry conditions, i.e., no liquid was added to the ball mill.

[0088] The deagglomerated powder SCCG-3 and SCCG-6 were used as-received after the deagglomeration step and the deagglomerated powder SCCG-4 and SCCG-5 were finally fraction sieved at 63μm and using the fraction <63 μm. Samples produced using SCCG-3 and SCCG-6 are comparative samples as the D50 size of the cemented carbide granules is too large.

[0089] The SCCG and the diamond powder was then blended by using a Caulk VARI-MIX II vibrating unit for 2.5 minutes. Then the powder blend was poured into a titanium refractory metal cup with a wall thickness of 127μm. This was followed by providing a sintered cemented carbide base on top of the powder blend to close the cup and thus containing the assembly. The powder blend in the refractory metal cup was pre-compacted by pushing the cemented carbide body on the powder blend. The contained assembly was then surrounded by a pressure media being hexagonal Boron Nitride (hBN); a Carbon Foil Heater, and a cylinder made up of a mixture of carbon lampblack and sodium chloride. These internal components where then contained within a reconstituted pyrophyllite pressure media container; the pressure media container was then inserted into a high pressure high temperature cubic press and sintering at high pressure and high temperature to form a domed shaped insert. The HPHT sintering was conducted at 52 kbars pressure. The sintering temperature used is detailed in the Table 3.

[0090] The samples were then HPHT sintered to form dome shaped inserts. Table 4 shows the properties of the HPHT sintering conditions used and the yields post sintering. Following the HPHT sintering the inserts were then ground and / or blasted with SiC to clean the dome. Table 4 also reports the homogeneity of the wear on the dome following the SiC blasting.

Table 4: Comments following HPHT sintering and blasting / cleaning of the dome.

Sample	HPHT max. temp (°C)	Number of samples	Number of samples failed after HPHT and sort of failure	Thickness of the diamondhm layer (mm)	Homogenous wear during SiC-blasting/cleaning of dome	Comments if not homogenous wear
A Invention)	1350	2	0	4.5	Yes	n/a
B (invention)	1350	2	0	5	Yes	n/a
C (comparison)	1350	2	0	5	No	Uneven wear on microscale
D (comparison)	1350	2	0	n/a	Yes	n/a
E (invention)	1350	1	0	2.5	Yes	n/a
	1400	1	0	2.5	Yes	n/a
F (invention)	1350	2	0	2.5	Yes	n/a
G (invention)	1350	3	0	2.5	Yes	n/a
	1400	1	0	2.5	Yes	n/a
H (comparison)	1375	2	0	n/a	Yes	n/a
I (comparison)	1425	2	0	4 mm but only 3 mm Diamond-CC layer	No	Uneven wear during blasting of dome

(continued)

Sample	HPHT max. temp (°C)	Number of samples	Number of samples failed after HPHT and sort of failure	Thickness of the diamondhm layer (mm)	Homogenous wear during SiC-blasting/cleaning of dome	Comments if not homogenous wear
J (comparison)	1375	2	0	5	No	Uneven wear during SiC-blasting of dome
K (invention)	1375	2	0	n/a	Yes	n/a
L (invention)	1375	2	0	2	Yes	n/a
O (comparison)	1375	6	4 delaminatio n	3-4 mm due to uneven wear during cleaning (blasting)	No	Uneven wear on one side of the dome on the samples

[0091] Table 4 shows that the comparative samples suffer from either or both failure after HPHT sintering due to delamination or non-homogenous wear during SiC-blasting/cleaning of dome, whereas the inventive samples had no failures post HPHT sintering and homogenous wear during SiC-blasting/cleaning of dome. The homogeneous wear during SiC blasting of the dome is an indication of homogeneous material properties, which will lead for better insert performance and provide inserts that will be less prone to cracking.

[0092] Tables 5 and 6 show the properties of commercially available samples that are considered to be the state of the art and the "benchmark" for the properties for the inserts produced according the invention disclosed herein. These samples have high diamond contents and therefore a much more expensive to produce than the inventive samples. These samples are produced with three layers of diamond in the dome, with each layer having a different concentration of diamond.

Table 5: Cemented carbide properties for the comparative benchmark samples

Sample	D50 WC grain size in diamond layer 1 (cutting layer) from EBSD	Co (wt%) content from EDS on WC-Co area in diamond layer 1	D50 diamond grain size by EBSD (μm)	D50 WC grain size in cemented carbide substrate from EBSD	Composition of cemented carbide substrate from EDS (wt%)
Q (benchmark)	0.39	5.4	8.32	1.89	8.8 Co, balance WC
R (benchmark)	0.45	8.2	9.98	1.64	8.2 Co, Balance WC

Table 6: Diamond properties of the comparative benchmark samples

Sample	Diamond content in layer 1 (cutting layer) by Image analysis on 500X SEM image (area %)	Diamond content in layer 2 (middle) by Image analysis on 500X SEM image (area %)	Diamond content in layer 3 (next to substrate) by Image analysis on 500X SEM image (area %)	Thickness ofdiamond layer 1 (μm)	Thickness ofdiamond layer 2 (μm)	Thickness ofdiamond layer 3 (μm)
Q	74	50	36	630	470	430
R	74	55	33	990	400	360

[0093] Table 7 shows the properties of the inserts post HPHT sintering.

Table 7: Composite of material post HPHT sintering

Sample	Number of entities/ grains in image	Magnification of SEM-image (X)	% of diamond entities that contain pores and/ or defects and /or cracks that are filled with constituents of the cemented carbide
A (invention)	40	200	48
B (invention)	21	370	52
F (invention)	85	140	60
R (layer 3) (benchmark)	60	1000	7

[0094] Table 7 shows that the benchmark comparison, R, has a much lower percentage of diamond entities that contain pores and/ or defects and /or cracks that are filled with constituents of the cemented carbide and thus falls outside of the scope of the claims. Figures 1a and 1b show SEM images at x140 and x450 magnification respectively of the structure of sample F, wherein there is a high percentage of diamond entities that contain pores and/ or defects and /or cracks that are filled with constituents of the cemented carbide. This can be compared to figure 3 that is an SEM image of sample R, which has a much lower percentage of diamond entities that contain pores and/ or defects and /or cracks that are filled with constituents of the cemented carbide.

[0095] Table 8 shows further comparative samples. The samples shown in this table are produced by conventional sintered at a temperature of 1410 °C in vacuum and applying an argon pressure at 60 bars at maximum temperature.

Table 8: Composition of cemented carbide samples used for comparison.

Sample	Grain size of the metal carbide (WC) (μm)	Nominal composition (wt%)	HV20	K1C (MPa/m)
S	1.72[2]	6.0 Co, balance WC	1450	11.2[4]
T	1.4[1]	8 Co, 0.8 Cr, balance WC	1520	12.0[3]
U	5.02[2]	11 Co, 1.11 Cr, balance WC	1060	13.3[4]
V	1.08	7.6 Co, 0.27 Nb 1.15 Ta, balance WC	1490	11.4[3]
W	2.4[1] 2.48[2]	6Co, 0.6 Cr, balance WC	1420	11.20[3] 11.69[4]

[1]By Jeffries
[2]050 by EBSD
[3]K1C from Palmqvist indents using Shetty's formula and 30 kg load
[4]K1C from SEVNB

[0096] Table 9 shows example of the hardness and fracture toughness of the sintered SCCGs and cemented carbides.

Table 9: Hardness and toughness properties

Sample	Average grain size of the metal carbide(WC) (μm)	Nominal binder content in SCCG	HV20	K1C[3] (MPa/m)
SCCG-1	0.75[1]	13 Co, 0.56 Cr	1380	17.08[3]
SCCG-2	1.2[1]	12.0 Co	1300	18.30[3]
SCCG-5	1.1[1]	8 Co, 0.8 Cr	1560	11.74[3]
SCCG-4	2.8[1]	11 Co, 1.11 Cr	1350	12.84[3]
S	1.72[2]	6.0 Co	1450	11.2[4]
T	1.4[1]	8 Co, 0.8 Cr	1520	12.0[3]

(continued)

Sample	Average grain size of the metal carbide(WC) (μm)	Nominal binder content in SCCG	HV20	K1C[3] (MPa/m)
U	5.02[2]	11 Co, 1.11 Cr	1060	13.3[4]
[1]By Jeffries [2] D50 by EBSD [3]K1C from Palmqvist indents using Shettys formula and 30 kg load [4]K1C from SEVNB				

[0097] Table 9 shows that all the SCCGs have a K1C > 10 mPa/m and that the hardness and toughness of the sintered SCCG and cemented carbides can be controlled by their composition. It was not possible to measure the hardness and toughness of the composite materials comprising the diamond and the high hardness would break the measuring equipment.

Example 4 - Homogeneity

[0098] The samples were ground, lapped and ion polished until the cemented carbide matrix and the diamond entities are in the same height level and thereafter SEM images was taken about 100 microns from the top of the dome (surface) and about 100 microns above the substrate. The images were analyzed with ImageJ program from Fiji. The scale in the images was set in the program prior to the image analysis. The area of interest was set to be the carbide matrix and the diamond entities was regarded as background. The threshold was set so that the diamonds entities that contained no CC constituents were black and the diamond entities that contained CC constituents also contained white areas or spots. The same threshold was used on both images. The images were then converted to binary images and the "particle size" was measured and the area of the "particles" (CC-matrix) was achieved. The area% of the diamond entities was calculated as 100% minus the area of the CC-matrix %. The area% is regarded to correspond with the volume% of the phases. Table 10 shows that homogenous samples were only achieved when blending diamonds with SCCG having a high powder density and a low D50 and D90. The results are shown in table 10.

Table 10: Homogeneity of distribution of cemented carbide and diamond entities.

Sample	Area % of the diamond entities ~100 μm below the top of the dome (cutting edge) as 100- area% of CC matrix	Analysed area (close to edge) (10^6 μm^2)	Area % of the diamond entities ~100 μm above the cemented carbide substrate as 100- area% of CC matrix	Analysed area (close to substrate) (10^6 μm^2)
A (invention)	27	0.76	28	0.72
B (invention)	49	0.52	52	0.50
I (comparison)	2.5	1.0	10.9	1.1
O (comparison)	23	0.7	13.3	2.5

[0099] The results from table 10 show that the distribution of the diamond entities in inventive samples is homogeneous and the distribution of diamond entities in the comparative samples is nonhomogeneous.

Example 5 - Lathe wear test

[0100] The samples tested in an abrasion wear test, wherein the sample tips are worn against a rotating granite log counter surface in a turning operation. The test parameters used were as follows: 100 N load applied to each insert, granite log rpm ~190, log diameter ranging from 130 to 150 mm, and a horizontal feed rate of 0.339 mm/rev. As much

of the length of the log (max 300 mm) was used in each test to remove that difference in composition in the rock have a significant impact on the results. If large piece broke out from the log this area was avoided and therefore the length in some tests were shorter than 300 mm. The sliding distance varied due to the difference in diameter and length of the part of the rock that could be used but were around 330-460 m and the mass loss versus sliding distance was approximately linear between the three samples of each grade that was tested. The sample was cooled by a continuous flow of water. Each sample was carefully cleaned and weighed prior to and after the test. Mass loss of one sample per material was evaluated, the sample volume loss for each of the tested materials was calculated from the measured mass loss and sample density, the results are presented in table 11.

Table 11: Wear test results

Sample	Diamond content (vol% or area%)	Calculated sintered density (g/cm^3)	Mass loss (g)	Estimated volume loss / meter (mm^3/m)
A (invention)	30	10.968	0.0008	1.51×10^{-4}
B (invention)	50	8.84	0 (-0.0001)	0
D (comparison)	0	14.16	0.0047	1.01×10^{-3}
E (invention)	15	12.76	0.0018	2.90×10^{-4}
F -(invention)	30	11.13	0.0007	1.43×10^{-4}
G (invention)[1]	30	11.13	0.0002	3.69×10^{-5}
H (comparative)	0	14.4	0.0064	1.01×10^{-3}
K (invention)	30	11.024	0.0008	1.72×10^{-4}
L (invention)	30	11.416	0 (-0.0003)	0
Q (benchmark)	74 (outer layer 1)		0.0000	0
R (benchmark)	74 (outer layer 1)	6.4	0.0001	3.26×10^{-5}
T (comparison)	0	14.24	0.0068	1.13×10^{-3}
Sample G was HPHT at 1350 C.				

[0101] Table 11 shows that the inventive samples have a lower wear rate that the comparative samples and similar wear to the benchmark examples even though they been produced with lower diamond content and consequently at lower cost. Moreover, the mismatch in CTE (thermal expansion coefficient) of the diamond containing layer in the invention compared to the cemented carbide base portion is significantly reduced with a lower diamond content and with separated diamond entities. Thus, the need for a layered structure with lower diamond composition towards the carbide base is removed.

Example 6 - Insert compression test

[0102] The insert compression test method involves compressing a drill bit insert between two planeparallel hard counter surfaces, at a constant displacement rate, until the failure of the insert. A test fixture based on the ISO 4506:2017 (E) standard "Hardmetals - Compression test" was used, with cemented carbide anvils grade H6F from Hyperion having a hardness exceeding 2000 HV, while the test method itself was adapted to toughness testing of rock drill inserts. The fixture was fitted onto an Instron 5989 test frame.

[0103] The loading axis was identical with the axis of rotational symmetry of the inserts. The counter surfaces of the fixture fulfilled the degree of parallelism required in the ISO 4506:2017 (E) standard, i.e. a maximum deviation of 0.5 μm / mm. The tested inserts were loaded at a constant rate of crosshead displacement equal to 0.6 mm / min until failure, while recording the load-displacement curve. The compliance of the test rig and test fixture was subtracted from the measured load-displacement curve before test evaluation. One diamond composite inserts was tested but at least three cemented carbide inserts per run. The counter surfaces were inspected for damage before each test. Insert failure was defined to take place when the measured load suddenly dropped by at least 1000 N. Subsequent inspection of

tested inserts confirmed that this in all cases this coincided with the occurrence of a macroscopically visible crack. The load at fracture was measured and the material strength was calculated and is characterized by means of the total absorbed deformation energy until fracture and calculated from the maximum load at fracture and the displacement of the insert. The summary inserts crushing strength (IC) and fracture energy (Ec), in Joules (J), required to crush the samples is shown in table 12 below:

Table 12: Crush test results

Sample	IC (kN)	Fracture energy Ec (J)
G (invention)	>89[1]	>8[1]
W (comparison)	35	3.0
[1] The test was stopped at 89 kN due to risk of failure of the counter plates.		

[0104] Both samples tested were "as-sintered" 10 mm spherical dome inserts. The crushing tests show that the strength of the inventive insert was significantly improved compared to the cemented carbide reference inserts even though the substrate in G is the same cemented carbide grade as used in sample W.

Example 7 - EBDS

[0105] Each sample was analyzed in the same way where a plane parallel section was cut from an insert using EDM cutting and mechanically polished. Thereafter, the samples were ion-polished using the flat mode until the diamonds and the cemented carbide were on the same height level after approximately 200-300 min at 6V and 20 min at 2V with 4° sample angle.

[0106] Three large maps for diamond analysis and two small maps for WC analysis was done using Aztec 6.0 software. In Table 13 the microscope and analysis set-up can be seen.

Table 13: Settings for the EBSD analysis in Aztec 6.0.

Phase and map	Diamond - Map 1	Diamond Map 2	WC- Map 1	WC- Map 2
Binning mode	2x2	4x4	4x4	4x4
Speed of acquisition	37.09 Hz	139.97 Hz	232.31 Hz	213.94Hz
Area	330x380 μm	260x450 μm	60x60 μm	60x60 μm
Step size	0.3 μm	0.3 μm	0.05 μm	0.05 μm
Included Phases	Diamond (HKL database)	Diamond (HKL database)	WC	WC, Co FCC and Co HCP

[0107] The post-processing was performed using AztecCrystal 2.2 software. For diamond map wild spike removal and zero solution removal down to 5 neighbours with 10 iterations per step. Additional Pseudo-symmetry rotations was removed, of axis [111] with an angle of 60 degrees (allowed deviating angle 5 degrees). Diamond-diamond boundaries were defined as having a misorientation angle larger than 10 degrees and boundaries was closed. Boarder grains were excluded. Smallest grain was defined as having size of 50 pixels in area. For WC auto-cleaning was used with an addition of Pseudo-symmetry rotations removal of axis [0001] with and angle of 30 degrees (allowed deviating angle 5 degrees). WC-WC boundaries were defined as having a misorientation angle larger than 3 degrees and boundaries was closed. Boarder grains were excluded. Smallest grain was defined as having size of 13 pixels in area.

Table 14: EBSD results

Sample	Area d50 grain size of diamond by EBSD	Area d10 grain size of diamonds by EBSD	Area d90 grain size of diamond by EBSD	% of the diamond entities comprising a plurality of crystals having a two or more different orientations wherein different orientations is defined as two or diamonds crystals having diamond to diamond bonding with at least 10 degrees difference in orientation by EBSD
G (inventive)	30.72	12.2	45.1	54
F (inventive)	48.3	25.9	62.0	52
R -layer 3 (benchmark)	10.0	6.5	13.2	14

Claims

1. A composite material (2) comprising a cemented carbide matrix (4) embedded with diamond entities (6) which are homogeneously distributed throughout;

   wherein the constituents of the cemented carbide comprise a tungsten carbide and a binder phase;
   wherein the grain size of the metal carbide is 0.6-8 microns;
   wherein the material comprises 5-65 vol% diamond entities;
   characterized in that:
   at least 20% of the diamond entities (6) contains pores and / or cracks (8) that are filled with constituent(s) of the cemented carbide.

2. The composite material (2) according to claim 1 wherein the binder content of the cemented carbide matrix is between 5 - 20 weight percent (wt%).

3. The composite material (2) according to any of the previous claims wherein the average diameter of the diamond entities (6) is between 10 - 500 μm.

4. The composite material (2) according to any of the previous claims wherein at least 25% of the diamond entities comprise a plurality of crystals having a two or more different orientations wherein different orientations is defined as two or more substantially neighbouring diamonds crystals having at least 10 degrees difference in orientation.

5. An insert (10) for a mining or rock cutting or wear part application comprising the material (2) according to any of claims 1 - 4.

6. The insert (10) according to claim 5 comprising a domed tip portion (14) comprising the composite material (2) according to any of claims 1-5 and a base portion (12) comprising cemented carbide.

7. The insert according to claim 6 wherein the base portion (12) comprises chromium.

8. The insert according to claim 6 or 7 wherein the base portion (12) comprises 4-15 wt% Co and has a room temperature hardness between 900 - 1650 Vickers.

9. A method for making a material (2) according to any of claim 1-4 comprising the steps of:

   a) providing friable diamond grains;
   b) providing sintered cemented carbide granules (SCCG);
   c) blending the diamond grains with the sintered cemented carbide granules to form a homogenous powder blend;
   d) placing the powder blend into preformed refractory metal cup;
   e) providing a refractory metal lid, or, a pre-sintered or sintered cemented carbide base on top of the powder blend to close the cup;
   f) pre-compacting the powder in the refractory metal cup;
   g) surrounding the cup with a pressure media;

h) inserting the pressure media surrounded cup into a high pressure high temperature container;
i) placing the above container in a high pressure high temperature press and sintering at high pressure and high temperature to form a composite material.

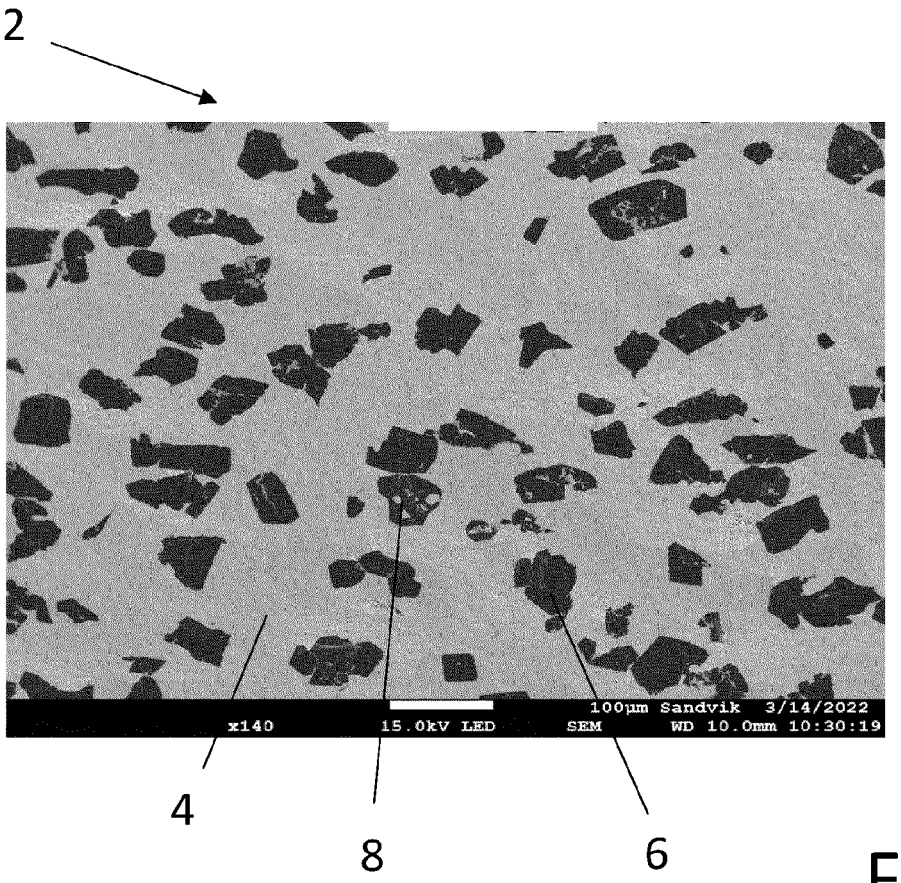
10. The method according to claim 9 wherein the D50 size of the SCCG is in the range of 5-60 microns.

11. The method according to claim 9 or 10 wherein the average tungsten carbide grain size within the SCCG is between 0.6 - 8 μm.

12. The method according to any of claims 9-11 wherein the relative powder density of the SCCG is >35% compared with the density of the fully sintered body of such granules.

13. The methods according to any of claims 9-12 wherein the SCCG has a K1C fracture toughness >10 MPa/m.

14. The method according to any of claim 9-13 wherein the SCCG have graphite on their surface prior to HPHT step.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SEM
100µm Sandvik 3/14/2022
WD 10.0mm 10:30:19
4
8
6

Fig 1a

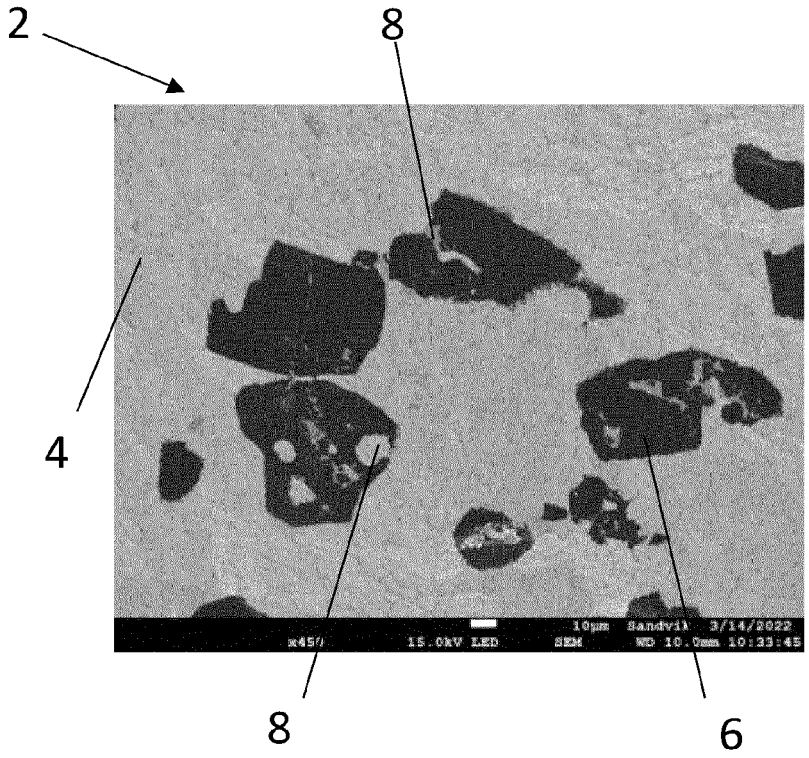
2
8
4
8
6

Fig 1b

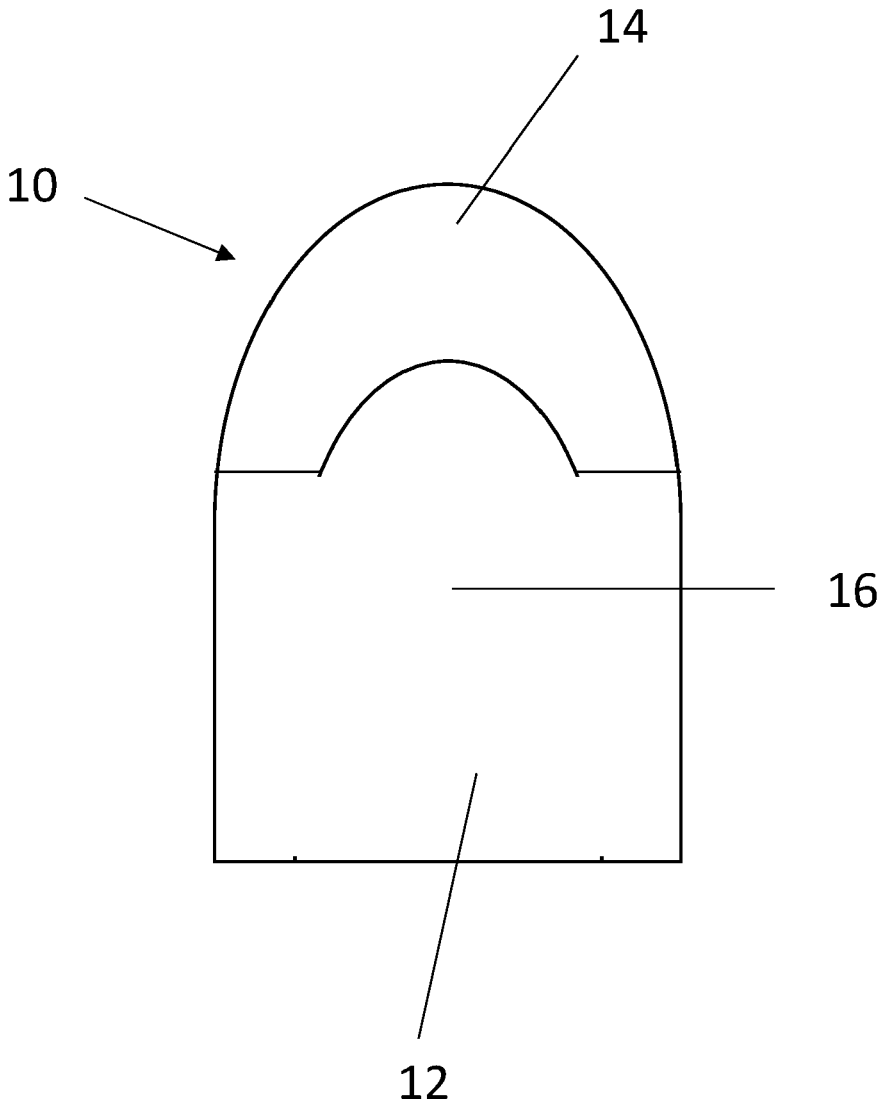

Fig 2

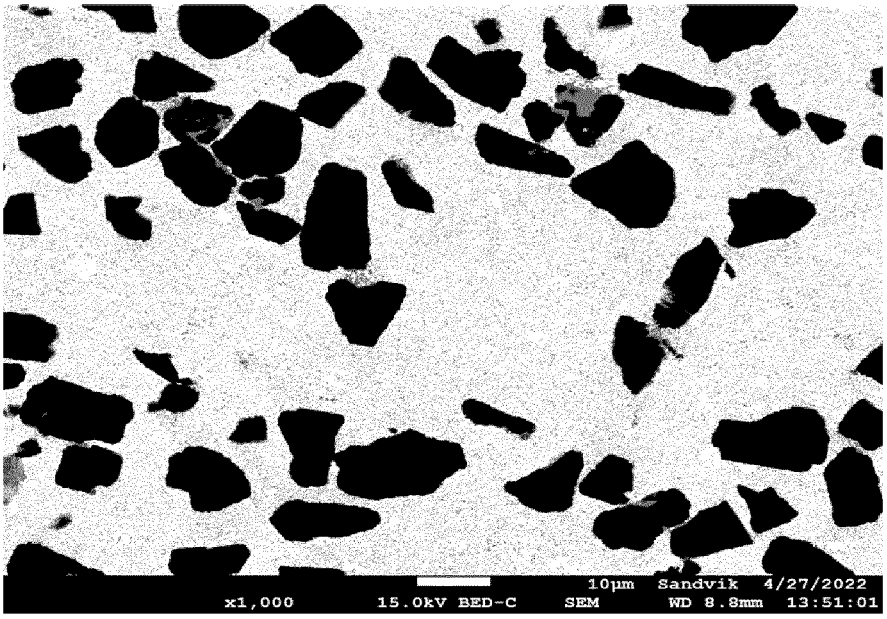

Fig 3

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The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
The Hague	4 August 2023	Momeni, Mohammad

CATEGORY OF CITED DOCUMENTS

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Place of search	Date of completion of the search	Examiner
The Hague	4 August 2023	Momeni, Mohammad

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