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(54) **CUTTING TOOL**

(57) The invention relates to a cutting tool comprising a cutting element; wherein the cutting element is made of a composite material comprising diamond entities embedded in a cemented carbide matrix where the diamond entities are homogeneously distributed throughout the cemented carbide matrix and wherein the composite material comprises 5 - 65 volume % diamond entities and

wherein at least 20% of the diamond entities contains pores and/or cracks that are filled with constituent(s) of the cemented carbide matrix. The cutting tool shows improved properties such as toughness and hardness. The invention also relates to a method of making such a composite.

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Description

[0001] The present invention relates to a composite material comprising diamond entities in a cemented carbide matrix for use as cutting tools in cutting operations like e.g. drilling, milling and turning 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 industrial applications like metal cutting etc. However, for toughness demanding cutting applications, PCD works less well due to its brittle behavior, which limits the lifespan of the cutting tools. 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 cutting tools. 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 cutting tools.

[0004] One objective of the present invention is to obtain a cutting tool that have both good wear resistance and toughness.

Definitions

[0005] By "cemented carbide" is herein meant a material that comprises a hard phase and a metal binder where at least 50 wt% of the hard phase is tungsten carbide. The cemented carbide can possibly also comprise 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 at or above the diamond stable region (>50 kBar) and with temperatures of at least about 1000°C.

[0007] By "diamond entity" is herein meant either a single diamond grain or two or more diamond grains bonded together, i.e. a diamond cluster.

[0008] 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.

[0009] By "cutting tool" is herein meant any tool used in cutting applications such as milling, turning, drilling etc. The cutting tool comprises at least one cutting edge. The cutting tool can have different shapes, depending on the application. The cutting tool can either be completely made from the composite material or the cutting tool can comprise at least one cutting element comprising at least one cutting edge made from the composite material.

[0010] By "cutting element" is herein meant the part of the cutting tool that is engaged in the cutting operation, i.e. the part that comprises the at least one cutting edge and is in contact with the work piece. A cutting element can for example be a tip for an insert, a solid top for a drill etc. The remaining part of the cutting tool, i.e. that is not the cutting element, is called the carrier body.

Detailed description of the invention

[0011] The present invention relates to a cutting tool comprising a cutting element; wherein the cutting element is made of a composite material. The composite material comprises diamond entities embedded in a cemented carbide matrix where the diamond entities are homogeneously distributed throughout the cemented carbide matrix. The cemented carbide matrix comprises tungsten carbide and a metal binder, wherein the metal binder content is between 4 and 20 wt% and a mean grain size of the tungsten carbide is 0.4-8 μm ; - wherein the composite material comprises 5-65 volume % 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 matrix.

[0012] 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.

[0013] The cemented carbide matrix comprises tungsten carbide and a metal binder.

[0014] In one embodiment of the present invention, the cemented carbide can also comprise other components common in the art of cemented carbides, i.e. one or more elements selected from Cr, Ta, Ti, Nb, Mo, Zr and V present as elements or as carbides, nitrides or carbonitrides. These elements can be added either to form a gamma phase (also called a cubic phase) or as grain growth inhibitors to limit the grain growth of the WC grains during HPHT or during the sintering of the cemented carbide granules. The amount of the added elements will depend on the specific element and the purpose of the addition but will be between 100 ppm up to 15 wt%.

[0015] The addition of a grain growth inhibitor will control the WC grain growth in the 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 Cr it also provides the advantage of increasing the plastic deformation and corrosion resistance of the material.

[0016] The presence of gamma phase increases the wear resistance of the cemented carbide matrix. Most commonly, gamma phase contains carbides and/or nitrides of tantalum, niobium and titanium. Presence of gamma phase increases the plastic deformation resistance at elevated temperatures.

[0017] The metal binder of the cemented carbide matrix is selected from cobalt, nickel, iron or a mixture thereof, preferably cobalt. The binder phase may also contain other elements such as Cr, V, Ti, Ta, W, Nb if such elements are added, since they will inevitably be dissolved in the binder during sintering. The binder will be one of the constituents that fills the pores and/or cracks in the diamond comprising components. The amount of binder is between 4 to 20 wt%, preferably between 5 and 15 wt%. This is measured by using wavelength dispersive quantitative methods such as microprobe analysis on cemented carbide areas of the sintered sample. Preferably it is measured by using energy dispersive spectroscopy (EDS) on cemented carbide areas of the sintered composite.

[0018] The mean grain size of the tungsten carbide in the cemented carbide matrix is between 0.4-8 μm, more preferably between 0.5 - 6 μm, most preferable 0.5 - 5 μm.

[0019] The WC grain size is evaluated either using the Jeffries method described below, from at least two different micrographs for each material. 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 appropriate size was selected within the SEM micrograph to contain at least 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 = mean 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 the WC-grain size could also be determined using EBSD on a cross-section of an ion polished sintered sample. This method is preferably used for other binder metals than Co. This is more precise, but also more time-consuming method that gives information regarding grain size distribution. When comparing Jeffries and EBSD grain size the area D50 value from EBSD corresponds well with the Jeffries value.

[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

[0023] The hit rate for WC phase on the raw data is preferable at least 50 %.

[0024] 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).

[0025] 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.

[0026] The diamond entities can be in the form of single diamond grains or diamond clusters or a mixture thereof. A diamond cluster being defined as two or more diamond grains bonded together.

[0027] The diamond entities could have either a regular or irregular shape.

[0028] In the composite material, the diamond entities are embedded in a cemented carbide matrix. The cemented carbide matrix provides toughness to the composite material. Hence it is preferred that the cemented carbide matrix is a continuous phase. The continuity of the cemented carbide matrix could for instance be investigated using scanning electron microscopy (SEM) or light optical microscopy (LOM). The cemented carbide matrix is considered continuous if a path of cemented carbide matrix (wider than a thin film between diamond grains) is present continuously throughout the part or from the surface of the composite material to the base of the part in a LOM or SEM image(s).

[0029] The amount of diamond entities in the composite is between 5-65 vol%, preferably 10-60 vol% and more preferably 15-55 vol%. Advantageously, this range provides the optimal balance between hardness and toughness.

[0030] The vol% of diamond entities are e.g. measured by SEM-image. This is analyzed from a SEM-image = or < 1kX on an ion polished surface with only a small difference in height between the diamond and the cemented carbide preferable using back scattered electrons. The images are imported to the Fiji ImageJ software and the threshold are set so that the cemented carbide phase is the targeted phase, and the surroundings are calculated as background. The area% of the background is regarded to correspond to the vol% of diamond entities. Alternately, the threshold is set so that the diamond phase is the targeted phase, and the surroundings are calculated as background. The area% of the targeted phase is regarded to correspond to the vol% of diamond entities. A filter(s) may be applied (e.g. a Despeckle filter to reduce noise) prior and or after the thresholding.

[0031] 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.

[0032] The diamond entities are homogenously distributed throughout the cemented carbide matrix in three dimensions in terms of distance between the neighboring diamond entities throughout the material. The volume of the diamond entities throughout the material is homogenously distributed. The size distribution of the diamond entities 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 on either size, volume or number of 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 analyzed by comparing SEM or LOM images from an area 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 $1/10^{\text{th}}$ 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 $1/10^{\text{th}}$ of the distance from the substrate if there is a substrate present or $1/10^{\text{th}}$ of the distance from the bottom of the insert if there is no substrate present.

[0033] 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 by using the Fiji Image J software using the same methodology as when measuring the amount of diamond entities, see above, but with the addition that the particle size function with the diamonds as the targeted phase, is used on a SEM-image of an ion polished close to planar surface. 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 filled by the constituents of the cemented carbide thus further increasing the toughness of the diamond entities.

[0034] According to the present invention the pores and / or cracks in the diamond entities are filled with constituent(s) from the cemented carbide matrix. 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] According to 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.

[0036] 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 neighboring diamonds crystals having at least 10 degrees difference in orientation.

[0037] In one embodiment the diamond entities include single crystal diamonds. The D50 of the diamond single crystals are between 8-100 μm , preferable 8-80 μm , more preferably 10-60 μm , even more preferably 12-80 μm , most preferably 15- 80 μm or 12-60 μm . The diamond single crystal grain size is analyzed 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.

With and without Carbide back/carrier body/insert design

[0038] In one embodiment of the present invention, when the cutting tool comprises a cutting element of the composite material, the composite material is bonded to a cemented carbide base, sometimes also called "carbide back". The bonding is done already during the manufacturing of the composite material. A cemented carbide base makes it easier to handle the cutting element during the manufacturing process, e.g. during cutting off the cutting elements to the final shape, but a cemented carbide base also makes it easier to braze the cutting element to a carrier body.

[0039] In one embodiment of the present invention, the cutting tool has a cylindrical shape and contains only the cutting element and the cemented carbide base.

[0040] In one embodiment of the present invention, the cutting element is a so called "cutting tip" with or without a cemented carbide base, that is placed in a pocket on a carrier body.

[0041] By "carrier body" is herein meant the tool body, that do not constitute the cutting element, with or without the carbide base.

[0042] In one embodiment of the present invention the cutting tool is an insert and the carrier body then comprises at least one pocket (in the art also called recess, notch, seat etc.) where a cutting element is situated. The carrier body can have any shape of a cutting tool insert. The cutting tip is usually fastened by brazing.

[0043] In one embodiment of the present invention the cutting tool is a rotary cutting tool, i.e. a drill, end mill or reamer etc. Then the cutting element can e.g. be a solid head or a vein.

[0044] In one embodiment of the present invention the cutting element is a so called "free standing" composite, and by that is meant that no cemented carbide base is present and the cutting element either constitutes the complete cutting tool or, is brazed directly to the carrier body.

[0045] The carrier body can be made of any material common in the art of cutting tools, the most commonly used is cemented carbide. Suitably the cemented carbide can have the composition as described above for the cemented carbide matrix in the composite according to the present invention. The material, size and shape of the base portion can

be tailored to suit the application.

[0046] In one embodiment of the present invention, the cutting tool is provided with a coating. The coating can be any coating suitable for cutting operations, such as a CVD (Chemical vapor deposition) or PVD (Physical vapor deposition) coating. It is common in the art to provide cutting tools with a coating in order to increase the tool life.

[0047] In one embodiment of the present invention, the cutting tool is provided with a CVD coating comprising several layers, suitably at least a carbonitride layer of e.g. Ti and an Al₂O₃ layer, preferably at least one Ti(C,N) layer, an α-Al₂O₃ and an outer TiN layer.

[0048] In one embodiment of the present invention, the cutting tool is provided with a CVD diamond coating.

[0049] In one embodiment of the present invention, the cutting tool is provided with a PVD coating, suitably being a nitride, oxide, carbide or mixtures thereof of one or more of the elements selected from Al, Si and groups 4, 5 and 6 in the periodic table.

[0050] The present invention also relates to a method of making a cutting tool at least partly comprising the composite as described above.

[0051] The method comprises the steps of:

- a) providing friable diamond grains
- b) providing sintered cemented carbide granules;
- 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 make a closed 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.

[0052] 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. Using cemented carbide granules is beneficial because you get better control of the final WC-Co structure in terms of grain size, binder content and properties of the cemented carbide matrix.

[0053] 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.

[0054] 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.

[0055] Examples of diamond feedstock with more features making them friable 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.

[0056] In one embodiment of the present invention the d50 size of the diamond grains is between 6-150 μm, preferably between 6-100 μm, more preferably between 6 - 80 μm, even more preferably between 6-50 μm, even more preferably between 6-40 μm.

[0057] The particle size distribution was measured using laser diffraction fully compliant with ISO 13320 for the complete size range from 0.1 μm to 8750 μm from Sympatec GmbH. The instrument was a Helos BR with Rodos M/Vibri dry sampling unit. The results (D10, D50 and D90) presented in this filing are calculated by the software. D10, D50, or D90 is defined as the size value corresponding to cumulative size distribution at 10%, 50%, or 90%, which represents the size of particles below which 10%, 50%, or 90% of the sample lies. Alternative notations are x10, x50 and x90, which the Windox software uses.

[0058] The grain size of the sintered cemented carbide granules expressed as D50 is between 5-60 μm. Advantageously, this range provides good flowability and high powder density and the mass of each sintered cemented carbide granules is more equal to the mass of a diamond particle. The D50 size of the cemented carbide granules is preferably

between 5-40 μm , even more preferably 8- 30 μm , most preferably 8-25 μm . The particle size distribution was measured using laser diffraction fully compliant with ISO 13320 for the complete size range from 0.1 μm to 8750 μm from Sympatec GmbH using a Helos BR instrument with Rodos M/Vibri dry sampling unit. The powder is analyzed with a combination of R3 (0.9 to 175 μm) and R5 (4.5 to 875 μm) measuring ranges. For each measuring range the samples are analyzed 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 .

[0059] In one embodiment the D90 size of sintered cemented carbide granules 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 feedstock 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.

[0060] 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.

[0061] The D10, D50 and D90 are calculated using Windox software. D10, D50 and D90 is defined as the size value corresponding to the 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.

[0062] In one embodiment the powder density of the sintered cemented carbide granules is at least 35%, preferably at least 40%, more preferably at least 50% compared with the fully dense sintered bodies of such granules. The powder density (or apparent density) is measured using a Hall flow meter and filling a known volume (hall density cup) using a funnel placed above the cup.

[0063] In one embodiment the sintered granules of cemented carbide particles are fully dense.

[0064] In one embodiment the cemented carbide granules have a tap density > 40% or >50% or >60% relative to 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.

[0065] The WC grain size in the granules can be modified by the choice of raw material, milling time, addition of grain size inhibitors, sintering temperature and time in liquid state and by making this in a separate process it is much easier to steer compared to the prior art.

[0066] The WC grain size within the sintered cemented carbide granules is between 0.4 - 8 μm . Advantageously, this grain size range provides the means to balance and optimize the hardness and toughness for cutting applications. The grain size is measured by image analysis on SEM images either from secondary or back-scatter electron images using Jeffries method giving an mean grain size or from analysis of an EBSD-image on an ion polished surface using the area D50-value.

[0067] Preferably, the mean WC grain size in the sintered cemented carbide granules is between 0.4-8 μm , more preferably between 0.5- 6 μm , most preferably 0.5 - 5 μm .

[0068] 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.

[0069] 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 $^{\circ}\text{C}$, more preferably between 1270-1500 $^{\circ}\text{C}$, most preferably between 1300-1500 $^{\circ}\text{C}$. 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 .

[0070] The sintering process are usually started with a debinding step where the organic binder is removed. The debinding step is usually performed at a temperature between 300 and 600 $^{\circ}\text{C}$.

[0071] 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.

[0072] In one embodiment the sintered granules of cemented carbide particles are fully dense.

[0073] 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 cemented carbide which is a heavier material. The cemented carbide granule size also influences the smallest distance to the next diamond entity.

[0074] The binder phase content, preferably Co, in the sintered carbide granules prior to HPHT is between 4-20 wt%, more preferably 6-15 wt% Co, most preferably 8-13 wt% Co. In the composite after HPHT the binder content in the cemented carbide part can thus range from about 4 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 analyzed by EDS (energy dispersive spectroscopy) or more preferably WDS (wavelength dispersive spectroscopy) on a sufficient large ion polished area where only cemented carbide is present.

[0075] 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).

[0076] For step c) the blending of the diamond grains with the sintered cemented carbide granules could for example be done by vibrating, turbula blending or shaking for example in a commercial paint shaker.

[0077] 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.

[0078] 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.

[0079] For step g) the pressure media could for example be hBN or an NaCl mixture that melts during the high temperature high pressure stage at or above the diamond stable region.

[0080] For step h) the high pressure container could for example be, but is not limited to, a natural or synthetically reconstituted pyrophyllite cube or cylinder.

[0081] 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.

[0082] 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.

[0083] Following HPHT, and after breaking the high pressure high temperature container apart and removing the pressure media, the refractory metal can is removed from the sintered compact e.g. by means of grinding or blasting. The sintered compact may then be further ground to a desired dimension. This may be the final dimension or the dimension of a blank. In the case of a blank, a section from such a blank may be cut off to a final dimension of a cutting tool or be joined to a carrier body. In the case when joined to a carrier body, additional operation such as electro discharge machining (EDM), grinding, brushing etc. may be applied.

[0084] In one embodiment of the present invention the cutting tool made according to the above, is provided with a wear resistant coating using CVD or PVD-technique.

[0085] In one embodiment of the present invention, the cemented carbide substrate is provided with a wear resistant PVD coating, suitably being a nitride, oxide, carbide or mixtures thereof of one or more of the elements selected from Al, Si and groups 4, 5 and 6 in the periodic table.

[0086] In one embodiment of the present invention a CVD coating is deposited comprising a first TiCN layer deposited by MTCVD and a second α -Al₂O₃ layer deposited by CVD. Possibly an outermost color layer for wear detection, e.g. a TiN layer, can also be deposited.

[0087] In one embodiment of the present invention, the cutting tool is provided with a CVD diamond coating.

[0088] The coating can also be subjected to additional treatments, such as brushing, blasting, shot peening etc.

Drawings

[0089]

Figure 1a shows a SEM Image of the composite material F at x140 magnification where A is the cemented carbide matrix, with embedded diamond entities Band C, where C clearly shows diamond entities having pores/cracks filled with cemented carbide matrix.

Figure 1b shows a section of the SEM Image in Figure 1a at a higher x450 magnification where A is the cemented carbide matrix, with embedded diamond entities B and C, where C clearly shows diamond entities having pores/cracks D filled with cemented carbide matrix.

5 Figure 2 shows a relative performance comparison from working example 3 and 4. Example 1

[0090] Samples according to the invention as well as comparative examples were produced by blending diamonds and sintered cemented carbide granules (SCCG) in the desired composition. Cylindrical cemented carbide base portions (substrates) were manufactured using conventional methods. For examples A, B and C, the cemented carbide base was manufactured from the powder being 12 wt% Co, 0.5 wt% TiC, 2.5 wt% (Ta,Nb)C with WC as a balance. For examples F, G and O the cemented carbide base was manufactured from the powder being 6 wt% Co, 0.6 wt% Cr with WC as a balance. For sample I the cemented carbide base was manufactured from the powder being 6 wt% Co with WC as a balance. The desired geometry of the composite material portion was formed on top of the cemented carbide base portion.

15 [0091] The diamond source for samples A, B, F, G, I and O was Resin bond RVG810 from Hyperion Materials & Technologies with the feedstock size according to Table 1.

[0092] For sample C, soft freeze spray dried diamond containing granules was used in the powder mixture having a D100 diamond granule size of 500 μm (MBM diamonds from Hyperion Materials & Technologies), having a bi-modal distribution of the diamond granule sizes with a maximum at 6 μm and 25μm, a powder density of the granules of 1.11 g/cm³ 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 at a debinding step prior to the HPHT step. The debinding was performed in a mixture of hydrogen and nitrogen gas up to 500 °C.

Table 1

Sample	SCCG	Diamond feedstock sizes	Vol% of the diamond
A (invention)	SCCG-1	230/270 US mesh	30
B (invention)	SCCG-1	230/270 US mesh	50
C (comparative)	SCCG-1	Mixture of 20 wt% 4-8 micron & 80 wt% 20-30 microns	30
F (invention)	SCCG-2	230/270 US mesh	30
G (invention)	SCCG-2	325/400 US mesh	30
I (comparative)	SCCG-3	230/270 US mesh	10
O (comparative)	SCCG-6	230/270 US mesh	30

[0093] The sintered cemented carbide granules SCCG-1 and SCCG-2 have been manufactured by spray drying a slurry comprising powders with the composition according to Table 2, a liquid (water/ethanol) and an organic binder (PEG 2% of dry powder weight). The spray dried granules were mixed with graphite powder and then debinded in hydrogen flow between 300 and 500 C. This was followed by sintering in vacuum at 1350 and 1360 °C, respectively (see Table 2) for 90 minutes. After sintering the graphite powder was removed by air classification to obtain a concentrated cemented carbide powder. The total carbon content of the sintered cemented granules is shown in Table 2.

[0094] The sintered cemented carbide granules SCCG-3, and SCCG-6 have been manufactured using soft spray dried cemented carbide granules with a relative density of about 25% and granule size D50 around 80-100 μm and with a maximum size of 250 μm. 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 debinding 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.

[0095] The deagglomerated powder SCCG-3 and SCCG-6 were used as-received after the deagglomeration step.

[0096] The properties of the sintered cemented carbide granules according to Tables 2 and 3.

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Table 2

	Mean WC grain size in the sintered granules (μm) ¹	wt% Co	wt% Cr	wt% Nb	wt% Ta	Sintering temperature ($^{\circ}\text{C}$)
SCCG-1	0.75	13	0.56	-	-	1360
SCCG-2	1.1	12	-	-	-	1350
SCCG-3	1.3	6	-	-	-	1275
SCCG-6	0.9	7.6	-	0.27	1.15	1275

¹ By Jeffries

Table 3

	Theoretical sintered density (g/cm^3) ²	D50 (μm)	D10 (μm)	D90 (μm)	Powder density (g/cm^3)	Relative density (%)	Tap density (g/cm^3)
SCCG-1	14.16	16.7	9.1	28.5	7.9	56	9.0
SCCG-2	14.4	18.1	11.0	29.2	7.2	50	8.1
SCCG-3	14.95	97.5	47.5	162.1	6.30	42	7.18
SCCG-6	14.7	72.9	31.9	138.3	6.09	41	6.69

² The theoretical sintered density can be calculated from the nominal composition. Carbide forming metals are included in the form of their carbides; Cr is regarded as fully dissolved in the binder phase and included as an element.

[0097] 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 $127\mu\text{m}$. This was followed by placing a cemented carbide base body on top of the powder blend to close the cup. The powder blend in the Ti cup was pre-compacting by pressing on the cemented carbide body. The filled Ti cup was then surrounded by hexagonal Boron Nitride (hBN) as pressure media; a Carbon Foil Heater, and a cylinder made up of a mixture of carbon lampblack and sodium chloride. These internal components were 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 kBar pressure. The sintering temperature used is detailed in the Table 4.

[0098] The samples were then HPHT sintered to form dome shaped inserts. Following the HPHT sintering the inserts were then blasted with SiC to clean the composite.

Table 4

Sample	HPHT max. temp ($^{\circ}\text{C}$)	Number of samples	No of samples failed after HPHT	Homogenous wear during SiC-blasting
A (invention)	1350	2	0	yes
B (invention)	1350	2	0	yes
C (comparison)	1350	2	0	No
F (invention)	1350	2	0	yes
G (invention)	1350	3	0	yes
	1400	1	0	yes

(continued)

Sample	HPHT max. temp (°C)	Number of samples	No of samples failed after HPHT	Homogenous wear during SiC-blasting
I (comparison)	1425	2	0	No
O- (comparison)	1375	6	4 delamination	No

[0099] The diamond entities in some of the samples were analyzed using SEM and back scatter electrons as described in the description. The results can be seen in Table 5.

Table 5

Sample	Number of diamond entities in image	Magnification of SEM-image	% of diamond entities that contain pores and/ or defects and /or cracks that are filled with constituent(s) of the cemented carbide
A (invention)	40	200	48
B (invention)	21	370	52
F (invention)	85	140	60

[0100] Figures 1a and 1b show SEM images 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 constituent(s) of the cemented carbide.

Homogeneity

[0101] Samples A, B, I and O 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 cemented carbide constituents were black and the diamond entities that contained cemented carbide constituents also contained white areas or spots. The same threshold was used on all images. The images were then converted to binary images and the "particle size" was measured and the area of the "particles" (cemented carbide-matrix) was achieved. The area% of the diamond entities was calculated as 100% minus the area of the cemented carbide matrix % (CC-matrix %). The area% is regarded to correspond with the volume% of the phases. Table 6 shows that homogenous samples were only achieved when blending diamonds with sintered cemented carbide granules having a high powder density and a low D50 and D90.

Table 6

	Area ~100 μm from the surface		Area ~100 μm above the CC base	
Sample	Area % of diamond entities	Analysed area (10 ⁶ μm ²)	Area % of diamond entities	Analysed area (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

[0102] The results from Table 6 show that the distribution of the diamond entities in inventive samples is homogeneous and the distribution of diamond entities in the comparative samples is non-homogeneous.

[0103] Samples F and G (sintered at 1350°C) were analyzed in the same way where a plane parallel section was cut from an insert using EDM cutting and mechanically polished. The samples were then ion-polished using the flat mode

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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. Three large maps for diamond analysis and two small maps for WC analysis was done using Aztec 6.0 software. In Table 7 the microscope and analysis set-up can be seen.

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Table 7

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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

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[0104] The post-processing was performed using AztecCrystal 2.2 software. For diamond map wild spike removal and zero solution removal down to 5 neighbors 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. The results are shown in Table 8.

Table 8

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Sample	Area d50 grain size of diamond by EBSD (μm)	Area d10 grain size of diamonds by EBSD (μm)	Area d90 grain size of diamond by EBSD (μm)	% 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

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Example 2

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[0105] Cutting tool inserts with the geometry ISO RNGN060300 were manufactured as described below by first mixing powder blends comprising diamonds and sintered cemented carbide granules (SCCG) which is then sintered using HPHT.

[0106] The diamonds used for Invention 1-2 and Comparative 1 were of grade RVG810 supplied by Hyperion Materials & Technologies and have a US mesh size of 325/400.

[0107] The diamonds used for Reference 1 were of grade MBM supplied by Hyperion Materials & Technologies.

[0108] The sintered cemented carbide granules used in these set of examples is SCCG-1 from Tables 2 and 3 in Example 1.

[0109] In Table 9, the compositions of the different powder blends and the applicable diamond powder properties are shown.

Table 9

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	Vol% SCCG	Diamond powder (US Mesh size except for Ref.1)
Invention 1	75	325/400
Invention 2	50	325/400

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(continued)

	Vol% SCCG	Diamond powder (US Mesh size except for Ref. 1)
Comparative 1	25	325/400
Comparative 2	100	n.a.
Reference 1	0	Mixture of 20 wt% (4-8 μm) + 80wt% (20-30 μm)
Reference 2	n.a	n.a.

[0110] For Invention 1-2 and Comparative 1, the different powders were mixed for 2.5 min using a Caulk VARI-MIX II. For Reference 1, the diamond powder was prepared (granulated) as described in EP2797851B1.

[0111] The prepared powders for Invention 1-2, Comparative 1-2 and Reference 1 were then placed into Ti cups with a wall thickness of 127 μm, respectively. This was followed by providing a sintered cemented carbide base on top of the powder blend to close the cup. The mentioned cemented carbide body had a weight composition of 12 % Co, 0.5% TiC, 2.5% (Ta,Nb)C and the remainder WC. The powder blend in the Ti cup was pre-compacting by pressing on the cemented carbide body. The filled Ti cup was then surrounded by hexagonal Boron Nitride (hBN) as pressure media; a Carbon Foil Heater, and a cylinder made up of a mixture of carbon lampblack and sodium chloride. These internal components were then contained within a reconstituted pyrophyllite pressure media container. Reference 1 was subjected to a thermal debinding step prior to being surrounded by the internal components. The reconstituted pyrophyllite pressure media container was further placed into a furnace under a vacuum and a temperature of 200°C for 30 min.

[0112] The samples were then all subjected to a treatment in a HPHT apparatus. The cycle in the apparatus comprised a fast ramp for 55 seconds to a max pressure of 52 kBar and a temperature of 1225°C and then a smooth transition into a slower ramp of 300 seconds at 52 kBar gradually climbing up to a sintering soak temperature of 1400 °C. The soaking temperature was held for 200 seconds and followed by a sharp transition into a down ramp with maintained pressure of 52 kBar for 200 seconds; an instant cut of electrical power and a natural cooling ramp with cooling water jackets dissipating the heat for 40 seconds and a 125 second gradual release of the applied pressure. After removing the components and material surrounding the Ti cup, the Ti cup were then removed by grinding to retrieve the sintered compact. The sintered compact was then further ground and finally a top section in the size of a ISO RNGN060300 insert was cut off from the sintered compact.

[0113] Reference 2 was made from a ground blank in commercially available grade AF K40 UF from Hyperion Materials & Technologies with 10 wt. % Co and 90 wt.% WC including Doping (wt%) that were cut off in the size of ISO RNGN060300 inserts.

[0114] The composites Invention 1-2 and Comparative 1-2 were investigated further to analyze the WC grain size, continuity of the Cemented Carbide (CC) matrix and the area % of the cemented carbide matrix (% CC). For Invention 1-2 and Comparative 1, the size of the diamond entities and the degree of diamond entities that have cracks/pores filled with cemented carbide material were also investigated. For the composites Invention 1-2 and Comparative 1-2, cross-section samples were prepared by means of Wire Electro Discharge Machining, grinding and lapping. For Invention 1-2 and Comparative 1, an added step in terms of ion polishing was applied until the cemented carbide matrix and the diamond entities was at the same height level. Thereafter SEM images to be analyzed were obtained for composites Invention 1-2 and Comparative 1-2. The WC grain size was analyzed using Jeffries method.

[0115] The continuity of the CC matrix was determined based on whether the CC material was continuous (wider than a thin film between diamond grains) from the surface to the substrate on the SEM images. The results can be seen in Table 10.

Table 10

	WC mean grain size (μm)	Continuous CC matrix
Invention 1	0.74	Yes
Invention 2	0.74	Yes
Comparative	0.74	No
Comparative 2	0.75	Yes

[0116] The area % of the diamond entities for Invention 1-2 and Comparative 1 was analyzed by processing backscatter SEM images in the Fiji Image J software (version 1.53t). First a Despeckle filter was applied to reduce noise followed by applying a threshold function with a default setting, with the diamond as the targeted phase. Thereafter the analyze

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particle function was applied with outlined particles selected. This returned the area % of the targeted phase (the diamond), as well as, outlined the target particles (the diamond). The area% of the diamond is regarded to correspond to the vol% of diamond entities. For Comparative 2, the % diamond entities were determined based on the input material (100 % Cemented Carbide). The degree of diamond entities that have cracks/pores filled with cemented carbide material was determined by reviewing the corresponding area of the outlined particles (as described above) and its vicinity on the backscatter SEM image. A diamond entity was considered having cracks/pores filled with cemented carbide material if cemented carbide material could be seen on the backscatter SEM image inside the particle or inside a pore in periphery of the particle (to distinguish between roughness/unevenness and a pore in the periphery, it was regarded as a pore if it was about at least as deep as wide). The results are shown in Table 11.

Table 11

	Diamond entities near surface ¹		Diamond entities near substrate ²	
	Area %	Filled %	Area %	Filled %
Invention 1 ³	22 ^{3a}	44 (18) ^{3a}	21 ^{3b}	56 (18) ^{3b}
Invention 2 ⁴	44 ^{4a}	43 (30) ^{4a}	45 ^{4b}	80 (15) ^{4b}
Comparative 1 ⁵	78 ^{5a}	20 (5) ^{5a}	70 ^{5b}	22 (9) ^{5b}
Comparative 2	0	n.a.	0	n.a.

¹ In an area centered at around a distance of 1/10th of the total thickness of the composite material from the surface
² In an area centered at around a distance of 1/10th of the total thickness of the composite material from the substrate
³ Magnification of acquired SEM-image (X): 70.
^{3a} Analyzed area: 500 x 160 μm
^{3b} Analyzed area: 550 x 149 μm
⁴ Magnification of acquired SEM-image (X): 95.
^{4a} Analyzed area: 575 x 140 μm
^{4b} Analyzed area: 500 x 160 μm
⁵ Magnification of acquired SEM-image (X): 70.
^{5a} Analyzed area: 576 x 140 μm
^{5b} Analyzed area: 500 x 160 μm

Example 3 (Working example)

[0117] The inserts made according to Example 2 was then tested in a face turning operation in carbon fiber.
[0118] The following cutting parameters were used.

Feed rate 0.04 mm/rev
 Depth of cut 0.051 mm
 Cutting speed 158 m/min
 Dry

[0119] After 135 passes, the edge rounding was measured with the results according to Table 12. The values are an average of two edges.

Table 12

	Edge rounding (μm)
Invention 1	121
Invention 2	118
Comparative 1	116
Comparative 2	152
Reference 1	113

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(continued)

	Edge rounding (μm)
Reference 2	142

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[0120] The degree of edge rounding gives an indication of the wear resistance of the cutting tool insert.

Example 4 (Working example)

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[0121] The inserts made according to Example 2 was then tested in a face milling operation in Ti6AlV4.

[0122] The following cutting parameters were used.

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Cutting speed	31 m/min
a_p	0.51 mm
f_z	0.46 mm/tooth
Coolant:	Emulsion

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[0123] After 10 passes, the flank degradation was measured, and the results are shown in Table 13.

Table 13

	Flank degradation (μm)
Invention 1	103
Invention 2	186
Comparative 1	814
Comparative 2	61
Reference 1	345
Reference 2	11

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[0124] The degree of flank degradation gives an indication of the toughness of the cutting tool insert.

Relative performance comparison

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[0125] As one objective of the present invention was to obtain a cutting tool that have both good wear resistance and toughness, the results from Example 3 Working example and from Example 4 Working example was combined on a relative basis as shown in Figure 2. For this, the "Wear resistance" (relative) axis was based on assigning Reference 1 to 100 % and Reference 2 to 0%. Vice versa, the "Toughness" (relative) axis was based on assigning Reference 1 to 0 % and Reference 2 to 100 %. Additionally, a linear trendline was added between Reference 1 and 2 to show a proportional loss and gain in the properties.

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[0126] Compared to the linear trade off in properties between Reference 1 and 2, Invention 1 and 2 clearly demonstrates an improvement whereas Comparative 1 and 2 do not show such improvement.

Claims

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1. A cutting tool comprising a cutting element; wherein the cutting element is made of a composite material; the composite material comprises diamond entities embedded in a cemented carbide matrix where the diamond entities are homogeneously distributed throughout the cemented carbide matrix, wherein the cemented carbide matrix comprises tungsten carbide and a metal binder and wherein the metal binder content is between 4 and 20 wt% and the mean grain size of the tungsten carbide is 0.4-8 μm ; and

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- wherein the composite material comprises 5 -65 volume % 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 matrix.

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2. A cutting tool according to any of the previous claims wherein the average diameter of the diamond entities is between 10-500 μm .
3. A cutting tool according to any of the previous claims wherein the composite material comprises 10-60 vol% diamond entities.
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4. A cutting tool according to any of the previous claims wherein at least 25% of the diamond entities contains pores and/or cracks that are filled with constituent(s) of the cemented carbide matrix.
5. A cutting tool according to any of the previous claims wherein the cemented carbide matrix comprises one or more of elements selected from Cr, Ta, Ti, Nb, Mo, Zr and V present as elements or as carbides, nitrides or carbonitrides.
- 15
6. A cutting tool 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 and wherein the diamond comprising entities comprise two or more substantially neighbouring diamonds crystals with at least 10 degrees difference in orientation.
7. A cutting tool according to any of the previous claims where the cutting element is provided with a carbide base.
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8. A cutting tool according to any of the previous claims where the cutting tool is provided with a PVD or CVD coating.
9. A method for making a cutting tool according to any of claim 1-x comprising the steps of:
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- a) providing friable diamond grains;
 - b) providing sintered cemented carbide granules;
 - c) blending the diamond grains with the sintered cemented carbide granules to form a 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 make a closed cup;
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 - 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
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 - high temperature to form a composite material.
10. A method according to claim 9 wherein the composite material is provided with a carrier.
11. A method according to any of claims 9-10 wherein a coating is deposited onto the cutting tool.
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12. The method according to any of claims 9-11 wherein the D50 size of cemented carbide granules is between 5-60 microns.
13. The method according to claim 9-12 wherein the mean WC grain size in the sintered cemented carbide granules is between 0.4 - 8 μm .
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14. The method according to any of claims 9-13 wherein the powder density is at least 35% compared with the density of the fully sintered bodies of such granules.
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15. The method according to any of claim 9-14 wherein the cemented carbide granules have graphite on their surface prior to HPHT step.
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Figure 1a

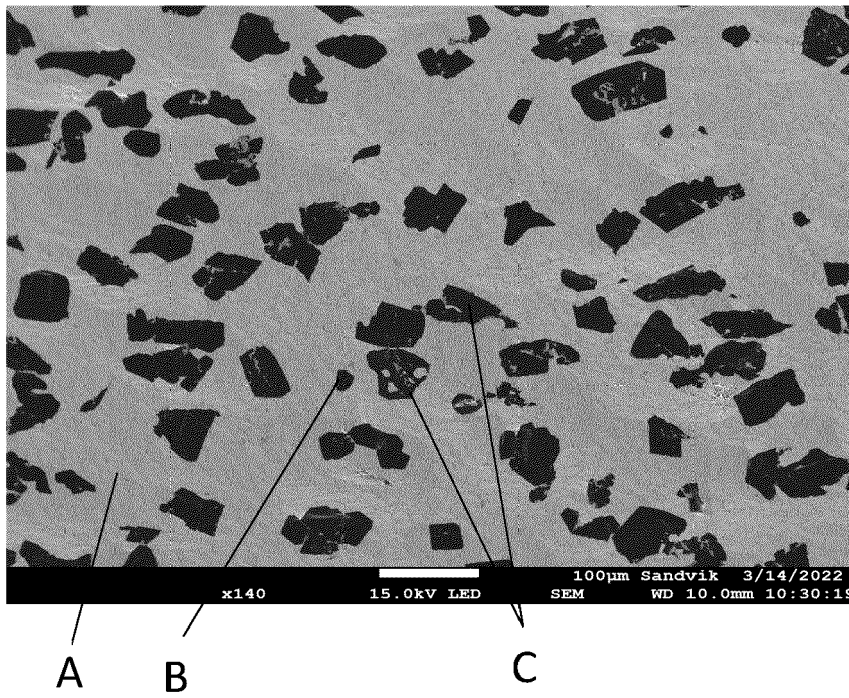


Figure 1b

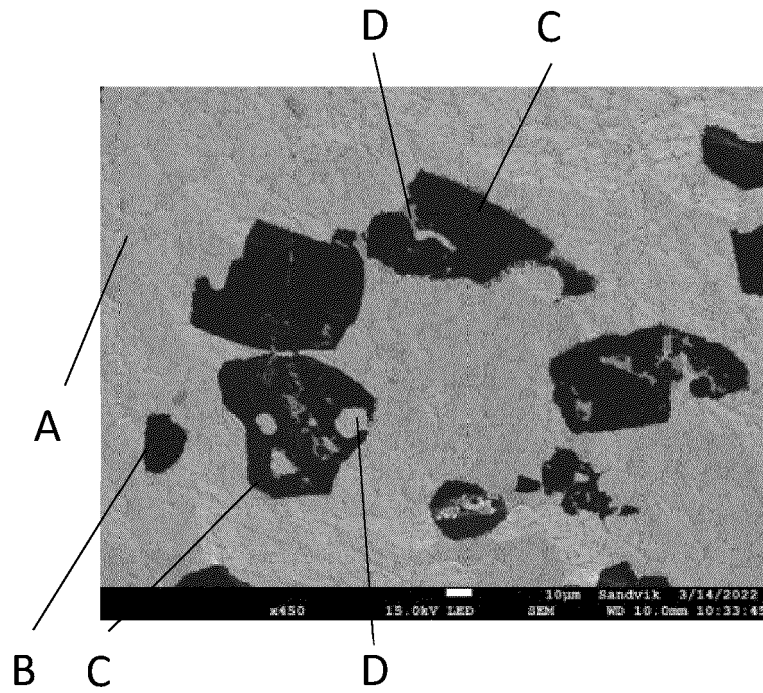
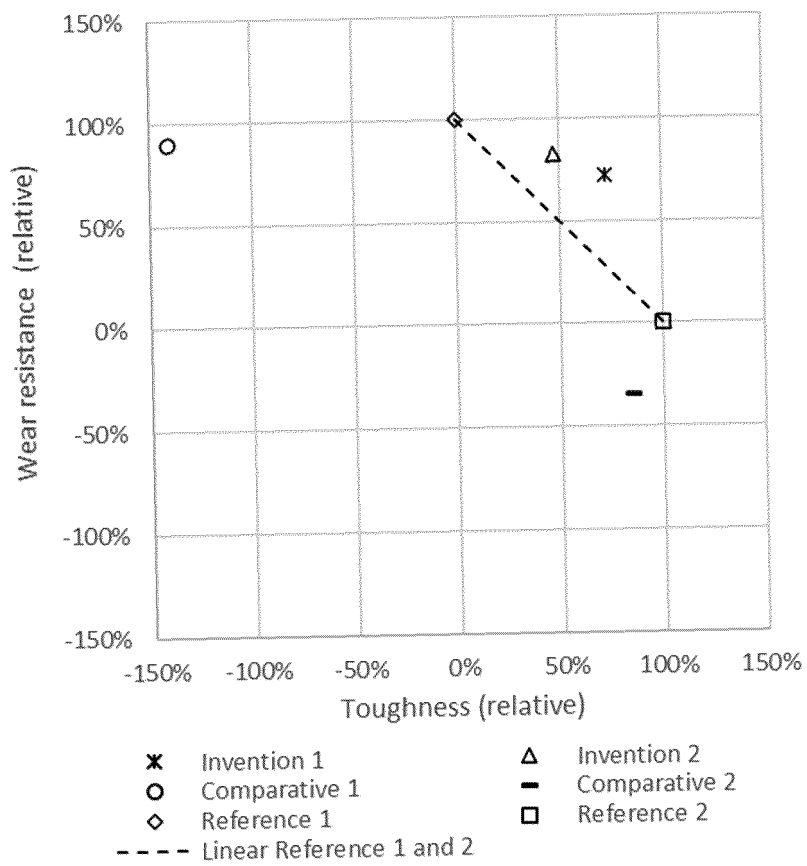


Figure 2





EUROPEAN SEARCH REPORT

Application Number

EP 23 15 4269

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DOCUMENTS CONSIDERED TO BE RELEVANT

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Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (IPC)
X	US 2011/020163 A1 (NILEN ROGER WILLIAM NIGEL [ZA]) 27 January 2011 (2011-01-27) * examples 3-5 *	1-15	INV. B22F3/15 B22F3/23 C22C26/00 C22C29/08
X	YEHA H M ET AL: "Effect of diamond additions on the microstructure, physical and mechanical properties of WC- TiC-Co/Ni Nano-composite", INTERNATIONAL JOURNAL OF REFRACTORY METALS AND HARD MATERIALS, ELSEVIER, AMSTERDAM, NL, vol. 71, 22 November 2017 (2017-11-22), pages 198-205, XP085328405, ISSN: 0263-4368, DOI: 10.1016/J.IJRMHM.2017.11.018 * pages 203-204; "3.6 Hardness" and "3.7 Fracture toughness" * * page 199; "2.1 Materials" * * figure 5 * * Abstract *	1-15	ADD. B22F5/00
X	EP 1 028 171 A1 (SUMITOMO ELECTRIC INDUSTRIES [JP]) 16 August 2000 (2000-08-16) * paragraphs [0034], [0092]; tables 1,14 *	1-15	TECHNICAL FIELDS SEARCHED (IPC) B22F C22C
X	EP 1 231 288 A1 (SUMITOMO ELECTRIC INDUSTRIES [JP]) 14 August 2002 (2002-08-14) * paragraphs [0024], [0025], [0032], [0038]; tables 1,5-9 *	1-15	
X	US 2011/226532 A1 (JONKER CORNELIS ROELOF [ZA] ET AL) 22 September 2011 (2011-09-22) * paragraphs [0079], [0081], [0101]; figures 2,3 *	1-15	

The present search report has been drawn up for all claims

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55

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Application Number
EP 23 15 4269

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20

25

30

35

40

45

50

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DOCUMENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (IPC)
X	US 2013/213721 A1 (NILEN ROGER WILLIAM [ZA] ET AL) 22 August 2013 (2013-08-22) * paragraph [0094]; claim 1; figure 3 * -----	1-15	
			TECHNICAL FIELDS SEARCHED (IPC)
The present search report has been drawn up for all claims			
Place of search The Hague		Date of completion of the search 4 August 2023	Examiner Momeni, Mohammad
CATEGORY OF CITED DOCUMENTS X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document		T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons & : member of the same patent family, corresponding document	

2
EPO FORM 1503 03:82 (F04C01)

ANNEX TO THE EUROPEAN SEARCH REPORT
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EP 23 15 4269

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Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US 2011020163 A1	27-01-2011	AU 2009237260 A1	22-10-2009
		CA 2713595 A1	22-10-2009
		CN 101952468 A	19-01-2011
		EP 2265738 A1	29-12-2010
		GB 2459272 A	21-10-2009
		JP 2011520031 A	14-07-2011
		KR 20100134117 A	22-12-2010
		RU 2010145994 A	20-05-2012
		US 2011020163 A1	27-01-2011
		WO 2009128034 A1	22-10-2009
		ZA 201005785 B	26-10-2011
EP 1028171 A1	16-08-2000	DE 69621564 T2	09-01-2003
		DE 69627053 T2	25-09-2003
		EP 0774527 A2	21-05-1997
		EP 1028171 A1	16-08-2000
		JP 3309897 B2	29-07-2002
		JP H09194978 A	29-07-1997
		KR 970027339 A	24-06-1997
		US 5889219 A	30-03-1999
EP 1231288 A1	14-08-2002	EP 1231288 A1	14-08-2002
		IL 148377 A	11-02-2007
		KR 20020013581 A	20-02-2002
		WO 0132947 A1	10-05-2001
US 2011226532 A1	22-09-2011	AU 2009305930 A1	29-04-2010
		BR PI0919645 A2	08-12-2015
		CA 2741197 A1	29-04-2010
		CL 2011000883 A1	02-09-2011
		CN 102223973 A	19-10-2011
		CN 102224317 A	19-10-2011
		EP 2342418 A1	13-07-2011
		EP 2358490 A1	24-08-2011
		JP 2012506493 A	15-03-2012
		JP 2012506508 A	15-03-2012
		KR 20110099684 A	08-09-2011
		PE 20120222 A1	19-03-2012
		RU 2011119897 A	27-11-2012
		US 2011226532 A1	22-09-2011
		US 2011253459 A1	20-10-2011
		US 2016207168 A1	21-07-2016
		US 2018333825 A1	22-11-2018
US 2020353591 A1	12-11-2020		
US 2022226965 A1	21-07-2022		
WO 2010046860 A1	29-04-2010		

EPO FORM P0459

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**ANNEX TO THE EUROPEAN SEARCH REPORT
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EP 23 15 4269

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The members are as contained in the European Patent Office EDP file on
The European Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

04-08-2023

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
		WO 2010046863 A1	29-04-2010
		ZA 201103168 B	26-09-2012

US 2013213721 A1	22-08-2013	CN 103210172 A	17-07-2013
		EP 2582905 A2	24-04-2013
		GB 2481313 A	21-12-2011
		GB 2491306 A	28-11-2012
		US 2013213721 A1	22-08-2013
		WO 2011158190 A2	22-12-2011

EPO FORM P0459

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Patent documents cited in the description

- US 7647992 B **[0003]**
- WO 2009128034 A **[0003]**
- EP 2797851 B1 **[0110]**