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(71) Applicant: **Sandvik Intellectual Property AB
811 81 Sandviken (SE)**

(72) Inventors:
- **YIN, Lei**
  Hampton, Middlesex TW12 2RF (GB)
- **HIRSCH, Alexander**
  Oxford, OX2 0AG (GB)
- **CUI, Yi**
  Coventry, CV3 5PT (GB)

(74) Representative: **Klöfver, Jörgen et al
Sandvik Intellectual Property AB
811 81 Sandviken (SE)**

(54) Sintered composite body comprising cemented carbide and cBN grains

(57) The present invention relates to a sintered composite body comprising cemented carbide and cBN grains, wherein the cBN grains are dispersed in a cemented carbide matrix. Said body further comprises a cBN depleted zone extending from the surface of the body and 50-400 μm towards the core of the body and the mean cBN grain size outside the depleted zone is 1-20 μm and the cBN content outside the depleted zone is 0.3-4 wt%.

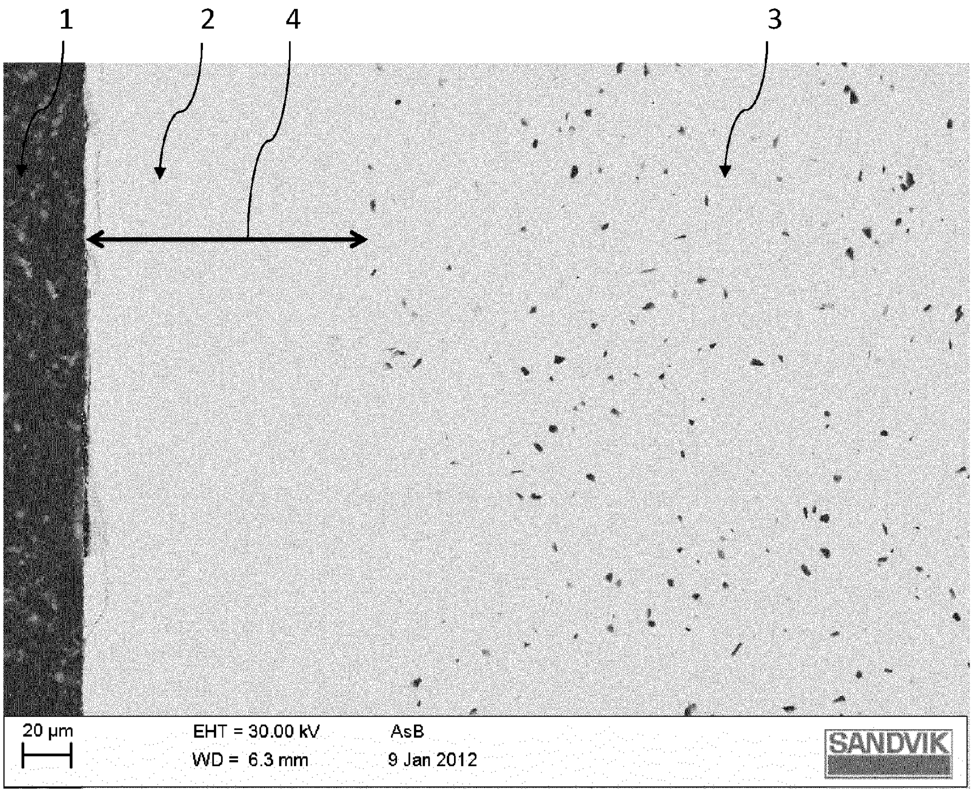

FIG. 1

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Description

Technical field

[0001] The present invention relates to a sintered composite body comprising cemented carbide and cBN grains, wherein the cBN grains are dispersed a cemented carbide matrix and wherein the mean cBN grain size is 1-20 μm and the cBN content is 0.3-4 wt%.

Background

[0002] Cemented carbide components are used in a wide range of applications, especially in components subjected to extreme wear under abrasive conditions. In the oil, gas and mining industry it is a commonly used material in several important components, from drilling bits to general wear parts. The most important features of such components are a combination of high surface hardness and high toughness.

[0003] Cubic boron nitride (cBN) is a superhard material surpassed only by diamond in hardness, which is widely used in demanding applications such as machining tools. cBN is generally crystallographic stable at temperatures below 1400°C.

[0004] One way of increasing the wear resistance of cemented carbide in for example cutting applications is to add particles of a hard material like cBN grains. This has been disclosed for example in EP 0256829 where a high pressure of 50-70 kilo bars is applied during the sintering.

[0005] A problem with a material with increased wear resistance is that also the wear resistance during grinding is increased. Grinding is a common final treatment during production, aimed to achieve a desired shape and surface finish of a product, for example a cutting tool or a saw tooth. Due to an improved wear resistance this step can be costly and also demanding for certain geometries.

Summary

[0006] It is an object of the present invention to provide a cemented carbide body comprising grains of cBN, which is easier to grind into its final shape compared to prior art. It is a further object of the present invention to provide a cemented carbide body comprising grains of cBN with a predetermined grinding property. It is a further object of the present invention to provide a cemented carbide body comprising grains of cBN, with an improved joinability compared to prior art.

[0007] At least one of these objects is achieved by a sintered body according to claim 1. Preferred embodiments are disclosed in the dependent claims.

[0008] The present invention relates to a sintered composite body comprising cemented carbide and cBN grains, wherein the cBN grains are dispersed in a cemented carbide matrix , wherein said body comprises a cBN depleted zone 2 extending from the surface of the body and 50-400 μm, preferably 100-300 μm, towards the core of the body and the mean cBN grain size outside the depleted zone is 1-20 μm, preferably 1-10 μm, more preferably 2-8 μm, and the cBN content outside the depleted zone is 0.3-4 wt%, preferably 0.3-2 wt%, more preferably 0.5-1 wt%.

[0009] The cemented carbide matrix comprises hard constitutes in a metallic binder phase. The binder phase can comprise one or more selected from the group of Co, Ni and Fe and the hard constitutes can comprise WC. The cemented carbide can further comprise hard constitutes selected from borides, carbides, nitrides or carbonitrides of metals from groups 4, 5 or 6 of the periodic table, preferably tungsten, titanium, tantalium, niobium, chromium and/or vanadium.

[0010] The manufacturing of the sintered composite body typically comprises mixing and milling powders of the cemented carbide and cBN, pressing bodies of the powder to a desired shape and finally to sinter the pressed bodies to form dense bodies comprising cBN grains in a cemented carbide matrix. During the sintering process the binder phase liquidize and enclose the hard constitutes and the cBN grains. The depleted zone forms during the sintering step, which is disclosed in more detail below. The milling, mixing and pressing steps can be performed with conventional methods as known in the art.

[0011] The cBN grain size does typically not change during the mixing, milling and pressing steps. The surface of the cBN grain can be coated with a thin layer of a metal element, for example a thin Ti coating, to increase the wetting performance of the grain surface during the sintering step.

[0012] The body of the present invention can be of any shape, for example in the shape of a saw tooth, a drilling button or a wire drawing nib. The core in the body is located inside the body. The shape and extension of the core depends on the shape of the body. For example, in a spherical body the core can be a central point, in a body extended in on direction, the core can be extended, and in a ring shaped body, the core can be ring shaped or cylindrically shaped.

[0013] The cBN depleted zone is hereby meant an area that in SEM analyze at 750x magnification does show that the cBN grains, that normally appear as black spots or small areas in an otherwise continuous matrix of cemented carbide, are missing or depleted. The depleted zone is essentially free of cBN grains and it extends from the surface of the body and down below the surface towards the core of the body. The area where the cBN grains are not depleted extends outside the depleted zone for example towards and through the core of the body.

[0014] One advantage with the sintered composite body according to the present invention is that the grinding of the surface of the body, i.e. grinding of the depleted zone, is more easy to perform due to that the hard particles of cBN are missing in the outer surface area of the material. The depleted zone is less wear resistant than the cBN containing material in the core of the body.

[0015] Another advantage with the sintered composite body according to the present invention is that joining of the body to another body or material can be improved. At brazing or welding the strength of the weld, i.e. the melt region, is dependent of the strength of the material in the weld. And cBN grains are not preferred in a weld due to that cBN grains have a thermal mismatch with the cemented carbide matrix. An advantage with an absence of cBN grains in the weld is that it leads to an absence of stresses due to said thermal mismatch. Also, the wettability of materials in the weld could be improved if no cBN is present. Additionally, cBN as a brittle phase is not present in the joint area. All of these facts lead to an improved joining strength if no cBN is present in the joint. In general, absence of cBN in the welding zone makes it possible to use existing production process parameters for welding and plating, thus reducing the production costs.

[0016] In one embodiment of the present invention, the extension of the depleted zone is 50-200 μm, preferably 100-200 μm. This is preferred in applications focusing on achieving a good surface finish and/or a small radius after surface or cutting edge grinding operations.

[0017] In one embodiment of the present invention, the extension of the depleted zone is 200-400 μm, preferably 200-300 μm. This is preferred in applications requiring high toughness to withstand initial impact. If such a body withstands the initial impact, it thereby has an increased chance to wear with a stable wear rate.

[0018] In one embodiment of the present invention, the cemented carbide comprises 6-16 wt% binder phase. In one embodiment of the present invention the binder phase comprises Co. In one embodiment of the present invention the cemented carbide comprises 10-14 wt% Co.

[0019] In one embodiment of the present invention, the cemented carbide comprises WC. In one embodiment of the present invention, the cemented carbide comprises 80-94 wt% WC. In one embodiment of the present invention the mean WC grain size is 0.5 to 8 μm, preferably 0.5 to 4 μm, most preferably 0.8 to 1.2 μm, as measured with linear intercept method in the sintered material.

[0020] In one embodiment, the present invention relates to a wear part comprising the sintered composite body according to above.

[0021] In one embodiment, of the present invention relates to saw tooth comprising the sintered composite body according as disclosed above.

[0022] The present invention further relates to the use of the sintered composite body in oil or gas applications, for example as a drilling button or an insert for a drilling head.

[0023] The present invention further relates to the use of the sintered composite body in wire drawing applications, for example as a wire drawing nib.

[0024] The composite body according to the present invention can be sintered in a sintering process in accordance with the settings as indicated below.

[0025] The sintering temperature is preferably 1250- 1360 °C, preferably 1300-1360 °C. At a too low sintering temperature, the material will not sinter. It is important to reach the melting point of the binder. A too high sintering temperature results in that the cBN grains decomposes into hBN, which is a less hard phase of BN. The sintering temperature is preferably chosen to achieve full densified bodies and a gradient zone of a preferable depth.

[0026] The sintering can be performed in vacuum. Vacuum sintering is a standard process of production for many cemented carbide manufacturers.

[0027] The sintering can be performed using HIP (hot isostatic pressing). Sintering using a HIP is advantageous in that it leads to higher densities of the materials. It also enables a lower sintering temperature compared to what is possible at vacuum sintering, maintaining full densification of the material.

[0028] The sintering can for example be performed in a gas comprising Ar and/or N_2.

[0029] The temperature is hold at a sintering temp during a dwelling time of preferably 10-80 minutes. A too long sintering time can result in undesired grain growth of the cemented carbide. A too short sintering time can result in not completely sintered material at the centre of a body. The sintering time is suitably adjusted with regards to batch size, sintering equipment, cemented carbide composition, size of bodies, etc. to achieve dense sintered bodies with a preferable gradient depth.

[0030] Other objects, advantages and novel features of the invention will become apparent from the following detailed description of the invention when considered in conjunction with the accompanying drawings and claims.

Brief description of drawings

[0031] Embodiments of the invention will now be described with reference to the accompanying drawings, wherein:

FIG. 1 is a SEM picture of a polished through cut of a sintered body according to a preferred embodiment of the present invention, wherein the grain size of the cBN is 4-8 μm (Sample C). The epoxy resin 1, the depleted zone 2 and an area in the material that is not depleted 3 are indicated. The cBN grains 5 appear as dark spots. The width of the depleted zone is indicated with an arrow 4.

FIG. 2 is a SEM picture corresponding to FIG. 1, but wherein the grain size of the cBN is 2-4 μm (Sample A).

Detailed description

[0032] In the following, examples of bodies according to different embodiments of the invention will be presented, and the method of making the bodies will be disclosed in detail.
[0033] cBN powder comprising cBN grains was milled for 1 hour in a 250 ml laboratory ball mill. After milling of the cBN, cemented carbide powder comprising 86.98 wt% WC, 0.62 wt% Cr_3C_2 and 12.4 wt% Co was added. The amount of cBN was adjusted to equal 0.78 wt% cBN in the mixture with cemented carbide. After the addition of cemented carbide powder, the mixture was milled for another 30 minutes. During the whole process, a mixture of ethanol and water was used as a milling liquid. After milling, the slurry was poured onto a tray and dried over night at 70°C. After that, the powder was sieved using a 500 micron mesh.
[0034] Two cBN grain sizes were analyzed: 2-4 my and 4-8 my respectively. The cBN grains were, as delivered from the manufacturer, coated with a thin coating of Ti. The given size of the cBN grains is the size specified by the manufacturer.
[0035] The dry powder was pressed to a body of a bar with rectangular cross-section. As-pressed dimensions were about 25.5x8x6.5 mm.
[0036] The bodies were sintered in sintering steps defined below. The following sintering parameters were analyzed: temperature, time, pressure and sintering gas, see Tables 2-5 below.
[0037] After the sintering each body were cut through and the through cut was studied in SEM whereby the depth of the depleted zone was studied. Examples of SEM pictures of the depleted zone are shown in FIG. 1 and 2.

Table 1. (grain size)

Sample	cBN grain size (μm)	Pressure (mbar)	Gas	Temperature (°C)	Time (min)	Depleted zone (μm)
A	2-4	5	Ar	1360	70	220
B	4-8	5	Ar	1360	70	180

[0038] As can be seen in Table 1, the extension of the depleted zone is dependent on the grain size of the cBN particles. After a sintering at equal conditions, the sample A with smaller grains of 2-4 μm had a larger width of the depleted zone compared to the sample B with the larger grains of 4-8 μm.

Table 2. (sintering temp)

Sample	cBN grain size (μm)	Pressure (mbar)	Gas	Temperature (°C)	Time (min)	Depleted zone (μm)
C	4-8	5	Ar	1300	70	110
B	4-8	5	Ar	1360	70	180

[0039] As can be seen in Table 2, the extension of the depleted zone is dependent on the sintering temperature. After a sintering at equal conditions but at different temperatures, the sample C sintered at 1300°C had a smaller width of the depleted zone compared to the sample B sintered at 1360°C.

Table 3 (sintering time)

Sample	cBN grain size (µm)	Pressure (bar)	Gas	Temperature (°C)	Time (min)	Depleted zone (µm)
D	2-4	50	Ar	1360	70	280
E	2-4	50	Ar	1360	35	240

[0040] As can be seen in Table 3, the extension of the depleted zone is also dependent on the sintering time. After a sintering at equal conditions but at different sintering times, the sample D sintered at 70 minutes had a larger width of the depleted zone compared to the sample E sintered at 35 minutes.

Table 4 (sintering pressure)

Sample	cBN grain size (µm)	Pressure (bar)	Gas	Temperature (°C)	Time (min)	Depleted zone (µm)
A	2-4	0.005	Ar	1360	70	220
D	2-4	50	Ar	1360	70	280
F	2-4	1	N_2	1360	70	165
G	2-4	50	N_2	1360	70	275

[0041] As can be seen in Table 4, the extension of the depleted zone is also dependent on the sintering pressure. After a sintering at equal conditions in Ar gas, but at different pressures, the sample A sintered at 5mbar had a smaller width of the depleted zone compared to the sample D sintered at 50 bar. The same relation is valid for sintering in N2 gas: the sample F sintered at atmosphere pressure had a smaller width of the depleted zone compared to the sample G sintered at 50 bar.

Table 5 (sintering gas)

Sample	cBN grain size (µm)	Pressure (bar)	Gas	Temperature (°C)	Time (min)	Depleted zone (µm)
D	2-4	50	Ar	1360	70	280
G	2-4	50	N_2	1360	70	275

[0042] As can be seen in Table 5, the sintering gas Ar or N2 did not have any clear diverging effect on the width of the depleted zone. At the sintering conditions shown in Table 5, the depleted zone for sample D and G had about the same width of the depleted zone.

[0043] While the invention has been described in connection with various exemplary embodiments, it is to be understood that the invention is not to be limited to the disclosed exemplary embodiments, on the contrary, it is intended to cover various modifications and equivalent arrangements within the appended claims.

Claims

1. A sintered composite body comprising cemented carbide and cBN grains, wherein the cBN grains are dispersed in a cemented carbide matrix, wherein said body comprises a cBN depleted zone (2) extending from the surface of the body and 50-400 µm towards the core of the body and the mean cBN grain size outside the depleted zone is 1-20 µm and the cBN content outside the depleted zone is 0.3-4 wt%.

2. The sintered composite body according to claim 1, wherein the cBN depleted zone (2) extending from the surface of the body and 50-200 µm towards the core of the body.

3. The sintered composite body according to claim 1, wherein the cBN depleted zone (2) extending from the surface of the body and 200-400 µm towards the core of the body.

4. The sintered composite body according to any of claims 1-3, wherein the cemented carbide comprises 6-16 wt% binder phase.

5. The sintered composite body according to claim 4, wherein the binder phase comprises Co.

6. The sintered composite body according to claim 4, comprising 10-14 wt% Co.

7. The sintered composite body according to any of claims 1-6, wherein the cemented carbide comprises WC.

8. The sintered composite body according to claim 7, wherein the cemented carbide comprises 80-94 wt% WC.

9. The sintered composite body according to claim 7 or 8, wherein the mean WC grain size is 0.5 - 8 μm.

10. A wear part comprising the sintered composite body according to any of claims 1-9.

11. A saw tooth comprising the sintered composite body according to any of claims 1-9.

12. Use of the sintered composite body according to any of claims 1-9 in oil or gas applications.

13. Use of the sintered composite body according to any of claims 1-9 in wire drawing applications.

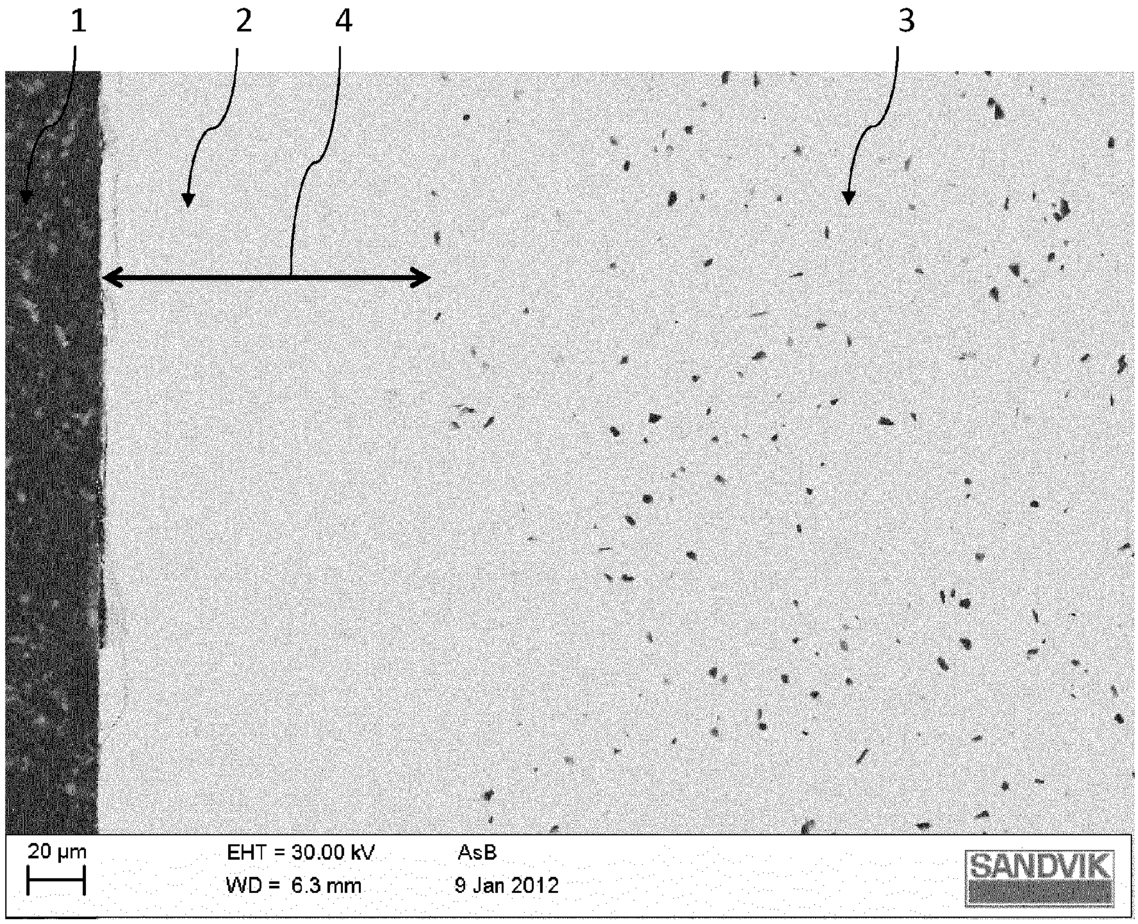

FIG. 1

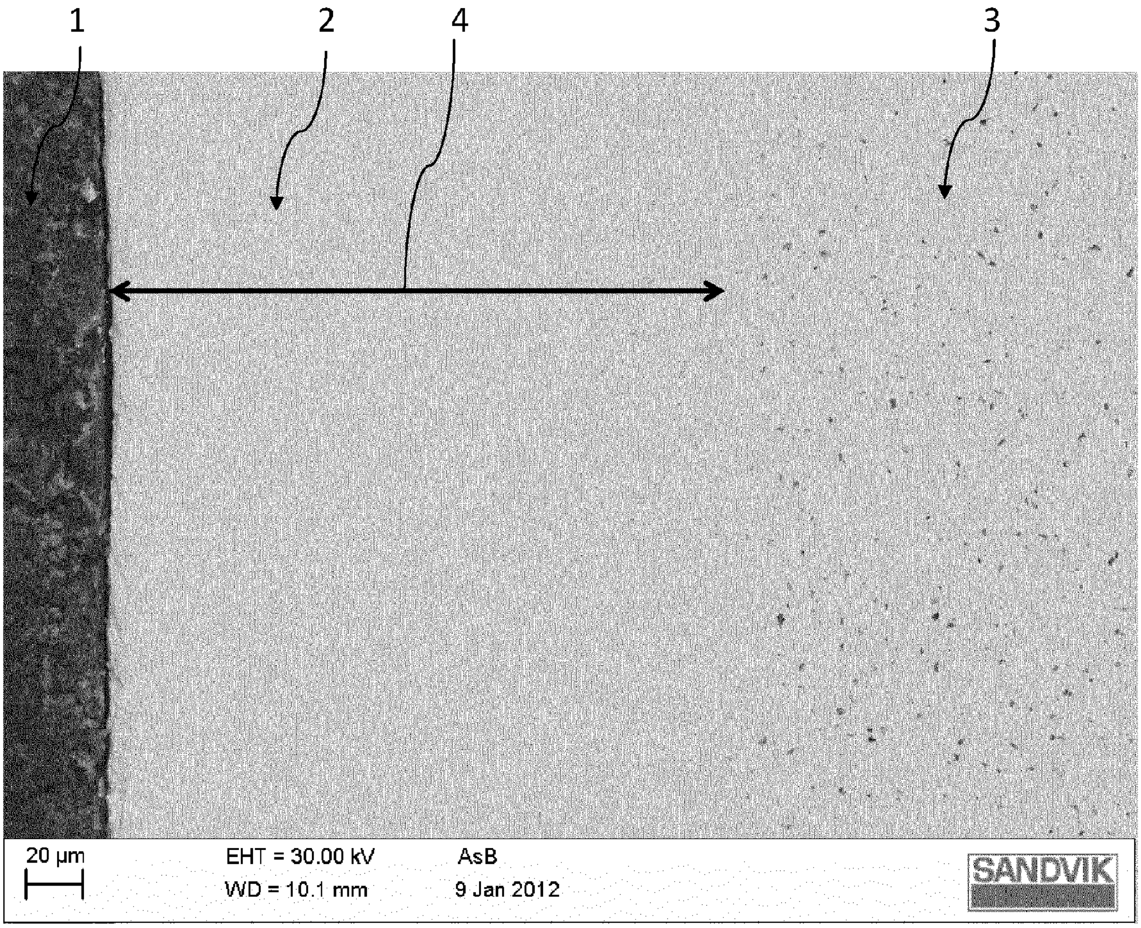

FIG. 2

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EUROPEAN SEARCH REPORT

Application Number
EP 12 16 0972

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (IPC)
E	EP 2 433 727 A1 (SANDVIK INTELLECTUAL PROPERTY [SE]) 28 March 2012 (2012-03-28) * paragraphs [0001], [0002], [0009] - [0012], [0014], [0015], [0017] - [0022], [0026], [0030], [0031], [0033], [0042], [0043], [0047] * -----	1-12	INV. B22F7/06 B22F7/08 B23B27/02 B23B27/14 C22C29/08

TECHNICAL FIELDS SEARCHED (IPC)

C22C
B23B
B22F

The present search report has been drawn up for all claims

1

Place of search	Date of completion of the search	Examiner
Munich	23 August 2012	Brown, Andrew

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

EPO FORM 1503 03.82 (P04C01)

ANNEX TO THE EUROPEAN SEARCH REPORT ON EUROPEAN PATENT APPLICATION NO.

EP 12 16 0972

This annex lists the patent family members relating to the patent documents cited in the above-mentioned European search report.
The members are as contained in the European Patent Office EDP file on
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23-08-2012

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
EP 2433727 A1	28-03-2012	EP 2433727 A1	28-03-2012
		WO 2012038529 A2	29-03-2012

EPO FORM P0459

For more details about this annex : see Official Journal of the European Patent Office, No. 12/82

REFERENCES CITED IN THE DESCRIPTION

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- EP 0256829 A **[0004]**