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(54) Title: COATED CUTTING TOOL INSERT

(57) Abstract: The present invention relates to a CVD-coated cutting tool insert with improved toughness properties having the ability to withstand high temperatures without sacrificing edge line security. The insert coating comprises a TiC_xN_y-layer with a low tensile stress level of 50-500 MPa and an α-Al_20_3 -layer with a high surface smoothness with a mean Ra < 0.12 μm as measured by AFM- technique, obtained by subjecting the coating to an intensive wet blasting operation.

COATED CUTTING TOOL INSERT

The present invention relates to a high performance coated cutting tool insert particularly useful for turning in low alloyed steel, carbon steel and tough hardened steels in the area from finishing to roughing in wet and dry conditions at high cutting speed, having the ability to withstand high temperatures without sacrificing edge security. The insert is based on WC, cubic carbides and a Co-binder phase with a cobalt enriched surface zone giving the cutting insert an excellent resistance to plastic deformation and a high toughness performance. Furthermore, the coating comprises a number of wear resistance layers which have been subjected to a surface post treatment giving the tool insert a surprisingly improved cutting performance.

The majority of today's cutting tools are based on a cemented carbide insert coated with several hard layers like TiC, TiC_xN_y, TiN, $TiC_xN_yO_z$ and Al_2O_3. The sequence and the thickness of the individual layers are carefully chosen to suit different cutting application areas and work-piece materials. The most frequent employed coating techniques are Chemical Vapour Deposition (CVD) and Physical Vapour Deposition (PVD). CVD-coated inserts in particular have a tremendous advantage in terms of flank and crater wear resistance over uncoated inserts.

The CVD technique is conducted at a rather high temperature range, 950-1050 °C. Due to this high deposition temperature and to a mismatch in the coefficients of thermal expansion between the deposited coating materials and the cemented carbide tool insert, CVD can lead to coatings with cooling cracks and high tensile stresses (sometimes up to 1000 MPa). The high tensile stresses can under some cutting conditions be a disadvantage as it may cause the cooling cracks to propagate further into the cemented carbide body and cause breakage of the cutting edge.

In the metal cutting industry there is a constant striving to increase the cutting condition envelope, i.e., the ability to withstand higher cutting speeds without sacrificing the ability to resist fracture or chipping during interrupted cutting at low speeds.

Important improvements in the application envelope have been achieved by combining inserts with a binder phase enriched surface zone and optimised, thicker coatings.

However, with an increasing coating thickness, the positive effect on wear resistance is out-balanced by an increasing negative effect in the form of an increased risk of coating delamination and reduced toughness making the cutting tool less reliable. This applies in particular to softer work piece materials such as low carbon steels and stainless steels and when the coating thickness exceeds 5-10 µm. Further, thick coatings generally have a more uneven surface, a negative feature when cutting smearing materials like low carbon steels and stainless steel. A remedy can be to apply a post smoothing operation of the coating by brushing or by wet blasting as disclosed in several patents, e.g., EP 0 298 729, EP 1 306 150 and EP 0 736 615. In US 5,861,210 the purpose has, e.g., been to achieve a smooth cutting edge and to expose the Al_2O_3 as the top layer on the rake face leaving the TiN on the clearance side to be used as a wear detection layer. A coating with high resistance to flaking is obtained.

Every post treatment technique that exposes a surface, e.g., a coating surface to a mechanical impact as, e.g., wet or dry blasting will have some influence on the surface finish and the stress state (σ) of the coating.

An intense blasting impact may lower the tensile stresses in a CVD-coating, but often this will be at the expense of lost coating surface finish by the creation of ditches along the cooling cracks or it can even lead to delamination of the coating.

A very intensive treatment may even lead to a big change in the stress state, e.g., from highly tensile to highly compressive as disclosed in EP-A-1 311 712, in which a dry blasting technique is used.

It has now surprisingly been found that a cutting tool insert having a combination of a certain cemented carbide substrate composition and a certain coating structure and thickness, and being post treated by wet-blasting under controlled conditions obtains excellent cutting properties over a broader range of applications than prior art cutting tool inserts.

The cobalt binder phase is highly alloyed with W. The content of W in the binder phase can be expressed as the CW-ratio:

CW-ratio = M_s/(wt-%Co*0.0161)

wherein M_s = measured saturation magnetization in hAm^2/kg and wt-% Co is the cobalt content in the cemented carbide. A low CW-ratio corresponds to a high W-content in the Co binder phase. The employed post treatment will give the coating a favourable tensile stress level, the Al_2O_3 layer a certain important crystallographic feature and a top surface with an excellent surface finish.

The mentioned combination with the blasting technique effectively expands the limitations of what coating thickness that can be applied without performance penalty. As a result of the invention application areas of unsurpassed width is now possible. The significant improvements achieved with respect to toughness behaviour and coating adhesion was surprising.

To significantly change the stress state of a coating by blasting, the blasting media, e.g., Al_2O_3 grits have to strike the coating surface with a high impulse. The impact force can be controlled by, e.g., the blasting pulp pressure (wet blasting), the distance between blasting nozzle and coating surface, grain size of the blasting media, the concentration of the blasting media and the impact angle of the blasting jet.

It is an object of the present invention to provide CVD-coated tool inserts with improved toughness properties having the ability to withstand high temperatures without sacrificing edge security or toughness.

Fig. 1 shows a goniometer set-up for the evaluation of residual stress by X-ray measurements in which

E = Euler ¼-cradle

S = sample

I = incident X-ray beam

D = diffracted X-ray beam

θ = diffraction angle

$\omega = \theta$

ψ = tilt angle along the Euler ¼-cradle

Φ = rotation angle around the sample axis

The present invention thus relates to coated cutting tool inserts comprising a body of generally polygonal or round shape having at least one rake face and at least one clearance face, comprising a coating and a carbide substrate. The body has a composition of 4.4-6.6, preferably 5.0-6.0, most preferably 5.0-5.8, wt-% Co, 4-8.5 wt-% cubic carbides, balance WC, preferably 85-91 wt-% WC, most preferably 87-90 wt-% WC, preferably having an average grain size of 1-4 µm, a CW-ratio in the range 0.78-0.92 and a surface zone of a thickness of 15-40 µm, preferably 15-35 µm, most preferably 25-35 µm, depleted from the cubic carbides TiC, TaC and/or NbC. The coating comprises at least one TiC_xN_y-layer and one well-crystalline layer of 100 % α-Al_2O_3. One such α-Al_2O_3 layer is the top visible layer on the rake face and along the cutting edge line and the layer can be intensively wet blasted with a sufficiently high energy to create tensile stress relaxation in both the Al_2O_3 and the TiC_xN_y-layers. The Al_2O_3 top layer has a very smooth surface at least in the chip contact zone on the rake face.

It has surprisingly been discovered that a significant improved toughness performance can be achieved if a coated cutting tool insert with a generally polygonal or round shape having at least one rake face and at least one clearance face, said insert being at least partly coated, produced to possess the following features:

- a penultimate TiC_xN_y layer with a thickness of 5-15 µm, preferably 6-13 µm, most preferably 7-13 µm, where $x \geq 0$, $y \geq 0$ and $x+y=1$, preferably produced by MTCVD, with tensile stresses of 50-500 MPa, preferably 50-400 MPa, most preferably 50-300 MPa and

- an outer α-Al_2O_3-layer with a thickness of 3-12 µm, preferably 3.5-8 µm, most preferably 4-8 µm, being the top layer on the rake face and along the edge line having a mean roughness Ra < 0.12 µm, preferably ≤ 0.10 µm, at least in the chip contact zone of the rake face, measured over an area of 10 µm x 10 µm by Atomic Force Microscopy (AFM) and an XRD-diffraction intensity (peak height minus background) ratio of $I(012)/I(024) \geq 1.3$, preferably ≥ 1.5.

Preferably there is a thin 0.2-2 µm bonding layer of $TiC_xN_yO_z$, $x \geq 0$, $z>0$ and $y \geq 0$ between the TiC_xN_y-layer and the α-Al_2O_3-layer. The total thickness of the two layers is ≤25 µm.

Also according to the present invention, additional layers can be incorporated into the coating structure between the substrate and the layers, composed of metal nitrides and/or carbides and/or oxides with the metal elements selected from Ti, Nb, Hf, V, Ta, Mo, Zr, Cr, W and Al to a total coating thickness of <5 µm.

It is preferred to have some tensile stresses left in the TiC_xN_y layer since it was found that such induced compressive stresses were not as stable with respect to temperature increase, which occurs in a cutting operation, as compared to if the coating has some tensile stresses still present. It was also found, that if compressive stresses were to be induced by blasting, a very high blasting impact force was required and under such conditions flaking of the coating frequently occurred along the cutting edge.

The residual stress, σ, of the inner TiC_xN_y layer is determined by XRD measurements using the well known $\sin^2\psi$ method as described by I.C. Noyan, J.B. Cohen, Residual Stress Measurement by Diffraction and Interpretation, Springer-Verlag, New York, 1987 (pp 117-130). The measurements are performed using CuK_α-radiation on the TiC_xN_y (422) reflection with a goniometer setup as shown in Fig. 1. The measurements are carried out on an as flat surface as possible. It is recommended to use the side-inclination technique (ψ-geometry) with six to eleven ψ-angles, equidistant within a $\sin^2\psi$-range of 0 to 0.5 (ψ=45°). An equidistant distribution of Φ-angles within a Φ-sector of 90° is also preferred. To confirm a biaxial stress state the sample shall be rotated for Φ=0° and 90° while tilted in ψ. It is recommended to investigate possible presence of shear stresses and therefore both negative and positive ψ-angles shall be measured. In the case of an Euler ¼-cradle this is accomplished by measuring the sample also at Φ=180° and 270° for the different ψ-angles. The $\sin^2\psi$ method is used to evaluate the residual stress preferably using some commercially available software such as DIFFRAC^{Plus} Stress32 v. 1.04 from Bruker AXS with the constants Young's modulus, E=480 GPa and Poisson's ratio, ν=0.20 in case of a MTCVD Ti(C,N)-layer and locating the reflection using the Pseudo-Voigt-Fit function. In the case of the following parameters are used: E-modulus=480 GPa and Poisson's ratio ν=0.20. In case of a biaxial stress state the tensile stress is calculated as the average of the obtained biaxial stresses.

For the α-Al_2O_3 it is in general not possible to use the $\sin^2\psi$ technique since the required high 2θ angle XRD-reflections are often too weak. However, a useful alternative measure has been found which relates the state of the α-Al_2O_3 to cutting performance.

For an α-Al_2O_3 powder the diffraction intensity ratio I(012)/I(024) is close to 1.5. Powder Diffraction File JCPDS No 43-1484 states the intensities $I_0(012)=72$ and $I_0(024)=48$. For tensile stressed (with σ about > 350 MPa) CVD α-Al_2O_3-layers on cemented carbide, the intensity ratio I(012)/I(024) is surprisingly significantly less than the expected value 1.5, most often < 1. This may be due to some disorder in the crystal lattice caused by the tensile stresses. It has been found that when such a layer is stress released by, e.g., an intense blasting operation or if it has been completely removed from the substrate and powdered, the ratio I(012)/I(024) becomes closer, equal or even higher than 1.5. The higher the applied blasting force the higher the ratio will be. Thus, this intensity ratio can be used as an important state feature of an α-Al_2O_3 layer.

According to the present invention a cutting tool insert is provided with a CVD-coating comprising a penultimate TiC_xN_y-layer and an outer α-Al_2O_3-layer. The Al_2O_3 can be produced according to patent EP 603 144 giving the Al_2O_3-layer a crystallographic texture in 012-direction with a texture coefficient TC(012)>1.3, preferably > 1.5 or produced according to patents US 5,851,687 and US 5,702,808 giving a texture in the 110-direction with texture coefficient TC(110)> 1.5. In order to obtain a high surface smoothness and low tensile stress level the coating is subjected to a wet blasting operation with a slurry consisting of F150 grits (FEPA-standard) of Al_2O_3 in water at an air pressure of 2.2-2.6 bar for about 10-20 sec/insert. The spray guns are placed approximately 100 mm from the inserts with a 90° spray angle. The insert has a different colour on the clearance side than on the black rake face. An outermost thin 0.1-2 µm colouring layer of TiN (yellow), TiC_xN_y (grey or bronze), ZrC_xN_y (reddish or bronze), where $x \geq 0$, $y \geq 0$ and $x+y=1$ or TiC (grey) is preferably deposited. The inserts are then blasted removing the top layer exposing the black Al_2O_3 layer. The coating on the rake face will have the low desired tensile stress 50-500 MPa while the clearance side will have high tensile stresses in the range 500-700 MPa, the tensile stress on the rake face being lower than the tensile stress on the clearance face, dependent on the choice of coating and the coefficient of Thermal Expansion (CTE) of the used cemented carbide insert. In an other embodiment of the invention the coated insert is blasted both on the rake face and the clearance side and a coloured heat resistant paint is sprayed on the clearance side or a coloured PVD layer is deposited there in order to obtain a possibility to identify a used cutting edge.

<u>Example 1</u>

A) Cemented carbide cutting inserts with the composition 5.5 wt-% Co, 2.9 wt-% TaC, 0.5 wt-% NbC, 1.4 wt-% TiC, 0.9 wt-% TiN, balance WC, having an average grain size of about 2 µm, with a surface zone, 29 µm thick, depleted from cubic carbides. The saturation magnetization, M_s, was measured to be 0.077 hAm^2/kg giving a CW-ratio of 0.87. The inserts were coated with a 0.5 µm thick layer of TiN using conventional CVD-technique at 930 °C followed by a 7 µm TiC_xN_y layer employing the MTCVD-technique using $TiCl_4$, H_2, N_2 and CH_3CN as process gases at a temperature of 885 °C. In subsequent process steps during the same coating cycle a layer of TiC_xO_z about 0.5 µm thick was deposited at 1000 °C using $TiCl_4$, CO and H_2, and then the Al_2O_3-process was stared up by flushing the reactor with a mixture of 2 % CO_2, 3.2 % HCl and 94.8 % H_2 for 2 min before a 7 µm thick layer of α-Al_2O_3 was deposited. On top was a thin approx. 0.5 µm TiN layer deposited. The process conditions during the deposition steps were as below:

	TiN	TiC_xN_y	TiC_xO_z	Al_2O_3-start	Al_2O_3
Step	1 and 6	2	3	4	5
$TiCl_4$	1.5 %	1.4 %	2 %		
N_2	38 %	38 %			
CO_2:				2 %	4 %
CO			6 %		
$AlCl_3$:					3.2 %
H_2S	-				0.3 %
HCl				3.2 %	3.2 %
H_2:	balance	balance	balance	balance	balance
CH_3CN	-	0.6 %			
Pressure:	160 mbar	60 mbar	60 mbar	60 mbar	70 mbar
Temp.:	930°C	885°C	1000°C	1000°C	1000°C
Time:	30 min	4.5 h	20 min	2 min	7 h

B) Cemented carbide cutting inserts of the same type as in A) differing only in TiC_xN_y and α-Al_2O_3 layer thickness, being 6 µm and 10 µm thick respectively, were manufactured using the same processing conditions except for the TiC_xN_y and Al_2O_3 depositing times being 4 h and 10 h, respectively.

XRD-analysis of the deposited Al_2O_3 layer of the inserts according to A) an B) showed that it consisted only of the α-phase with a texture coefficient TC(012)=1.4 defined as below:

$$TC(012) = \frac{I(012)}{I_o(012)} \left\{ \frac{1}{n} \sum \frac{I(hkl)}{I_o(hkl)} \right\}^{-1}$$

where

I(hkl) = measured intensity of the (hkl) reflection

I_O(hkl) = standard intensity of Powder Diffraction File JCPDS No 43-1484.

n = number of reflections used in the calculation

(hkl) reflections used are: (012), (104), (110), (113), (024), (116).

The coated inserts according to A) and B) were post treated by the earlier mentioned blasting method, blasting the rake face of the inserts, using a blasting pressure of 2.4 bar and an exposure time of 20 seconds.

The smoothness of the coating surface expressed as a well known roughness value Ra was measured by AFM on an equipment from Surface Imaging System AG (SIS). The roughness was measured on ten randomly selected plane surface areas (10µmx10µm) in the chip contact zone on rake face. The resulting mean value from these ten Ra values, MRa, was 0.11 µm.

X-ray Diffraction Analysis using a Bragg-Brentano diffractometer, Siemens D5000, was used to determine the I(012)/I(024)-ratio using Cu Kα-radiation.

The obtained I(012)/I(024)-ratio on the clearance side was about 1.4. A corresponding measurement on the rake face showed that the obtained I(012)/I(024)-ratio was about 2.2.

The residual stress was determined using ψ-geometry on an X-ray diffractometer Bruker D8 Discover-GADDS equipped with laser-video positioning, Euler 1/4-cradle, rotating anode as X-ray source (CuK_α-radiation) and an area detector (Hi-star). A collimator of size 0.5 mm was used to focus the beam. The analysis was performed on the TiC_xN_y (422) reflection using the goniometer settings 2θ=126°, ω=63° and Φ=0°, 90°, 180°, 270°, Eight ψ tilts between 0° and 70° were performed for each Φ-angle. The $\sin^2\psi$ method was used to evaluate the residual stress using the software DIFFRAC^{Plus} Stress32 v. 1.04 from Bruker AXS with the constants Young's modulus, E=480 GPa and Poisson's ratio, ν=0.20 and locating the reflection using the Pseudo-Voigt-Fit function. A biaxial stress state was confirmed and the average value was used as the residual stress value. Measurements were carried out both on the rake face and the clearance side. The obtained tensile stress on the clearance side was about 640 MPa for both the inserts according to A) and B). A corresponding measurement on the rake face showed that a tensile stress of about 450 MPa was obtained for the inserts according to A) and a tensile stress of about 480 MPa was obtained for the inserts according to B).

Example 2

Inserts A) from Example 1 were tested and compared with commercially available, nonblasted inserts (high performance inserts in the P15 area) with respect to toughness in a longitudinal turning operation with interrupted cuts.

Material: Carbon steel SS1312.

Cutting data:

Cutting speed = 120 m/min

Depth of cut = 1.5 mm

Feed = Starting with 0.15 mm and gradually increased by 0.08 mm/min until breakage of the edge

10 edges of each variant were tested

Inserts style: CNMG120408-PM

Results:

	Average feed at breakage
Commercially available inserts	0.244 mm/rev
Inserts A) from Example 1	0.275 mm/rev

Example 3

Inserts B) from Example 1 were tested and compared with the same commercially available inserts as in Example 2 with respect to toughness in a longitudinal turning operation with interrupted cuts.

Material: Carbon steel SS1312.

Cutting data:

Cutting speed = 140 m/min

Depth of cut = 1.5 mm

Feed = Starting with 0.15 mm and gradually increased by 0.08 mm/min until breakage of the edge

10 edges of each variant were tested

Inserts style: CNMG120408-PM

Results:

	Average feed at breakage
Commercially available inserts	0.232 mm/rev
Inserts B) from Example 1	0.315 mm/rev

Example 5

Inserts A) from Example 1 were tested with respect to resistance to gross plastic deformation in a facing operation of SS2541.

Cutting data:

Cutting speed= 220 m/min
Feed= 0.35 mm/rev.
Depth of cut= 2 mm

Tool life criteria: flank wear >= 0.5 mm

Results:

	Number of machining cycles needed to reach tool life
Commercially available inserts	65
Inserts A) from Example 1	85

Example 6

Inserts A) from Example 1 were also tested with respect to resistance to plastic deformation close to the edge in turning of SS2244-05.

Cutting data:

Cutting speed: 200 m/min
Feed: 0.35 mm/rev.
Depth of cut: 2.5 mm

Tool life criteria: flank wear >= 0.4 mm

Results:

	Number of machining cycles needed to reach tool life
Commercially available inserts	19

Inserts A) from Example 1 27*

* The test for inserts A) was terminated prematurely, after 27 cycles, with the defined tool life criteria still not being reached.

Examples 3-6 show that the inserts A) and B) from Example 1 and according to the invention exhibit much better plastic deformation resistance in combination with better toughness behaviour in comparison to the inserts according to prior art.

Claims

1. A coated cutting tool insert of cemented carbide comprising a body of generally polygonal or round shape having at least one rake face and at least one clearance face characterised in said insert having a composition of 4.4-6.6, preferably 5.0-6.0, most preferably 5.0-5.8, wt-% Co, 4-8.5 wt-% cubic carbides, balance WC, a CW-ratio in the range 0.78-0.92 and having a surface zone of a thickness of 15-40 µm, preferably 15-35 µm, most preferably 25-35 µm, depleted from the cubic carbides TiC, TaC and/or NbC, said insert being at least partly coated with a 10-25 µm thick coating including at least one layer of TiC_xN_y, where $x \geq 0$, $y \geq 0$ and $x+y=1$, preferably TiC_xN_y deposited by MTCVD, and an α-Al_2O_3-layer being the outer layer at least on the rake face, and that on said at least one rake face

- the TiC_xN_y-layer having a thickness of 5-15 µm, preferably 6-13 µm, most preferably 7-13 µm and a tensile stress level of 50-500 MPa, preferably 50-400 MPa and

- the α-Al_2O_3-layer having a thickness of 3-12 µm, preferably 3.5-8 µm, most preferably 4-8 µm, being the outermost layer with an XRD-diffraction intensity ratio I(012)/I(024) $\geq$1.3, preferably $\geq$1.5 and having a mean Ra value MRa < 0.12 µm, preferably $\leq$ 0.10 µm, at least in the chip contact zone on the rake face, and on said clearance face

- the TiC_xN_y-layer having a tensile stress in the range 500-700 MPa and that

- the α-Al_2O_3-layer has an XRD-diffraction intensity ratio I(012)/I(024)<1.5, preferably covered with a thin 0.1-2 µm TiN, TiC_xN_y, ZrC_xN_y or TiC layer giving the insert a different colour on that face

or on said at least one rake face and said at least one at least clearance face

- the TiC_xN_y-layer has a thickness of 5-15 µm, preferably 6-13 µm, most preferably 7-13 µm and a tensile stress level of 50-500 MPa, preferably 50-400 MPa and

- the α-Al_2O_3-layer with a thickness of 3-12 µm, preferably 3.5-8 µm, preferably 4-8 µm has an XRD-diffraction intensity ratio I(012)/I(024) $\geq$1.3, preferably $\geq$1.5 and on the rake face is the outermost layer with a mean Ra value MRa < 0.12 µm, preferably $\leq$ 0.10 µm, at least in the chip contact zone on the rake face, and on that said clearance face the top layer consists of a coloured heat resistant paint or a coloured PVD-layer.

2. A cutting tool insert according to the preceding claim characterized in having a thin 0.2-2 µm $TiC_xN_yO_z$ bonding layer, $x \geq 0$, $z>0$ and $y \geq 0$, between the TiC_xN_y- and the Al_2O_3-layer.

3. A cutting tool insert according to any of the preceding claims characterized in the α-Al_2O_3-layer having a texture in the 012-direction with a texture coefficient TC(012)>1.3, preferably TC(012)> 1.5.

4. A cutting tool insert according to any of the preceding claims characterized in the α-Al_2O_3-layer having a texture in the 110-direction with a texture coefficient TC(110)>1.5.

5. A cutting tool insert according to any of the preceding claims characterized in the coating containing additional layers composed of metal nitrides and/or carbides and/or oxides with the metal elements selected from Ti, Nb, Hf, V, Ta, Mo, Zr, Cr, W and Al to a total layer thickness of <5 µm.

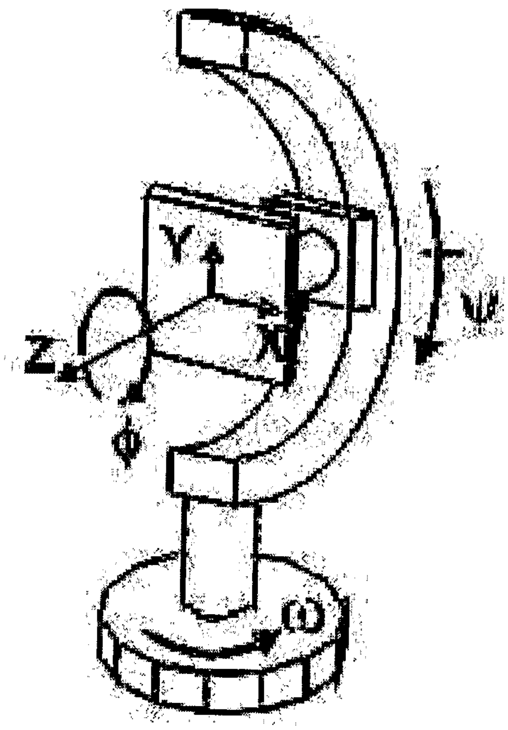

Fig. 1

INTERNATIONAL SEARCH REPORT

International application No.
PCT/SE2006/000736

A. CLASSIFICATION OF SUBJECT MATTER

IPC: see extra sheet

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC: C23C

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

SE,DK,FI,NO classes as above

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

EPO-INTERNAL, WPI DATA, PAJ

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	EP 0603144 A1 (SANDVIK AKTIEBOLAG), 22 June 1994 (22.06.1994), page 2, line 47 - page 3, line 34; page 5, line 11 - line 34, claims 1-7 --	1-5
A	EP 1464727 A2 (SANDVIK AB), 6 October 2004 (06.10.2004), figure 4, paragraphs (0031)-(0036) --	1-5
A	EP 0753603 A2 (SANDVIK AKTIEBOLAG), 15 January 1997 (15.01.1997), page 2, line 39 - page 3, line 32, claim 1, abstract --	1-5

[X] Further documents are listed in the continuation of Box C. [X] See patent family annex.

* Special categories of cited documents:
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Date of the actual completion of the international search	Date of mailing of the international search report
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International patent classification (IPC)

C23C 30/00 (2006.01)
C23C 16/30 (2006.01)
C23C 16/36 (2006.01)
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