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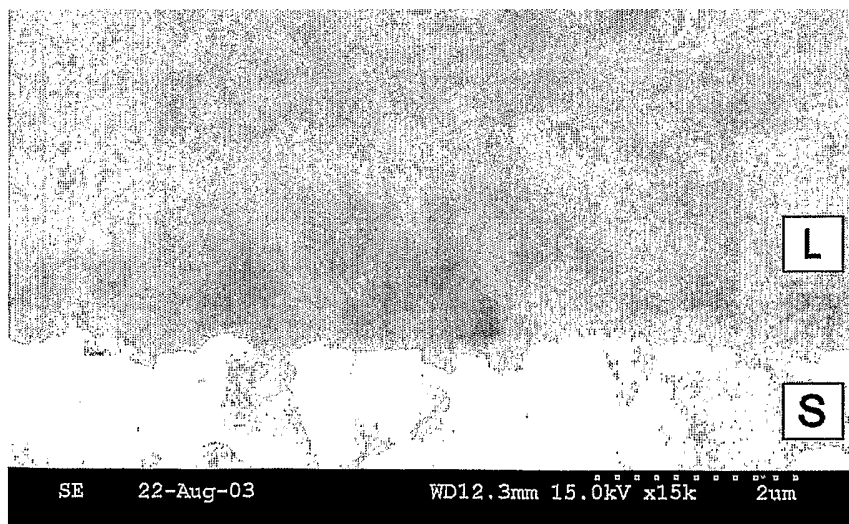
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For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

(54) Title: PVD-COATED CUTTING TOOL INSERT



(57) Abstract: The present invention relates to a coated cutting tool insert of cemented carbide with a coating including at least one layer of $Ti_{1-x}Al_xN$ deposited by PVD-technique. The layer has $x=0.4-0.6$ with a compressive residual stress of $>4-6$ GPa and a thickness of 1.5-5, preferably 2.5-4 μm . Both the I(111) and I(200) are <7.5 , preferably <5 , times, the intensity average noise level. Good adhesion despite high compressive stress is obtained if the layer is deposited by cathodic arc evaporation technique at a bias in the range $-90 < U < -50V$, preferably $-80V < U < -60V$.

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PVD-coated cutting tool insert

The present invention relates to a PVD coated cutting insert suitable for machining of metals by turning, milling, drilling or by similar chip forming machining methods. The invented coated insert shows maintained adhesion despite a relatively high compressive stress state of the (Ti,Al)N-layer.

Modern high productivity chip forming machining of metals requires reliable tools with high wear resistance, good toughness properties and excellent resistance to plastic deformation.

This has so far been achieved by employing a cemented carbide tool body coated with a wear resistant coating. The cemented carbide tool body is generally in the shape of an indexable insert clamped in a tool holder, but can also be in the form of a solid carbide drill or a milling cutter.

Cemented carbide cutting tools coated with various types of hard layers like TiC, Ti(C,N), TiN, $TiC_xN_yO_z$ and Al_2O_3 have been commercially available for many years. Such tool coatings are generally built up by several hard layers in a multilayer structure. The sequence and the thickness of the individual layers are carefully chosen to suit different cutting application areas and work-piece materials.

Tool coatings are most frequently deposited by using Chemical Vapour Deposition (CVD) or Physical Vapour Deposition (PVD) techniques. In some rare cases also Plasma Assisted Chemical Vapour Deposition (PACVD) has been practised.

The CVD-technique is often preferred over PVD as it has several advantages. It allows larger coating batches, produces coatings with good coating thickness distribution on complex shaped tools, has a high throw-

ing power, can be used to deposit electrical non-conducting layers like Al_2O_3 and ZrO_2 and many different materials can be deposited in the same coating run like e.g. Al_2O_3 , TiC , $\text{Ti}(\text{C},\text{N})$, TiN , $\text{TiC}_x\text{N}_y\text{O}_z$, $\text{Zr}(\text{C},\text{N})$ and ZrO_2 .

5 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 thermal coefficients of expansion between the deposited coating materials and the cemented carbide, CVD produces layers with
10 cooling cracks and tensile stresses.

PVD processes run at a significantly lower temperature, 450-650°C and are performed under strong ion bombardment which leads to crack free layers with high compressive stresses. The high compressive stresses and the
15 absence of cooling cracks makes PVD coated tools much tougher than CVD-coated tools and are therefore often preferred in interrupted cutting operations like in milling. The cathodic arc evaporation method is a successful physical vapour deposition (PVD) technique. It
20 offers possibilities to deposit a wide range of materials with high deposition rate and good adhesion to the substrate. In the cathodic arc evaporation deposition process material is evaporated from the target with a vacuum arc. The basic principles are illustrated in Fig.
25 1. The basic components consists of a vacuum chamber, a cathode and an arc power supply, a means of igniting an arc on the cathode surface, an anode and a substrate bias power supply. Arcing is initiated by applying a high bias and/or by mechanical ignition. The evaporation
30 is the result of the arc spot which moves randomly over the target source and melts/evaporates material. The material moves towards the substrates and forms a layer. The properties of this layer is influenced by e.g. the gases in the chamber and applied substrate bias.

The high levels of residual stress in PVD layers can have the beneficial effect of increasing the wear resistance and the hardness, but may on the other hand reduce the adhesion. The stresses may tend to push off the layer at the cutting edgeline curvature. Therefore high compressive stresses are avoided in PVD-layers. In JP-A-08-209334 the residual compressive stress of the (Ti,Al)N layer is reduced from about 3 GPa to 1.5-2 GPa and adhesion is improved as compared with the conventional (Ti,Al)N by adding low-hardness elements Co, Ni or Fe and allowing them to enter into solid solution. JP-A-07-188901 is similar. JP-A-05-177413 discloses a compressive stress of 0.2-2.0 GPa. JP-A-04-300104 discloses adjusting the residual tensile and compressive stresses in layer to below 0.1 GPa by a treatment such as sand blasting, etc. JP-A-10-140330 improves the adhesion of a layer by interposing a relatively soft layer under the layer having high compressive stress such as (Ti,Al)N-layer thus absorbing and relaxing the high compressive stress of the layer. US-A-2002/0051885 discloses a tool with a tool body and a wear resistant layer system, which layer system comprises at least one layer of MeX. Me comprises titanium and aluminium and X is nitrogen or carbon. The tool has a tool body of high speed steel (HSS) or of cemented carbide. In the MeX layer the quotient Q_I as defined by the ratio of the diffraction intensity $I(200)$ to $I(111)$ assigned respectively to the (200) and (111) planes in the X-ray diffraction of the material using θ - 2θ -method is selected to be >1 . Further, the $I(200)$ is at least twenty times larger than the intensity average noise value, both measured with a well-defined equipment and setting thereof.

It is an object of the present invention to provide a (Ti,Al)N coated cutting tool insert with relatively high compressive stress and maintained adhesion.

5 Fig. 1 shows the basic principles of the arc evaporation technique in which

- a) = Arc supply
- b) = Anode
- c) = Arc source
- 10 d) = Substrate table
- e) = Gas inlet
- f) = Vacuum pumps
- g) = Bias supply

15 Fig. 2 shows a SEM-image of the (Ti,Al)N-layer on the clearance side of the insert deposited at bias=-70 V in which

- S = substrate and
- L = layer.

20 Fig. 3 shows X-ray diffraction patterns, intensity(I) versus diffraction angle(2θ), of the (Ti,Al)N layers deposited at substrate bias between -40 and -200 V.

25 Fig. 4 shows the goniometer setup

- E = Euler 1/4-cradle
- S = sample
- I = incident X-ray beam
- 30 D = diffracted X-ray beam
- θ = diffraction angle
- ω = θ
- ψ = tilt angle along the Euler 1/4-cradle
- Φ = rotation angle around the sample axis

35

According to the present invention there is provided a coated cutting tool insert for metal machining such as turning, milling and drilling comprising a body of cemented carbide onto which a hard and wear resistant refractory coating is deposited by PVD technique. The wear resistant coating is composed of one or more layers of refractory compounds of which at least one layer consists of $Ti_{1-x}Al_xN$ deposited by PVD-technique and that the other layer(s), if any at all, comprise(s) wear resistant nitrides, carbides and/or carbonitrides as known in the art. The (Ti,Al)N-layer has a composition of $Ti_{1-x}Al_xN$ with $x=0.4-0.6$ and a compressive stress of $>4-6$ GPa at a thickness of 1.5-5, preferably 2.5-4, μm . The layer is of a glassy type (Fig. 2) with no pronounced diffraction peaks (Fig. 3). Both the intensities of the (111) and (200) reflections, $I(111)$ and $I(200)$ respectively, should be <7.5 , preferably <5 , times the intensity average noise value (150 counts). The average noise value shall be included in the values of $I(111)$ and $I(200)$ and measured with a Bragg-Brentano diffractometer, Siemens D5000 with CuK_{α} radiation at an operating voltage of 40 kV and current of 50 mA, a 2θ angular speed of $0.02^{\circ}/min$ and a time constant of 1 s.

The residual stress, σ , of the (Ti,Al)N-layer is evaluated 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 shall be performed using CuK_{α} -radiation on the (Ti,Al)N 200-reflection. The goniometer setup is shown in Fig. 4. It is recommended to use the side-inclination technique (ψ -geometry) with six to eleven, preferably eight, ψ -angles, equidistant within a $\sin^2\psi$ -range of 0 to 0.5 ($\psi=45^{\circ}$). An equidistant distribution of ϕ -angles

within a Φ -sector of 90° is preferred. To confirm a biaxial stress state the sample shall be rotated for $\Phi=0^\circ$ and 90° while tilted in ψ . It is recommended to investigate the possible presence of shear stresses and therefore both negative and positive ψ -angles shall be measured. In the case of an Euler 1/4-cradle this is accomplished by measuring the sample also at $\Phi=180^\circ$ and 270° for the different ψ -angles. The measurement shall be performed on an as flat surface as possible, preferably on a clearance side of an insert. For the calculations of the residual stress values the Possion's ratio, $\nu=0.20$ and the Young's modulus, $E=450$ GPa are to be used. Alternatively, Young's modulus, E , can be determined e.g. by nano-indentation technique. Preferably the data are evaluated using commercially available software such as DIFFRAC^{Plus} Stress32 v. 1.04 from Bruker AXS preferably locating the (200) reflection by the Pseudo-Voigt-Fit function. In case of a biaxial stress state the total stress is calculated as the average of the obtained biaxial stresses.

According to the method of the present invention the (Ti,Al)N coated cutting tool inserts with high compressive stress and still good adhesion are obtained if the layers are deposited by cathodic arc evaporation technique at a bias of -70 V.

Preferably said layer is deposited with a bias, U , in the range $-90 < U < -50$ V, preferably $-80 < U < -60$ V; with a nitrogen pressure in the range of $20-40$ μ bar; arc current in the range $160-220$ A and temperature in the range $400-600^\circ\text{C}$.

Example 1

Cemented carbide cutting inserts of geometry CNMG120408-MM with the composition 10 wt-% Co and as rest WC were coated with an about 3 μm layer of (Ti,Al)N

using arc evaporation technique in a Balzers Rapid Coating System (RCS) equipment with a target composition of Ti50Al50 (at-%) in a nitrogen atmosphere at the following process conditions.

- 5 Temperature: 450 °C
 Pressure: 32 μ bar
 Arc current: 200 A
 Substrate bias, U: -40, -70, -100, -140 and -200 V

- 10 The composition of the (Ti,Al)N-layers was estimated by electron probe microanalysis (EPMA) with an analysis area of 0.5-1 μ m using a Jeol JXA 8900 R at 15 kV.

- 15 Structure of the layers were analyzed using a Hitachi S-4300 FEG-SEM at 15 kV.

- Hardness and Young's modulus of the layers were evaluated from nano-indentation measurements made on a
20 CSEM nano-hardness tester. The data were obtained for a load of 50 mN. The depths of all indents were less than 10% of the layer thickness.

- Texture, residual stress and phases present in the
25 layers were evaluated by XRD measurements. The equipment used for phase and texture analysis was a Bragg-Brentano diffractometer, Siemens D5000 with CuK_α radiation at an operating voltage of 40 kV and current of 50 mA. The 2 θ angular speed was 0.02°/min and the time constant was 1
30 s. The residual stress was evaluated using ψ -geometry on a 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 0.8 mm was used to
35 focus the beam on to the clearance side of the insert.

The analysis was performed on the (Ti,Al)N (200) reflection using the goniometer settings $2\theta=50^\circ$, $\omega=25^\circ$ and $\phi=0^\circ, 90^\circ, 180^\circ, 270^\circ$. Eight ψ tilts between 0° and 70° were performed for each ϕ -angle. The $\sin^2\psi$ method was used to evaluate the data using the software DIFFRAC^{Plus} Stress32 v. 1.04 from Bruker AXS. The (200) reflection was located using the Pseudo-Voigt-Fit function. The residual stress values were calculated using a Poisson's ratio of $\nu=0.20$ and values of Young's modulus, E , determined with the nano-indentation technique for each layer (given in the table). For the 200 V layer, $E=450$ GPa was used as no nano-indentation data could be collected due to spontaneous spalling. A biaxial stress state was confirmed and the total compressive stress was calculated as the average of the obtained biaxial stresses.

Spalling resistance of the layers was evaluated by an edge line delamination test in wet turning of a Sanmac 304L stainless steel. The cutting speed was 200 m/min, the feed rate 0.3 mm/rev and the depth of cut 3 mm. The results are summarised in the Table.

Table

Layer, Negative substrate bias, -U (V)	Thick- ness (μm)	I (111) (counts) *	I (200) (counts) *	Young's Modulus (GPa)	Hard- ness (GPa)	Composition (atom-%)		Residual stress, σ (GPa)	Edge delami- nation
						Ti	Al		
40	3.2	678	6008	493	33.8	26.6	23.6	-1.7	None
70	3.0	382	625	445	37.0	25.8	23.3	-4.4	None
100	3.0	423	394	454	35.9	26.0	23.1	-5.1	100 %
140	1.6	1831	356	446	36.2	26.9	23.1	-5.1	100 %
200	1.8	6216	353	**	**	27.0	24.0	-5.6	**

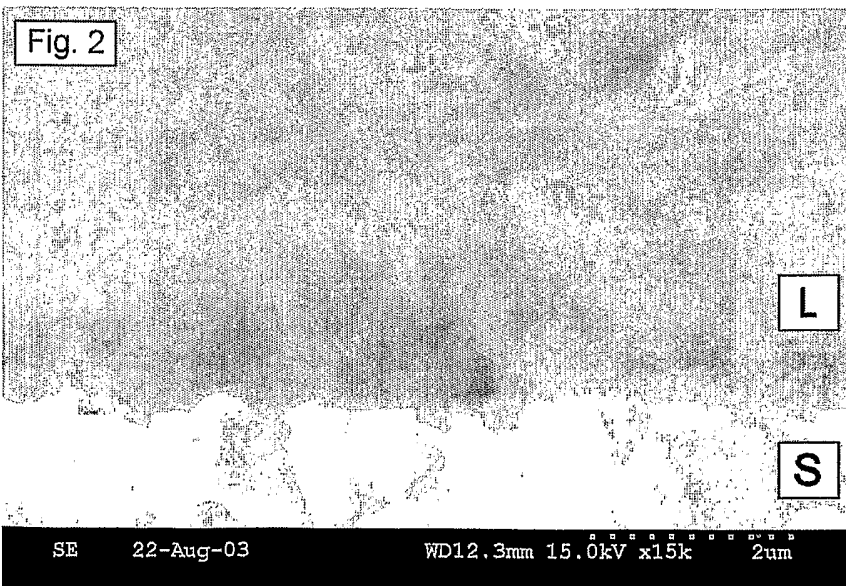
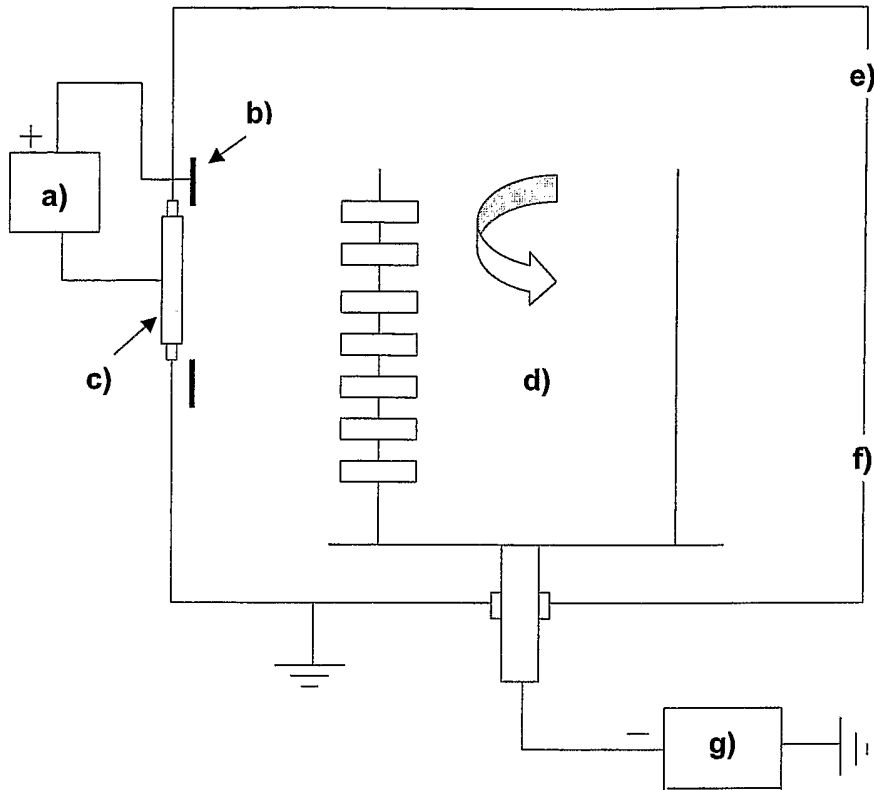
* Including the intensity average noise level (150 counts).

** No data could be collected due to spontaneous delamination.

Claims

1. A coated cutting tool insert of cemented carbide with a coating including at least one layer of $Ti_{1-x}Al_xN$ deposited by PVD-technique c h a r a c t e r i s e d in
5 that $x=0.4-0.6$ with a compressive residual stress of $>4-6$ GPa and a thickness of $1.5-5$, preferably $2.5-4$, μm ; both the intensities of the (111) and (200) reflections, $I(111)$ and $I(200)$, are <7.5 , preferably <5 times, the intensity average noise level.
- 10 2. Method of making a coated cutting tool insert of cemented carbide with a coating including at least one layer of $Ti_{1-x}Al_xN$ deposited by PVD-technique
c h a r a c t e r i s e d in depositing the layer with a bias, U , in the range $-90 < U < -50V$, preferably $-80V < U < -$
15 $60V$; with a nitrogen pressure in the range of $20-40$ μbar ; arc current in the range $160-220$ A and temperature in the range $400-600$ $^{\circ}C$.

Fig. 1



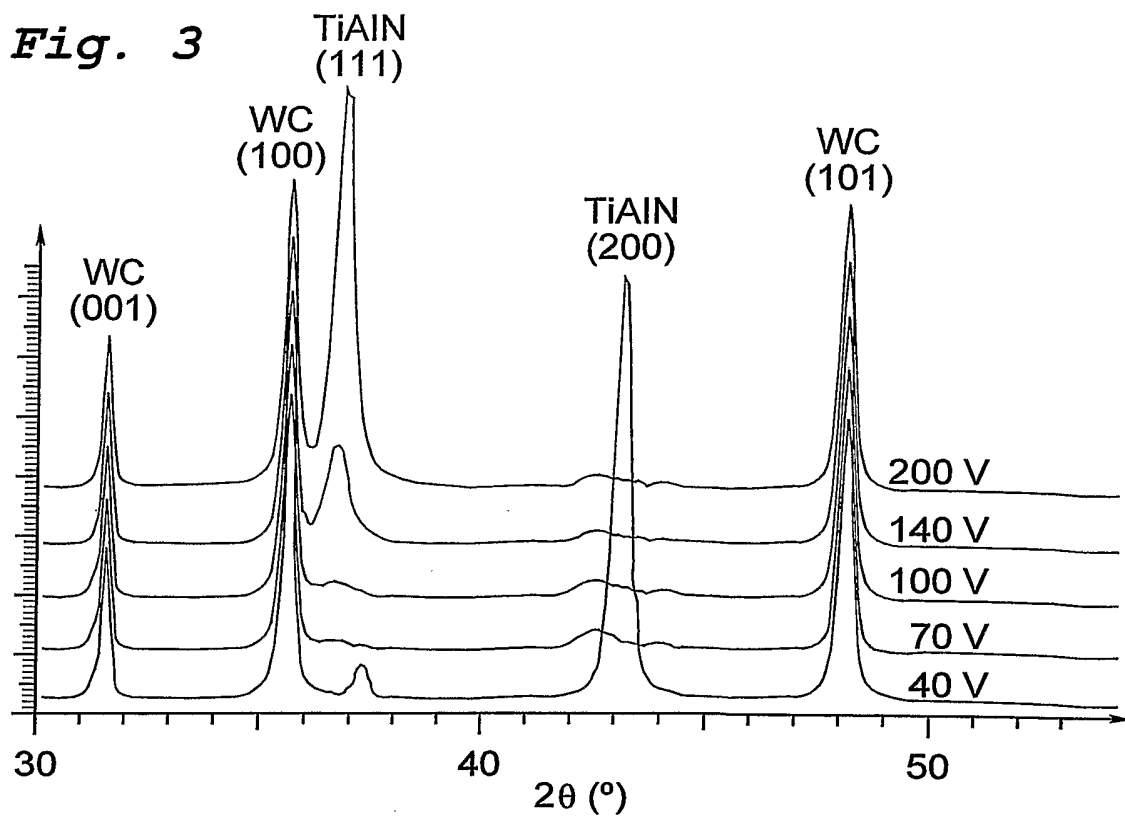
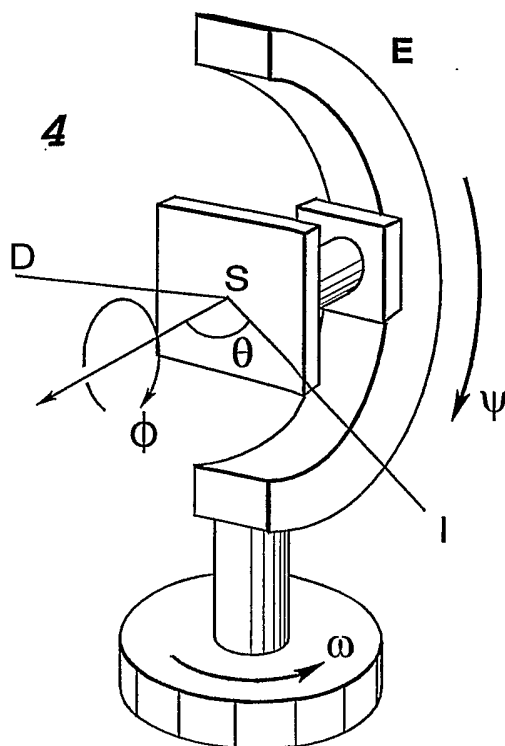


Fig. 4



INTERNATIONAL SEARCH REPORT

International application No.

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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	US 20020051885 A1 (HANS BRAENDLE ET AL), 2 May 2002 (02.05.2002), abstract, paragraphs (0011) - (0039) --	1-2
A	PATENT ABSTRACTS OF JAPAN vol. 1996, no. 12, 26 December 1996 (1996-12-26) & JP 8 209 335 A (SADOHARA TAKESHI), 13 August 1996 (1996-08-13) abstract --	1-2

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

* Special categories of cited documents:

"A" document defining the general state of the art which is not considered to be of particular relevance

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"P" document published prior to the international filing date but later than the priority date claimed

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INTERNATIONAL SEARCH REPORT

International application No.

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C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	PATENT ABSTRACTS OF JAPAN vol. 1997, no. 08, 29 August 1997 (1997-08-29) & JP 9 095 763 A (MITSUBSHI HEAVY IND LTD), 08 April 1997 (1997-04-08) abstract --	1-2
A	PATENT ABSTRACTS OF JAPAN vol. 1998, no 10, 31 August 1998 (1998-08-31) & JP 10 140330 A (HITACHI TOOL ENG CO LTD), 26 May 1998 (1998-05-26) abstract -- -----	1-2

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C23C 14/32 (2006.01)

C23C 30/00 (2006.01)

INTERNATIONAL SEARCH REPORT
Information on patent family members

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PCT/SE 2005/001311

US	20020051885	A1	02/05/2002	US	6844069	B	18/01/2005
				AU	4028197	A	05/04/1999
				BR	9714868	A	25/07/2000
				EP	1021584	A,B	26/07/2000
				SE	1021584	T3	
				PT	1021584	T	30/04/2004
				US	6071560	A	06/06/2000
				WO	9914391	A	25/03/1999
