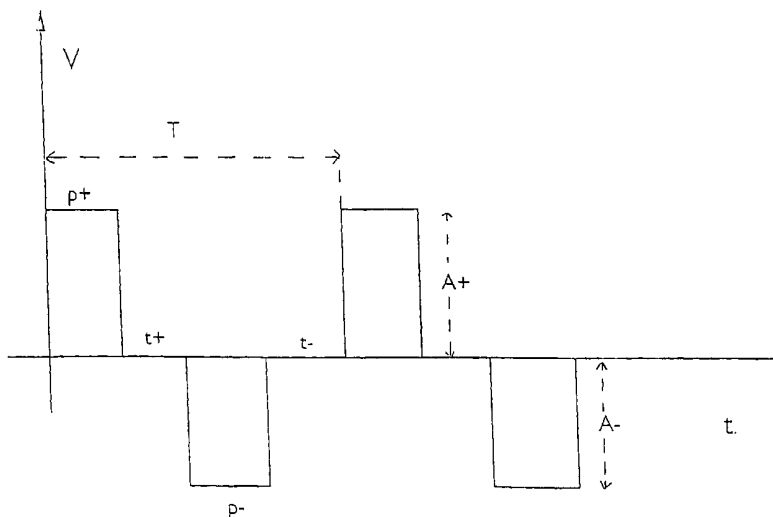




## INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

<div>(51) International Patent Classification <sup>7</sup> : C23C 16/40, 16/515, 16/30, 30/00, C04B 41/89, B23B 27/14</div>	<div>A1</div>	<div>(11) International Publication Number: WO 00/70120</div> <div>(43) International Publication Date: 23 November 2000 (23.11.00)</div>
<div>(21) International Application Number: PCT/SE00/00974</div> <div>(22) International Filing Date: 16 May 2000 (16.05.00)</div> <div>(30) Priority Data: 9901823-6 19 May 1999 (19.05.99) SE</div> <div>(71) Applicant: SANDVIK AB (publ) [SE/SE]; S-811 81 Sandviken (SE).</div> <div>(72) Inventors: SÖDERBERG, Staffan; Mossvägen 112, S-141 44 Huddinge (SE). LITTECKE, Peter; Sjöstigen 31, S-141 72 Huddinge (SE).</div> <div>(74) Agent: TÅQUIST, Lennart; Sandvik AB, Patent Dept., S-811 81 Sandviken (SE).</div>		<div>(81) Designated States: CA, JP, KR, ZA, European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE).</div> <div>Published With international search report.</div>

**(54) Title:** Al<sub>2</sub>O<sub>3</sub> COATED CUTTING TOOL**(57) Abstract**

The present invention describes a coated CBN cutting tool for metal machining. The tool consists of one or more CBN bodies with or without cemented carbide backing. The coating is composed of one or more layers of refractory compounds of which at least one layer consists of fine-grained, crystalline  $\gamma$ -phase alumina, Al<sub>2</sub>O<sub>3</sub>, with a grain size less than 0.1  $\mu$ m. The  $\gamma$ -phase alumina is deposited with a Plasma Activated Chemical Vapor Deposition (PACVD) process in which the plasma is produced by applying a bipolar pulsed DC voltage across two electrodes to which the tool substrates to be coated are fixtured and electrically connected.

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Al<sub>2</sub>O<sub>3</sub> COATED CUTTING TOOL

The present invention relates to a cutting tool for metal machining, comprising at least one body containing PCBN, (polycrystalline cubic boron nitride), with or without cemented carbide backing, and on the surface of said body a hard and wear resistant refractory coating. The coating is adherently bonded to the body and covers all functional parts thereof. The coating is composed of one or more layers of refractory compounds of which at least one layer consists of fine-crystalline  $\gamma$ -Al<sub>2</sub>O<sub>3</sub>, deposited by Plasma Activated CVD-methods (PACVD).

Cutting tools having cutting edges formed of a superhard abrasive such as a cubic boron nitride (CBN) based material are manufactured by powder metallurgical techniques and are mainly used for the machining of cast iron and hardened steel. Several types of CBN cutting tools are known, the majority of which consisting of a CBN tip that has been brazed onto a cemented carbide insert. Others have the CBN sintered directly to a cemented carbide backing of sufficient thickness to produce an insert while yet others consist of solid CBN without any cemented carbide backing.

Subjecting a sintered CBN body to temperatures over 1000 °C may result in undesirable structural changes in the material. Furthermore, in the case of a brazed insert the braze joint will be destroyed.

Swedish patent application 9704387-1 discloses a method for depositing refractory alumina (Al<sub>2</sub>O<sub>3</sub>) thin layers on cutting tools made of cemented carbide, cermet, ceramics or high speed steel. The invented method is a Plasma Activated Chemical Vapor Deposition (PACVD) process in which the plasma is produced by applying a bipolar pulsed DC voltage across two electrodes to which the tool substrates to be coated are fixtured and electrically connected.

WO 98/28464 discloses in the example that by applying a coating including a MTCVD-TiCN and a CVD-Al<sub>2</sub>O<sub>3</sub>-layer to such a CBN tool material, substantial advantages in tool life can be achieved when machining  
5 hardened ball bearing steel.

US 5,503,913 proposes for the improvement of the wear properties of tools with cutting edge of cubic boron nitride or polycrystalline cubic boron nitride to coat the superhard body with a 0.5-6  $\mu\text{m}$  thick coating  
10 of one or more oxides of the metals Zr, Y, Mg, Ti or Al. The coating is deposited from the gas phase at temperatures up to 800 °C, preferably using a pulse plasma CVD-process.

According to the present invention there is provided a cutting tool comprising at least one body containing at least 35 vol-% cubic boron nitride, CBN, onto which a hard and wear resistant refractory coating is deposited. Said wear resistant coating is composed of one or more layers of refractory compounds of which at  
20 least one layer, preferably the outermost layer, consists of  $\gamma\text{-Al}_2\text{O}_3$  deposited by PACVD techniques at substrate temperatures of 500 to 800 °C. Said  $\gamma\text{-Al}_2\text{O}_3$ -layer consists of high-quality, dense, fine-grained crystalline Al<sub>2</sub>O<sub>3</sub>. The layer is transparent and has a  
25 very smooth surface finish. It is further characterized by a high hardness larger than or equal to 19 GPa and a grain size estimated between 5 and 200 nm. Furthermore, the  $\gamma\text{-Al}_2\text{O}_3$ -layer is virtually free of cracks. The inner layer(s), if any at all, between the tool body and the  
30 Al<sub>2</sub>O<sub>3</sub>-layer, is/are composed of metal nitrides and/or carbides with the metal elements selected from Ti, Nb, Hf, V, Ta, Mo, Zr, Cr, W and Al.

In a first embodiment the PCBN material according to the invention contains more than 70 vol-% CBN with 5-  
35 10 wt% AlN and a metallic binder phase, generally Co, originating from the cemented carbide backing of the

tool. This material is particularly useful for the machining of cast iron.

In a second embodiment the PCBN-material contains less than 70 vol-% CBN the remainder being other hard wear resistant constituents such as carbides, nitrides, carbonitrides, oxides or borides of the metals of groups IVA to VIA of the periodic table usually TiC, TiN or Ti(C,N). An example of such a material is disclosed in US 5,639,285. This kind of PCBN-material is mainly used in cutting tools for machining of hardened steel.

Often, the PCBN material also contains smaller amounts (typically <10 wt% each) of other components, e.g. Co, Ni, WC, Al, AlN and Al<sub>2</sub>O<sub>3</sub>.

In a third embodiment PCBN cutting tool is manufactured without cemented carbide backing or support. Usually such a product consists of more than 80 vol-% CBN the remaining portion being a metallic binder phase generally Co.

In a fourth embodiment the PCBN cutting tool consists of a CBN-containing material attached to a substrate, preferably cemented carbide, by brazing, sintering or in any other way. The cemented carbide is preferably WC-Co with 10-20, preferably 15-17, wt-% Co.

The  $\gamma$ -Al<sub>2</sub>O<sub>3</sub>-layers according to the invention further give the cutting edges of the tool an extremely smooth surface finish which results in an improved surface finish also of the work piece being machined. The very smooth surface finish can be attributed to the very fine crystallinity of the coating. The " $\gamma$ -Al<sub>2</sub>O<sub>3</sub>"-layers may also partially contain other phases from the " $\gamma$ -series" like  $\theta$ ,  $\delta$  and  $\eta$ . Identification of the  $\gamma$ - and/or  $\theta$ -phases in the Al<sub>2</sub>O<sub>3</sub>-layers according to the invention can preferably be made by X-ray diffraction. Reflexes from the (400) and (440) planes of the  $\gamma$ -Al<sub>2</sub>O<sub>3</sub>-layers occurring at the  $2\theta$ -angles 45.80 and 66.80 when using CuK $\alpha$  radiation, unequivocally identifies the  $\gamma$ -phase.

Weaker reflexes from the (222), (200) and (311) planes of the  $\gamma$ -phase can occasionally be identified. When the  $\theta$ -phase is present in the  $\text{Al}_2\text{O}_3$ -layers according to the invention, said phase is identified by the reflexes from the (200, 20-2) planes.

The fine-grained, crystalline  $\gamma$ - $\text{Al}_2\text{O}_3$  according to the invention is strongly textured in the [440]-direction. A Texture Coefficient, TC, can be defined as:

$$\text{TC}(\text{hkl}) = \frac{I(\text{hkl})}{I_0(\text{hkl})} \left\{ \frac{1}{n} \sum \frac{I(\text{hkl})}{I_0(\text{hkl})} \right\}^{-1}$$

where

$I(\text{hkl})$  = measured intensity of the (hkl) reflection

$I_0(\text{hkl})$  = standard intensity from the ASTM standard powder pattern diffraction data

n = number of reflections used in the calculation

(hkl) reflections used are: (111), (311), (222), (400) and (440) and whenever the  $\text{TC}(\text{hkl}) > 1$ , there is a texture in the [hkl]-direction. The larger the value of  $\text{TC}(\text{hkl})$ , the more pronounced is the texture. According to the present invention, the TC for the set of (440) crystal planes is  $\geq 1.5$ .

A further improvement in cutting performance can be anticipated if the edges of the  $\gamma$ - $\text{Al}_2\text{O}_3$  coated cutting tools according to the invention are treated by a gentle wet-blasting process or by edge brushing with brushes based on e.g. SiC as disclosed in US 5,861,210.

The total coating thickness according to the present invention varies between 0.5 and 20  $\mu\text{m}$ , preferably between 1 and 15  $\mu\text{m}$ , with the thickness of the non- $\text{Al}_2\text{O}_3$ -layer(s) varying between 0.1 and 10  $\mu\text{m}$ , preferably between 0.5 and 5  $\mu\text{m}$ . The fine-grained  $\gamma$ - $\text{Al}_2\text{O}_3$ -coating can also be deposited directly onto the CBN cutting tool and the thickness of said  $\gamma$ - $\text{Al}_2\text{O}_3$  varies then between 0.5 and 15  $\mu\text{m}$ , preferably between 1 and 10  $\mu\text{m}$ . Likewise further coatings of metal nitrides and/or carbides with

metal elements selected from Ti, Nb, Hf, V, Ta, Mo, Zr, Cr, W and Al can be deposited on top of the  $\text{Al}_2\text{O}_3$ -layer, preferably TiN.

The method is based on a plasma activation of a reaction mixture of the chemical reactants  $\text{AlCl}_3$ ,  $\text{H}_2$  and Ar together with an oxygen-donor  $\text{O}_2$ ,  $\text{CO}_2$ , CO and  $\text{N}_2\text{O}$ . Preferably the oxygen-donor is  $\text{O}_2$ . Applying a bipolar pulsed DC voltage across two electrodes produces the plasma or two sets of electrodes to which the substrate bodies to be coated are fixtured and electrically connected. Alternatively, the reactor wall may serve as an electrode. The two electrodes or the two sets of electrodes alternately act as anode(s) and cathode(s). The alternation of the voltage pulse applied to the electrodes between positive and negative potential has several advantages. Firstly, the undesirable built-up electrical charge on non-conducting surfaces during the negative pulse period will be discharged during the positive pulse period and, by selecting a high enough frequency, >5 kHz, of the bipolar pulsed DC voltage, arcing can be eliminated. This will permit stable, long-term processing. Secondly, if no pause time between the positive and the negative pulses is allowed for, the plasma will constantly be activated resulting in a higher deposition rate compared to prior art unipolar pulsed DC voltage techniques. A further advantage of the method is that the growth rate of the coating is practically constant on all surfaces alike, edges, corners and flat surfaces of the body subjected to coating. The method, bipolar pulsed DC voltage PACVD, can also successfully be used for the deposition of non-insulating coatings such as TiC, TiN, TiCN and  $\text{TiAlN}$ , or other carbides and/or nitrides with the metal element chosen from Nb, Hf, V, Ta, Mo, Zr, Cr and W.

In order to attain optimum coating quality and growth rate when applying the bipolar pulsed DC voltage

technique, the frequency, pulse amplitudes, positive and negative pulse on/off times can be varied which is illustrated in Fig. 1. A frequency in the range 5-100 kHz can be used, preferably 8-20 kHz. The pulse amplitudes can vary between 300 and 1000 Volts, preferably between 600 and 900 Volts, and the negative and positive pulse on times as well as the negative and positive pulse off times (zero potential) within the period T of the pulsed DC voltage can also be varied in order to modify the coating properties and the stability of the process. Following the definitions of the parameters  $P_+$ ,  $P_-$ ,  $t_+$ ,  $t_-$ ,  $A_+$  and  $A_-$  below:

Pulse on time positive= $P_+$   
Pulse on time negative= $P_-$   
Pulse off time positive= $t_+$   
Pulse off time negative= $t_-$   
Positive pulse amplitude= $A_+$   
Negative pulse amplitude= $A_-$   
where  $P_- \geq P_+ \geq 0.1P_-$ , preferably  $0.5P_- \geq P_+ \geq 0.1P_-$ , and where also  $P_- \geq 0.1T$ . The negative and positive pulse off times should be set to larger or equal to zero i.e.  $t_- \geq 0$  and  $t_+ \geq 0$ . The amplitude  $A_+$  is essentially of equal magnitude as amplitude  $A_-$ .

The following gas composition, process pressure and substrate temperature may be used:

	Possible range	Preferred range
$AlCl_3$	0.1-2 %	0.2-0.4 %
$O_2$	0.1-3.9 %	0.1-2.0 %
$H_2$	25-95 %	70-80 %
Ar	5-75 %	20-30 %
$O_2/AlCl_3$ -ratio	>1.5	1.5-2.5
Process		
pressure:	0.05-1 kPa	0.1-0.4 kPa
Substrate		
temperature:	500-800 °C	600-700 °C



It is within the purview of the skilled artisan to determine whether the requisite grain size and phase compositions have been obtained and to modify the deposition conditions in accordance with the present specification, if desired, to effect the structure of the  $\text{Al}_2\text{O}_3$  coating within the frame of the invention.

Claims

1. Cutting tool comprising a substrate and a coating said substrate comprising a body containing at least 35 vol-% cubic boron nitride and said coating comprising  
5 at least one thin layer of alumina  
c h a r a c t e r i s e d in that said alumina layer essentially consists of  $\gamma$ - $\text{Al}_2\text{O}_3$  with a grain size between 5 and 200 nm.

2. Cutting tool according to claim 1  
10 c h a r a c t e r i s e d in that the  $\text{Al}_2\text{O}_3$ -layer exhibits significant X-ray diffraction reflexes from at least one of the (440) and (400) crystal planes, said at least one or more layers having a hardness of at least 19 GPa.

3. Cutting tool according to claim 1  
15 c h a r a c t e r i s e d in that the  $\text{Al}_2\text{O}_3$ -layer has a preferred growth orientation in the [440]-direction with a texture coefficient  $\geq 1.5$  defined as below

$$\text{TC}(\text{hkl}) = \frac{I(\text{hkl})}{I_0(\text{hkl})} \left\{ \frac{1}{n} \sum \frac{I(\text{hkl})}{I_0(\text{hkl})} \right\}^{-1}$$

20 where

$I(\text{hkl})$  = measured intensity of the (hkl) reflection

$I_0(\text{hkl})$  = standard intensity from the ASTM standard powder pattern diffraction data

$n$  = number of reflections used in the calculation

25 (hkl) reflections used are: (111), (311), (222), (400) and (440).

4. Cutting tool according to any of the previous claims c h a r a c t e r i s e d in that the fine grained crystalline  $\gamma$ - $\text{Al}_2\text{O}_3$ -layer contains portions (detectable by XRD technique) of additional alumina phases  
30 from the  $\gamma$ -series of the  $\text{Al}_2\text{O}_3$ -polymorphs, preferably  $\theta$ -phase.

5. Cutting tool according to any of the previous claims c h a r a c t e r i s e d in having at least one  
35 layer of thickness 0.1-10  $\mu\text{m}$ , preferably 0.5-5  $\mu\text{m}$ , com-

prising metal nitrides and/or carbides with the metal element selected from Ti, Nb, Hf, V, Ta, Mo, Zr, Cr, W and Al, preferably TiC, TiCN, TiN or TiAlN.

6. Cutting tool according to any of the previous  
5 claims c h a r a c t e r i s e d in that the outer layer is  $\text{Al}_2\text{O}_3$ .

7. Cutting tool according to any of the previous claims c h a r a c t e r i s e d in that the outer layer is TiN.

10 8. Cutting tool according to any of the previous claims c h a r a c t e r i s e d in that the PCBN material contains more than 80 vol-% CBN, the remaining portion being a metallic binder phase generally Co.

9. Cutting tool according to claims 1-7  
15 c h a r a c t e r i s e d in that the PCBN material contains <70 vol-% CBN, the remainder being other hard wear resistant constituents such as carbides, nitrides, carbonitrides, oxides or borides of the metals of groups IVA to VIA of the periodic table, preferably TiC, TiN or  
20 Ti(C,N).

10. Cutting tool according to any of the previous claims c h a r a c t e r i s e d in that said tool consists entirely of the CBN-containing material.

11. Cutting tool according to any of claims 1-9  
25 c h a r a c t e r i s e d in that said tool consists of the CBN-containing material attached to a substrate, preferably cemented carbide, by brazing, sintering or in any other way.

12. Cutting tool according to claim 11  
30 c h a r a c t e r i s e d in that the cemented carbide is WC-Co with 10-20, preferably 15-17, wt-% Co.

13. Method of making a cutting tool according to claim 1 by a Plasma Activated CVD (PACVD) method  
c h a r a c t e r i z e d in that the plasma is produced by  
35 a bipolar pulsed DC voltage applied between two electrodes or two sets of electrodes to which the substrate

bodies to be coated are fixtured and electrically connected, and said electrodes or set of electrodes alternately act as anode(s) and cathode(s), that the chemical reactants are  $\text{AlCl}_3$ ,  $\text{H}_2$  and Ar together with an oxygen-donor  $\text{O}_2$ ,  $\text{CO}_2$ , CO or  $\text{N}_2\text{O}$ , that the pulse frequency is set between 5 and 100 kHz, preferably between 8 and 20 kHz, that the pulse amplitudes are set in the range 300 to 1000 Volts, preferably in the range 600 to 900 Volts and that the substrate temperature is in the range 500 to 800 °C, preferably 550 to 650 °C, depending on the material of the tool body being coated.

14. A method according to claim 13 characterized in that the oxygen-donor is  $\text{O}_2$ .

15. A method according to claim 14 characterized in that the chemical reactants are set to in vol-%:

$\text{AlCl}_3$ : 0.1-2.0, preferably 0.2-0.4,

$\text{O}_2$ : 0.1-3.9, preferably 0.1-2,

$\text{H}_2$ : 25-95, preferably 70-80 and

Ar: 5-75, preferably 20-30

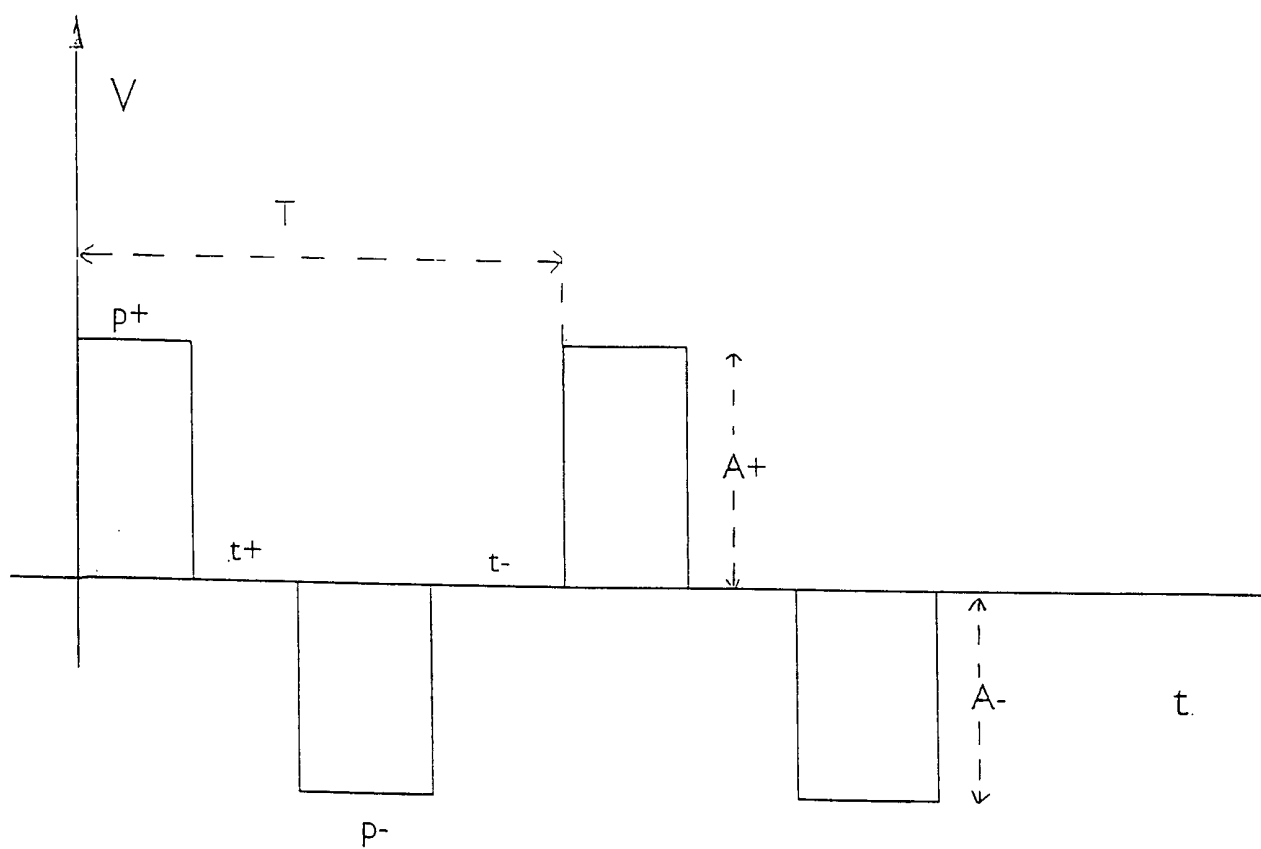
$\text{O}_2/\text{AlCl}_3$ -ratio: >1.5, preferably 1.5-2.5

16. A method according to claim 15 characterized in that the total process pressure is set in the range 0.05 to 1 kPa, preferably in the range 0.1 to 0.4 kPa.

17. A method according claim 16 characterized in that the pulse on time negative ( $P_-$ ) is larger or equal to the pulse on time positive ( $P_+$ ), and the latter is larger or equal to 10% of the pulse on time negative, i.e.  $P_- \geq P_+ \geq 0.1P_-$ , and further the pulse on time negative is larger or equal to 10% of the period T, i.e.  $P_- \geq 0.1T$ , and that the negative and positive pulse off times are set to be larger or equal to zero, i.e.  $t_- \geq 0$  and  $t_+ \geq 0$ .

18. A method according to claim 17 characterized in that the amplitudes of the

positive( $A_+$ ) and negative( $A_-$ ) pulses of the bipolar pulsed DC voltage are of equal magnitude.

**Fig. 1**

## INTERNATIONAL SEARCH REPORT

International application No.

PCT/SE 00/00974

## A. CLASSIFICATION OF SUBJECT MATTER

IPC7: C23C 16/40, C23C 16/515, C23C 16/30, C23C 30/00, C04B 41/89, B23B 27/14  
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)

IPC7: C23C, C04B, B23B

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)

WPIL, EDOC, JAPIO, INSPEC, EI COMPENDEX, METADEX, PASCAL, ENERGY SCITEC, CA

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	Surface and Coatings Technology; 108-109, Volume, 1998, Ch. Täschner et al, "Deposition of hard crystalline Al <sub>2</sub> O <sub>3</sub> coatings by bipolar pulsed d.c. PACVD", see especially the abstract; page 258 and page 262 - 264 --	1-18
X	US 5879823 A (JOHN J. PRIZZI ET AL), 9 March 1999 (09.03.99), column 4, line 34 - line 61; column 7, line 6 - line 46, claims 5, 8, 19, 20 --	1-12
A	US 5516588 A (HENDRIKUS VAN DEN BERG ET AL), 14 May 1996 (14.05.96), column 6, line 45 - line 67; column 8, line 25 - line 40 --	5-7

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

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Date of the actual completion of the international search

4 July 2000

Date of mailing of the international search report

27.07.2000

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## INTERNATIONAL SEARCH REPORT

International application No.

PCT/SE 00/00974

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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Information on patent family members

02/12/99

International application No.

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