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DECISION of 2 June 2005

Case Number:	T 1185/02 - 3.2.2			
Application Number:	93905706.3			
Publication Number:	0627016			
IPC:	C22C 29/02			

Language of the proceedings: EN

Title of invention: Cemented carbide with binder phase enriched surface zone

Applicant: SANDVIK AKTIEBOLAG

Opponent:

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Headword:

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Relevant legal provisions: EPC Art. 54, 56

Keyword:
"Inventive step (yes) after amendment"

Decisions cited:

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Catchword:

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Boards of Appeal

Chambres de recours

Case Number: T 1185/02 - 3.2.2

D E C I S I O N of the Technical Board of Appeal 3.2.2 of 2 June 2005

Appellant:	SANDVIK AKTIEBOLAG S-811 81 Sandviken (SE)
Representative:	Weber, Roland, DiplChem. Weber, Seiffert, Lieke Taunusstrasse 5a D-65183 Wiesbaden (DE)
Decision under appeal:	Decision of the Examining Division of the European Patent Office posted 12 July 2002 refusing European application No. 93905706.3 pursuant to Article 97(1) EPC.

Composition of the Board:

Chairman:	т.	к.	н.	Kriner
Members:	R.	Ries		
	Ε.	Dufrasne		

Summary of Facts and Submissions

I. This appeal is against the decision of the examining division dated 12 July 2002 to refuse European patent application No. 93 905 706.3.

The ground of refusal was that the subject matter of claim 1 lacked an inventive step having regard to the documents

D1: US-A-4 649 084 and

D2: US-A-4 913 877

- II. On 9 September 2002 the appellant (applicant) lodged an appeal against the decision and paid the prescribed fee on 20 September 2002. On 8 November 2002 a statement of the grounds of appeal was filed.
- III. The Board's provisional opinion was expressed in the official communication of 21 February 2005. In addition to the prior art D1 and D2, document

D3: US-A-4 548 786

already acknowledged as technical background in the introductory part of the description was referred to.

IV. At the end of the oral proceedings which took place on 2 June 2005, the appellant requested that the decision under appeal be set aside and a patent be granted on the basis of the following documents:

- claim 1 as filed during the oral proceedings;
- description pages 1 to 10 as published in WO-A-93/17140 and
- figures 1 to 3 as published in WO-A-93/17140.

V. The single claim reads as follows:

"1. Coated cemented carbide insert with improved toughness and resistance against plastic deformation consisting of

- a substrate consisting of WC, cubic phases of carbide and/or carbonitride, a binder phase based on Co and/or Ni, and optionally carbon, wherein the amount of cubic phase expressed as the total content of metallic elements, that form cubic carbides, is between 6 and 15 weight-%,

the substrate having

- a binder phase enriched surface zone being less than 50 μ m thick, the binder phase enriched surface zone being essentially free from cubic phase except for the very surface where the share of cubic phase is \leq 50 volume-%, and the binder phase content in the binder phase enriched zone having a maximum of more than 1.1 of the binder phase content in the inner portion and said maximum being at a distance of 10-30 μ m from the surface, - a zone of less than 300 µm below the binder phase enriched surface zone, wherein the binder phase content is 0.85 - 1 of the content in the inner portion of the substrate, and wherein the content of cubic phases is constant and equal to the content in the inner portion of the substrate,

- and at least one wear resistant coating deposited on the substrate with CVD- or PVDtechnique."

VI. In support of inventive step, the appellant drew attention to the features relating to the composition of the starting material and that of the outermost surface of the cemented carbide substrate. It also referred to the criticality of the process parameters necessary to adhere to so that the claimed concentration profiles for the binder phase and the cubic phase in the different zones at and below the surface of the cemented carbide substrate were successfully achieved.

Reasons for the Decision

- 1. The appeal is admissible.
- 2. Amendments

The single claim derives from a combination of claims 1 to 5 and the subject matter present in the passage bridging page 5, line 16 to page 6, line 7 of the published application (WO-A-93/17140).

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The wording "a substrate <u>comprising</u> of ..." has been replaced by "<u>consisting</u>" so that components other that those listed are excluded from the claimed cemented carbide substrate. For the sake of clarity, the relative term "essentially" before the word "constant" has been deleted to make plain that the concentration of the cubic phase (CP) in the zone below the binder enriched surface zone (BEZ) corresponds - within the accuracy typically resulting from the physical measuring method and the non-homogeneity of the material - to the concentration in the inner portion of the substrate.

The single claim therefore satisfies the requirements of Articles 123(2) and 84 EPC.

- 3. Novelty and inventive step, Articles 54 and 56 EPC
- 3.1 Document D1 is concerned with a coated cemented carbide substrate comprising tungsten carbide (WC) and cubic phase CP (called in D1: a B-1 phase which is a hard solid solution of carbides generally having a cubic crystal lattice structure) in a cobalt binder phase (cf. D1, column 4, lines 6 to 24). As shown in D1, example 5 and Figure 5, the insert exhibits a cobalt enriched subsurface zone (BEZ) having a thickness between 10 to 40 microns which is nearly depleted of the CP (cf. D1, column 10, lines 16 to 56; see also column 6, lines 2 to 12).

The claimed cemented carbide insert differs from this prior art in that in D1 a CP (B-1 phase) enriched surface layer typically exhibiting a thickness of about 1 to about 5 microns or even up to 10 microns is provided which, according to example 3, covers 95% of the surface. This surface structure is produced by the specific sintering process described in document D1, column 6, lines 34 to 40; lines 54 to 65; column 7, lines 4 to 7, and in claim 37. Due to the presence of this CP enriched surface layer and to the oxidation of the surface of the insert, the outermost metal oxide wear layer that is deposited on this surface becomes tightly adherent to the substrate (cf. D1, column 7, lines 50 to 56; column 10, lines 54 to 56).

By contrast, the presence of the CP on the surface of the claimed cemented carbide substrate is restricted to less than 50 volume% at maximum. More preferably, the formation of CP should be avoided since, as explained on page 6, lines 9 to 12 of the application, the cubic phase and possible graphite that may have been formed during sintering are essentially removed from the surface prior to the coating of the cemented carbide substrate. Hence, the technical teaching given in document D1 is leading away from producing the cemented carbide insert claimed in the present application.

3.2 Document D2 discloses a Co-based cemented carbide substrate comprising aluminium nitride (AlN) as an additional ingredient in an amount sufficient to enhance the surface toughness of the cemented carbide body by promoting an enrichment of cobalt and the depletion of AlN near the peripheral surface in the BEZ (cf. D2, column 1, lines 51 to 59; column 2, lines 36 to 45; claim 1). It is also apparent from document D2, example 3, Table 1 that in the zone adjacent to and below the BEZ the concentration of the Co binder is

depleted to a minimum value of 5.8%. Compared to the average concentration of about 7.2% Co determined inside the core of the insert, a relative Co concentration of 0.81 results. As can be seen, the depletion of the Co binder in the second zone below the BEZ of the known insert is high and outside the relative binder concentration range of 0.85 to 1 defined for this zone in the claimed insert. A high depletion of the cobalt binder in this zone, however, promotes the formation of cracks which adversely affect the cutting performance of the insert during machining. Given that in document D2 AlN is described as an indispensable ingredient which is, however, excluded from the claimed cemented carbide insert and nothing is disclosed in D2 as to how to prevent the depletion of the binder phase in the zone below the BEZ, the technical teaching given in document D2 could not lead a skilled person in an obvious way to the claimed subject matter.

3.3 Like the claimed cemented carbide insert, document D3 discloses a cemented carbide substrate of the general proportions: 2 to 5 wt% of TiC, 5 to 10 wt% TaC, 5 to 10 wt% Co, balance WC which comprises (a) a Co enriched zone, (b) an outer surface of the substrate with optimum cobalt dispersion and enrichment which supports (c) a multilayer coating combination of hard wear resistant materials including TiN and TiC (cf. D3, column 2, lines 46 to column 3, line 33). The graph given in Figure 2 indicates (compared to the nominal content of 6 wt% Co) the distribution of the cobalt binder phase and the CP concentration (expressed in wt% of Ti) in the BEZ and the zone adjacent to and below the BEZ. This type of cemented carbide substrate is

obtained by controllably injecting gaseous nitrogen into the sintering cycle manufacturing process. Therefore, this document which is already acknowledged as technical background in the introductory part of the application is regarded as representing the closest prior art.

Document D3 describes in column 4, lines 2 to 8 that varying the conditions of nitrogen partial pressure in the sintering atmosphere, the hold temperature and hold time affect the depth of the resulting CP (B-1 phase) depletion as well as the degree and depth of the cobalt enrichment in the BEZ. As has been mentioned in connection with the cemented carbides known from D1 and D2, a second zone below the BEZ exists in the insert given in D3 where the Co binder phase is considerably depleted (to about 4 wt%) below the average (or nominal) content of the binder phase (about 6 wt%) and where the amount of metallic element(s) present in the CP is enriched.

The present inventors have, however, realized that cracks grow easily in this second zone which has a decisive influence on the toughness and fracture frequency during machining. Starting from this prior art, the problem underlying the present application, therefore, resides in providing a cemented carbide substrate exhibiting an improved match in the above mentioned properties.

The solution to this problem consists in a cemented carbide substrate comprising an outermost surface where the share of CP is less than 50 volume-%, a BEZ and a second zone adjacent to and below the (BEZ) where the depletion of the binder phase is minimized to fall within the relative binder concentration range of 0.85 to 1. In addition, a constant concentration of the CP equal to that in the inner portion of the insert is obtained. This internal structure of the claimed cemented carbide substrate is achieved in that, after sintering in vacuum or in an inert atmosphere (gradient sintering), nitrogen gas is supplied to the sintering atmosphere at a temperature above T_{liguidus} of the binder phase and by maintaining the insert in this atmosphere until the binder has solidified (cf. the application page 4, lines 12 to 33). In so doing the cemented carbide substrate defined in claim 1 of the application does not comprise a zone which is prone to the propagation of cracks so that the substrate exhibits a unique balance of mechanical properties including a very good toughness behaviour and a high resistance to plastic deformation.

The examples according to the invention and the comparative examples show that the claimed object has been successfully achieved.

Document D3 does, however, not disclose the same process applied in the present application. Although nitrogen gas is actually introduced below the liquidus temperature of the binder phase, it is evacuated from the furnace before cooling the insert and solidifying the binder. More particularly, no gradient sintering is performed <u>prior</u> to the nitrogen gas treatment as it is specified in the process described in the present application and, consequently, a cemented carbide having a Co binder phase and CP concentration profile different from that claimed is obtained. Document D3 focuses essentially on providing in the cemented carbide substrate a subsurface zone highly enriched in cobalt (i.e. the BEZ) but, like D1 and D2, it does not pay any attention to the depletion of the binder phase in the second zone below the BEZ. Consequently, no information is found anywhere in document D3 as to how the depletion of the cobalt binder in the zone adjacent to and below that zone could be effectively reduced. Hence, document D3 fails to provide any incentive as to how the identified problem could be solved. Given this situation, the claimed coated cemented carbide insert also involves an inventive step vis-à-vis the technical teaching given in document D3.

Order

For these reasons it is decided that:

- 1. The decision under appeal is set aside.
- 2. The case is remitted to the first instance with the order to grant a patent on the basis of:
 - claim 1 as filed during the oral proceedings;
 - description pages 1 to 10 as published in WO-A-93/17140 and
 - figures 1 to 3 as published in WO-A-93/17140.

The Registrar:

The Chairman:

V. Commare

T. H. K. Kriner