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D E C I S I O N
of 16 November 2000

Case Number: T 0604/97 - 3.2.2

Application Number: 91101128.6

Publication Number: 0440157

IPC: C23C 16/36

Language of the proceedings: EN

Title of invention:
Process for producing a surface-coated blade member for cutting tools

Patentee:
MITSUBISHI MATERIALS CORPORATION

Opponent:
Widia GmbH

Headword:
-

Relevant legal provisions:
EPC Art. 56

Keyword:
"Inventive step (no)"

Decisions cited:
-

Catchword:
-

Case Number: T 0604/97 - 3.2.2

D E C I S I O N
of the Technical Board of Appeal 3.2.2
of 16 November 2000

Appellant: (Opponent)
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Respondent: (Proprietor of the patent)
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Decision under appeal: **Decision of the Opposition Division of the European Patent Office posted 2 April 1997 rejecting the opposition filed against European patent No. 0 440 157 pursuant to Article 102(2) EPC.**

Composition of the Board:

Chairman: W. D. Weiß
Members: R. Ries
J. C. M. De Preter

Summary of Facts and Submissions

I. European patent No. 0 440 157 was granted on 7 June 1995 on the basis of European patent application No. 91 101 128.6.

II. The granted patent was opposed by the present appellant (Widia GmbH) on the grounds that its subject matter lacked an inventive step with respect to the state of the art (Article 100(a) EPC). A second opponent (Sandvik AB) withdrew its opposition with letter of 6 August 1996.

III. With its decision posted 2 April 1997 the Opposition Division held that the patent could be maintained in the form as granted and rejected the opposition. Amongst others, the following documents were cited in the opposition proceedings:

D1: JP-A-60149775 and abstract

D2: DE-A-2 505 009

D3: MPR, April 1984, "Carbonitride Coatings at Moderate Temperature for Cemented Carbide Tools", two pages

D4: US-A-3 971 656

D5: 12th Int. Plansee Seminar '89, 8 to 12 May 1989, Proceedings volume 3, pages 13 to 25

D7: Cermets, Special publication from: Pulvermetallurgie in Wissenschaft und Praxis, volume 4, 1988, pages 1 to 31

D8: MPR,"New CVD Coatings for Carbide Inserts", December 1988, pages 832 to 834

D9: Industrie-Anzeiger 11/1988, pages 28 to 30

D10: Industrie-Anzeiger no. 31, 17 April 1985, pages 36 to 38

D15: Hartmetalle, R. Kieffer, F. Benesovsky, Springer Verlag 1965, pages 64, 534

IV. An appeal was lodged against this decision on 30 May 1997. In its letter of 20 October 2000, the appellant for the first time referred to document

D16: EP-A-0 263 747.

V. In an official communication, the Board referred to the document

D5a: M. Bonetti et al., Proceedings of the 8th International Conference on Chemical Vapour Deposition, Paris-Gouvieux 1981, pages 606 to 616

VI. Oral proceedings were held before the Board on 16 November 2000.

The appellant (opponent) requested that the decision under appeal be set aside and that the European patent be revoked.

The respondent (proprietor of the patent) requested that

- the appeal be dismissed (main request);

- the decision under appeal be set aside and that the patent be maintained on the basis of claims 1 to 3 submitted by letter of 16 October 2000 (first auxiliary request);

- or on the basis of claims 1 to 3 submitted at the oral proceedings (second auxiliary request).

Claim 1 of the main request reads as follows:

"1. A process for producing a surface-coated blade member for cutting tools, comprising the steps of:
(a) preparing cermet substrate, wherein
said cermet substrate contains 70 to 95% carbo-nitride by weight as a hard phase-constituting component,
said cermet substrate has the formula $(Ti_xM_y)(C_uN_v)$, wherein M is at least one metal selected from the group consisting of metals in groups IV_A,V_A and VI_A of the Periodic Table except for titanium, and wherein x, y, u and v are molar ratios, and $0.5 \le x \le 0.95$, $0.05 \le y \le 0.5$, $0.2 \le u \le 0.8$, $0.2 \le v \le 0.8$, $x+y=1$ and $u+v=1$, and
said cermet substrate contains a binder component of one or two metals selected from the group of cobalt and nickel; and,
(b) forming at least one layer of titanium carbo-nitride by a chemical vapor deposition process on said cermet substrate, wherein
said chemical vapor deposition process is conducted in a temperature range of 700 to 900°C,
said chemical vapor deposition process uses a reaction gas containing acetonitrile, and
said layer of titanium carbo-nitride has an average thickness of 0.5 to 20 µm.

Claim 1 of the first auxiliary request reads:

"1. A process for producing a surface-coated blade member for cutting tools, comprising the steps of:

(a) preparing cermet substrate, wherein

said cermet substrate contains 70 to 95% carbo-nitride by weight as a hard phase-constituting component,

said cermet substrate has the formula $(Ti_xM_y)(C_uN_v)$, wherein M is at least one metal selected from the group consisting of metals in groups IV_A, V_A and VI_A of the Periodic Table except for titanium, and wherein x, y, u and v are molar ratios, and $0.5 \le x \le 0.95$, $0.05 \le y \le 0.5$, $0.2 \le u \le 0.8$, $0.2 \le v \le 0.8$, $x+y=1$ and $u+v=1$, and

said cermet substrate contains a binder component of one or two metals selected from the group of cobalt and nickel; and,

(b) forming a layer of titanium nitride by a chemical vapor deposition process on said cermet substrate; and

(c) forming at least one layer of titanium carbo-nitride by a chemical vapor deposition process on said layer of titanium nitride, wherein

said chemical vapor deposition process is conducted in a temperature range of 700 to 900°C,

said chemical vapor deposition process uses a reaction gas containing acetonitrile, and

said layer of titanium carbo-nitride has an average thickness of 0.5 to 20 µm.

The wording of claim 1 of the second auxiliary request differs from that of the first auxiliary request in that it comprises an additional step d:

"1. ..20 µm; and
(d) forming a layer of titanium nitride by a chemical vapor deposition process on said at least one layer of titanium carbonitride."

VII. The appellant argued as follows:

A person skilled in this field of technology is aware of the compositions of the various cermet substrates and of the methods for depositing one or more coating(s) thereupon, e.g. chemical vapour deposition (CVD), physical vapour deposition (PVD) etc. In the chapter "Prior art", the patent itself mentions the CVD process as being conventional for producing hard reaction layers of TiC, TiCN etc or the cermet substrate. However, due to the high temperature of about 1000°C used in the CVD process, diffusion of the binder metals cobalt or nickel into the surface layer occurs and impairs the wear resistance of the hard coating. The diffusion problem as such is well known in the art and also to the patentee. Reference is made in this context to the surface coated WC-base hard metal disclosed in another patent application of the present patentee which comprises a diffusion preventing layer (cf. document D16, page 3, lines 28 to 41). Thus, faced with the problem of reducing the amount of binder which diffuses into the surface layer and bearing in mind the well known physical law that the diffusion velocity increases exponentially with temperature, the man skilled in the art would obviously look for a coating method which operates at lower working temperatures than those used in the conventional CVD method. Based on these considerations, he would inevitably be led to the moderate temperature CVD process (MT-CVD) which is well known in the art through numerous publications (cf. documents D2, D3, D5, D8 to D10). This process utilizes an organic compound, like acetonitrile, as the source of C and N rather than methane and nitrogen to deposit Ti(CN), TiC and TiN coatings in a temperature range of 700 to 900 °C (see in particular document D8, page 832).

As to the process given in claim 1 of the first and second auxiliary requests and including the step of forming a TiN layer on the substrate and on the TiCN layer, it is known from document D16 that the binder metal Co can be prevented from diffusing into the hard coating by providing a "barrier layer" on the substrate. Moreover, the sequence of coatings TiN-TiCN-TiN on a cutting tool substrate is known from document D5, Figure 8.

Consequently, the subject matter claimed in the main, first and second auxiliary request does not involve an inventive step.

The respondent argued as follows:

None of the documents cited by the appellant refers to a process of coating a cermet substrate with a hard TiCN layer that comprises the process steps and conditions reflected in claim 1 of the patent. Thus, the closest prior art is not represented by any of the cited documents, but is the technological background described on page 2 of the patent. Starting from this prior art, the problem underlying the opposed patent consists in providing a process which improves the wear resistance of the hard surface coating. The solution to this problem is a chemical vapour deposition process which is carried out at 900°C or lower and which uses a reaction gas containing acetonitrile. By the low process temperature the diffusion of the binder metals Co or Ni into the coating is effectively avoided.

This technical problem is not addressed in any of the above-mentioned documents. Although there are documents (D2, D3, D5, D5a, D8 to D10) which disclose the Moderate Temperature Chemical Vapour Deposition (MT-CVD), they are concerned with the coating of cemented carbides rather than cermets. Moreover, "nitriles" in general are used as a reaction gas, and there is no particular preference for acetonitrile as is in the patent. Hence, given that the prior art fails to give any clear hint to a person skilled in the art that - compared to high temperature CVD - the MT-CVD process using acetonitrile as the reaction gas results in any benefits for the coating of cermets, in particular in an improvement to the wear resistance of the hard coating by reducing the diffusion of the binder metal into the coating, a person skilled in the art would not be encouraged to transfer the MT-CVD process to the coating of cermets. Thus taking into consideration the could - would approach, the process claimed in the patent was not deducible in an obvious way from the prior art and, therefore, involves an inventive step.

With the help of the TiN intermediate layer claimed in claim 1 of the first and second auxiliary requests, the bonding strength between the cermet substrate and the TiCN is increased. Although document D5 discloses a multicoating comprising a sequence of layers of TiN-TiCN-TiN, these coatings are formed by a plasma activated process at a temperature between 450 and 600°C. Compared to the claimed process, this temperature range is much lower. Hence, the subject matter given in claims 1 of the first and second auxiliary is neither derivable in an obvious way from the teaching given in D5 alone nor from a combination of D5 with the teaching given in any other document.

Reasons for the Decision

1. The appeal is admissible.

2. *Main request*

2.1 The closest prior art

In the introductory paragraph entitled "Prior art" the patent at issue acknowledges that it is conventional practice to form hard surface coatings of TiCN by chemical vapour deposition on cermet substrates of the claimed type. This paragraph further reflects the fact that CVD normally is carried out with a $TiCl_4$-CH_4-N_2-H_2 gas mixture at a relatively high temperature of about 1000°C which entails the drawback of adversely affecting the wear resistance properties of the TiCN reaction coating because the binder metal atoms diffuse into the surface coating (cf. the patent, page 2, lines 15 to 51).

It is beyond dispute that cermets having a composition of the particular type acting according to claim 1 as a substrate for the production of cutting tools belong to the state of the art. Reference is made in this context for example to document D4 which discloses a composition comprising a $(Ti_xM_y)(C_uN_v)_z$ hard phase, where M is a Group VI metal with x+y=1, u+v=1, $0.8 \leq z \leq 1.07$ and y ranging from 0.04 to 0.40. The hard phase is embedded in a Co and/or nickel binder that makes up between 8 and 25 wt% of the composition (cf. D4, claims 1 and 4). More specifically, document D4 also discloses examples falling within the claimed ranges (see for instance Table 6, carbonitride D, consisting of $(Ti_{60}M_{40})(C_{80}N_{20})_z$ and 14 wt% Ni as a binder; column 3, lines 54/55; Table 7, designation H $(Ti_{70}M_{30})(C_{80}N_{20})_{92}$ comprising a binder metal selected from Ni, Co, Fe; example 8 in column 10 comprising $(Ti_{80}M_{20})(C_{70}N_{30})_{0.95}$ and 11 wt% Ni + 2.75 wt% Mo as a binder). Cermets which exhibit one or two TiCN coatings having a thickness 2 to 7 µm are disclosed in document D1 (see Table 1-1, examples 4 and 5, Table 1-3, example 2). The diffusion phenomenon, i.e. the migration of Co or W from the binder of the substrate into the TiC or TiCN layer during the formation of the coating had been observed before the priority date of the patent at issue and had been known to the expert to impair the resistance of the hard surface layer. In view of these considerations, the Board concurs with the respondent's position that the acknowledgement of the state of the art given the introductory part of the description of the patent at issue (see EP-A-0440157, page 2, lines 15 to 51) correctly summarizes the knowledge of a person skilled in this field of technology. It, therefore, represents the closest prior art.

2.2 The problem to be solved

Starting from this prior art, the fundamental problem underlying the patent in suit resides in providing a CVD process for producing surface coated cermet blade members or inserts which effectively minimizes or even avoids the diffusion of the binder metals Co or Ni into the hard TiCN coating so that the wear resistance of the hard layer is not impaired (cf. the patent page 2, lines 42 to 58).

The solution to this problem consists in a chemical vapour deposition process

- which uses a reaction gas comprising acetonitrile and

- which is carried out at a "low" temperature ranging from 700 to 900°C rather than 1000°C used in conventional CVD which in the following will be called High Temperature Chemical Vapour Deposition (HT-CVD) (cf. the patent, page 3, lines 1 to 10).

2.3 Inventive step

The claimed solution to the above-mentioned problem would be, however, derivable in an obvious manner for a person skilled in the field of chemical vapour deposition technology. Having regard to the fact that diffusion is strongly dependent upon temperature, it had already been envisaged to carry out the CVD process at temperatures of 900°C or below, but the formation of the TiCN reaction layer was found to be unsatisfactory (see EP-A-0440157, page 2, lines 48 to 51). Another state of the art approach to overcome the diffusion problem in the HT-CVD process consists in providing a "diffusion barrier layer" sandwiched between the substrate and the outer hard layer (see document D16).

Bearing in mind that the CVD-process temperature is the key feature to reduce diffusion, the skilled person in his search for a "low temperature process" would be immediately prompted to select the so called "Moderate Temperature Chemical Vapour Deposition" (MT-CVD) process. One of the essential advantages of this process is that, due to the use of organic C-N compounds (nitriles) substituted for the conventionally used chloride compounds, it can be carried out at relatively low temperatures. The MT-CVD process has been amply described in the prior art (cf. D2, D3, D5, D5a, D8 to D10) and thus belongs to the technical background knowledge of a skilled person. For instance document D8 mentions that by using acetonitrile as a source of C and N, hard TiCN coatings can be deposited in a temperature range of 700 to 900°C. When working in this temperature range, excessive thermal load on the substrate material is avoided, interfacial stress between the coating and the substrate and the risk of eta-phase formation are reduced (cf. D8, page 832). According to the technical teaching given in documents D9 and D5a, the MT-CVD process is not restricted to a specific substrate, but is applicable to various substrates including ceramics, cemented carbides, glass, copper, tungsten or steel (cf. D5a, page 608, lines 1 to 10; D9, page 30, left hand column lines 5 to 8). In a more detailed investigation, document D10 shows, how the properties of the resulting hard coating are affected by specific nitrile compounds and by the deposition temperature selected in the MT-CVD process. More particularly, it is mentioned that "by lower temperatures, the diffusion between the substrate and the coating is reduced" (cf. D10, page 37, column 2, lines 26 to 30).

Thus, contrary to the respondent's position, the skilled worker would in his search for a solution to the technical problem defined above seriously turn to the MT-CVD process because, by the reduced thermal load, the diffusion between the binder in the substrate and the hard coating encountered in the HT-CVD process can be effectively lowered.

Consequently, the subject matter of claim 1 of the main request does not involve an inventive step.

3. *First auxiliary request*

Claim 1 of the first auxiliary request requires in step (c) the deposition of a TiN layer by conventional HT-CVD on the substrate before depositing the TiCN top layer by MT-CVD. Although the function of this layer is not unambiguously described, it appears from the patent specification page 3, lines 42 to 48 that the intermediate TiN layer, at least to a certain extent, is intended to act as a barrier layer to additionally prevent the diffusion of cobalt into the TiCN layer. The application of a barrier layer, however, merely represents conventional practice as has been previously shown. Therefore, the process described in claim 1 of the first auxiliary request does not involve an inventive step either.

4. *Second auxiliary request*

The considerations above equally apply to claim 1 of the second auxiliary request which additionally provides in step (d) to deposit an outer surface layer so that the hard TiCN coating is sandwiched between two layers of TiN. Due to its golden colour such an outer TiN surface coating is frequently applied merely in order to improve the appearance of the final product. It does not add anything inventive to solve the diffusion problem underlying the patent at issue. At all events, such an outer coating of TiN would not be absolutely necessary to provide the cermet with a wear resistant coating. This assessment was not challenged by the patentee.

Consequently, the features incorporated into the claims according to the first and second auxiliary requests fail to add anything inventive to the subject matter of the claims according to the main request. Hence, the claims according to the auxiliary requests are not acceptable either.

Order

For these reasons it is decided that:

1. The decision under appeal is set aside.

2. The patent is revoked.

The Registrar: The Chairman:

V. Commare W. D. Weiß