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D E C I S I O N
of 25 October 1994

Case Number: T 0912/91 - 3.3.2

Application Number: 84308695.0

Publication Number: 0145496

IPC: C04B 35/56

Language of the proceedings: EN

Title of invention:

Sintered silicon carbide/graphite/carbon composite ceramic
body having ultrafine grain microstructure

Patentee:

The Carborundum Company

Opponent:

- 01) RHONE-POULENC CHIMIE
02) Stora Feldmühle AG
03) Hutschenreuther AG

Headword:

Relevant legal provisions:

EPC Art. 123(2) and (3), 114(2), 54, 56

Keyword:

"Change of claim category"
"Novelty and inventive step (yes)"
"Use of a compound in a composition for obtaining a specific
effect not suggested in the prior art"

Decisions cited:

Catchword:

Case Number: T 0912/91 - 3.3.2

D E C I S I O N
of the Technical Board of Appeal 3.3.2
of 25 October 1994

Appellant: The Carborundum Company
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Decision under appeal: Decision of the Opposition Division of the European
Patent Office of 3 July 1991 posted on 1 October
1991 revoking European patent No. 0 145 496
pursuant to Article 102(1) EPC.

Composition of the Board:

Chairman: P. A. M. Lançon
Members: M. M. Eberhard
J. A. Stephens-Ofner

Summary of Facts and Submissions

- I. European patent No. 0 145 496 based on application No. 84 308 695.0 was granted on the basis of 38 claims.
- II. The Respondents (Opponents 1, 2 and 3) filed a Notice of Opposition requesting the revocation of the patent on the grounds of lack of novelty, lack of inventive step and insufficiency of disclosure. Of the documents cited during the opposition procedure only the following ones were relied upon in the present appeal:
- (2) JP-A-58130165: English translation filed on 22.1.91
 - (9) DE-A-2 923 729
 - (10) DE-A-2 809 278
 - (14) US-A-4 346 049 equivalent to DE-A-2 624 641.
- III. The Opposition Division revoked the patent on the grounds that Claim 1 of the main request and Claim 1 of the first auxiliary request filed on 3 July 1992 did not meet the requirements of Article 123(3) EPC, and that the subject-matter of Claim 1 of the second auxiliary request filed at the same date did not involve an inventive step. The Opposition Division considered document (14) as being the closest prior art. It held that the use of graphite in a raw batch for forming a sintered silicon carbide body was known, or at least suggested, in the art (cf. documents (10), (9) and (2)). Thus, the replacement of a part of the carbonizable organic material of document (14) with graphite could only involve an inventive step if it was associated with a surprising technical effect. However, such an effect had not been demonstrated. Nor could it

be derived from the data and Figures of the patent in suit that the addition of graphite led to an improvement in microstructure. Furthermore, it has not been made clear in which respect the sintering conditions were simplified by the use of graphite. Therefore, so they held, the partial substitution of carbon derived from carbonizable material with graphite was an obvious alternative to a person skilled in the art.

IV. The Appellant (Patentee) lodged an appeal against this decision and submitted two additional analyses of the experimental data contained in the patent in suit, together with the Statement of Grounds of Appeal. On 4 October 1994 the Appellant filed an affidavit of Dr D. W. Kuhlmann. Oral proceedings were held on 25 October 1994 in the absence of Respondents II and III, who had informed the Board that they would not attend the hearing. During the oral proceedings Respondent I referred to US-A-4 135 938 which corresponded to document (10) except for the mention of a particle size of below 3 μ m for the carbon. The Appellant withdrew its request based on the product claims, and submitted a set of use claims as the sole request. Claim 1 of this sole request reads as follows:

"Use of from 1 to 13 per cent by weight of graphitic elemental carbon having an average grain size not in excess of 8 microns (μ m) in a pressureless-sintered silicon carbide/graphite/carbon composite ceramic body essentially free of uncombined silicon consisting moreover essentially of:

- (a) from 0.5 to 5.0 percent by weight of amorphous carbon, with the body having a total uncombined carbon content of from 1.5 to 15 percent by weight;
- (b) 0.15 to 5 percent by weight in total of a sintering aid selected from the group of boron, aluminium, beryllium in elemental or compound form; and
- (c) a balance of silicon carbide;

the body having a density of at least 75 percent of its theoretical density based on the law of mixtures,

for obtaining a composite body having a homogeneous fine grain microstructure of silicon carbide grains and graphite grains with at least 50 percent of its silicon carbide grains on an area basis having a size not exceeding 8 microns (μm) and an aspect ratio of less than 3."

V. The Appellant put forward the following main arguments:

Claim 1 related to the **use** of graphite **in a composition** for a certain **purpose**, i.e. for obtaining a homogeneous fine grain microstructure of silicon carbide grains and graphite grains in the composite body. This use claim included the mention of an effect, and was therefore a use claim of the kind dealt with in decision G 2/88 of the Enlarged Board.

Starting from document (14) as the closest prior art, the technical problem was to provide means for keeping the microstructure fine. The cited documents neither disclosed the use of graphite for the purpose indicated

in Claim 1, nor gave any hint of the improvement of the microstructure.

The Opposition Division had not appreciated that even though experimental error might exist, a clear trend could still be ascertained from the data and Figures of the patent in suit by one skilled in the art. The additional analyses enclosed with the Statement of Grounds of Appeal confirmed that there was a definite inverse correlation between graphite content and particle size, the largest average particle size being obtained at the lowest graphite content. This surprising effect was also clearly shown by the affidavit of Dr D. W. Kuhlmann. The simple linear regression performed by Respondent I improperly excluded those samples without graphite. A valid statistical analysis also had to look at the grain size data of samples without graphite for the sake of comparison. The second surprising effect consisted in that the addition of an effective amount of graphite allowed the green body to be sintered under conditions which did not need be controlled as precisely as was necessary without the presence of graphite. This effect was demonstrated by the reheat results reported in Table II of the patent in suit and by the corresponding Figures. The time/temperature window for sintering, while maintaining a relatively small average grain size, was widened by the use of added graphite. This made the sintering operation less critical, which was an expected and commercially important benefit of the claimed invention.

VI. Respondent I contested the admissibility of the request based on the use claims submitted by the Appellant

during the oral proceedings. He further argued that the affidavit of Dr Kuhlmann was submitted so late that it should be disregarded, as the very short time available up to the oral proceedings did not enable the Respondent to obtain the comments of an external independent expert in statistical analysis.

The arguments put forward by Respondents I and II as regards novelty can be summarised as follows:

The starting composition of document (10) comprised 0.45 to 15 wt% of carbon either in the form of free carbon such as graphite, or in the form of any carbonizable organic compound and silicon carbide with a particle size of below 3 μm . The liquid used to make the slurry could also be a compound which gave carbon by heating. Therefore, document (10) disclosed the use of a combination of graphite and carbon in the sintered body. As the raw batch and the process of (10) were the same as those of the patent in suit, the resulting sintered bodies did not differ from each other. The statement that the carbon was chemically combined with the silicon carbide was only an assumption of the author.

The sintered composite body of document (14) was also prepared by the same process as in the patent in suit. This body had a fine grain microstructure of silicon carbide with an average grain size of 7.5 μm and an aspect ratio of less than 3. The use of graphite was not mentioned in (14), however the comparative tests of Respondent I enclosed with the letter dated 27 May 1991 have shown that starting mixtures containing 2 or 5 wt% of carbon led to sintered bodies having graphite

contents of 1.1 or 2.7 wt% respectively. Moreover, according to (14) the calculated carbon content could be 6.6 wt%. Even with a conversion of the organic material to graphite of only 50%, the graphite content already lay within the claimed range.

As regards obviousness, Respondent I submitted that (14) aimed at obtaining a sintered body having a fine-grained equiaxed microstructure. In 1980 silicon carbide was mainly used in sliding seals for pumps, and this use required that the material should exhibit good frictional properties as well as a great resistance to thermal shock. As indicated in the Statement of Grounds of Appeal, a small grain size improved the tribological properties and the strength of the body. Therefore, the achievement of a fine-grained microstructure was desirable. Both documents (2) and (10) taught the use of graphite in combination with a carbonizable resin and (2) further disclosed that the frictional properties of the sintered body were improved by the addition of graphite. Thus, it would have been obvious to the skilled person to combine the teaching of (14) and (2) and to use graphite in the sintered bodies of (14). Furthermore, according to (10) the electrical properties and the resistance to thermal shock were also enhanced. In view of (2) or (10), the skilled person would have expected that the use of graphite in the composite body of (14) would improve its mechanical properties.

In his written submissions, Respondent II contended that document (9) not only dealt with the same problem that the patent in suit did, but also concerned the same kind of sintered body, since aluminium nitride was

also mentioned as sintering aid in the patent in suit. The skilled person would have inferred from (9) that the addition of graphite was equivalent to the use of carbon derived from organic compounds. Since, on the one hand, document (9) addressed the question of thermal stability and wear resistance and, on the other hand, it was well known to the skilled person that graphite was suitable as a lubricant in solid bodies, it would have been obvious to replace a part of the carbon derived from organic compounds by graphite in the composition of document (14). Even if the improvement concerning the SiC grain size would have been achieved, this would not have been at all surprising.

Respondents I and II contested that the results of the image analysis and of the regression analysis enclosed with the Statement of Grounds of Appeal demonstrated the first of the alleged surprising effects. Respondent I argued that its own regression analysis showed that with the products containing at least 1% of graphite there was no strong inverse correlation between the grain size and the graphite content, and that the grain size varied inversely to the content of amorphous carbon. Furthermore, he argued, these analyses were based on the experimental data of the patent in suit and these examples did not prove the alleged effect. Nor did the reheat tests have establish the second of the alleged surprising effects, instead they have merely shown that the time period of the sintering step could be prolonged, but not that a treatment at a higher temperature would have had no influence on the grain size.

Respondent III did not submit any arguments or requests.

VII. The Appellant requested that the decision under appeal be set aside, and that a patent be granted on the basis of the single request submitted during oral proceedings. Respondents I and II requested that the appeal be dismissed.

Reasons for the Decision

1. The appeal is admissible.
2. The use claims of the sole request were submitted by the Appellant during the oral proceedings. In the present case the Board has decided to admit this new request, since, although filed at a very late stage of the procedure, it represents a genuine attempt to overcome the objections raised by the Respondents in connection with the patentability of the product claims. In addition, it meets the requirements of Articles 123(2) and (3) EPC for reasons which will be set out below.
3. The affidavit of Dr D. W. Kuhlmann was filed on 4 October 1994, i.e. a mere three weeks before these oral proceedings, although it was executed as far back as 16 February 1994. The change of representative, put forward by the Patentee's new representative at the hearing as the reason for such a long delay cannot excuse such tardiness. Furthermore the short time between receipt of this affidavit and the appeal hearing was clearly insufficient to enable Respondent I

to deal with it adequately or at all. In these circumstances, and taking into account that the content of this late filed affidavit does not appear to be so relevant that it might influence the outcome of this appeal, the Board has decided to disregard it (cf. Article 114(2) EPC).

The late submitted US-A-4 135 938, based on the same priority document as (10), might have been relevant to the product claims on file at the beginning of the oral proceedings. However, these claims have been abandoned and replaced by the set of use claims. In view of the fact that this US patent, like the corresponding document (10), is totally silent about the effect of the graphite on the properties or characteristics of the composite body, it can have no influence on the outcome of this appeal insofar as it concerns the new use claims. Therefore, this document is likewise disregarded.

4. In view of the limitation of the claims to the use of 1 to 13 wt% of graphite in the sintered composite body, the Respondent's objection that the invention cannot be carried out by the skilled person throughout the whole range of the claimed subject-matter (originally 1 to 48 wt% of graphite in a sintered body having a density of at least 75%) has been overcome. Therefore, the Board finds that the objection of insufficiency of disclosure has been overcome.

5. There are no objections on the basis of Article 123(2) and (3) to the amended claims of the sole request. This is because the subject-matter of Claim 1 is supported by the product Claims 1, 2, 4 and 15 as originally

filed and the original description page 27, lines 17 to 30, page 28, lines 10 to 14 and 23 to 29; page 29, lines 6 to 10. Furthermore it is directly and unambiguously derivable from these passages that the use of graphite leads to a fine microstructure as defined in Claim 1.

The change of claim category from product claims to use claims does not broaden the scope of protection of the granted Claims 1, 15 and 29. Even if it were considered that the use claim is notionally equivalent to a claim to a process including the step of using the graphite in the sintered body and that the effect of Article 64(2) EPC is to confer protection to the product resulting from this process as well, this would not represent an extension of protection in the sense of Article 123(3) EPC, since the sintered composite body is defined in the use Claim 1 in a more restricted way than the composite body of the granted Claim 1: see the narrower range of the graphite content, the indication of the amount of sintering aid and the limitation to particular sintering aids in the use claim (cf. G 2/88 OJ EPO 1990, 093, point 5.1).

6. Turning, next, to the issue of novelty, the subject-matter of Claim 1 is novel since none of the cited documents discloses the use of 1 to 13 wt% of graphite with an average grain size not in excess of 8 μm in the sintered composite body in order to obtain a homogeneous fine grain microstructure as defined in Claim 1. Thus, in particular:

6.1 In document (2) the particle size of the graphite which is added to the raw composition is greater than the

average grain size stated in Claim 1. Furthermore, graphite is used in the composition in order to decrease the friction coefficient, i.e. for obtaining a different effect (cf. page 3, line 15 to page 4, line 2; examples 3 and 4, Fig. 1).

6.2 Document (14) neither mentions the use of graphite in the raw batch nor does it explicitly disclose the presence of graphite having an average grain size not in excess of 8 μm in the sintered composite body. As shown by Respondent I in the Annex I of its letter dated 27 May 1991, it appears that during a sintering step at 2100°C for 60 minutes a part of the carbonizable resin present in the raw batch is converted into graphite. In view of these results it cannot be excluded that the sintered composite bodies of Table 1 of document (14) might contain a small amount of graphite. However, the Respondents have not shown that under the sintering conditions used in experiment 1 of Table 1 of (14), i.e. 2120°C for 30 minutes, **at least 1 wt% graphite** having an average grain size not in excess of 8 μm was formed. In these circumstances, the Board cannot conclude that the sintered body defined in Claim 1 does not differ from that of said experiment 1. Furthermore document (14) does not teach the effect of graphite on the grain growth.

6.3 Document (10) contains the following statement: "most of the additional carbon presumably chemically combines with the silicon carbide and the said additive during the heating of the shaped body in the inert environment" (cf. page 8, 2nd paragraph). The Respondents have not shown that this statement is

incorrect. Furthermore, there is no disclosure of the use of graphite in the sintered body in order to obtain a homogeneous fine grain microstructure of silicon carbide grains and graphite grains as defined in Claim 1.

6.4 The sintered ceramic body of document (9) contains 55 to 99.5 wt% of silicon carbide, 0.5 to 45 wt% of aluminium nitride and 0.5 to 6 wt% of carbon or a carbon containing material (cf. Claim 1). The carbon can be used in the raw batch in the form of a carbonizable organic material, coal or colloidal graphite. It is not disclosed in (9) that the use of graphite has the effect of achieving a homogeneous fine grain microstructure as defined in Claim 1.

7. Turning to the issue of obviousness, the parties and the Opposition Division both accepted document (14) as representing the closest prior art, as does the Board in this appeal.

This document discloses a pressureless sintered alpha silicon carbide ceramic body having a predominantly equiaxed microstructure, i.e. a crystalline microstructure in which the grains have an aspect ratio of less than 3:1. The sintered body consists essentially of from about 91 to 99.85 wt% silicon carbide, up to about 5 wt% carbonized organic material, from about 0.15 to 3.0 wt% boron and up to about 1.0 wt% additional carbon. Its preferred minimum density corresponds to about 75% of the theoretical density. The carbonized organic material is free carbon or uncombined carbon produced in situ by the carbonization of the organic material used in the raw

batch (cf. column 3, lines 3 to 11; column 4, lines 19 to 22; column 4, line 59 to column 5, line 18). This sintered ceramic body can be produced by (a) mixing together appropriate amounts of alpha silicon carbide powder, a carbonizable organic material, a boron source, a temporary binder, a solvent in which the carbonizable organic material is soluble, (b) drying the resulting mixture, (c) shaping the dried mixture so as to produce a shaped body with a density of at least 1.6 g/cm^3 (at least 75% of the theoretical density) and an equiaxed microstructure, the temperature being between about 1900 and 2250°C (cf. column 3, lines 11 to 37 and Claim 1). Example 1 of this document teaches the effect of the sintering temperature and time on the crystal size.

- 7.1 As pointed out in the patent in suit, document (14) shows that it is difficult to achieve the desired fine grain size, equiaxed microstructure unless close control over the sintering conditions is maintained, particularly as regards the temperature. It is clear from example 1 of (14) that the grain size or growth of grain size is very sensitive to the sintering **time and temperature** since a sintering temperature of 2120°C for 30 minutes leads to a mean grain size of 7.5 μm whereas at a temperature of 2140°C for 45 minutes the resulting mean grain size is of 10 μm . Starting from (14) as closest prior art, the problem underlying the patent in suit can thus be seen in providing means for avoiding this drawback, i.e. means which make it possible to produce a sintered body having a fine grain microstructure without requiring such an exact temperature/time control during sintering as in

document (14) (cf. page 2, lines 1 to 11 and 21 to 25 of the patent in suit).

The patent sets out to solve this problem by using from 1 to 13% by weight of graphite having an average grain size not in excess of 8 μm in a pressureless sintered silicon carbide/graphite/carbon composite ceramic body having the composition and density stated in Claim 1.

7.2 Examples 1 to 4 and 12 of the patent in suit were performed using either different amounts of graphite or no graphite at all (example 12) while maintaining constant the amount and type of amorphous carbon source resin, the amount and type of sintering aid and the sintering conditions. The comparison of examples 1 to 4 with example 12 shows that the inclusion of the graphite powder in combination with the resin carbon source in the raw batch results in a finer microstructure even when the samples are sintered at a temperature as high as 2150°C for 30 minutes in argon and then reheated and maintained at this temperature for one hour. It is evident from these examples that less grain growth occurs in those samples containing the added graphite, and that a very fine and even finer grain microstructure than in the samples without graphite can still be obtained at a temperature of 2150°C (cf. Tables 1 and 2 and page 11, lines 7 to 14), i.e. within ranges of sintering temperature and time which are widened in comparison with the data given in Table 1 of (14). The same conclusion can be drawn from the comparison of examples 2 and 16, which include the same 5% total carbon level.

The comparative examples of the Annex II enclosed with the Respondent's letter of 27 May 1991 do not show that the technical problem is not solved since the sintering step was carried out at a temperature of 2100°C, i.e. a sintering temperature which is lower than in the examples of the patent in suit or in example 1 (samples 1 and 2) of document (14) and, therefore, which is less critical as regards the influence on the grain growth.

Thus, in view of Examples 1 to 4, 12 and 16 of the patent in suit and in the absence of credible evidence to the contrary, the Board finds that the technical problem stated above has been solved by the claimed use of graphite.

7.3 Document (14) aims at obtaining a sintered ceramic body having a predominantly equiaxed microstructure and teaches that the sintering temperatures should be closely controlled if it is desired to maintain a fine grain size, equiaxed microstructure (cf. column 2, last paragraph; column 4, line 47 to column 5, line 8; column 10, lines 54 to 63; column 11, line 30 to column 12, line 18). This document contains no information which could give the skilled person an incentive to use 1 to 13% by weight of graphite having an average grain size not in excess of 8 µm in the composite body in order to reduce the grain growth at sintering temperatures as high as 2150°C and to solve the technical problem stated above.

7.4 Although document (2) discloses the use of graphite in combination with a carbonizable organic material in the raw batch composition, the purpose of the addition of

graphite is to decrease the friction coefficient. The graphite is used in the ceramic composite body as an antifriction or lubricating agent (cf. page 2, 2nd and 3rd paragraphs; page 3, 2nd paragraph; page 3, last line to page 4, line 2; page 6, 6th paragraph; Figure 1). This document is silent about the average grain size and the aspect ratio of the silicon carbide grains in the sintered composite body. It does not suggest that the use of graphite in the composite body might reduce the grain growth of the silicon carbide grains at sintering temperatures at which according to (14) a substantial grain growth occurs. It should be further noted that a combination of the teachings of (2) and (14) would not lead to the claimed subject-matter since document (2) involves the use of graphite having a higher grain size than in the present invention.

- 7.5 Document (10) deals with the problem of providing a dense, pressureless sintered silicon carbide body which exhibits a good thermal shock resistance and improved electrical properties. This is achieved by sintering a shaped body consisting of silicon carbide with a particle size below 3 μm , 0.3 to 3 wt% of a sintering additive selected from boron nitride, boron phosphide or aluminium diboride and 150 to 500% of carbon by weight of said additive, the carbon being either in the form of free carbon such as graphite or in the form of a carbonizable organic composition (cf. page 7, the three last paragraphs and the paragraph bridging pages 9 and 10). This document teaches that these sintering additives lead to ceramic bodies having improved thermal shock resistance and electrical properties over bodies prepared with boron carbide as

sintering aid (cf. paragraph bridging pages 9 and 10, page 13, Table 2). Graphite is not used in the examples and it is solely mentioned as possible carbon source without any information about its effect on the properties or characteristics of the sintered body. Document (10) also contains no indication as to the average grain size of the silicon carbide in the sintered ceramic bodies. Therefore, it could not be inferred from it that the use of graphite might render the sintering operation less critical as regards the grain growth. In these circumstances, the skilled person confronted with the problem stated above would not have been prompted to combine the teaching of (10) and (14) in the expectation of solving this problem.

- 7.6 The purpose of document (9) is to provide a co-sintered silicon carbide-aluminium nitride product which exhibits the positive properties of both silicon carbide and aluminium nitride and which is substantially insoluble in warm water (cf. page 8, first paragraph). Therefore, this document deals with a problem which completely differs from that of the patent in suit. The starting mixture contains a carbon component which is used in any form that facilitates its mixing with the silicon carbide and gives a uniform dispersion of the carbon component in the mixture, for example colloidal graphite or a carbonizable organic material or coal. However, the use of a carbon containing material, in particular of a carbonizable organic material, is considered to be more advantageous (cf. page 8, 2nd paragraph and page 13, last paragraph). The latter is also used in all the sixteen examples of (9). According to page 11, lines 5 to 9, the carbon component facilitates the sintering

operation, and aids the reduction of oxides which might otherwise remain in the finished sintered product. The grain size or grain structure of the sintered product is not indicated in (9). It is not derivable from this teaching that the use of graphite in combination with amorphous carbon would result in less grain growth and would thus enable the sintering operation to be carried out without exercising the close temperature/time control required in (14). The fact that document (9) also addresses the question of thermal stability and wear resistance of the sintered ceramic body would be of no assistance to the skilled person confronted with the problem stated above, all the more so as these properties are not attributed to the presence of colloidal graphite.

8. It follows from the above that it was not obvious to arrive at the claimed use in view of the cited prior art. Therefore, the subject-matter of Claim 1 is considered to meet the requirements of inventive step set out in Article 52(1) and 56 EPC.
9. Claim 1 being allowable, the same applies to the dependent Claims 2 to 13 whose patentability is supported by that of Claim 1.
10. Finally, the Board finds that considering and deciding in substance on the maintenance of the patent on the basis of the present claims as amended during oral proceedings in the absence of two of the Opponents does not conflict with the decision of the Enlarged Board of Appeal G 4/92 (OJ EPO 1994, 149). According to this decision, a party who fails to appear at oral proceedings must have the opportunity, in accordance

with Article 113(1) EPC, to comment on new (and therefore surprising) facts and evidence submitted in those proceedings. The submission during oral proceedings of auxiliary requests is, clearly, neither a "fact" nor can it be "evidence" within the meaning of the above decision, so that that decision does not apply in such a case. Were it otherwise, no decision could ever be issued at the end of an appeal hearing where, as is usually the case, auxiliary requests are filed and, as is also frequently the case, one or more of the Opponents does not attend the hearing. This would render such hearings pointless, as well as offend the general principle of legal certainty, i.e. the general interest of the public in the termination of legal disputes ("expedit reipublicae ut sit finis litium").

Order

For these reasons it is decided that:

1. The decision under appeal is set aside.
2. The case is remitted to the Opposition Division with the order to maintain the patent on the basis of the claims as set out in the sole request submitted during oral proceedings with the description to be amended and with the drawings of the patent as granted.

The Registrar:

The Chairman:

P. Martorana

P. A. M. Lançon