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D E C I S I O N
of 26 September 2001

Case Number: T 0256/99 - 3.3.5

Application Number: 90308188.3

Publication Number: 0414383

IPC: C04B 35/58

Language of the proceedings: EN

Title of invention:
Wear-resistant member

Patentee:
KABUSHIKI KAISHA TOSHIBA

Opponent:
SAINT-GOBAIN INDUSTRIAL CERAMICS, INC.

Headword:
Wear-resistant member

Relevant legal provisions:
EPC Art. 123(2), 54

Keyword:
"Novelty (yes) - evidence not sufficient to show that the claimed product is the inevitable result of carrying out the teaching of the prior art"

Decisions cited:
-

Catchword:
-



Case Number: T 0256/99 - 3.3.5

D E C I S I O N
of the Technical Board of Appeal 3.3.5
of 26 September 2001

Appellant:
(Proprietor of the patent)

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(Opponent)

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Decision under appeal:

Decision of the Opposition Division of the
European Patent Office posted 27 January 1999
revoking European patent No. 0 414 383 pursuant
to Article 102(1) EPC.

Composition of the Board:

Chairman: R. K. Spangenberg
Members: M. M. Eberhard
M. B. Günzel

Summary of Facts and Submissions

- I. European patent No. 414 383 based on application No. 90 308 188.3 was granted on the basis of 19 claims. Claim 1 reads as follows:

"1. A wear-resistant member comprising ceramic material containing silicon nitride as a main component and a sintering auxiliary component or composition comprising at least 0.1 to 5% by weight of yttrium oxide wherein said ceramic has an amorphous phase mainly consisting of said sintering auxiliary component or composition and said amorphous phase has a segregation size of 100 μm or less."

- II. The respondent (opponent) filed a notice of opposition requiring revocation of the patent on the grounds of lack of novelty and inventive step, insufficiency of disclosure, and extension of the patent beyond the content of the application as filed. The respondent relied inter alia on the following documents during the opposition proceedings:

D1: US-A-4 904 624

D11: DE-A-37 30 618

D14: US-A-4 407 970

D17: Affidavit of Dr Vimal Pujari dated 5 February 1998.

- III. The opposition division decided to revoke the patent. It took the view that granted claim 1 lacked novelty with respect to the disclosure of D14. D14 explicitly disclosed all the features of claim 1 except for the segregation size of 100 μm or less. However, reference example 12 of D14 had a composition as defined in

claim 1 and also met the two minimum conditions for achieving a segregation size of 100 μm or less, namely a porosity of 2% or below before HIP treatment (HIP = Hot Isostatic Pressing) and an amount of yttrium oxide of 0.1 to 5 wt%. Therefore, the product of reference example 12 of D14 fell within the definition of claim 1. This was confirmed by the affidavit D17 in which reference example 12 was reproduced.

- IV. The appellant lodged an appeal against this decision and filed eight sets of amended claims on 27 May 1999 as a main request and seven auxiliary requests. In reply thereto the respondent submitted a second affidavit from Dr Pujari on 4 January 2000, hereinafter D20. Oral proceedings were held on 26 September 2001. The appellant filed additional sets of claims during the oral proceedings and withdrew the previous requests. He finally maintained three sets of amended claims as the main request and two auxiliary requests which are designated "1st request", "2nd request" and "3rd request" respectively.

Claim 1 of the main request differs from granted claim 1 in that the segregation size of "100 μm " has been replaced by "30 μm " and the amount of sintering auxiliary component has been incorporated into the claim by adding the words "2 to 15% by weight of" after "as a main component and". Claim 1 of the "2nd request" differs from claim 1 of the main request only in that the segregation size is "50 μm " instead of 30 μm .

- V. The appellant's arguments can be summarised as follows:

The amendments in claim 1 of the main request were allowable. The segregation size of 30 μm was indicated in Table 1, sample 8, of the original application. Furthermore it was disclosed on page 5 thereof that the

segregation size is preferably 50 μm or less. The skilled reader would have understood that the values of 30 μm and less in Table 1 could characterise the whole invention and not just the formulations explicitly mentioned in Table 1. Associating the value of 30 μm with the whole teaching of the application did not constitute new matter.

Both D14 and affidavit D17 had been submitted at a very late stage of the opposition proceedings. They should not have been admitted into the proceedings. The same applied to affidavit D20. Claim 1 of the "2nd request" was novel with respect to D11 or D14 which nowhere disclosed the segregation size of the amorphous phase. The experiments in D17 and D20 were not a reproduction of reference example 12 and example 19 of D14. The conditions disclosed in D14 had been departed from and this was only for convenience but not for technical reasons. Therefore it had not been shown that the reproduction of reference example 12 or example 19 would inevitably have led to a segregation size of 50 μm or less. The question whether or not a porosity of 2% or below before HIP-treatment and an amount of 0.1 to 5 wt% Y_2O_3 were the minimum requirements to produce the claimed segregation size was not relevant. The burden of proof rested upon the respondent to reproduce the teaching of D14 in order to show that it inevitably resulted in a product having the claimed segregation size. Concerning D11, it was derivable from D20 itself that the batch LM10 was not prepared following exactly the process described in D11. A commercial product and its manufacture usually differed greatly from the disclosure in the corresponding patent. It was not sure that the commercial product was the same as that of D11. Therefore, it had not been proven that a product disclosed in D11 destroyed the novelty of claim 1 of the 2nd request.

VI. The respondent argued that claim 1 of the main request did not meet the requirements of Article 123(2) EPC. The value of 30 μm was disclosed in the original application only for a specific composition. The application as filed contained no broad disclosure showing that this value could be associated with the range 0.1 to 5 wt% Y_2O_3 . Comparison of example 2 and comparative example 1 of the patent showed that the segregation size was very sensitive to the kind and/or composition of the sintering aids. Claim 1 of the 2nd request was not novel with respect to D14. The skilled person would inevitably have produced a wear-resistant member having the segregation size stated in claim 1 when reproducing reference example 12 or example 19 of D14 as shown by affidavits D17 and D20. Slight changes had been made in the experimental procedure when reproducing the said examples. However, it was explained in D17/D20 that none of the modifications caused any substantial difference in the sintered or HIPped density. The explanations were well-founded and convincing. The alterations concerned only parameters which were not said to be important in the patent in suit. Although according to D17 the use of a mixture water/alcohol as the milling medium negatively affected the density, a porosity of less than 2% was obtained for the sintered product. As D14 did not disclose a period of time for the sintering and the HIP treatments, the time had to be chosen by the respondent. Two different times were used for the sintering step, 90 minutes in D20 and 2 hours in D17. A higher moulding pressure was used in the reproduction, however, the use of different moulding pressures in the patent in suit did not seem to have an influence on the segregation size. The reproduction of example 19 produced essentially the same porosity as that reported by D14 before and after HIP-treatment. This clearly showed that the alterations in the reproduction procedure were insubstantial. The minimum requirements

for achieving the segregation size of $\leq 50 \mu\text{m}$ were a porosity of 2% or less before the HIP treatment and a yttrium oxide content of 0.1 to 5 wt%. As these conditions were fulfilled in reference example 12 and example 19 of D14, a segregation size of $50 \mu\text{m}$ or less was automatically obtained. Claim 1 also lacked novelty with respect to D11 since the reproduction of D11 would inevitably have resulted in a product having the claimed segregation size as evidenced by affidavits D17 and D20. The latter showed that the composition of the NT154 material fell within the definition of claim 1 and was prepared according to the procedure disclosed in D11. The thermal treatment at a temperature of 1000 to 1500°C before the HIP-treatment was not mentioned in D20; however, the author of D20 might have thought that a summary of the process of D11 was sufficient.

VII. The appellant requested that the decision under appeal be set aside and that the case be remitted to the opposition division for further prosecution on the basis of the "1st request (main request)" submitted during the oral proceedings. As auxiliary requests, the appellant requested that the case be remitted to the opposition division for further prosecution on the basis of any of the 2nd or 3rd requests submitted during the oral proceedings, taken in their numerical order. The respondent requested that the appeal be dismissed.

Reasons for the Decision

1. The appeal is admissible.

"First request" (main request)

2. Claim 1 does not meet the requirements of Article 123(2)EPC for the following reasons. It is disclosed in the application as filed that the ceramic material contains 0.1 to 5 wt% of yttrium oxide as a sintering aid, that the total amount of sintering aids is in the range of 2 to 15 wt% and that the amorphous phase has a segregation size of 100 μm or less, preferably 50 μm or less: see original claims 1, 2 and 5; page 5, 2nd paragraph; paragraph bridging pages 6 and 7. The range of 30 μm or less for the segregation size is not explicitly disclosed in combination with the amounts of Y_2O_3 and sintering aids stated above. The upper limit of 30 μm is indicated in example 1, sample 8, of the original application (see page 11, Table 1). However, this segregation size is obtained using a specific combination of sintering aids, namely 5 wt% Y_2O_3 , 2 wt% Al_2O_3 and 1 wt% TiO_2 . The examples of the original application which lead to a segregation size of 30 μm or less all contain Al_2O_3 as a second sintering aid (1 or 2 wt%) in addition to Y_2O_3 and at least 1 wt% of a third sintering aid, namely TiO_2 or HfO_2 or a mixture of TiO_2 and AlN (see Table example 1, samples 9 to 12, and Table 2, example 3, samples 5 to 13). Therefore, it derives directly and unambiguously from the examples in which the segregation size is $\leq 30 \mu\text{m}$ that Y_2O_3 is used in combination with a minimum amount of the said additional sintering aids. Furthermore a comparison of example 2 and comparative example 1 of the original application shows that a ceramic material containing Y_2O_3 5 wt%, Al_2O_3 2 wt% and TiO_2 1 wt% as sintering aids gives a segregation size of

100 μm or below whereas a ceramic material prepared under the same conditions but containing no TiO_2 leads to a final product with a segregation size exceeding 100 μm . The application as filed thus shows that the segregation size is strongly influenced by the kind and composition of the sintering aids. It is therefore not directly and unambiguously derivable from the original application that a ceramic material containing silicon nitride as the main component and 2 to 15 wt% of a sintering aid composition comprising 0.1 to 5 wt% Y_2O_3 , ie a composition which may contain Y_2O_3 alone as the sintering aid, has an amorphous phase with a segregation size of 30 μm or less. Therefore claim 1 contravenes Article 123(2) EPC and the main request must fail.

"Second request" (ie first auxiliary request)

3. Claims 1 to 7 of this request fulfil the requirements of Article 123(2) and (3) EPC. Claim 1 is based on a combination of original claims 1, 2 and 5 with the total amount of 2 to 15 wt% of sintering aids disclosed in the application as filed (see paragraph bridging pages 6 and 7). Claims 2, 3 and 4 correspond to original claims 3, 4 and 6 respectively. The subject-matter of claims 5, 6 and 7 is disclosed in original claim 10 and in the original application, page 1, lines 1 to 3; page 12, second paragraph. The scope of protection of the amended claims is restricted compared to that of the granted claims.
4. As objected by the respondent claim 1 does not clearly indicate whether the yttrium oxide content is expressed with respect to the ceramic material or to the sintering aid composition. However this deficiency, ie lack of clarity, already existed in granted claim 1 and lack of clarity is not a ground of opposition. In accordance with the case law of the boards of appeal,

the board considers that in the present case where claims have been amended during the appeal proceedings, the board has in principle the power to deal with the issue of lack of clarity only if it arises out of the amendments introduced in claim 1. The deficiency indicated above clearly does not arise out of the amendments made to the claim. In these circumstances, the objection need not be investigated further than is necessary to enable assessment of the issues already at hand, and claim 1 has to be interpreted on the basis of the description. In the first alternative, ie if the amount of Y_2O_3 were expressed with reference to the ceramic material, then it would vary from 0.1 to 5 wt% and the examples in the description would be illustrative of the claimed subject-matter. In the second alternative, ie if the amount of 0.1 to 5 wt% of Y_2O_3 were referred to the sintering composition, the wear resistant member would then contain 0.002 to 0.75 wt% Y_2O_3 , as pointed out by the respondent. However, in this alternative all the examples of the patent in suit would have Y_2O_3 -contents falling outside the claimed range. The second alternative would thus be contrary to the teaching of the description whereas the first alternative would be in agreement therewith. In these circumstances, the board interprets the content of 0.1 to 5 wt% of Y_2O_3 in claim 1 as being referred to the ceramic material and not to the sintering aid composition.

5. Concerning the introduction of D14 and affidavit D17 into the opposition proceedings, it is observed that the opposition division has a discretion to decide whether or not evidence filed at a late stage of the proceedings should be introduced into the proceedings (Article 114(2) EPC). In the case of a document, relevance thereof is normally taken into consideration in the exercise of this discretion. The ground for the revocation of the patent in the decision under appeal

is lack of novelty in view of D14 completed by the affidavit D17. Therefore there is no doubt that both the disclosure of D14 and the attempt to reproduce one of the examples thereof in D17 were very relevant to the granted claims and had to be introduced into the proceedings.

Concerning affidavit D20 submitted at the appeal stage, this evidence is clearly a direct reply to the amended claims submitted by the appellant with the statement of grounds of appeal and in which the segregation size of 100 μm had been replaced by 30 μm . Therefore, affidavit D20 is not considered as a piece of evidence not submitted in due time.

6. Turning to the novelty issue with respect to D14, this document discloses sintered ceramic bodies which are prepared from starting materials whose compositions lie within the ranges stated in claim 1: see D14, Tables 1 and 2, reference example 12 and example 19. D14 does not disclose that these two bodies contain an amorphous phase having a segregation size of 50 μm or less. As not only the compositions of the starting materials in reference example 12 and example 19 of D14 but also the process for producing the resulting ceramic bodies are different from those exemplified in the patent in suit, no conclusion can be drawn as to whether or not the ceramic bodies of D14 contain an amorphous phase having a segregation size $\leq 50 \mu\text{m}$.

6.1 The respondent's arguments that the reproduction of reference example 12 and example 19 inevitably resulted in a ceramic body having an amorphous phase with a segregation size $\leq 50 \mu\text{m}$ are not convincing for the following reasons. D14 does not disclose all the operating conditions of the sintering step and of the subsequent HIP-treatment. Only the temperature and the pressure are reported but not the sintering time and

the hiping time. Therefore, the respondent selected a sintering time of 2 hours and a hiping time of 1 hour for the reproduction of reference example 12 and a sintering time of 90 minutes with a hiping time of 2 hours for example 19 which concerns a different composition (see D17 and D20). Sintering times and hiping times shorter or longer than the specific values chosen by the respondent would however be considered as possible by the skilled person reading D14 and are not at all excluded by the technical teaching of D14. Therefore, in order to prove that the reproduction of D14 inevitably results in the claimed segregation size the respondent should have shown that by selecting other sintering and hiping times, in particular shorter times than those selected in D17 and D20, a segregation size $\leq 50 \mu\text{m}$ would still be obtained. The respondent has, however, not provided such evidence. Furthermore in the experiments of D17 and D20 the respondent chose a temperature of 1800°C for the HIP-treatment, ie a temperature higher than in D14 (1750°C), and a moulding pressure which is about twice as high as in D14 (1360 atm instead of $700\text{Kg}/\text{cm}^2$). It is argued in D17 (point 5d) that the change in the hiping temperature would not affect the sintered density. According to D20 (point 16) the change in the cold isostatic pressure is insubstantial enough not to affect the final result in respect of segregation size. This might be true for the combination of parameters selected by the respondent; however, this does not demonstrate that the combination of shorter sintering and hiping times with a lower hiping temperature of 1750°C and a considerably lower moulding pressure would still lead to a ceramic body with a segregation size $\leq 50 \mu\text{m}$. According to page 4, lines 50 to 51, of the patent in suit the segregation size varies with the moulding, sintering and HIP conditions and Table 1 shows that the simultaneous variation of the moulding

pressure, sintering pressure, sintering temperature, sintering time, HIP-temperature and HIP-time may lead to segregation sizes smaller or higher than 50 μm for the same composition of sintering aids, all other conditions remaining identical. In view of this teaching, the board is not convinced on the basis of the evidence on file that the reproduction of reference example 12 and example 19 of D14 inevitably results in a product having a segregation size of 50 μm or less. In these circumstances, the question can be left open whether or not the respondent's experiments can be considered as a reproduction of example 19 and reference example 12 of D14 despite the numerous other changes in the attempt to reproduce these examples (e.g. the changes in the starting silicon nitride powder, milling medium, milling time, kind and amount of binder, and the spray-drying step).

- 6.2 The respondent further argued that the minimum requirements for achieving the segregation size of $\leq 50 \mu\text{m}$ were a porosity of 2% or less before the HIP-treatment and a Y_2O_3 content of 0.1 to 5 wt%. He contended that as these conditions were fulfilled in reference example 12 and example 19 of D14, a segregation size $\leq 50 \mu\text{m}$ was automatically obtained therein. These arguments cannot be accepted for the following reasons. The respondent has provided no evidence to support his affirmation concerning the said minimum requirements. The appellant's representative indicated at the oral proceedings that he did not know whether or not these two conditions were sufficient to obtain a segregation size $\leq 50 \mu\text{m}$. Furthermore, Table 1 of the patent in suit shows that for a composition containing 5 wt% Y_2O_3 , 2 wt% Al_2O_3 , and 1 wt% TiO_2 (ie a Y_2O_3 content lying within the range 0.1 to 5 wt%) the segregation size may be higher than 50 μm even if the porosity before HIP is 2% (see example 1, sample 1).

Therefore it derives from this example that fulfilment of the two said conditions does not inevitably result in an amorphous phase with a segregation size $\leq 50 \mu\text{m}$.

7. Concerning the novelty of claim 1 with respect to D11, it is observed that D11 does not disclose the segregation size of the amorphous phase. According to the affidavits D17 and D20, the material described in D1/D11 was the product "NT154" of the Norton Company and the sample LM10 prepared in 1988 and analysed later contained an amorphous phase having a segregation size not exceeding $50 \mu\text{m}$. It is further indicated in affidavit D20 that the batch LM10 was prepared as it is substantially described in D1/D11. The process used for the preparation of sample LM10 is then reported in point 13 of D20. It is inter alia stated that "the screened powder was cold isostatically pressed at 20 ksi and subsequently densified using glass encapsulation HIP technology at 1800°C for 1 hour". A comparison of the process described in point 13 with the process disclosed in D11 shows that one essential feature of the process of D11, namely the heat treatment at a temperature between 1000°C and 1500°C in a nitrogen atmosphere which is carried out before the HIP-treatment, is not mentioned in point 13. According to D11 this heat treatment is carried out for a time sufficient to reduce the amount of silicium oxide to less than 1% and the amount of the $\text{Y}_2\text{Si}_2\text{O}_7$ -phase to a value below the X-ray diffraction detection limit, this time being in general from 20 to 60 minutes (see claims 6 to 8; page 4, lines 5 to 13 and 47 to 49; page 5, Table 1). Taking into account that the said heat treatment is an essential feature of D11 which has an influence on the silica content and the phases present in the final product, the board is not convinced that the sample LM10 which was analysed by the respondent is a product according to D11. The respondent has provided no evidence showing that the

process of D11 including this heat treatment results in products having the claimed segregation size of 50 μm or less. The respondent's argument that the process reported in D20 was likely only to be a summary of the process of D11 is not convincing since one would expect an essential feature of a patent to be mentioned even in a summary thereof. Therefore, on the basis of the evidence provided by the respondent the board is not convinced that the reproduction of the teaching of D11 would inevitably result in a product having the features stated in claim 1.

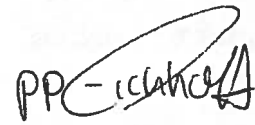
8. It follows from the above that, on the basis of the evidence on file, the board cannot consider that the subject-matter of claim 1 of the "2nd request" lacks novelty over the disclosure of D14 or D11.
9. The sole issue dealt with in the appealed decision is novelty. The issue of sufficiency of disclosure was in particular left open by the opposition division. The appellant requested at the oral proceedings that the case be remitted to the opposition division for further prosecution and the respondent did not object thereto. The board, in the exercise of its discretionary power pursuant to Article 111(1) EPC, finds it appropriate to remit the case to the opposition division for further prosecution.

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 for further prosecution on the basis of the "2nd request" filed during the oral proceedings before the board.

The Registrar:


U. Bultmann



The Chairman:


R. Spangenberg

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