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D E C I S I O N
of 25 August 1999

Case Number: T 0262/96 - 3.3.5

Application Number: 87300826.2

Publication Number: 0235936

IPC: C04B 35/48

Language of the proceedings: EN

Title of invention:
Magnesia partially-stabilized Zirconia

Patentee:
Didier-Werke AG

Opponent:
Cerasiv GmbH Innovatives Keramik-Engineering

Headword:
Mg-PSZ/DIDIER

Relevant legal provisions:
EPC Art. 54, 56

Keyword:
"Novelty (yes) - commercially available products not shown to have the claimed combination of features"
"Inventive step (yes) - teaching leading away from the claimed solution"

Decisions cited:
-

Catchword:
-



Case Number: T 0262/96 - 3.3.5

D E C I S I O N
of the Technical Board of Appeal 3.3.5
of 25 August 1999

Appellant:
(Opponent)

Cerasiv GmbH
Innovatives Keramik-Engineering
Postfach 11 49
73201 Plöchingen (DE)

Representative:

Uppena, Franz, Dr.
Dynamit Nobel AG
Patentabteilung
53839 Troisdorf (DE)

Respondent:
(Proprietor of the patent)

Didier-Werke
Lessingstrasse 16-18
65186 Wiesbaden (DE)

Representative:

Kyle, Diana
Elkington and Fife
Prospect House
8 Pembroke Road
Sevenoaks
Kent TN13 1XR (GB)

Decision under appeal:

Decision of the Opposition Division of the
European Patent Office posted 12 January 1996
rejecting the opposition filed against European
patent No. 0 235 936 pursuant to Article 102(2)
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. 0 235 936 based on application No. 87 300 826.2 was granted on the basis of 15 claims. Granted claim 1 reads as follows:

"1. A partially stabilized zirconia body formed of a molded uncalcined mixture of magnesia powder and zirconia powder containing silica, said body consisting of crystalline zirconia, silica and magnesia, the silica being 0.05-0.5% by weight of the body and the magnesia being 2.4-4.0% by weight of the body, and incidental constituents and impurities optionally including less than 0.05% by weight of oxides which form an insoluble zirconate that does not combine with magnesia;

said body having a crystalline microstructure consisting of (1) grains of cubic stabilized zirconia comprising zirconia and a major portion of the magnesia, (2) discrete precipitates of tetragonal zirconia within the cubic grains, (3) discrete precipitates of monoclinic zirconia within the cubic grains, and (4) monoclinic zirconia at the boundaries of the cubic grains, each of said tetragonal and monoclinic precipitates having a substantially ellipsoidal shape with a long dimension of 0.1-0.4 micrometres; and the partially stabilized zirconia material of the body having:

(a) a microstructure wherein the the monoclinic zirconia content is 10-20 volume percent determined on a polished surface, a thermal expansion coefficient over the range of 25-1000°C of $(65-105) \times 10^{-7}/^{\circ}\text{C}$, and a modulus of rupture of at least about 350 MPa, measured at 25°C, after exposure to thermal shock; or

(b) a microstructure wherein the monoclinic zirconia content is 20-60 volume percent determined on a polished surface, a thermal expansion coefficient over the range of 25-1000°C of $(10-65) \times 10^{-7}/^{\circ}\text{C}$, and a modulus of rupture of at least about 250 MPa, measured at 25°C, after exposure to thermal shock."

II. The Appellant (Opponent) filed a notice of opposition requesting revocation of the patent on the grounds of lack of novelty and lack of inventive step. The Appellant's arguments in support of these grounds were in particular based on a public prior use of two ceramic materials manufactured by the firm Feldmühle. It was alleged that these materials had been put on the market under the names ZN40 and ZN50 before the priority date of the patent in suit. The Appellant relied on the following documents:

D1: WO-83/04247

D2: EP-A-0 013 599

D3: Advances in Ceramics, vol. 12, Science and Technology of Zirconia II, 1984, pages 480-487

D4: NASA CR-174943, 1985

D5: DE-C-23 07 666

D6: Internes Datenblatt ZN40

D7: Advances in ceramics, vol. 3, Science and Technology of zirconia, 1981, pages 98-115

D8: J. Am. Ceram. Soc., vol. 69, No. 7, July 1986, pages 578-579

D9: "Konkurrenzuntersuchung vom 22.11.1985"

D10: J. of Mat. Science, vol. 19, 1984, pages 2934-2942

D11: J. Am. Ceram. Soc. 68 (4), 1985, pages 197-202.

III. The Opposition Division rejected the opposition. In its decision it held that the subject-matter of claim 1 was not anticipated by the alleged prior use of the products ZN40 and ZN50. The existence at the priority date of a magnesia partially-stabilized zirconia (Mg-PSZ) body as defined in claim 1 could not be unambiguously derived from a combination of pieces of information disclosed in the cited documents. D1 and D2 showed that a prejudice existed against the use of zirconia powders with a high silica content for providing a Mg-PSZ body exhibiting the desired properties without introducing special additives or further purifying the batch. D4, D6, D9 and D11 did not suggest that the use of zirconia with a silica content between 0.05 and 0.5 wt% would have led to products having the claimed structure and properties even in the absence of suitable additives.

IV. The Appellant lodged an appeal against this decision. Oral proceedings were held on 25 August 1999. At the appeal stage, the Appellant put forward the following arguments:

Mg-PSZ ceramics were commercially available under the name ZN40 and ZN50 a long time before the priority date. The requirements in point 1.4 of decision G 1/92 were met. The skilled person relying on his general technical knowledge would have been able to discover the composition and internal structure of these products without undue burden. The results would have been the information indicated in D3, D4, D6, D8 and D9. These documents showed that the ZN40 and ZN50 products exhibited practically the same properties, compositions and internal structures as the claimed

bodies. The silica content in D4 was 0.043 wt%. As this value was measured on the sintered body, it was lower than the actual value. The difference of only 0.007 wt% with respect to the claimed lower limit was therefore not significant. The explanation for the inconsistent silica contents in D4 und D6 was simple. The Appellant had realised that the artificially constructed prejudice in D1 and D2 did not exist in reality and that it did not depend on the silica content. Thus, he had used commercially available zirconias containing sometimes more and sometimes less silica for preparing ZN40 products without introducing additives. The small numerical difference between the monoclinic zirconia content in ZN40 and in embodiment (a) of claim 1 was merely a formal difference which was not sufficient to provide novelty since the bodies were characterised by a monoclinic zirconia content of **about** 10 to 20 vol.% in the patent in suit. Furthermore, the method of measurement of this parameter was hardly quantitatively reproducible taking into account the fact that the result depended, in a critical manner, on the way the body surface was polished. The ZN40 materials having the compositions indicated in D4 and D9 had a microstructure identical to that disclosed in D3 or in claim 1 of D2. The designation Mg-PSZ itself implied that their microstructure was quantitatively the same as in D2 and thus fell within the definition of claim 1. D7, Figure 8, also disclosed the claimed microstructure. The modulus of rupture (MOR) and thermal expansion coefficient of the ZN40 materials disclosed in D4 and D9 fell within the claimed ranges as shown by D3.

Concerning the commercial ZN50 ceramic, the difference between the thermal expansion coefficients was so small that novelty of the product according to embodiment (b) of claim 1 could not be acknowledged on this basis. Furthermore, the ZN50 material also destroyed the

novelty of embodiment (a) of claim 1 since the monoclinic zirconia content of 20%, the thermal expansion coefficient of about $7 \times 10^{-6}/K$ and the MOR stated in D8 all fell within the claimed ranges. It could be derived from D4, D3 and D8 that the products ZN40 and ZN50 were sold before the priority date.

The closest prior art was not D2 but the commercially available ceramic ZN40/ZN50. It was permissible to consider a commercially available product as closest prior art. The ZN40 ceramic disclosed in D4 had more features in common with the claimed product than the Mg-PSZ of D2. The alleged prejudice based on D1 and D2 had already been overcome by the Appellant with the commercial products ZN40 and ZN50 so that no inventive step could be derived from said prejudice. These products showed that Mg-PSZ ceramics exhibiting the required properties were also obtainable with zirconia having higher silica contents than in D2 without introducing special additives. The skilled person would have tried the thermal ageing disclosed in D3 in order to obtain another product.

V. The Respondent submitted the following arguments:

No information was provided to assist in determining (i) the date of the alleged use, (ii) what had been used and (iii) all the circumstances of the alleged use. In fact the information relating to ZN40 in the Table presented in the statement of grounds of appeal was not that of a single material for which a prior use was alleged but rather a combination of selected information from five documents. Of these documents D6, D8 and D9 did not form part of the state of the art. Furthermore, the documents contained no cross-reference and were mutually inconsistent with regard both to the chemical analysis and the properties of the materials ZN40 and ZN50. D4 and D6 showed that the amount of

silica in ZN40 could vary but did not disclose the physical properties of the resulting products and it could not be assumed that the different compositions led to the same physical properties. D3 and D8 disclosed MOR values but not the silica content.

D2 represented the closest prior art. The problem stated in the patent in suit was not an artificial one since according to D2 bodies prepared from a mixture having a high content of silica were badly cracked. None of the references D3 to D9 disclosed how a product having excellent properties could be made with zirconia containing relatively high levels of silica.

- VI. The Appellant requested that the decision of the Opposition Division be set aside and that the patent be revoked. The Respondent requested that the appeal be dismissed and the patent maintained as granted.

Reasons for the Decision

1. The appeal is admissible.
2. Regarding the issue of novelty, the Appellant contended that the ZN40 and ZN50 materials were commercially available before the priority date and that these products had the composition, microstructure and properties indicated in claim 1 of the patent in suit.
 - 2.1 It is assumed to the Appellant's benefit that the material ZN40 referred to in D4 as well as the ZN40 and ZN50 materials described in D3/D8 were available to the public before the priority date.

- 2.2 The question arises whether or not the ZN40 referred to in D4 and the ZN40 and ZN50 materials of D3/D8 possessed the combination of features stated in claim 1.

D4 discloses the spectrographic analysis of three Mg-PSZ materials which were provided by different ceramic producers and investigated at a Research Institute in Chicago. D4 shows that the analysed sample of the Mg-PSZ material designated "Feldmühle ZN-40" contains 1.85 wt% Mg and 0.02 wt% Si, which corresponds respectively to 3.07 wt% MgO as indicated in D4 and 0.043 wt% SiO₂ as calculated by the Appellant. The silica content of this sample of ZN40 material is thus lower than the lower limit of 0.05 wt% stated in claim 1. The Appellant's argument that the difference between the said numerical values was only 0.007 and, thus, not significant, is not convincing. As the silica content of this ZN40 material is itself relatively low, this difference represents in fact 16%. A difference of 16% in the silica content is sufficient to distinguish two products from each other if such low silica contents can be determined with sufficient accuracy by the used method of measurement. The Appellant did not provide information about the standard deviation or the degree of accuracy of the used method. Instead, he argued at the oral proceedings that the value of 0.043 wt% was in fact lower than the **actual** value since the analysis was effected on the sintered body and such an analysis was more problematic than an analysis performed on the starting powder because of the additional components formed during sintering. In the Board's view the fact that an analysis might be more difficult on the sintered product does not mean that the result of the analysis is necessarily too low. Furthermore, the Appellant's affirmation that the silica content measured in the sintered Mg-PSZ was lower than the actual value was not supported by

evidence and it was contested by the Respondent. If it were assumed for the sake of argument that the value given in D4 is too low, then the Appellant would still have had to prove that the **actual** value lies within the claimed range of 0.05 to 0.5 wt%. Evidence to this effect was not provided by the Appellant although the burden of proof rests with him. In these circumstances, the Board considers that, in the absence of evidence to the contrary, the silica content disclosed in D4 lies outside the claimed range. Furthermore, D4 is silent about the properties and microstructure of the ZN40 material having the specific composition disclosed therein.

As the silica content of the ZN40 material referred to in D4 lies outside the claimed range, even the combination of information given in D4 with the properties and microstructure of the ZN40 material indicated in D3, D8 or in other documents disclosing the microstructure of Mg-PSZ ceramics would not destroy the novelty of the claimed ceramic bodies.

- 2.3 The Appellant further relied on D6 and D9 to show that the ZN40 or ZN50 material may have a silica content falling within the claimed range of 0.05 to 0.5 wt%. D6 gives the analysis of several samples of starting powder mixtures suitable for preparing the ZN40 material. These analyses were carried out between October 1978 and August 1980. The silica content of the starting powders varies from 0.14 to 0.225 wt% and thus lies within the claimed range. However, D6 is an internal sheet, allegedly from the firm Feldmühle (the Appellant's legal predecessor), and it remains unclear whether the corresponding sintered materials were put on the market before the priority date. The Appellant has provided no evidence that ZN40 materials with these higher silica contents were sold or made available to the public in another way before the said date.

D9 refers to a ZN40 material containing 3.3 wt% MgO and 0.18 wt% silica, ie a MgO and silica content both falling within the claimed ranges. However, D9 is an internal report, allegedly also from Feldmühle, which is marked strictly confidential, and as in the case of D6, the Appellant has provided no evidence that a ZN40 material having the composition, monoclinic zirconia content and thermal expansion coefficient stated in D9 had been sold or made available to the public in any other way before the priority date.

Therefore, ZN40 materials having the compositions indicated in D6 or D9 are considered not to form part of the state of the art.

- 2.4 D3 refers to ZN40 and ZN50 ceramics and discloses that ZN40 is a Mg-PSZ ceramic having a thermal expansion coefficient of $9.8 \times 10^{-6}/K$ (RT {room temperature}-1000°C), ie a value lying within the claimed range for embodiment (a). This material has a MOR in flexure of 500 MPa (see page 482, Table II). The MOR after thermal shock (ie thermal shock resistance) and the content of monoclinic zirconia are not stated in D3. Furthermore, D3 does not disclose the composition of the Mg-PSZ body on which the thermal expansion coefficient, toughness and MOR were determined. The Appellant has provided no evidence showing that the data disclosed in D3 relate to a ZN40 material containing 0.05 to 0.5 wt% silica and less than 0.05% of the oxides as defined in claim 1 (hereinafter referred to as "additives"). As indicated above, the ZN40 material of D4 has a lower silica content, and even if ZN40 materials with a silica content of 0.05 to 0.5 wt% were prepared by the Appellant's predecessor before the priority date, it has not been proved that they were commercially available before the said date (see point 2.3 above).

- 2.5 D8, which was published after the priority date but whose text was approved before this date, discloses the same values of MOR, toughness and thermal expansion coefficient as in D3. The MgO content is said to be 3.2%. The thermal shock resistance is not given. D8, like D3, is silent about the possible presence and amounts of the other components of this Mg-PSZ ceramic, in particular the amount of silica and other impurities or "additives". Therefore, the Board's considerations in connection with the products described in D3 apply likewise to the ZN40 material of D8.

Furthermore, according to D8 the content of monoclinic zirconia in ZN40 is < 5%. Assuming to the Appellant's benefit that the monoclinic content of less than 5% was determined on a polished surface, then it is well below the claimed lower limit of 10 vol.% stated in claim 1 for embodiment (a). The Appellant's submission that this lower limit has to be construed as meaning **about** 10 vol.% since the bodies are characterised by a monoclinic zirconia content of about 10 to 20 vol.% on page 5 of the patent in suit cannot be accepted by the Board. The product according to the invention is defined on page 3, lines 11 to 36, of the patent in suit and the definition given in this passage is in agreement with the wording of claim 1, ie the word "about" is not used to characterise the monoclinic zirconia content in line 24 and in line 32 where this characterisation is repeated. In these circumstances, the mere fact that the word "about" is used on page 5, line 18, is not sufficient to justify an interpretation of claim 1 which would not be in agreement with the general definition of the invention on page 3 of the patent in suit and with the wording of claim 1. The Appellant further objected that the method of measurement of the monoclinic zirconia content led to results which were hardly reproducible. Although the monoclinic zirconia content depends on the conditions

used for polishing the surface, the Appellant has not shown that the method of measurement is so inaccurate that even in the case of identical polishing conditions it leads to meaningless values. The Board observes in this respect that not only the Respondent but also other ceramic producers such as the firm Feldmühle (the Appellant's predecessor) also use this parameter to characterise a Mg-PSZ.

2.6 Concerning the Appellant's arguments that the microstructure of the Mg-PSZ materials was already known, the Board observes that D7 (published in 1981, ie before D3/D8) indeed discloses that commercial Mg-PSZ materials contain cubic zirconia matrix grains, very fine intergranular tetragonal particles, and monoclinic zirconia particles with two morphologies, namely large, coarsely twinned grain boundaries and finer but still twinned intergranular precipitates. According to D7, the critical particle size at which particles are still tetragonal on cooling to room temperature, is $\approx 0.2 \mu\text{m}$ (see page 100, last paragraph; page 101, third paragraph). However, D7 does not refer to the ZN40 material and the content of monoclinic zirconia is not disclosed in this document. No information can be drawn from Figure 8 in this respect. Furthermore, D8 and D2 show that the content of monoclinic zirconia may lie outside the claimed range. Therefore, the Appellant's allegation that the microstructure of the ZN40 material of D3/D8 is also quantitatively the same as the claimed one is not convincing.

2.7 It follows from the above that the designation ZN40 covers materials having different compositions, in particular different silica contents which may fall outside or inside the claimed range. However, those products with a silica content lying inside this range were not shown to have been made available to the

public before the priority date. The samples of ZN40 material according to D3/D8, which were assumed to be commercially available before the said date but whose silica and "additives" contents are not indicated in D3/D8, were not shown to have silica and "additives" contents as well as a thermal shock resistance and monoclinic zirconia content falling within the claimed ranges. Therefore, the Appellant, who carries the burden of proof, has not provided evidence from which it could be directly and unambiguously derived that the samples of ZN40 material made available to the public before the priority date have silica and "additives" contents lying within the claimed ranges and possess the combination of characteristics stated in claim 1.

The Board observes that decision G 1/92 (OJ EPO 1993, 277) relied upon by the Appellant, does not address the question of whether or not it would be permissible to combine pieces of information relating to different materials covered by the same designation in order to prove that the subject-matter of a claim lacks novelty. Rather, the said decision deals with the question whether or not the chemical composition of a product which is available to the public forms part of the state of the art if the product can be analysed and reproduced by the skilled person. This question, however, is not relevant here.

- 2.8 The fact that the ZN40 material might have been prepared by the process of D5 cannot change the preceding conclusion since D5, which contains no reference to the material ZN40, does not mention the silica content of the ceramic bodies or starting mixtures, the monoclinic zirconia content of the sintered bodies, their microstructure, their resistance to thermal shock and their thermal expansion coefficient.

- 2.9 The Appellant further argued that the commercially available ZN50 product destroyed the novelty of both embodiments (a) and (b) of the claimed bodies.

Only D3 and D8 refer to the ZN50 product. According to D8 the material ZN50 is a Mg-PSZ ceramic having the same composition as ZN40. The sole information derivable from D3/D8 as regards the composition of the commercially available ZN50 material is therefore that it contains 3.2% MgO. As pointed out above, the silica content of this material is not mentioned in these documents, and it is unclear whether or not this material contains "additives". If it were assumed to the Appellant's benefit that the thermal expansion coefficient of $\approx 7.0 \times 10^{-6}/K$ (RT-1000°C) concerns a ZN50 material containing 20% of monoclinic zirconia, then both values would fall within the ranges defined in claim 1 for embodiment (a). However, it is not directly and unambiguously derivable from D3/D8 that the thermal expansion coefficient and the monoclinic zirconia content were determined on a ceramic body having a silica content of 0.05 to 0.5 wt% and containing less than 0.05 wt% of "additives". Furthermore, the Appellant has provided no evidence that a ZN50 material having the said silica and "additives" contents had been made available to the public before the priority date, let alone that such a material exhibited the claimed combination of features. Therefore, the Board considers in the absence of evidence to the contrary that the commercially available ZN50 referred to in D3/D8 does not destroy the novelty of embodiment (a) of claim 1. The preceding reasons in connection with the silica and "additives" contents of the ZN50 material apply likewise to embodiment (b) of claim 1.

- 2.10 It follows from the above that the subject-matter of claim 1 is new with respect to the ZN40 and ZN50 materials. It is also novel over the disclosure of the remaining documents which were published before the priority date. This was not disputed so that further considerations in this respect are not necessary.
3. Concerning the issue of inventive step, the Respondent considered at the oral proceedings that D2 represented the closest prior art, whereas, in the Appellant's view, the commercially available ZN40 material was the closest state of the art.
- 3.1 As pointed out by the Appellant a product which was available to the public before the priority date can be considered as the closest state of the art. It results from the preceding considerations that the commercially available ZN40 product referred to in D4 has a silica content lower than 0.05 wt%. The Appellant argued that the physical properties stated in D3/D8 were the same for all the ZN40 materials independently of their silica content and, thus, that the ZN40 material of D4 would also exhibit these properties. However, this allegation was supported by no evidence and was contested by the Respondent. In these circumstances, the burden of proof rests with the party who made this allegation and the Board cannot accept the Appellant's unproved affirmation. In the absence of evidence that the properties stated in D3/D8 were measured on a ceramic body having the composition indicated in D4, the Board considers that the resistance to thermal shock, thermal expansion coefficient and content of monoclinic zirconia of this product are not known. Regarding the commercially available ZN40 material having the properties stated in D3/D8, the silica and "additives" contents of the body on which these properties was determined are also considered as unknown in the absence of convincing evidence in this

respect. Furthermore, the monoclinic zirconia content is well below the claimed range (see points 2.2 to 2.8 above). Concerning the Mg-PSZ products of D2, their silica content is also lower than 0.05 wt%; however, this document further discloses the thermal shock resistance of the products, which falls within the claimed range. D2 also describes in detail the microstructure of the ceramic bodies, and the monoclinic zirconia content of the polished bodies lies within the claimed range. In addition, the problem of obtaining both high strength and good thermal shock resistance properties is discussed in detail in D2, which gives a detailed description of the preparation process. For these reasons, the Board considers that D2 is closer to the claimed subject-matter than the commercially available ZN40 material of D4 or D3/D8.

- 3.2 The Mg-PSZ material disclosed in D2 has a magnesia content from about 2.8 to 4.0 wt% and is prepared from a zirconia powder containing no more than about 0.03 wt% silica. This material has a microstructure comprising grains of cubic stabilised zirconia, each grain containing discrete precipitates of tetragonal zirconia having an elliptical shape and a mean length of about 1500 Å, and discrete precipitates of microcrystalline monoclinic zirconia. Monoclinic zirconia is present at the boundaries of the cubic grains in an amount of about 8 to 15% of the material. The material comprises from 0.5 to 20 wt% of the said monoclinic zirconia precipitates. The amount of monoclinic phase present in a polished surface is for example about 12.5% for an ageing time of 8 hours and this amount increases linearly for higher ageing times up to 16 hours. This ceramic material combines both high strength and good thermal shock resistance

properties: see claims 1 and 2; page 4, lines 1 to 12; page 9, lines 7 to 19; page 18, lines 12 to 17; page 21, lines 22 to 25; page 23, lines 22 to 26; whole page 27; page 28, Table 1; Figures 1, 2, 4 and 8.

The requirement concerning the purity of the zirconia powder in D2 can present practical disadvantages in that the more readily available commercial zirconia powders generally do not meet this purity requirement. The technical problem to be solved with regard to D2 can be seen in the provision of other Mg-PSZ bodies which can be prepared from readily available zirconia powders and which have a high strength and toughness while exhibiting a good thermal shock resistance.

It is proposed that this problem be solved by the Mg-PSZ bodies having the combination of characteristics stated in claim 1. These bodies differ from those of D2 at least by their silica content. In view of the examples of the patent in suit and of the physical properties and thermal shock resistance indicated on page 5 thereof, it is credible that the problem stated above has actually been solved by the claimed Mg-PSZ bodies. This was not disputed by the Appellant.

- 3.3 Concerning the silica content of the ceramic material, D2 teaches that it is necessary to use zirconia with a very low silica content (no more than about 0.03 wt%) as the starting material in the method of manufacture disclosed in D2. The use of Harshaw Electronic Grade zirconia, which contains from 0.05 to 0.1 wt% silica, was found to lead to a ceramic material having a substantially lower strength than the material according to D2 (see page 8, lines 16 to 23). D2 further discloses comparative examples illustrating the use of zirconia containing between 0.05 to 0.10 wt% silica instead of < 0.03 wt%. It is stated that the resulting sintered material, in common with other

materials prepared in accordance with the invention of D2 but using zirconia containing more than about 0.03 wt% silica, was badly cracked after the firing and/or ageing steps (see page 28, line 17 to page 29, line 9).

D1 discloses Mg-PSZ ceramics with excellent thermal shock resistance which have the microstructure of the material described in US-A-4 279 655 (hereinafter D2a) except for the amount of intragranular monoclinic zirconia. D2a is the US patent corresponding to D2. Regarding the composition, D1 requires that the Mg-PSZ material contains from 0.05 to 1.00 wt% of a special additive. The latter is a metal oxide which forms an insoluble zirconate that does not combine with magnesia and which may form a glass with silica. The additive is preferably strontia or baria or a rare earth oxide or a mixture thereof (see page 3, lines 2 to 25; claim 1). D1 teaches that the silica content of the new materials is not as critical as in the ceramic materials of D2a and that up to 1.0 wt% of silica is acceptable in the final products, although lower silica contents are preferred (see page 4, lines 8 to 13).

The skilled person seeking a solution to the technical problem stated above would have tried to prepare Mg-PSZ bodies using zirconias which are more readily available than the low content zirconia required in D2, for example zirconias with a silica content of 0.05 to 0.1 wt% mentioned on page 8 of D2. However, in view of the teaching of both D2 and D1, the skilled person would have expected that the desired strength and thermal shock resistance cannot be obtained without introducing at least 0.05 wt% of the additives required in D1 or other additives which might compensate the negative effect of silica illustrated in D2 (see pages 28 to 29). Therefore, the skilled person would have been led away from using such zirconia without

introducing at least 0.05 wt% of the said additives into the composition to scavenge the silica.

Furthermore, neither D1 nor D2 suggests how ceramic bodies containing 0.05 to 0.5 wt% silica and no additives of the kind described in D1 (or < 0.05 wt% thereof) should be prepared in order to obtain bodies having both high strength and good thermal shock properties.

- 3.4 The commercially available product ZN40 having the composition stated in D4² contains none of the specific additives required in D1 (ie strontia, baria or rare earth metal oxides). However, its silica content is also lower than the lower limit of 0.05 wt% indicated in claim 1 and the Appellant has not proved that the thermal expansion coefficient, monoclinic zirconia content and other properties disclosed in D3/D8 relate to a ZN40 material with the composition stated in D4. In the absence of information in this respect, it cannot be ascertained whether or not the said product exhibits both a high strength and a good thermal shock resistance. On the basis of the available data, the Board considers that the composition given in D4 and the information that the product is a Mg-PSZ material cannot suggest to the skilled person, who is aware of the teaching of D1 and D2, that a ceramic material containing more silica than indicated in D4 and none of the additives specified in D1 (or <0.05 wt%) would be able to solve the technical problem stated above.

Concerning the commercially available ZN40 and ZN50 Mg-PSZ materials referred to in D3/D8, the composition of these products is not indicated in D3/D8. As pointed out in items 2.4 and 2.5 above, no evidence was provided by the Appellant that their silica content lies within the claimed range. It is also unclear, in the absence of evidence concerning their composition, whether or not they contain special additives. In these

circumstances, the Board can only consider that the silica content of these products and the presence or absence of additives are unknown. Therefore, these products cannot give any pointers towards the claimed solution of the relevant technical problem.

- 3.5 With regard to the documents which were published before the priority date, the Board observes the following. D3 discloses that ZN40 has a MOR of 500 MPa and can withstand severe thermal shock conditions. However, as the composition of the ZN40/ZN50 material is not indicated in D3 the considerations in the preceding paragraph apply likewise to the disclosure of this document. Assuming to the Appellant's benefit that D4 was a document made available to the public, then the reasons given above in point 3.4 also apply to this document since it contains no information about the ZN40 product other than those already considered above. D5 is silent about the silica content of the ceramic bodies and their thermal shock resistance. D7 describes the microstructure of the Mg-PSZ ceramics **qualitatively** and mentions the dimension of the tetragonal precipitates but it gives no information about the silica content of these products and it does not disclose any value of both the strength and thermal shock resistance of the Mg-PSZ ceramics. D10 is a study of the microstructure evolution and ordering in a sintered commercial Mg-PSZ material which was heat treated at different temperatures. The forsterite which was found in the samples after heat treatment at 1400°C is said to be a product of silica impurities reacted at high temperature with MgO-rich regions. It is not indicated whether or not special additives were present and D10 is silent about the strength and thermal shock properties of the studied material. D11 discloses ceramic bodies containing about 0,2% silica, ie a value within the claimed range. However, these bodies are not Mg-PSZ having a magnesia content as stated in claim 1,

but ternary (Ca, Mg)-PSZ bodies containing 3.2 wt% CaO and 1.1 wt% MgO. The strength and resistance to thermal shock of these bodies is neither mentioned nor discussed. Therefore, the teaching of these documents taken in combination with the teaching of D1 and D2 would not give the skilled person an incentive to use zirconias with a silica amount of at least 0.05 wt% without introducing at least 0.05 wt% of the additives required in D1 in order to obtain Mg-PSZ bodies having a good thermal shock resistance while still exhibiting high strength and toughness.

- 3.6 The Appellant argued that an artificial prejudice was constructed in D1 and D2 and that the prejudice, if existing, had been overcome by the commercial products ZN40/ZN50. In the Board's view, the question whether or not D1 and D2 are sufficient to demonstrate the existence of a prejudice before the priority date can remain open since it is not decisive in the present case. D1 and D2 teach the negative effect of silica on the properties of the Mg-PSZ ceramics prepared according to D2 and the possibility of compensating this effect by the use of additives disclosed in D1. This teaching is supported by examples on pages 28 to 29 of D2. The Board has no reason to doubt the veracity of the teaching of D1/D2, all the more so in that one of the authors was acknowledged by both parties to be a leading expert in the field. As indicated above, the skilled person faced with the problem stated above would, in view of this teaching, have been led away from using zirconias with silica contents of at least 0.05 wt% without introducing silica-compensating additives. The commercially available ZN40 and ZN50 materials cannot be considered to point in an opposite direction to that of D1/D2 since they contain either less than 0.05 wt% silica (D4) or it was not proved that their silica content is at least 0.05 wt% (D3/D8).

3.7 As set out in point 3.1 above, the product described in D4 does not represent the closest prior art. However, even if this product were taken as starting point for the assessment of the inventive step, the outcome of the decision would be the same for the following reasons. The ZN40 material referred to in D4 is characterised therein only by its composition and by the fact that it is a Mg-PSZ ceramic. In the absence of evidence that the monoclinic zirconia content and properties stated in D3/D8 concern a ZN40 material having the composition stated in D4, the monoclinic content and properties of this material are considered to be unknown (see points 3.1 and 3.4 above). Starting from this product, the technical problem would be the same as that defined in point 3.2 above. The solution as defined in claim 1 differs from the said ZN40 material inter alia by the higher silica content of the ceramic body and its monoclinic zirconia content. The skilled person seeking for a solution to the stated problem was also aware of the teaching of D1 and D2 and, thus, of the negative effect of silica on the strength and thermal shock resistance, already reported above. Therefore, he would have been led away from increasing the silica content of the ZN40 product having the composition stated in D4 without introducing at least 0.05 wt% of the additives required in D1 or other silica-compensating additives. The reasons set out in points 3.3 to 3.6 above about the combination of information from the prior art do not depend on the sequence in which the pieces of information are combined and, thus, they apply likewise to the case where the ZN40 material is taken as starting point.

3.8 It follows from the above that the ceramic bodies as defined in claim 1 also meet the requirement of inventive step set out in Articles 52(1) and 56 EPC.

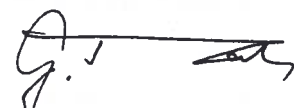
4. Claim 1 being allowable, the same applies to dependent claims 2 to 5 whose patentability is supported by that of claim 1. Claim 6, which is directed to "a method of making a partially stabilised zirconia body according to any one of claims 1 to 5" derives its patentability from that of the claimed ceramic body. The same applies to the dependent process claims 7 to 15.

Order

For these reasons it is decided that:

The appeal is dismissed

The Registrar:



G. Rauh

The Chairman:



R. Spangenberg