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DECISION
of 14 February 2001

Case Number: T 0868/97 - 3.3.5
Application Number: 88302967.0
Publication Number: 0286335
IPC: C04B 37/02

Language of the proceedings: EN

Title of invention:
Air-tight ceramic container

Patentee:
KABUSHIKI KAISHA TOSHIBA

Opponent:
ABB Patent GmbH

Headword:
Air-tight container

Relevant legal provisions:
EPC Art. 56

Keyword:
"Inventive step (yes)"

Decisions cited:
-

Catchword:
-
Case Number: T 0868/97 - 3.3.5

DECISION
of the Technical Board of Appeal 3.3.5
of 14 February 2001

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Composition of the Board:
Chairman: R. K. Spangenberg
Members: M. M. Eberhard
J. H. Van Moer
Summary of Facts and Submissions

I: European patent No. 286 335 based on application No. 88 302 967.0 was granted on the basis of eleven claims. 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. In support of his arguments, the appellant relied on the following documents:


D2: Report TC-1-A 325-86 of the "Nederlands Instituut voor Lastechnik", report No. 86M/0817/HAN/ESB, 10 February 1986

D3: FR-A-2 450 794

II. The opposition division decided that the patent in an amended form met the requirements of the EPC. The decision was based on amended claims 1 to 10 filed on 29 March 1996 with a correction in claim 5 made on 18 February 1997. Claims 1 and 7 thereof read as follows:

"1. A method of manufacturing an air-tight ceramic container, the method comprising brazing a peripheral end face of a metal cover member (2a, 2b) to an opening end face of a ceramic tubular member (1), characterised in that the opening end face of the ceramic tubular member (1) is coated with an active metal consisting of Ti and/or Zr in an amount of from 0.1 to 10 mg/cm² and, having an average grain size of less than 40 µm to form an active metal layer, said
active metal powder being coated either by spraying on the opening end face of the ceramic tubular member which has been previously applied with an organic adhesive or by applying a mixture containing the active metal powder and an organic binder onto the opening end face,

a brazing filler metal is placed on said active metal layer,

and said peripheral end face of said metal cover member (2a, 2b) is placed in contact with said brazing material, the arrangement being such that said active metal layer and said brazing filler metal are sandwiched between said end face of said ceramic tubular member (1) and said peripheral end face of said metal cover member (2a, 2b);

and heating to simultaneously react the active metal layer with the ceramic base to form a metal-ceramic bond and to melt said brazing material to braze said cover thereto."

"7. A method of manufacturing a vacuum interrupter, the vacuum interrupter comprising a ceramic tubular member (1) having metal cover members (2a,2b) brazed to opening end faces thereof by the method of claim 1,

a pair of contact rods (4a,4b) extending through the respective metal cover members, ends of the rods being arranged opposite each other and at least one of said contact rods (4b) being moveable in an axial direction so that said contact rods (41[sic],4b) can be opened and closed;

metal contact members (3a,3b) arranged at the adjacent ends of said contact rods (4a,4b);

a bellows (7) for moving said at least one moveable contact rod in the axial direction while maintaining a vacuum tight connection between the rod
(46[sic]) and the respective metal cover member (2b), and

a metal arc-shield (6) positioned around said contact members (3a,3b) to prevent the metal evaporated from said contact members from adhering to an inner surface of said tubular member, said method comprising the steps of:

coating a powder of an active metal consisting of Ti and/or Zr to an inner surface of the ceramic tubular member in an amount of from 0.1 to 10 mg/cm² and, having an average grain size of less than 40 µm to form an active metal layer, said active metal powder being coated either by spraying on the opening end face of the ceramic tubular member which has been previously applied with an organic adhesive or by applying a mixture containing the active metal powder and an organic binder onto the opening end face;

placing a brazing filler metal on said active metal layer;

placing said arc-shield to be in contact with the brazing filler metal; and

heating to simultaneously react the active metal layer with the ceramic base to form a metal-ceramic bond and to melt said brazing material to braze said arc-shield to the inner surface of said tubular member."

The opposition division took the view that D1 represented the closest prior art. The object of the invention was to improve an already known one-step bonding method so as to obtain a preferred bonding structure as shown in Figure 1B of the patent in suit. The claimed solution was not rendered obvious by the cited documents. According to page 11 of D1 previous one-step methods were less successful than the two-step
method, and the merit of the invention was to have adapted the grain size of the active metal in a manner which allowed a *simultaneous* reaction of the active metal layer with the ceramic base and the melting of the brazing material.

III. The appellant lodged an appeal against this decision and relied on a new document in the statement of grounds of appeal, namely DE-B-14 33 158 (hereinafter D4). In reply to a communication of the board requiring a translation of D1 and D2 from Dutch into one of the official languages, the appellant filed translations of parts of these documents. These partial translations are referred to hereinafter as D1a and D2a. The respondent filed two auxiliary requests on 12 January 2001. Oral proceedings were held on 14 February 2001. At the oral proceedings the respondent submitted an amended main request. Claims 1 to 6 and 8 to 10 of this request are identical to those of the request forming the basis of the decision appealed. Claim 7 differs from previous claim 7 in that the term "opening end face" was replaced twice by "inner surface" in the coating step with the powder of the active material. Furthermore, reference numbers (41) and (46) were corrected to (4a) and (4b) respectively.

IV. The appellant put forward the following arguments:

The claimed process differed from that of D1a only by the particle size of the Ti powder. However this feature was already well-known to the skilled person. It was also disclosed in D4 which taught a grain size of the active component of maximum 50 µm, preferably 0.1 to 10 µm. The skilled person would have inferred from D4 that this particle size was not only suitable
for a compacted brazing mixture but also for a process according to D1 since, in the paragraph bridging columns 1 and 2 of D4, reference was made to a process where the active metal and the brazing material were applied separately to the ceramic surface. The skilled person was therefore told in D4 that if the Ti powder was homogeneously dispersed in the coating he could take another particle size even when the Ti powder and the brazing material were deposited separately. In view of D4, it was obvious to the skilled person to decrease the particle size of the active material to values lower than 50 µm in the process of D1 and to check whether the process could be optimised or improved with such a known particle size. The appellant contested the correctness of the statements on page 11 of D1 which were relied upon in the decision under appeal. He pointed out that they were only based on some test samples which were not reliable, and that the skilled person would, however, have tried to optimise the one-step bonding method which was less expensive than the two-step method. In reply to the question of the board whether the improvement acknowledged in the decision under appeal was contested, the appellant indicated that he did not know whether or not an improvement had been achieved by reduction of the particle size. He further argued that a bonding profile similar to that of Figure 1B of the patent in suit could also be obtained with the known one-step bonding method as shown on Figure 2 of D2. The subject-matter of claim 7 also lacked an inventive step taking into account that a vacuum interrupter was already known from D3. It was obvious to the skilled person to apply the same brazing method to the vacuum interrupter of D3.

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

Starting from D1, the technical problem was to improve the one-step bonding method of D1 to produce the profile shown in Figure 1B of the patent in suit, which profile was necessary to obtain an air-tight ceramic container. D1 seemed to suggest that the one-step bonding method led to poor results and did not suggest any variation of the particle size. D4 could not be combined with the teaching of D1 for several reasons. First, the active components and the brazing metal were combined to form a compacted brazing mixture. Therefore, D4 was concerned with an entirely different type of process. Secondly D4 discussed the problems occurring in a brazing process in which a layer of active metal and a layer of brazing filler metal were applied separately and overcame these problems by producing the compacted homogeneous powder mixture formed from the filler metal and the active metal. Thus, D4 taught away from the use of a sandwich structure for performing brazing. Finally even if the skilled person had looked at D4, he would not have produced the present invention since the requirement concerning the formation of a homogeneous dispersion of the active metal in the filler metal could not be applied to single layers. The skilled person, on realising that D1 did not exhibit particularly good results would not have turned to the teaching of D4 to reduce the grain size.

Reasons for the Decision

1. The appeal is admissible.
2. The amended claims of the main request meet the requirements of Articles 123(2) and (3) EPC. Claim 1 is based on a combination of original claims 1 and 3 with the passage on page 10, line 28 to page 11, line 12, of the application as originally filed. The feature concerning the sandwich structure of the arrangement is directly and unambiguously derivable from the Figures and from the whole description. It can be directly derived from page 9, lines 16 to 22, of the original description that simultaneous reaction of the active metal layer with the ceramic base to form a metal-ceramic bond and melting of the brazing material occur during the heating step. The amendments in claim 7 are disclosed in original claim 11 and on page 10, lines 20 to 24; page 11, lines 1 to 13 and lines 21 to 28 of the original description in addition to the passages already mentioned above. Dependent claims 2 to 6 and 8 to 10 correspond respectively to original claims 2 to 4, 6, 7, 10 to 12. The scope of protection of amended claims 1 and 7 has clearly been restricted over that of granted claims 1 and 8.

3. The processes according to claims 1 and 7 of the main request are novel over the disclosure of the cited documents. As this was not in dispute, further considerations in this respect are not necessary.

4. In agreement with both parties and the opposition division, the board considers that D1/D1a represents the closest prior art. D1a, which is a translation in German of only parts of the report D1, discloses experiments concerning the formation of a brazing bond between a ceramic ring and a metal cap. Experiments were performed using either metallized ceramic rings or non-metallized ceramic rings. In a first series of
experiments (points 3 and 4 on page 4 and 5 of D1a) a paste containing a binder and a titanium hydride powder having a particle size of 60 µm was applied to non-metallized alumina rings. After drying, a foil of brazing material was placed on each coated alumina ring between the ceramic ring and the metal cap. Each sample had two ceramic-metal bonds, thus forming a container after brazing (see page 5 of D1a, fourth paragraph; photograph on page 20 of D1). The samples were then brazed in a vacuum oven at temperatures in the range of 830°C to 980°C (see Tables 3 to 8 of D1). The tightness and bonding strength of the test samples were tested. The structure of the brazing joint was also examined microscopically for a lot of samples (see page 5, last paragraph of D1a). The results of these experiments and the conclusions are given on pages 9 to 11 of D1, but were not translated. According to page 11 of D1 which the opposition division relied on in the decision under appeal, all the ceramic-metal bonds on a metallized ceramic base were visually tight and good whereas the ceramic-metal bonds on a non-metallized base exhibited a rather thin and irregular meniscus and in one case the bond was loose. All the bonds on metallized ceramics were tight before and after thermic cycles (tightness measured by the helium leakage test). Of the twelve ceramic-metal bonds on non-metallized ceramic bases, five samples were tight before thermic cycles while, thereafter, only 3 remained tight. The TiH₂-paste method was not found reliable compared to the reproducible and tight ceramic-metal bonds on metallized ceramics. The ceramic-metal bonds on metallized ceramic also gave the highest strength. The direct brazing of the metal cap on the ceramic base coated with an active material such as a TiH₂ or Ti coating with no independent metallizing step is
referred to hereinafter as the one-step bonding method in opposition to the case where a metallization is performed in advance.

4.1 Starting from this prior art, the technical problem underlying the patent in suit was to improve the one-step bonding method of D1 so as to produce the desired bonding structure shown in Figure 1B, which is necessary to obtain a ceramic container having a highly reliable air-tightness.

Figure 1B shows the spreading of the brazing metal like an unfolded fan from the end portion of the metal cover toward the end face of the ceramic tubular member (see the sentence bridging columns 1 and 2 of the patent in suit). Alternatively this desired profile can also be referred to as the spreading of the brazing metal in the form of a meniscus on each side of the brazing joint (see the reference to the meniscus in D1, page 11, lines 3 to 4).

It is proposed that this problem be solved by the process comprising the combination of features defined in claim 1 of the main request. This process differs from the TiH$_2$ method of D1 at least by the use of a Ti and/or Zr powder instead of a TiH$_2$ powder and by the lower average particle size of the powder. According to page 7, lines 22 to 27, of the patent in suit an air-tight ceramic container having a highly reliable air-tightness can be obtained by applying the bonding method of the invention without increasing a bonding area between the ceramic tubular member and the metal cover. In example 1 no substantial leakage and a bonding structure as shown in Figure 1B were obtained (see the very low He leakage value). In examples 2 and
3 internal high vacuum was maintained. In view of the results indicated in the patent in suit, it is credible, in the absence of evidence to the contrary, that the technical problem has actually been solved by the process as defined in claim 1.

4.2 The appellant indicated at the oral proceedings that he did not know whether an improvement had been obtained over the one-step bonding method of D1. The appellant's doubts as to whether or not the claimed process brought about an improvement were, however, not supported by any evidence. Taking into account that according to page 11 of D1 the one-step bonding method leads to a rather thin and irregular meniscus and that the TiH$_2$-paste method was not found reliable as regards the tightness of the samples, whereas, on the contrary, according to the patent in suit the desired structure shown in Figure 1B and an excellent tightness are obtained with the claimed process, it is plausible that the said improvement has actually been achieved. In these circumstances, if the appellant had doubts in this respect, the burden of proof rested on him to show that the combination of features as defined in claim 1 does not lead to the said improvement.

The appellant further argued that the disclosure on page 11 of D1 was based only on some samples which were not reliable. The board is not convinced by these arguments since the disclosure on page 11 does not relate to some samples but seems in fact to represent the conclusions drawn from several series of experiments insofar as the incomplete translation of D1 makes it possible to understand correctly the disclosure of this document.
The appellant further argued that a bonding structure or profile as shown in Fig 1B could also be obtained by the known one-step bonding method of D1/D1a and made reference to Figure 2 of D2 to support this argument. This argument is not convincing for the following reasons. D2 is a document which was made available to the public more than four years later than D1 and the appellant has not shown that Figure 2 of D2 concerns a sample prepared in the same way as those of D1. From the preparation of the samples described on page 14, point 4.1, of D2a, it can be inferred that it was not identical to that of D1. Furthermore, the substrate "Macor" used in the sample shown in Fig. 2 of D2 is a glass-ceramic containing 45% of a glass phase and 55% of a mica phase, which exhibits properties very different to those of an alumina support (see for example the softening point of 1000°C on page 12 of D2a). The appellant's affirmation at the oral proceedings that it made no difference whether the non-metallic part was a glass-ceramic or an alumina ceramic cannot be accepted by the board since it appears not to be in agreement with the results discussed on page 17 of D2a (see first and second paragraphs) or with the conclusions on page 19 thereof (see second and third paragraphs).

4.3 D1 itself discloses further experiments using Ti instead of TiH₂ as the active material (see enclosure headed "FOM report" on pages 6 to 8 of D1a). In these experiments the alumina rings were coated with a mixture containing a Ti powder and a binder such that the amount of Ti on the alumina base was at least 2 mg/cm². The metal caps were brazed to the coated ceramic rings using a foil of brazing material interposed between the alumina ring and the metal cap.
Brazing was performed in a vacuum oven by slow heating to 750°C, then rapid heating to 950°C and brazing for 5 minutes at 925°C. This minimum amount of Ti falls within the claimed range. However, the particle size of the titanium powder used in these experiments is not disclosed. D1/D1a does not contain any information suggesting that the particle size of the active material powder might be important in combination with the amount of active material per unit surface of the ceramic base in order to achieve the bonding structure as shown in Figure 1B.

4.4 D4 discloses a brazing powder mixture in compacted form and a process for bonding a material having low wettability such as alumina with a metal. The compacted mixture consists of at least one of the active materials Ti-hydride, Zr-hydride, Ti or Zr in powder form and a powder of a usual brazing material. The particles of the active component, which have a particle size of at most 50 µm, preferably 0.1 to 10 µm, are so homogeneously distributed in the brazing powder that they did not touch each other but are on all sides surrounded by the particles of the brazing powder. The powder mixture is composed of three to five different particles sizes in order to ensure the highest void filling and is compacted to at least 65% of its theoretical density (see column 3, lines 3 to 20 and claim 1). This brazing powder mixture makes it possible to reduce or avoid the formation of zones with different compositions during the brazing process, which lead to differences in the wettability of the surface (see column 2, lines 13 to 32, and column 2, line 59 to column 3, line 2).

The appellant's arguments that, in view of this
teaching, it would have been obvious to use a particle size of less than 40 µm for the Ti or Zr powder in the one-step bonding method of D1 are not convincing for the following reasons. The bonding method of D4 does not involve separate deposition of a layer of the active component and of the brazing filler metal onto the ceramic material like D1 but placing the compacted homogeneous powder mixture of active component and brazing metal between the ceramic and the metal piece to be bonded together. These two methods represent two entirely different types of processes, and, therefore, it cannot be predicted whether or not the particle size of the active component which is suitable for the method of D4 might bring about an improvement in the process of D1. D4 makes reference in the paragraph bridging columns 1 and 2 to the known process in which the active component and the brazing metal are applied separately to the ceramic surface. This process is said to give unreliable results which are difficult to control. The drawbacks of this process are discussed in detail in column 2, lines 13 to 32: see in particular the differences in the concentrations (or compositions) at different locations of the hydride layer, which can lead to differences in the wettability of the surface. D4 proposes to use the compacted homogeneous mixture of active component and brazing metal in order to overcome these drawbacks. It is not the small particle size of the active component but the combination of this feature with the homogeneous distribution of the active component in the brazing powder, the maximum void occupation and the compaction of the powder to a certain degree, which leads to the desired results. This teaching cannot suggest to the skilled person that an improvement of the bonding structure might be achieved by applying only one of
these features, namely the small particle size of the active component, to the completely different process involving separate deposition of the active component layer and of the brazing metal. As the other essential features of D4, in particular the homogeneous distribution of the active component in the brazing metal and the maximum void occupation cannot be applied to this process, the skilled person would have had no reasons to expect an improvement of the bonding structure. The appellant’s argument that the use of active components having the claimed particle size was not specific to the process of D4 but was part of the general knowledge of the skilled person in this technical field, was not supported by evidence although the burden of proof lies on the appellant in this respect. Furthermore, this argument is not in agreement with the particle size of 60 µm disclosed in D1 for the TiH₂ powder. In this context, it is observed that although D4 was published in 1968, ie more than 11 years before the date of the report D1, the experiments reported in D1 were carried out with coarser particles. For the preceding reasons the board considers that picking out only one of the essential features stated in D4 and applying it to a completely different method such as the method of D1 to arrive at the claimed subject-matter can only be the result of an ex-post facto analysis.

4.5 In the report D2/D2a, which was established about five years after D1, neither Ti nor Zr was used as the active component but TiH₂. The particle size of the TiH₂ powder used to coat the ceramic and the glass ceramic plates is not mentioned in D2a. This document also contains no information from which it might be inferred that an improvement of the bonding structure might be
obtained by reducing the particle size of the active component to values less than 40 µm. D3 is also silent in this respect. Therefore, the disclosure of these documents cannot point toward the claimed solution.

4.6 It follows from the above that the subject-matter of claim 1 according to the main request meets the requirement of inventive step set out in Articles 52(1) and 56 EPC.

5. Claim 7 of the main request comprises the different steps indicated therein for bonding the arc-shield to the inner surface of the ceramic tubular member. It also implicitly contains the steps recited in claim 1 for brazing the peripheral end face(s) of the metal cover(s) to the opening end face(s) of the ceramic tubular member when all other elements are mounted inside the tubular member. This was confirmed by the respondent at the oral proceedings. It follows that the subject-matter of claim 7 is also considered to involve an inventive step for the reasons given above in connection with claim 1.

Claims 1 and 7 being allowable, dependent claims 2 to 6 and 8 to 10 derive their patentability from that of claims 1 and 7 and are, therefore, also allowable. The description has been brought into conformity with the amended claims.

Order

For these reasons it is decided that:
1. The decision under appeal is set aside

2. The case is remitted to the first instance with the order to maintain the patent with the following documents:

- claims 1 to 10 as filed during the oral proceedings

- description pages 3 to 8 and 10 as indicated in the appealed decision and page 9 submitted during the oral proceedings

- drawings as granted

The Registrar: 

The Chairman:

S. Hue 

R. Spangenberg