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D E C I S I O N
of 18 April 1997

Case Number: T 0765/94 - 3.3.2

Application Number: 90102224.4

Publication Number: 0385132

IPC: C04B 35/00

Language of the proceedings: EN

Title of invention:
Superconducting wire

Applicant:
SUMITOMO ELECTRIC INDUSTRIES, LTD.

Opponent:
-

Headword:
Superconductive wire/SUMITOMO

Relevant legal provisions:
EPC Art. 56

Keyword:
"Inventive step - no"

Decisions cited:
-

Catchword:
-



Case Number: T 0765/94 - 3.3.2

D E C I S I O N
of the Technical Board of Appeal 3.3.2
of 18 April 1997

Appellant: SUMITOMO ELECTRIC INDUSTRIES, LTD.
5-33, Kitahama 4-chome
Chuo-ku
Osaka-shi, Osaka 541 (JP)

Representative: Kuhnen, Wacker & Partner
Alois-Steinecker-Strasse 22
D-85354 Freising (DE)

Decision under appeal: Decision of the Examining Division of the
European Patent Office posted 6 May 1994 refusing
European patent application No. 90 102 224.4
pursuant to Article 97(1) EPC.

Composition of the Board:

Chairman: F. Antony
Members: G. J. Wassenaar
R. E. Teschemacher

Summary of Facts and Submissions

- I. European patent application No. 90 102 224.4 was refused by a decision of the Examining Division announced at the oral proceedings held on 25 March 1994 and dispatched with reasons on 6 May 1994.

The decision was taken on the basis of Claim 1 filed on 7 June 1993 and Claims 2 to 6 filed on 11 August 1992 as main request, and alternative Claim 1 filed on 7 June 1993 together with Claims 2 to 6 filed on 11 August 1992 as auxiliary request.

- II. The Examining Division held that the subject-matter of the independent Claim 1 (main and auxiliary requests) did not meet the requirements of Article 56 EPC. In the grounds of the decision, the Examining Division held that EP-A-280 292 (D4) was the most relevant prior art, disclosing superconducting wires comprising a flexible base material and an oxide superconducting layer formed thereon. According to the decision under appeal it was obvious to use a base material having a roughness of not more than 0.05 μm in view of the disclosure in D4 that, if the superconducting material was applied by vapour phase deposition, as known e.g. from EP-A-298 461 (D5), the surface of the base material had to be polished to a high degree as pretreatment.

- III. The Applicant (Appellant) lodged an appeal against that decision on 15 July 1994, with payment of the fee. With the statement of grounds, filed on 15 September 1994, the Appellant submitted three new sets of claims and the technical specification of a contour measuring apparatus available under the tradename DEKTAK 3030, together with a graph of a roughness measurement of a silicon substrate. The Appellant essentially argued that the roughness of the base material used in D4 is

much higher than required by the present claims and that there was no indication in the prior art that the critical temperature and the critical current density could be markedly increased by reducing the surface roughness.

IV. During the oral proceedings, which were held on 18 April 1997, the Appellant filed a set of new Claims 1 and 2, replacing all earlier sets of claims. New Claim 1 reads as follows:

"A superconducting wire comprising a flexible base material of metal, Ni base alloy or yttrium stabilized zirconia having an average surface roughness of not more than $0.05 \mu\text{m}$ and a high T_c oxide superconducting layer of about $1 \mu\text{m}$ thickness or less formed on said base material by vapor phase deposition."

According to the Appellant, the problem underlying the alleged invention was, to provide a wire with a superconductive oxide coating with a high critical temperature, a high critical current density and a thickness of not more than $1 \mu\text{m}$. He stressed that, although the skilled person was aware of the influence of the surface structure of a substrate on the electrical properties of a thin layer coated thereon, it was not obvious for him to polish the surface to the smoothness required by Claim 1. Such a treatment was time consuming and expensive and the skilled person would not expect a substantial improvement from such a treatment. In the application it had been shown by experimental evidence that, contrary to the skilled person's expectation, the critical temperature T_c and critical current density J_c abruptly increased if the roughness R_a of the substrate surface was reduced from 0.1 to $0.04 \mu\text{m}$.

- V. The Appellant requested that the decision under appeal be set aside and that a patent be granted on the basis of Claims 1 and 2 as submitted during oral proceedings.

Reasons for the Decision

1. The appeal is admissible.
2. The amendments are allowable under Article 123(2) EPC.
3. The subject-matter of claims 1 and 2 is novel.
4. *Inventive step*
 - 4.1 It is out of dispute that of the documents on file D4 represents the closest prior art. This document discloses a superconducting wire comprising a flexible base material and a high T_c oxide superconducting layer formed on said base material by sol-gel coating (column 2, lines 22-48, column 5, lines 4-8, column 7, lines 34-39). One of the base materials mentioned and employed in the examples is yttrium stabilized zirconia (YSZ); see column 5, lines 49-54. In Example 12 a superconducting oxide layer on YSZ is disclosed with a layer thickness of 0.5 to 1.0 μm having a T_c of 68 K. The roughness of the YSZ base material is not disclosed in Example 12; D4 does, however, disclose a surface roughness R_a of about 0.3 μm for zirconia tape (column 6, lines 5-12), it being not entirely clear whether this value also applies to YSZ base material, because a distinction is made between "zirconia having flexibility" and "YSZ" (column 5, lines 49-54).

4.2 In agreement with the statement in the application (page 3, line 23 to page 4, line 2 of the original text) and the statement made by the Appellant during oral proceedings, the technical problem underlying the invention was to improve the superconductive properties of a coated wire having an oxide superconductive layer with a thickness of not more than 1 μm .

According to Claim 1 this problem is solved by polishing the surface of the base material to an average roughness of not more than 0.05 μm and forming the coating by vapour phase deposition.

According to Example I, an oxide superconductive film with a thickness of 0.5 μm , having a critical temperature of about 80 K was obtained by vapour phase deposition on a YSZ substrate having an average roughness R_a of 0.04 μm (400 Å). The critical temperature is clearly higher than that of oxide films of comparable thickness disclosed in D4. Also according to the said Example I, a sample with a thickness of 1 μm and a roughness R_a of 0.04 μm had a critical current density of 10^2 A/cm^2 at 77.3 K. D4 does not disclose critical current densities, which according to the Appellant would suggest that they were not very high. Anyhow, there is at least no reason to believe that the critical current densities of the thin oxide superconductive films according to D4 were higher than the values given in the present application. Therefore, the Board is satisfied that the claimed wire actually solves the above mentioned technical problem.

4.3 It remains to be decided if, for solving the above-mentioned problem, it was obvious to apply the oxide film by vapour phase deposition and, in pretreating, the substrate to polish it to the smoothness as required by Claim 1.

- 4.3.1 Document D4 indicates in the description of the prior art that, before the invention disclosed therein, elongated bodies having a coating of oxide superconductive material were manufactured by a vapour phase method, such as CVD (chemical vapour deposition) and PVD (physical vapour deposition). The vapour phase method is said to have several disadvantages. In particular, it requires specific equipment such as a plasma generator and an apparatus for heating raw materials, and film formation has to be performed under a high-temperature atmosphere. The vapour phase method further requires high cleanliness and hence the surface must be polished in a high degree as pretreatment (column 1, lines 29-48 and column 6, lines 48-55).
- 4.3.2 As against that, the sol-gel method of D4 leads indeed to a simplification of the coating method, though at the expense of reduced critical temperature. All of the critical temperatures of the coated zirconia tape or YSZ according to Examples 9 and 10 (77 K), Example 11 (50 K), Example 12 (68 K), Example 14 (30 K) and Example 15 (65 K) of D4 are lower than the critical temperature of the uncoated wire of Example 6 (79 K). For the relatively thick coating of Examples 9 and 10, having a thickness of 5 μm , the reduction in critical temperature is only moderate, but for the thin coatings of Examples 11, 12 and 14, having a thickness of 0.5 to 1.0 μm , the reduction is dramatic because the above values imply that no superconductivity occurs at the boiling temperature of liquid nitrogen. Thus it became clear to a skilled person that the sol-gel method was not suitable for making superconductive coatings having a thickness of about 1 μm or less.
- 4.3.3 Since the sol-gel coating method proposed by D4 thus turned out to be unsatisfactory for thin coatings, it was obvious for the skilled person wishing to improve the superconductive properties of thin coatings, to

return to the earlier vapour phase method, seeking to improve the same. From D4 the skilled person knew that for the vapour phase method it was essential to polish the surface of the base material to a high degree. It is not disclosed in D4 to what degree the said substrate should be polished, but this could be easily determined by trial and error. The skilled person would perform a test series, just as taught in the present application whereby T_c is determined as a function of the average roughness R_a , and would then find that in the range between 0.1 and 0.04 μm , T_c would significantly increase, while below 0.04 μm no substantial further improvement could be achieved.

- 4.3.4 The Appellant's contention that the skilled person, although aware of the beneficial effect of polishing the substrate on the electrical properties of the coating, would not polish it to a substantial degree as required by present Claim 1, because this step is time consuming and expensive and he would not expect a substantial improvement, cannot be accepted. The skilled person would know from D4 that for thicker oxide superconductive layers, T_c is in the neighbourhood of the theoretical value for unsupported oxide superconductors. He would be aware of the most obvious explanation that T_c for thin layers is substantially lower by the influence of the substrate on the crystalline quality of the oxide superconductor, which influence would decrease as the layer became thicker. An obvious means for suppressing the influence of the substrate would be to reduce its surface as far as possible, i.e. to polish it to a high degree. The skilled person would thus expect that with a "perfectly" smooth surface the T_c of thin layers would also be in the neighbourhood of the theoretical value,

hence if the T_c of a thin layer was substantially below the theoretical T_c of the oxide superconductor under consideration, a substantial improvement could be expected to result from further polishing. Whether the result would be worth the effort, could perhaps not be precisely foreseen, but could easily be determined by obvious exploratory testing (see point 4.3.3 above).

4.3.5 The Appellant's further argument that, even if the skilled person would try to improve T_c by polishing he would not polish below an R_a of $0.05 \mu\text{m}$ (since no substantial improvement would be observed by reducing R_a from 1 to $0.1 \mu\text{m}$) and would therefore not detect the surprising increase in T_c between 0.1 and $0.04 \mu\text{m}$, cannot be accepted either. The advantage of the sol-gel method of D4 was that, contrary to the vapour phase method, no polishing to a high degree was necessary, the surface roughness R_a of a base material used in the sol-gel method of D4 being about $0.3 \mu\text{m}$; this means that, according to D4, for vapour phase deposition R_a must be reduced substantially below $0.3 \mu\text{m}$. The skilled person would thus contemplate a reduction of at least one order of magnitude, which would bring down R_a to something like $0.03 \mu\text{m}$. The substantial increase in T_c between 0.1 and $0.04 \mu\text{m}$ would thus be observed by routine experimentation.

4.3.6 The Board has also noted in passing that the priority date of the application in suit is less than 6 months after the publication date of D4 so that it could not be argued that a seemingly apparent measure for some considerable time escaped the attention of skilled workers and therefore might involve an inventive step.

4.4 In summary, the claimed solution of the above mentioned technical problem, viz. the improvement of the superconductive properties of thin oxide films by vapour phase deposition on a surface having an average

roughness R_a below $0.05 \mu\text{m}$, is considered to be the result of routine experimentation based on suggestions given in D4, and which required no more than ordinary technical skill, without involving any inventive step in the meaning of Article 56 EPC.

Order

For these reasons it is decided that:

1. The appeal is dismissed.

The Registrar:

The Chairman:

P. Martorana

F. Antony