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Case Number: T 1623/08 - 3.4.03

Application Number: 02806112.5

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IPC: H01L 39/14, H01L 39/24, H01F 6/00, C22C 27/02

Language of the proceedings: EN

Title of invention:
Zirconia-stabilized multi-filamentary niobium-tin superconducting wire

Applicant:
GENERAL ELECTRIC COMPANY

Headword:
-

Relevant legal provisions:
EPC Art. 123(2)
RPBA Art. 15(3)(5)(6)

Relevant legal provisions (EPC 1973):
EPC Art. 56
EPC R. 71(2)

Keyword:
"Inventive step (no)"

Decisions cited:
-

Catchword:
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Beschwerdekammern | Boards of Appeal | Chambres de recours

Case Number: T 1623/08 - 3.4.03

D E C I S I O N
of the Technical Board of Appeal 3.4.03
of 13 November 2012

Appellant: (Applicant)
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Decision under appeal:
Decision of the Examining Division of the European Patent Office posted 18 February 2008 refusing European patent application No. 02806112.5 pursuant to Article 97(2) EPC.

Composition of the Board:

Chairman: G. Eliasson
Members: V. L. P. Frank
T. Karamanli

Summary of Facts and Submissions

I. This is an appeal against the refusal of European patent application No. 02 806 112 for the reason that the application was amended in such a way that it contained subject-matter which extended beyond the content of the application as filed (Article 123(2) EPC).

II. On appeal the applicant requested in writing that the decision under appeal be set aside and that a patent be granted on the basis of the main request, or of the 1st or 2nd auxiliary request, all submitted with the statement of grounds of appeal. The 1st auxiliary request was formed by claims 3 and 4 of the main request; the 2nd auxiliary request was formed by claims 5 and 6 of the main request.

Auxiliarily oral proceedings were requested.

III. Claims 1, 3 and 5 of the appellant applicant's main request read as follows:

1. "A superconducting wire (100), said superconducting wire (100) comprising:
a) at least one filament (110) having a filament diameter, wherein said at least one filament (110) is continuous and comprises a niobium alloy having a plurality of Nb_3Sn grains having a plurality of ZrO_2 precipitates disposed therein, wherein said plurality of Nb_3Sn grains has an average grain size of less than about 10 percent of said filament diameter and the zirconium comprises up to 8 atomic percent of the alloy; and

b) a metallic matrix surrounding and contacting said at least one filament (110), wherein said metallic matrix is electrically conductive at temperatures below about 77 K and has a coefficient of thermal expansion that is substantially the same as or greater than that of Nb_3Sn."

3. "A preform for forming a superconducting wire (100), wherein the superconducting wire (100) comprises at least one filament (110), wherein said at least one filament (110) comprises a plurality of Nb_3Sn grains having a plurality of ZrO_2 precipitates disposed therein and a metallic matrix surrounding and contacting said at least one filament (110), said preform comprising: at least one niobium alloy rod, said at least one niobium alloy rod comprising a niobium alloy having zirconium and oxygen in solid solution, wherein zirconium and oxygen are present in an atomic ratio of about 1:2 and the zirconium comprises up to 8 atomic percent of the alloy; and a metallic preform matrix surrounding and contacting said at least one niobium alloy rod, wherein said metallic preform matrix comprises copper and between 5 weight percent and 13 weight percent tin."

5. "A method of making a superconducting wire (100), the superconducting wire (100) comprising at least one filament (110), wherein said at least one filament (110) is continuous and comprises a niobium alloy having a plurality of Nb_3Sn grains with a plurality of ZrO2 precipitates disposed

therein, and a metallic matrix surrounding and contacting said at least one filament (110), the method comprising the steps of:
providing the niobium alloy having zirconium and oxygen in solid solution,
wherein zirconium and oxygen are present in an atomic ratio of about 1:2 and the zirconium comprises up to 8 atomic percent of the alloy;
forming at least one niobium alloy rod from the niobium alloy;
providing a metallic preform matrix material comprising copper and between 5 weight percent and 13 weight percent tin surrounding and contacting the at least one niobium alloy rod;
forming a wire from the metallic preform matrix material and the at least one niobium alloy rod;
and heat treating the wire at a temperature of between 700°C and 1100°C for a predetermined time, thereby forming the superconducting wire (100), with the matrix material having a coefficient of thermal expansion that is substantially the same as or greater than that of Nb_3Sn, and wherein the Nb_3Sn grains have an average size that is less than 10 percent of the diameter of the filament.

IV. The following documents are mentioned in this decision:

D1 = US 4 324 842 A

D2 = US 3 838 503 A

D4 = "The role of Oxygen and Zirconium in the formation and growth of Nb_3Sn grains", Met. and Mat. Trans. A, Vol. 25A, pp. 213-219, January 1994

D5 = "Effect of Oxygen and Zirconium on the growth and superconducting properties of Nb_3Sn", Met. and Mat. Trans. A, Vol. 25A, pp. 203-212, January 1994

V. The examining division found that:

- In claims 1 and 7 of the main and the auxiliary request the feature the "metal matrix comprising tin" introduced subject-matter not originally disclosed in the application. It was clear from the application that the metal preform matrix comprised tin. It was, however, not explicitly disclosed in the application that also the metal matrix (i.e. the metal surrounding the filaments comprising the Nb_3Sn grains with precipitates of ZrO_2 after the heat treatment of the preform to form the Nb_3Sn) comprised tin. In case of a sufficient reaction time and temperature, the tin would completely diffuse into the Niobium to form Nb_3Sn. The application hence did not meet the requirements of Article 123(2) EPC.

VI. The appellant applicant argued essentially as follows:

- The new claims overcame the objections of the examining division since claim 1 did not specify tin as a component of the matrix of the wire. New claims 3 and 5 specified tin as a component of the matrix of the preform.

- The present invention, as claimed in new claims 1, 3 and 5, concerned the problem of shortening the length of time taken for heat treating the wire

formed from the preform. This was achieved by incorporating zirconium and oxygen into the niobium alloy of the filament, thereby producing smaller grain size in the Nb_3Sn grains, enabling heat treatment at a higher temperature and thus a shorter processing time.

– Document D1 dealt with the problem of improving strain characteristics by incorporating a small amount of beryllium into the matrix. It was submitted that a skilled person, looking to shorten the manufacturing process, would not be led to D4 (or D5), which dealt with improving the critical current density in a niobium foil. In fact, D1 taught away from considering document D4 because it related to modifying the matrix rather than the filament. D4 taught the use of zirconium and oxygen to improve the electrical properties of a superconducting foil of niobium which had been coated in tin, while D1 disclosed a niobium filament in a bronze matrix in which beryllium had been included in the matrix to improve its (physical) strain characteristics. There was no incentive for a skilled person, starting from D1, to take part of the teachings of D4 and apply them to D1. There was nothing in D1 or D4 to suggest that the manufacturing time could be reduced. It was accordingly submitted that the present invention, as claimed in the claims of the main request were patentable.

– Regarding independent claim 3, the arguments presented above with regard to claim 1 were applicable to claim 3 as well. Moreover, claim 3

specified zirconium and oxygen in solid solution in the niobium rod, and tin in the copper matrix surrounding it. Although D1 used a preform from which a wire was produced, D4 related to an entirely different system using a tin-coated niobium foil.

– Independent claim 5 was directed to a method of making a superconducting wire. The arguments presented above with regard to claim 1 applied also to this claim. In addition, the claimed method used a preform comprising a niobium rod having zirconium and oxygen in solid solution, in a bronze matrix. Tin from the matrix diffused into the rod, and the resulting Nb_3Sn grains grew slowly due to the presence of the zirconium and oxygen, resulting in smaller grains which allowed subsequent heat treatment at a higher temperature than hitherto, thus shortening the manufacturing time compared with that of known processes, such as that of D1. The process of D4 was entirely different, with no rod in a matrix, but rather a niobium foil containing zirconium, the surface of which was oxidized and then pulled through a bath of molten tin. There was nothing in either D1 or D4 to suggest that the heat treatment time could be shortened by adopting any or all of their teachings.

VII. In a communication pursuant to Article 15(1) RPBA annexed to the summons to oral proceedings, the board informed the appellant of its provisional opinion that the superconducting wire of claim 1, the preform for forming a superconducting wire of claim 3 and the method of making a superconducting wire of claim 5 of the main request did not involve an inventive step over

a combination of documents D2 and D5. As the 1st and 2nd auxiliary requests were based, respectively, on claims 3 and 5 of the main request, these comments applied as well to these requests. Hence none of the appellant's requests appeared to be allowable.

VIII. The appellant's representative announced with letter dated 20 September 2012 that he would not attend the oral proceedings.

IX. Oral proceedings were held on 13 November 2012 in the absence of the applicant.

Reasons for the Decision

1. The appeal is admissible.

2. As announced in advance, the duly summoned appellant did not attend the oral proceedings. According to Rule 71(2) EPC 1973, the proceedings could however continue without him. In accordance with Article 15(3) RPBA, the board relied for its decision only on the appellant's written submissions. The board was in a position to decide at the conclusion of the oral proceedings, since the case was ready for decision (Article 15(5) and (6) RPBA), and the voluntary absence of the appellant was not a reason for delaying a decision (Article 15(3) RPBA).

3. The board considers that the objection under Article 123(2) EPC raised by the examining division is overcome by the amendments made to the claims.

4. *Inventive step (Article 56 EPC 1973).*

4.1 The board considers that document D2 represents the closest state of the art. It discloses a multifilament superconducting Nb_3SN wire in a bronze-tin matrix (Example VI, Figure 1). The superconducting Nb_3SN wire was formed from a Nb rod containing 1% Zr dopant which was inserted in a pure copper tube. This assembly was then drawn to a composite size of 60 mils. A plurality of these individually drawn composites were collectively packaged in a copper tube and again drawn to form a composite assembly having thin core wires 13 in a pure copper matrix 15 (Figure 1a). This assembly was then coated with tin 19 and heat treated at 650°C for 100 hours to produce Nb_3Sn filaments 27 around the Zirconium containing Nb cores 13 (Figure 1d). The small amount of Zr (1-5%) made the grain size of the Nb_3Sn very small, which improved the critical current.

4.2 The superconducting wire of claim 1 of the main request differs from the superconducting wire of document D1 in that

(a) the Nb_3Sn grains have a plurality of ZrO_2 precipitates disposed therein, and in that

(b) the Nb_3Sn grains have an average grain size of less than about 10 percent of the filament diameter.

4.3 The appellant argued that the problem addressed by the application was how to maintain a fine grained structure when annealing at temperatures higher than the ones previously used and not at improving the critical current itself.

4.4 Hence the board considers that the problem addressed by the application can be seen in maintaining a fine grained structure in a multifilament Nb_3Sn when annealing at higher temperatures than the ones previously used, eg the annealing temperature of 650°C disclosed in document D2. The fine grained structure is required for maintaining high critical currents, but not necessarily improving its value beyond the ones obtained in the prior art.

4.5 Document D5 discloses the effect of oxygen and zirconium on the growth and superconducting properties of Nb_3Sn. In the reported experiments a Nb foil with 1 and 2 at % Zr were used. The annealing was done at 1050 °C in an argon atmosphere. The best results were obtained at an O:Zr ratio just over 2:1. This ratio allowed obtaining a very small Nb_3Sn grain size of about 0.2-0.5 µm and high critical currents. The addition of oxygen to the Nb-1 at% Zr foil altered dramatically the Nb_3Sn grain size. The rate of grain growth in Nb_3Sn was significantly reduced by the presence of ZrO_2 precipitates within the Nb_3Sn grains (page 203, left hand column, 2nd paragraph; page 203, right hand column, 1st paragraph; page 204, right hand column, "Reaction anneal"; page, 205, right hand column, "Results", 2nd paragraph; Figure 3; page 206, left hand column; Figure 8 and 9; page 209, right hand column, "Discussion"; page 211, right hand column, "Conclusions").

4.6 Hence the board considers that the skilled person would have applied the conclusions of the experiments made in D5 on Nb foils to the fabrication of a superconducting wire as disclosed in document D2, since it is evident

to the skilled person that the experiments of D5, although performed on Nb foils, are relevant to the manufacture of superconducting Nb_3Sn wires. He thus would have adjusted the Zr:O ratio to about 1:2 and kept the amount of dopant at about 1-2 at% Zr. In this manner the annealing temperature could be increased to about 1050°C , ie at the temperatures specified in the method of claim 5 (between 700 and 1100 °C) and the annealing times accordingly reduced.

4.7 The above observations were communicated to the appellant applicant in the annex to the summons to oral proceedings. The appellant did not file any substantive response to the board's provisional view and neither he nor his representative attended the oral proceedings. Hence the board sees no reason to depart from them.

4.8 The board finds, for these reasons, that the superconducting wire of claim 1, the preform for forming a superconducting wire of claim 3 and the method of making a superconducting wire of claim 5 of the main request do not involve an inventive step over a combination of documents D2 and D5.

As the 1st and 2nd auxiliary requests are based, respectively, on claims 3 and 5 of the main request, the above conclusions apply as well to the corresponding claims of these requests.

Hence none of the appellant's requests is allowable.

Order

For these reasons it is decided that:

The appeal is dismissed.

Registrar: Chair:

S. Sánchez Chiquero G. Eliasson