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Datasheet for the decision of 26 March 2013

Case Number: T 0086/11 - 3.2.08

Application Number: 01201703.4

Publication Number: 1225243

IPC: C22F 1/18, C22C 16/00, G21C 3/07

Language of the proceedings: EN

Title of invention:
Method for manufacturing a tube and a sheet of niobium—containing zirconium alloy for a high burn-up nuclear fuel

Patent Proprietor:
Korea Atomic Energy Research Institute
Korea Electric Power Corporation

Opponent:
FRAMATOME ANP

Headword:
-

Relevant legal provisions:
EPC Art. 84, 100(a)(b)(c)
EPC R. 42(1)(2)

Keyword:
"Main request: clarity (yes)"
"Sufficiency of disclosure (yes)"
"Novelty (yes)"
"Inventive step (yes)"

Decisions cited:
-

Catchword:
-

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Case Number: T 0086/11 - 3.2.08

D E C I S I O N
of the Technical Board of Appeal 3.2.08
of 26 March 2013

Appellant: (Opponent)	FRAMATOME ANP Tour Areva 1 place de la Coupole D-92400 Courbevoie (FR)
Representative:	Neyret, Daniel Jean Marie Cabinet Lavoix 2, place d'Estienne d'Orves F-75441 Paris Cedex 09 (FR)
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Representative:	Kortekaas, Marcel C.J.A. Exter Polak & Charlouis B.V. (EP&C) P.O. Box 3241 NL-2280 GE Rijswijk (NL)
Decision under appeal:	**Interlocutory decision of the Opposition Division of the European Patent Office posted 19 November 2010 concerning maintenance of European patent No. 1225243 in amended form.**

Composition of the Board:

Chairman: T. Kriner
Members: R. Ries
C. Schmidt

Summary of Facts and Submissions

I. By its interlocutory decision dispatched on 19 November 2010, the opposition division held that the subject matter of the claims according to the main request then on file met the requirements of the EPC and that the patent could be maintained in amended form on the basis of this request.

II. The appellant (opponent) lodged an appeal against this decision on 7 January 2011, paying the appeal fee on the same day. The statement setting out the grounds of appeal was filed on 17 March 2011.

III. On appeal, the parties referred in particular to the following documents:

D1: US-A-6 125 161;

D2: JP-A-8067954 (and D2' a computer-generated English translation);

D4: US-A-5 844 959;

D10: EP-A-0 198 570.

IV. Oral proceedings took place before the Board on 26 March 2013. The following requests were made:

- The appellant requested that the decision under appeal be set aside and that European patent No. 1 225 243 be revoked.

- Respondents I and II (patent proprietors) requested that the decision under appeal be set aside and that
the patent be maintained on the basis of the set of claims according to the main request
or, alternatively, on the basis of one of the first to fourth auxiliary requests, all requests filed with letter dated 20 February 2013.

V. Claim 1 of the main request reads as follows:

"A method for manufacturing a tube or a sheet of niobium-containing zirconium alloy for high burn up nuclear fuel, consisting of the following steps:
melting a metal mixture comprising zirconium and alloying elements to obtain an ingot (first step);
forging the ingot in the β phase range (second step);
β-quenching the forged ingot after performing a solution heat-treatment at 1015-1075° (third step);
hot-working the quenched billet (fourth step);
cold-working the hot-worked ingot three to five times, with intermediate vacuum annealing (fifth step); and
final vacuum annealing the cold-worked billet (sixth step);
characterized in that
said niobium-containing zirconium alloy is selected from
a) a niobium-containing zirconium alloy comprising Nb 0.3-0.6 wt%, Sn 0.7-1.0 wt%, Fe 0.2-0.5 wt%, Cr 0.05-0.25 wt%, one element of Mn and Cu 0.05-0.4 wt%, Si 80-120 ppm, O 600-1400 ppm and Zr the balance;
b) a niobium-containing zirconium alloy comprises Nb 0.15-0.25 wt%, Sn 0.9-1.40 wt%, Fe 0.2-0.4 wt%, Cr

0.10-0.25 wt%, Cu 0.05-0.12 wt%, Si 80-120 ppm, O 600-1400 ppm and Zr the balance;
c) a niobium-containing zirconium alloy comprises Nb 0.05-0.3 wt%, Sn 0.3-0.7 wt%, Fe 0.2-0.4 wt%, one element of Cr and Cu 0.05-0.2 wt%, Si 80-120 ppm, O 600-1400 ppm and Zr the balance;
d) a niobium-containing zirconium alloy comprises Nb 1.3-1.8 wt%, Sn 0.2-0.5 wt%, Fe 0.1-0.3 wt%, one element of Cr, Mn and Cu 0.05-0.3 wt%, Si 80-120 ppm, O 600-1400 ppm and Zr the balance;
e) a niobium-containing zirconium alloy comprises Nb 0.8-1.2 wt%, Sn 0.8-1.2 wt%, Fe 0.2-0.4 wt%, Cr 0.10-0.25 wt%, one element of Mn and Cu 0.05-0.3 wt%, Si 80-120 ppm, O 600-1400 ppm and Zr the balance;
f) a niobium-containing zirconium alloy comprises Nb 0.8-1.2 wt%, Fe or Cu 0.05-0.3 wt%, Si 80-120 ppm, O 600-1400 ppm and Zr the balance;
the fourth step is performed at 600-650°C,
the sixth step is performed at 440-600 °C for 2-4 hours,
wherein the cooling rate on β-quenching, and temperatures of intermediate vacuum annealing and final vacuum annealing at α phase range after the β-quenching are changed so as to attain the condition under which precipitates in the alloy matrix are limited to an average diameter of 80 nm or smaller and the accumulated annealing parameter ∑A, represented by the following equation 1 (∑A), is limited to 1.0×10^{-18} hr or less:

$$\sum A = \sum_i t_i \times \exp(-Q/RT_i) \quad \text{Equation 1}$$

wherein t_i is annealing time (hr) of i-th annealing step after β-quenching, T_i is annealing temperature (K) of

i-th annealing step after β-quenching, R is gas constant, Q is activation energy and Q/R equals about 40,000 K."

VI. The arguments of the appellant relevant to the present decision can be summarized as follows:

Clarity; Article 84 EPC

In paragraphs [0027], [0030] and [0057], the patent specification taught that controlling the accumulated annealing parameter ∑A resulted in obtaining precipitates having a diameter of 80 nm or smaller. This wording was to be interpreted as defining the maximum size of the precipitates. Contrary thereto, the size of the precipitates was limited in claim 1 to an average diameter of 80 nm or smaller. Given this contradiction between the claims and the description, an objection arose under Article 84 EPC.

Sufficiency of disclosure; Article 100(b) EPC

β-quenching was known in the art to have an influence on the size of the precipitates. However, the patent specification did not provide any technical information about the specific process parameters (temperature level, cooling rate etc) which were used in the β-quenching step. Hence no guidance was given to the skilled person on how to achieve successfully the desired size of 80 nm of the precipitates when putting into practice the claimed method.

Furthermore, the patent was silent on the carbon level in the alloys. In Zr-Nb alloys of the claimed type,

carbon was an important component for grain size control and was added to this end in amounts between 50 and 200 ppm. Carbon therefore had an influence on the mechanical properties of the alloy.

Moreover, the process described in the patent specification was insufficiently disclosed since it failed to fix a lower limit for the accumulated annealing parameter ∑A. The patent specification thus kept the skilled person guessing as to how to perform the claimed process at low values of ∑A and how to achieve the desired alloy properties.

An objection therefore arose under Article 100(b) EPC.

Novelty; Article 100(a) EPC

Should the term "*method consisting of the following steps*" featuring in claim 1 of the main request be interpreted as including only the process steps which were specified in the claim, the novelty objection would be waived.

Inventive step

After hot working and before cold rolling of the alloy, the process disclosed in document D2 (and also that in D1) stipulated an annealing step which was excluded from the claimed process. The patent specification did not, however, disclose any reasoning as to why the intermediate beta-annealing step was omitted and what technical effect was associated therewith. Document D10, which dealt with the same type of Zr-Nb alloys as the patent-in-issue, taught that β-annealing between hot

extrusion and cold working "may" be effected, depending upon the alloy (D10, page 2, lines 56 to 58). It would be therefore clear to the skilled person that intermediate β-annealing was purely optional. Moreover, the process of producing Zr-Nb-Sn alloys described in document D4, column 6, lines 28 to 38 did not provide a mandatory β-annealing step after hot working and before cold rolling.

Starting from D2 (or D1) and having regard to the technical teaching of D10 or D4 and depending on the alloy composition, β-annealing was purely an optional step. It would therefore be obvious to the skilled practitioner to dispense with the intermediate β-annealing step, if it was not needed.

Moreover, documents D2 and D1 disclosed neither the grain size of the precipitates nor the amounts of silicon and oxygen which were specified in claim 1 of the main request.

However, as to the size of the precipitates, the patent specification stipulated clearly and precisely in paragraph [0030] that controlling the accumulative $\sum A$ to 1.0×10^{-18} hr or less resulted in obtaining precipitates with 80 nm in diameter. Hence, the grain size of the precipitates was a direct consequence of $\sum A$. In conclusion, the precipitates having a grain size of 80 nm was not an independent distinguishing feature.

Finally, the effect of silicon and oxygen as β-grain refiners and their influence on the mechanical properties were known to the skilled person for instance from document D4, column 6, lines 53 to 67,

which disclosed the typical ranges for Si and O. Adding Si and O within the claimed amounts would be obvious to the skilled person, all the more so since the patent specification failed to give any detailed reasons as to why the claimed ranges for silicon (80 to 120 ppm) and oxygen (600 to 1400 ppm) should be adhered to.

The subject matter of claim 1 of the main request therefore lacked an inventive step.

VII. The arguments of respondents I and II relevant to the present decision can be summarized as follows:

Clarity; Article 84 EPC

Process claim 1 of the main request was restricted in scope by use of the expression "method *consisting of the steps*" because this wording excluded from the claimed method process steps other than those specified in claim 1. Furthermore, claim 1 defined clearly and unambiguously that the precipitates in the alloy matrix were limited to an average diameter of 80 nm or less for the claimed method. Consequently, a contradiction did not exist between the claims and the description of the patent, contrary to the appellant's doubts. The requirement of Article 84 EPC was therefore met.

Sufficiency of disclosure; Article 100(b) EPC

The numerous exemplifying experiments, which were amply disclosed in the patent specification, described in detail the claimed process and, therefore, could be repeated without problem by the person skilled in the art. As regards the β-quenching step in the patent,

which was objected to by the appellant, the typical and commonly known quenching parameters for Zr-alloys were used.

The appellant's argument that claim 1 and the patent specification provided insufficient disclosure because they failed to include a lower limit for the accumulated annealing parameter ∑A was unjustified, since no such lower limit was required. Without the lower limit being explicitly mentioned, the patent specification provided sufficient technical information about the heat-treatments so that a skilled person could carry out the invention. Moreover, the appellant's argument that mild accumulated annealing the Zr-Nb alloys with low values for ∑A would not solve the problem underlying the present patent was unfounded, since no evidence in the form of comparative tests was provided in support of the appellant's argument.

Novelty; Article 100(a) EPC

An essential difference between the process of the prior art and that claimed in the patent resided in the beta-annealing step which was performed in the processes of the prior art D1 or D2 after the hot extrusion and before the subsequent cold rolling. Furthermore, neither D1 or D2 nor any other document provided a basis for restricting the accumulated annealing parameter to ∑A to 1.0×10^{-18} hr or less. Moreover, D1 and D2 did not specify the oxygen content and the silicon content in the Zr-Nb alloy, and both documents were silent on the size of the precipitates. The subject matter of claim 1 of the main request was therefore novel.

Inventive step; Article 100(a) EPC

Starting from document D2 as the closest prior art, the problem to be solved by the patent was to provide a method of preparing tubes or sheet of Zr-Nb alloys having an improved strength and an increased resistance to aqueous corrosion when irradiated to high fluency. Neither D4 or D10 nor any other of the cited documents suggested selecting the claimed alloy composition, the accumulated annealing parameter ∑A and the general principle of dispensing with the beta annealing step between hot extrusion and cold rolling, as required by the claimed process.

The subject matter of claim 1 of the main request therefore involved an inventive step.

Reasons for the Decision

1. The appeal is admissible.

2. Interpretation of claim 1

Claim 1 of the main request has been amended to include the wording "Method for manufacturing... , *consisting of* the following steps:...".

If a claim for a chemical compound or alloy composition refers to it as "consisting of the components A, B and C" by their proportions expressed in percentages, the presence of any additional component is excluded and the percentages add up to 100%. The same strictly

exclusionary view of the phrase "*consisting of*" is taken for claims, which define a method or process consisting of the steps A, B and C in that steps other than those specifically mentioned in the claim are excluded from such a process. Compared to other phrases such as "consisting essentially of" or "comprising", the term "consisting of" means in the present case a strict limitation and clarification of the claimed subject matter.

3. Clarity; Article 84 EPC

Turning to the appellant's doubts whether the "maximum" or "average" diameter of the precipitates was actually meant in the patent in issue, the description of the patent merely refers to precipitates with 80 mn in diameter or smaller (patent specification, paragraphs [0027], [0030], [0057]). Claim 1 as granted (as well as claim 1 as originally filed) makes it clear for the skilled reader, however, that the "average diameter of 80 nm or smaller" is to be used when determining the grain size of the precipitates. Hence, a contradiction between the description and the claims does not exist, contrary to the appellant's view.
Moreover, since claim 1 as granted already refers to an average diameter of 80 nm or smaller, this objection does not result from an amendment to the granted claims and therefore cannot be considered with respect to clarity.

The patent specification and the amended claims according to the main request thus meet the requirement of clarity pursuant to Article 84 EPC.

4. Sufficiency of disclosure; Article 100(b) EPC

4.1 Article 100(b) EPC stipulates that the patent shall disclose the invention in a manner sufficiently clear and complete for it to be carried out by a person skilled in the art. Within the meaning of this Article, sufficiency of disclosure is not confined to the claims, but must be assessed on the basis of the application as a whole, including the description, claims and figures - supplemented by the common general knowledge of the person skilled in the art. According to the established jurisprudence of the boards of appeal (Case Law, 6th edition, 2010, II.A.3 b), c), 4.1 and 4.2), the requirements of Article 100(b) EPC are satisfied if it is possible to reproduce the claimed subject matter, i.e. in the present case the claimed process, using the original application documents without any inventive effort over and above the ordinary skills of a practitioner.

According to Rule 42(1)(e) EPC, the description must describe in detail at least one way of carrying out the claimed invention, using examples where appropriate and referring to the drawings, if any.

4.2 In the present case, the description of the patent in suit discloses numerous working examples which describe in detail the claimed process. Specifically, the examples disclose the composition of the Nb-containing alloys which are used for the claimed process and the sequence of steps to be carried out, including the mechanical working steps and the temperature level and duration of the heat-treatments. The examples also disclose the mechanical properties and resistance to

corrosion of the Nb-containing zirconium alloys obtained by the claimed process. Where the patent specification does not provide specific technical details for a step of the claimed process, such as for the beta-quenching step and the carbon content of the Zr-Nb alloy tubes, the skilled practitioner will use the parameters which are typical and commonly known in the technical field of producing Zr-Nb alloy tubes or sheet. With respect to the carbon content, this means that for the alloys used in the claimed process this element represents a residual impurity rather than a compulsory component which is added on purpose.

4.3 The appellant objected to the absence of a lower limit for the accumulated annealing parameter ∑A and of how the desired properties of the alloy could be achieved at low values of ∑A.

It is, however, considered that the sequence of process steps, including the hot working and annealing steps, already limits the window of operation to such an extent that a lower limit is not needed for ∑A. It is also noted in this context that the appellant did not provide any convincing evidence in support of its allegation.

4.4 In conclusion, the patent specification discloses the claimed process in a manner sufficiently clear and complete for it to be carried out by a person skilled in the art and, therefore, meets the requirements of Article 100(b) EPC.

5. Novelty

None of the cited documents discloses all the technical features set out in claim 1 of the main request. Given the strictly exclusionary view of the wording "method consisting of the steps A, B and C" referred to in paragraph 2 of this decision, which was confirmed by the Board during the oral proceedings, novelty of the subject-matter of claim 1 was no longer objected to by the appellant.

6. Inventive step

6.1 The Board concurs with the appellant's assessment that either D1 or D2 could qualify as representing the closest prior art. In contradistinction to the patent in issue, the process disclosed in document D1 requires that an intermediate annealing step is performed in a vacuum furnace after the hot rolling and before the subsequent cold rolling (D1, column 4, lines 12 to 14). Likewise, the process disclosed in document D2 specifies an intermediate annealing step after hot working and before cold working. According to the example given in Table 2 this step is carried out at 649°C/1.5 h (D2, paragraph [0020]; Table 2).

Moreover, neither D1 nor D2 discloses the presence of silicon and oxygen in amounts falling within the claimed ranges in the Zr-Nb alloy and therefore the alloys used in D1 or D2 do not match the compositional requirements of the Zr-Nb alloys used for the claimed process. Furthermore, D1 and D2 do not indicate the average diameter of the precipitates and fail to

provide a basis for selecting the claimed accumulated annealing parameter $\sum A$ to be 1.0×10^{-18} hr or less.

6.2 Starting from the technical teaching given in D2 or in D1, the problem underlying the claimed process resides in providing a method which results in Zr-Nb alloy tubes or sheet which, compared to commercial Zr alloy tubes, exhibit superior corrosion properties and equal or even better mechanical properties, in particular a better tensile strength and creep rate (patent specification, [0063[, [0066]).

This problem is solved by choosing the narrowly defined compositions of the Zr-Nb alloys and performing the process steps specified in claim 1 of the main request, including the omission of the annealing step which is described in D1 and D2 and which for this reason has already a technical effect.

As mentioned above, the crucial difference between the claimed process and D1 or D2 is seen to reside in the intermediate annealing step after hot working and before cold rolling, which is excluded from the claimed process. It is clearly discernible from the context of the prior art D1 or D2 that intermediate annealing is an essential step in the processes disclosed in D1 or D2. Omitting this process step would mean acting against the teaching given in these documents.

6.3 The appellant argued that, according to document D4, column 6, lines 28 to 34 or D10, page 2, lines 56 to 58, the intermediate annealing step was taught as being merely optional which, depending on the alloy composition, could be dispensed with. In the

appellant's view, it would therefore be obvious to the skilled person to omit an optional step which was not needed.

The Board disagrees. It is true that the passage in D4, column 6, lines 28 to 34 referred to by the appellant does not mention an intermediate annealing step. However, the process described in D4, column 7, lines 43 to 51 indicates that the hot extruded hollow tube or coextruded composite or lined hollow tube of the Zr-Nb-Sn alloy is optimally annealed for several hours to approximately 590°C or below, depending on the desired precipitate size, and is then subjected to a series of alternating pilgering and annealing steps at or below 590°C to form a tube. Therefore, document D4, like D2 or D1, teaches away from omitting the intermediate annealing step.

Turning to document D10, it is true that a β-anneal "may" be effected depending upon the alloy (D10, page 2, lines 56 to 58). In practice, however, as document D10 teaches on page 3, lines 27 to 29, the hot extruded hollow tube was β-annealed (step 4) at 954°C for a period of fifteen minutes in preparation of a first cold working step (i.e. before a pilgering reduction, step 5). Reference is also made to the flow diagram of the known process depicted in D10, Figure 1, which specifically provides β-annealing after hot extrusion and before cold working. Since document D10 does not teach the general principle of dispensing with the intermediate β-annealing step, this document also points away from the claimed process.

6.4 The appellant further alleged that omitting intermediate β-annealing in the claimed process did not result in a technical effect on the mechanical and anti-corrosion properties of the final product. However, the appellant did not submit any evidence in support of its allegation.

6.5 Consequently, the technical teaching of D2 (or D1) taken individually or in combination with the teaching given in document D4 or, alternatively, in document D10 would not lead in an obvious way to the claimed process.

Given that, based on this technical difference, the process set out in claim 1 of the main request involves an inventive step over the relevant prior art, there is no need to deal with the remaining technically distinguishing parameters such as the specific contents of silicon and oxygen which are required in the Zr-Nb alloys, the average diameter of the precipitates and the restriction of the accumulated annealing parameter $\sum A$ to 1.0×10^{-18} hr or less.

Order

For these reasons it is decided that:

1. The decision under appeal is set aside.

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

   Claims: 1 to 5 according to the main request filed with letter dated 20 February 2013;

   Description: pages 3 to 7 of the patent specification, pages 2, 8 to 11 filed on 3 November 2011;

   Drawings: Figures 1 to 8 of the patent specification.

The Registrar: The Chairman:

V. Commare T. Kriner