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Datasheet for the decision of 25 October 2011

Case Number: T 2321/09 - 3.2.03

Application Number: 03760334.7

Publication Number: 1519805

IPC: B22F 3/00, B22F 9/16

Language of the proceedings: EN

Title of invention:
Method for preparing metallic alloy articles without melting

Applicant:
GENERAL ELECTRIC COMPANY

Opponent:
-

Headword:
-

Relevant legal provisions:
EPC Art. 54, 56, 83

Relevant legal provisions (EPC 1973):
-

Keyword:
"Novelty (yes)"
"Inventive step (yes)"
"Remittal for consideration inter alia of Article 83 EPC"

Decisions cited:
-

Catchword:
-

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Beschwerdekammern | Boards of Appeal | Chambres de recours

Case Number: T 2321/09 - 3.2.03

D E C I S I O N
of the Technical Board of Appeal 3.2.03
of 25 October 2011

Appellant: (Applicant)
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Decision under appeal:
Decision of the Examining Division of the European Patent Office posted 13 July 2009 refusing European patent application No. 03760334.7 pursuant to Article 97(2) EPC.

Composition of the Board:

Chairman: U. Krause
Members: G. Ashley
K. Garnett

Summary of Facts and Submissions

I. European patent application EP-A-03 760 334 originates from international application WO-A-03/106080 and concerns a method for making metallic articles based on titanium.

II. The claims of the main request were considered by the examining division as being filed late and to contain added subject-matter contrary to Article 123(2) EPC, hence were not admitted into the proceedings. Claim 1 of the first auxiliary request was considered to lack clarity (Article 84 EPC), and to lack novelty (Article 54 EPC). Claim 1 of the second auxiliary request was considered to contain added subject-matter and also to lack novelty.

The examining division thus decided to refuse the application.

III. The decision was posted on 13 July 2009. Notice of appeal was filed by the applicant (here the appellant) on 11 September 2009 and the appeal fee was paid on the same day. A statement containing the grounds of appeal was filed on 18 November 2009.

IV. In accordance with Article 15(1) of the Rules of Procedure of the Boards of Appeal (RPBA), the Board issued a preliminary opinion of the case, together with a summons to attend oral proceedings. In response, the appellant filed with the letter of 23 September 2011 four sets of claims as its main and auxiliary requests.

V. Oral proceedings were held on 25 October 2011.

VI. Requests

The appellant requested that the decision under appeal be set aside and that a patent be granted on the basis of the main alternatively the first, second or third requests. In the event of the main request not being granted, the appellant requested that the case be remitted to the examining division for further prosecution.

VII. Claims

Claim 1 of the main request reads as follows:

"1. A method for preparing an article (20) of a base metal alloyed with an alloying element, comprising the steps of

providing (40) a chemically reducible nonmetallic base-metal precursor compound of a base metal;

providing (42) a chemically reducible nonmetallic alloying-element precursor compound of an alloying element; thereafter

mixing (44) the base-metal precursor compound and the alloying-element precursor compound to form a compound mixture; thereafter

chemically reducing (48) the compound mixture to a metallic alloy powder, without melting the metallic alloy powder, such that it is not macroscopically or

grossly melted so that it liquefies and loses its shape; and thereafter

consolidating (50) the metallic alloy powder material to produce a consolidated metallic article (20), without melting the metallic alloy powder and without melting the consolidated metallic article (20), such that it is not macroscopically or grossly melted so that it liquefies and loses its shape;

wherein the article (20) is made of titanium alloyed with an alloying element, wherein:

the chemically reducible nonmetallic base-metal precursor compound is a compound of titanium base metal, and

the chemically reducible nonmetallic alloying-element precursor compound is a compound of an alloying element that is thermophysically melt incompatible with the titanium base metal."

Dependent claims 2 to 7 concern a preferred embodiment of the method of claim 1.

VIII. Prior Art

The following documents are of relevance for this decision.

WO-A1-00/76698 (D1) is referred to in the decision of the examining division.

US-A-3 909 247 (D2) is cited in the international search report.

US-A-5 779 761 (D4) and US-A-5 958 106 (D5) are cited in the application.

IX. Submissions of the Party

(a) Article 123(2) EPC

The appellant submits that the amendments made to the claims address the objections raised by the examining division under Article 123(2) EPC.

(b) Novelty

Document D1:

The appellant argued that D1 does not disclose the chemical reduction of a mixture of nonmetallic precursor compounds to produce an alloy powder. In the case of titanium, the precursor compound is a hydride, which is thermally decomposed rather than chemically reduced to form the metallic powder. There is also no clear teaching in D1 that melting of the alloy powder should be avoided. For example, the list of elements at the bottom of page 13 of D1 includes copper, which is said to be reduced below 1350°C; this indicates that there is no concern as to whether or not an alloy powder containing copper is melted.

Document D2:

There is no disclosure in D2 of the formation of alloys based on titanium; the example given (example 5) only concerns the making of pure titanium metal. The method of D2 involves the reduction of a cyanide complex to a hydride, which is then decomposed to yield metallic powder. Therefore, as in D1, the step of chemically reducing nonmetallic precursor compounds to produce an alloy powder is not disclosed. In addition, the method of D2 starts with a metal cyanide complex of the metals in question, which the appellant sees as being a single precursor compound rather than a mixture of precursor compounds, as required by claim 1. There is also no requirement in D2 that melting should be avoided, and examples in column 5 include magnesium and aluminium, which could melt under the process conditions described in D2.

Documents D4 and D5:

These documents describe similar processes. The claimed process differs principally in that D4/D5 do not disclose a process in which the initial alloy powder is produced without melting. For example the processes of D4/D5 are said to be suitable for making gallium, which has a melting point of about 30°C and would inevitably melt. There is therefore no preference expressed in these documents as to whether melting does or does not occur.

The appellant submitted that there is no enabling disclosure in D4/D5 for making an alloy. Although Table 1 provides a list of metal halides that can be

used in the process of D4/D5, there is no example of an alloy being made. There is no indication of how to combine the metal halides and produce an alloy whilst avoiding melting, and in particular, there is no indication as to how to integrate thermophysically different elements into the alloy.

At column 5, lines 29 to 32 it is said that the form of metallic material produced is a titanium sponge that requires jackhammering from the collection vessel; this indicates that the resulting material is not a powder, as required by claim 1.

No consolidation step is disclosed for the powder made by the processes of D4/D5, hence the requirement that such a step must take place without melting is also not disclosed.

(c) Inventive Step

The appellant submits that starting from the cited prior art, one of the problems to be addressed is how to produce more reliable larger components.

The features of carrying out the process without melting and the inclusion of thermophysically incompatible elements in the titanium base material has a synergistic effect in improving the properties of articles made from the material.

By avoiding melting, the microstructure present in articles made by the claimed process allows defects to be detected more easily and more reliably. In addition, precipitates such as borides are much finer than if

formed in the liquid phase. It is therefore possible to manufacture larger components more reliably.

Since there is no indication in the prior art of avoiding melting in order to gain these benefits, the method of claim 1 has an inventive step.

Reasons for the Decision

1. The appeal is admissible.

2. Article 123(2) EPC

2.1 The expression "without melting" in claim 1 of the application as originally filed (WO-A-03/106080) is defined further in claim 1 of the main request as taking place so that macroscopic or gross melting such that liquefying and loss of shape does not occur.

2.2 The examining division argued that the application does not disclose a method in which a metallic alloy powder is obtained by chemically reducing the compound mixture such that it is not macroscopically or grossly melted so that it liquefies and loses its shape. The reason given by the examining division is that the latter feature is only referred to in the context of a consolidated article and not for the metallic alloy powder.

2.3 In the second paragraph on page 14 of the application it is said that on completion of reduction (step 48 in Figure 2), the physical form of the metallic material depends on that of the precursor compounds. Thus

starting with precursor powders or vapours leads to metallic material in powder form. The need to avoid melting is emphasised throughout the application, for example at page 5 (last paragraph) and page 15 (lines 1 and 2, and last paragraph). The concept of "without melting" is specified on page 13 (second paragraph) as meaning that the material is not macroscopically or grossly melted so that it liquefies and loses its shape.

The skilled person would thus derive the features of the amendment from the application, and hence the requirements of Article 123(2) EPC are met.

3. Novelty (Article 54 EPC)

3.1 Document D1

3.1.1 Claim 1 relates to the preparation of articles made of titanium together with an alloying element.

3.1.2 D1 describes a process for making metal articles by the conversion of precursor powders that have been formed into the shape of the required article. This occurs either by the reduction of articles made from metal oxide, or by the decomposition of those made from metal hydrides (see page 5, lines 16 to 23).

3.1.3 Articles made from titanium, along with vanadium and zirconium, are only disclosed in D1 as being made from hydride precursors (see page 2, lines 17 to 24; page 12, lines 10 to 16 and claim 14). Such hydride precursors are not converted to the metals by reduction but by heating at a temperature sufficiently high enough to decompose the hydride (page 2, lines 18 to 20).

3.1.4 Since in D1 the precursor powders are formed into an article prior to conversion, and in case of titanium there is no chemical reduction, the claimed step of "chemically reducing the compound mixture to a metallic alloy powder " is not disclosed, and hence the method of claim 1 is novel with respect to D1.

3.2 Document D2

According to the process disclosed in D2, a metallic cyanide complex is formed which is then reduced by hydrogen. In the case of titanium (see Example V in column 6) the product is a hydrided titanium powder which is further subjected to a vacuum and a high temperature to yield the metal. The product of the reduction step is therefore not a metallic powder but a hydride which, as in the method of D1, requires further processing to produce the metal. Since there is no indication in D2 that an alloy powder containing titanium could be made without the intermediate step of forming a hydride, the claimed method is novel with respect to this document.

3.3 Documents D4 and D5

3.3.1 D4 discloses a process in which vapours of metal halides are reduced to pure metals or alloys. In particular, D4 describes a method in which titanium tetrachloride is used as the precursor compound.

3.3.2 On the basis of column 5, lines 29 to 32 of D4, the appellant argues that the method of D4 does not result in a powder, as it is a batch process resulting in a

sponge that requires jackhammering from the sides of the collection vessel. However this is described in D4 in the context of a discussion of the prior art process. The process of D4 is continuous and is said to avoid the problems associated with the known batch process (see column 5, lines 54 to 58). The product of the process of D4 is clearly a powder (see, for example, claims 21, 28 and 30).

3.3.3 The process of D4 involves injecting titanium tetrachloride vapour into a flowing stream of sodium. A reduction reaction takes place which is highly exothermic, resulting in molten reaction products of titanium and sodium chloride. These molten reaction products are quenched in the bulk sodium stream (column 3, lines 49 to 51) and, by varying the process conditions, are maintained below the sintering temperature, ie below the melting point of the produced metal (column 3, lines 61 to 62 and column 4, lines 4 to 6).

3.3.4 D4 also teaches that alloys can be made by providing a suitable mixture of metal halides at the start of the process (column 3, lines 18 to 21; column 6, lines 9 to 13); examples of suitable halides are given in Table 1. The melting point of some of the elements in Table 1 is relatively low (for example, aluminium, antimony and gallium have melting points of about 660°C, 631°C and 30°C respectively). Given that the reduction reaction delivers a substantial amount of heat, the Board agrees with the appellant's submission that there would inevitably be melting of the initial reaction products when producing alloys containing such metals.

3.3.5 The general teaching of D4 is that the titanium produced by the process is in solid form and is maintained well below its sintering temperature. This applies to both the "low temperature" and the "high temperature" embodiments of the process shown in Figures 2 and 3 respectively (see column 4, lines 4 to 6 and column 5, lines 36 to 42).

However, it cannot be said with certainty that in the initial stages of the process of D4 the metal is not melted; in fact the contrary appears to be the case. Since claim 1 requires that an initial metallic alloy powder is produced without melting, this is a novel feature over D4. There is also no disclosure in D4 of a consolidating step, and whilst such a step is inevitable for turning the powder into a useful object, there is no indication that it would be carried out without any significant melting taking place.

The claimed process is thus novel over D4 and also over D5, which discloses a similar process.

4. Inventive Step (Article 56 EPC)

4.1 One of the ways that the method of claim 1 can be put into practice is by vapour-phase reduction, and reference is made in the application to D4 and D5 as providing a fuller description of this approach (see the application, page 3, lines 3 to 5 and page 9, second paragraph). Consequently, the process of D4 (or D5) is a relevant starting point for the assessment of inventive step. The methods of D1 and D2 result in formation of the alloy material via the thermal decomposition of titanium hydride rather than by

chemical reduction, hence are further away from the claimed method than the process of D4.

4.2 As set out above in the section concerning novelty, the claimed method differs from that of D4 principally in that the initial metallic alloy powder produced by the chemical reduction is not melted.

4.3 Starting from D4, the problem to be solved is seen as providing an improved method for making titanium based materials, particularly with the view of reliably manufacturing large components.

4.4 The solution is to prevent melting both during production of the initial alloy material and during consolidation of the powder.

According to the application (see page 2, "summary of the invention") the claimed method provides a convenient way of making alloys of titanium and other elements that thermophysically incompatible, but which enhance the mechanical properties of the alloy. The appellant explained that, for example, borides formed in a process that avoids melting are much finer and have a more uniform distribution than those formed from the liquid phase; this in turn leads to improved strength and fatigue properties.

In addition, articles produced by the claimed invention exhibit significantly reduced noise level during ultrasonic inspection; this allows smaller defects to be detected, or larger articles to be reliably fabricated and inspected. Coarse beta phase formed from molten alloy lead to coarse alpha-phase colonies in the

final material, which give rise to the high background noise level. By avoiding melting, such alpha colonies are prevented; any beta phase that forms via a solid state transformation is much finer and does not mask the presence of small defects.

4.5 None of the available prior art documents addresses the objective problem, and none suggest the avoidance of melting as a means for improving the properties and the ability to detect defects in articles produced by a chemical reduction process. The method of claim 1 of the main request therefore has an inventive step.

5. Articles 83 EPC

5.1 The application makes it clear that the important aspect of the invention is that there is no melting of the metal. For example, on page 2 the summary of the invention states that

"The present approach permits a uniform alloy to be prepared without subjecting the constituents to the circumstance which leads to the incompatibility, specifically the melting process".

Claim 1 defines the chemical reduction as taking place without melting the metallic alloy powder and consolidating without melting either the alloy powder or the article.

5.2 Vapour phase reduction is cited as a particularly suitable method for carrying out the invention, and reference is made to the process described in documents

D4 and D5 as being appropriate (see page 14, first paragraph of the application).

As set out above (see points 3.3.3 and 3.3.4) the method of D4 aims to produce titanium in solid form, but the reduction of metal halides to metals is highly exothermic, such that the initial reaction products are molten.

5.3 The appellant suggests that the skilled person would adjust the process parameters accordingly, and although this would require some trials, it would be well within the skill of the average practitioner.

However, the Board notes that in D4 the flow of sodium is controlled to maintain the titanium in a solid phase, but nevertheless the reaction generates so much heat that it seems that initial melting of the metal takes place.

Bearing in mind that the invention requires that the metal is never melted, the application provides no explanation as to how melting is prevented in the very early stages of the process when the heat developed by the exothermic reaction is significant. Consequently, it is questionable if the disclosure is sufficiently complete for the skilled person to carry out the invention.

6. Remittal

6.1 The above issue concerning Article 83 EPC arose for the first time during the oral proceedings before the Board, and is of importance in determining whether or not the

patent should be granted. In such a case, it is appropriate for the appellant to have the opportunity to be heard before two instances. The Board therefore agrees to the request of the appellant for the case to be remitted to the examining division for further prosecution.

6.2 If it is found that the invention is sufficiently disclosed, then extensive amendment of the description will be required in order to meet the requirements of Article 84 EPC. Attention is also drawn to application EP-A-03 739 116, which is the subject of appeal T 2395/09. This application is also from the appellant and is directed to a similar process as claimed in the present case, hence the issue of double-patenting may arise.

Order

For these reasons it is decided that:

1. The decision under appeal is set aside.

2. The case is remitted to the examining division for further prosecution.

The Registrar: The Chairman:

D. Hampe U. Krause