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Datasheet for the decision of 26 March 2009

Case Number: T 1781/07 - 3.2.08

Application Number: 03021784.8

Publication Number: 1405927

IPC: C22C 1/04

Language of the proceedings: EN

Title of invention:
Method for preparing cryomilled aluminium alloys and components extruded and forged therefrom

Applicant:
The Boeing Company

Headword:
-

Relevant legal provisions:
-

Relevant legal provisions (EPC 1973):
EPC Art. 54, 56

Keyword:
"Novelty and inventive step (yes) - claims according to the 3rd auxiliary request"

Decisions cited:
-

Catchword:
-

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Case Number: T 1781/07 **-** 3.2.08

D E C I S I O N
of the Technical Board of Appeal 3.2.08
of 26 March 2009

Appellant: The Boeing Company
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Decision under appeal: **Decision of the Examining Division of the European Patent Office posted 02. April 2007 refusing European application No. 03021784.8 pursuant to Article 97(1) EPC 1973.**

Composition of the Board:

Chairman: T. Kriner
Members: R. Ries
A. Pignatelli

Summary of Facts and Submissions

I. The appellant (applicant) lodged an appeal against the decision of the examining division dated 2 April 2007 to refuse European patent application No. 03021784.8. The appeal was received at the European Patent Office on 11 June 2007 and the appeal fee was paid on the same date. The statement setting out the grounds of appeal was received on 10 August 2007.

II. In an official communication, the Board gave its provisional view on the case, in particular with respect to the following prior art:

D1: Z. Lee et al.: "Microstructural evolution of cryomilled nanocrystalline Al-Ti-Cu alloy", Ultrafine Grained Materials II, Edited by Y.T. Zhu et al., TMS, (The Minerals, Metals & Materials Society), February 2002, pages 653 to 659

D2: J. Hoon Choi et al.: "Consolidation behaviour of nanocrystalline Al-5at%Ti alloys synthesized by cryogenic milling", Journal of Alloys and Compounds, 315, (2001), 178 to 186

D3: X. Z. Liao et al.: "Deformation mechanisms at different grain sizes in a cryogenically ball-milled Al-Mg alloy", Ultrafine Grained Materials II, Edited by Y.T. Zhu et al., TMS, (The Minerals, Metals & Materials Society), February 2002, pages 323 to 330

D4: Improvements in Cryomill Processing, B. J. M Aikin and J.J. Juhas, in: Advanced Particulate Materials and Processes, Proceedings of the fifth International Conference on Advanced Particulate Materials and Processes, April 7-9, 1997, Sheraton West Palm Beach, Metal Powder Industries Federation, 105 College Road East, Princeton, New Jersey 08540-6692 USA, pages 287 to 294

III. Oral proceeding before the Board took place on 26 March 2009.

The appellant (applicant) requested that
- the decision under appeal be set aside and
- the patent be granted on the basis of the main request or, alternatively, according to one of the auxiliary requests 1 to 3, all requests filed during the oral proceedings before the Board.

IV. Independent claims 1 and 9 of the main request read as follows:

"1. Use of an aluminum alloy as a rocket propulsion system component, the component comprising a cryomilled alloy made by cryomilling from a base alloy comprising:

89 atomic% to 99 atomic% aluminum;

1 atomic% to 11 atomic% of a secondary metal selected from the group consisting of magnesium, lithium, silicon, titanium, zirconium, and combinations thereof; and

a tertiary metal selected from the group consisting of Be, Ca, Sr, Ba, Ra, Sc, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, Y, Nb, Mo, Tc, Ru, Rh, Pd, Ag, Cd, W, and

combinations thereof, the tertiary metal comprising up to 10 atomic%;

the base alloy having been cryomilled so that at least 0.3 % nitrogen by weight has been added to the alloy;

wherein the alloy has an average grain size of less than 0.5 µm; and

wherein the alloy does not contain any refractory materials, added by intention."

"9. A method of producing a component of a rocket propulsion system, the method comprising the preparation of an aluminum alloy comprising the steps of:

providing a metal powder comprising:

89 atomic% to 99 atomic% aluminum;

1 atomic% to 11 atomic% of a secondary metal selected from the group consisting of magnesium, lithium, silicon, titanium, zirconium, and combinations thereof; and

a tertiary metal selected from the group consisting of Be, Ca, Sr, Ba, Ra, Sc, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, Y, Nb, Mo, Tc, Ru, Rh, Pd, Ag, Cd, W, and combinations thereof, the tertiary metal comprising up to about 10 atomic%;

processing the metal powder by cryomilling such that at least 0.3 weight% nitrogen by weight is added to the metal; and

forming said aluminum alloy into a rocket propulsion system component;

wherein refractory material is not added to the metal during processing."

Claim 1 of the first auxiliary request reads as follows (the differences over claim 9 of the main request in bold letters):

"1. A method of producing a component of a rocket propulsion system, the method comprising the preparation of an aluminum alloy comprising the steps of:
providing a metal powder **consisting essentially of**:
89 atomic% to 99 atomic% aluminum;
1 atomic% to 11 atomic% of a secondary metal selected from the group consisting of magnesium, lithium, silicon, titanium, zirconium, and combinations thereof; and
a tertiary metal selected from the group consisting of Be, Ca, Sr, Ba, Ra, Sc, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, Y, Nb, Mo, Tc, Ru, Rh, Pd, Ag, Cd, W, and combinations thereof, the tertiary metal comprising up to about 10 atomic%;
processing the metal powder by cryomilling **in a liquid nitrogen atmosphere** such that at least 0.3 weight% nitrogen by weight is added to the metal;
removing gaseous components from the cryomilled powder;
consolidating the cryomilled powder into a metallic billet (52); and
extruding the metallic billet (52); wherein refractory material is not added to the metal during processing."

Claims 1 and 9 of the second auxiliary requests differ from claims 1 and 9 of the main request by the addition of the following expressions (in bold letters):

"1. Use of an aluminum alloy as a rocket propulsion system component, the component comprising a cryomilled **tertiary** alloy made by...
... added by intention, **wherein the secondary metal includes at least magnesium."**

"9. A method...preparation of **a tertiary** aluminum alloy.. about 10 atomic%; **wherein the secondary metal includes at least magnesium;**
processing the metal...during processing."

Independent claims 1 and 5 according to the third auxiliary request read as follows:

"1. An aluminum alloy for a rocket component, the alloy apart from impurities comprising:
a base alloy consisting of
1 to 4.6 wt.-% of magnesium;
6 to 9 wt.-% of zinc;
2 wt.-% of copper;
2 wt.-% of cobalt;
0.2 wt.-% of zirconium;
0.2 wt.-% of nickel; and
the reminder being aluminum;
processed by cryomilling whereby at least 0.3 wt.-% of nitrogen is added to the base alloy;
wherein the alloy has an average grain size of less than 0.5 µm; wherein refractory materials are present in amounts less than 0.5 vol.-%."

"5. A method of producing a rocket component, the method comprising the preparation of an aluminum alloy comprising the steps of:
providing a metal powder consisting of

1 to 4.6 wt.-% of magnesium, 6 to 9 wt.-% of zinc, 2 wt.-% of copper, 2 wt.-% of cobalt, 0.2 wt.-% of zirconium, 0.2 wt.-% of nickel, the reminder being aluminum;

processing the metal powder by cryomilling in a liquid nitrogen atmosphere such that at least 0.3 wt.-% of nitrogen is added to the metal and that said alloy has an average grain size of less than 0.5 µm;

removing gaseous components from the cryomilled powder;

consolidating the cryomilled powder into a metallic billet (52); and
extruding the metallic billet; wherein refractory materials are present in amounts less than 0.5 vol.-%."

V. The appellant's arguments are summarized as follows:

None of the cited documents D1 to D4 disclosed the use of the cryomilled Al-alloy composition for producing components of a rocket propulsion system or made it obvious to do so.

The cited documents remained silent about the claimed minimum nitrogen content of at least 0.3 wt.% N that was to be introduced by cryomilling the powder in liquid nitrogen. Moreover, the absence of refractory materials from the powder, or the limitation of their presence to less than 0.5 vol.-%, as set out in claim 1 of all requests, required a particular preparation method to prevent their formation during handling. None of the cited documents disclosed that the cryomilled Al-powder was substantially free of refractory materials.

Independent claims 1 and 9 of the second auxiliary request were limited to a tertiary cryomilled Al-alloy including at least one of the tertiary metals selected from the group defined in these claims and by the condition that the secondary metal included at least magnesium as a compulsory component. By contrast, documents D1, D2 and D4 related to Mg-free Al-alloys whereas D3 was concerned with a binary Al-Mg alloy rather than a ternary Al-alloy as claimed.

Hence, the claimed subject matter of the claims according to the main, first and second auxiliary requests was novel and involved an inventive over the cited prior art.

Reasons for the Decision

1. The appeal is admissible.

2. Main request and auxiliary request 1

2.1 Claim 9 of the main request and claim 1 of the first auxiliary request relate to a "method of producing a *component of a rocket propulsion system*". It is noted that the component referred to in these claims is not specified by its particular shape or design, by the mechanical and physical properties, or by the environment and the function the component is actually provided for in the rocket propulsion system. Hence, this technical feature is non-limitative and, therefore, fails to effect a patentable distinction from structural parts of the prior art which are produced by the same method but are provided for a different use. In consequence thereof, claim 9 of the main request and

claim 1 of the first auxiliary request are essentially concerned with the method steps set out in these claims.

2.2 Document D3 discloses a spray-atomized Al - 8.3 at.% Mg alloy powder (grain size 10 to 40 µm) as a starting material for cryogenic ball milling (CBM). The milling is carried at a rate of 180 rpm for 8 hours in liquid nitrogen added to the mill to maintain complete immersion of the milling media and keeping the temperature at -190°C. Prior to CBM, stearic acid is added to the powder as a process control agent. The powder is then compacted to form small pellets (see D3, page 324, Experimental procedure).

D3 does not mention the addition of refractory materials on purpose. It therefore can be assumed that this requirement of the claimed process is met by D3. It is also noted that the milling (180 rpm/8h in liquid nitrogen) in document D3 essentially corresponds to the parameters (liquid nitrogen, stearic acid as process control agent, 100 rpm/8h) reported for the exemplifying process in the application (see e.g. paragraph [0060]). It is therefore justified to assume that at least 0.3 wt.% of nitrogen is likewise incorporated into the known Al-Mg alloy powder so that this technical feature is also satisfied by D3.

2.3 Document D3 fails to mention the removal of gaseous components from the cryomilled powder (e.g. by degassing the powder) and the extrusion of a metallic billet formed from the powder.

These steps are however known in the art as typical and indispensable for producing a high density structural

component from a powder and are, therefore, without inventive merit. Reference is made in this context to document D1, page 654, Experimental procedure, first paragraph, disclosing the steps of packing a cryomilled Al-Ti-Cu powder into a can to prevent oxidation, eliminating any entrapped gases and stearic acid by degassing the powder in a vacuum, consolidating the powder by HIPping and extruding the article obtained.

In conclusion, the claimed processes amount to nothing more than conventional practice when producing a component from the cryomilled Al-Mg powder disclosed in document D3. Put the other way, the claimed processes are obvious from document D3 taken either individually or, alternatively, in combination with the technical teaching given in document D1.

2.4 The subject matter of claim 9 of the main request and of claim 1 of the first auxiliary request therefore lacks inventive step.

3. Auxiliary request 2

Claim 9 of the second auxiliary request additionally defines that the alloy is (a) a tertiary Al alloy which comprises (b) at least Mg as a compulsory component.

3.1 As to feature (a), claim 9 defines the tertiary component to be present in a range "up to 10 at.%" which means "from 0 at.% to 10 at.%". Given that a lower limit (other than 0 at.%) of the range for the tertiary component is absent, the alloy referred to in claim 9 cannot be clearly and unambiguously distinguished form the known cryomilled Al-8.3 at.% Mg

alloy of D3 which typically comprises at least one of the plethora of listed elements within the impurity level. Hence, the "tertiary" element referred to in the claimed Al-Mg alloy represents merely an optional feature which does not provide a patentable difference to the alloys described in document D3.

3.2 Consequently, claim 1 of the second auxiliary request is not allowable for lack of inventive step of its subject matter either.

4. Third auxiliary request

4.1 Claim 1 of the third auxiliary request results from the technical disclosure in paragraph [0059] of the application as published (page 15, lines 10 to 15 of the originally filed application) in combination with the subject matter of claims 1 and 9 as originally filed. Dependent claims 2 to 4 are based on original claims 6 to 8.

Independent claim 5 is based on original claims 9, 11, 12, 15 and 26 in combination with the technical disclosure of the originally filed description, page 15, and lines 10 to 15.
Dependent claims 6 to 14 find support in claims 13, 16, 18, 19, 21, 27 to 30.

Hence, there are no formal objections to the amended claims with respect to Article 123(2) EPC.

4.2 Novelty

None of the documents D1 to D4 discloses the composition of the cryomilled Al alloy defined in claim 1, which is worked in a metallic billet according to the method set out in claim 5. Document D1 discloses a nanocrystalline Al-10%Ti-2%Cu alloy (see D1, page 654, first full paragraph) whereas D2 is concerned with a binary Al-Ti alloy with has been cryomilled in liquid methanol rather than liquid nitrogen (see D2, page 179, experimental details). Document D3 relates to a cryogenic ball-milled binary Al-Mg alloy which does not comprise Zn, Cu and Co as compulsory elements. Document D4 is more remote since it relates to a cryomilled NiAl alloy (see D4, page 287, abstract).

The subject matter of independent claim 1 and 5 is, therefore, novel.

4.3 Inventive step

Starting from the Al-8.3 at.% Mg alloy in document D3 as the closest prior art, the problem underlying the present application resides in providing an Al-Mg alloy that is capable of withstanding the extremely low temperatures and extreme mechanical stress occurring in high performance rocket propulsion systems (see the application as filed, page 3, lines 17 to 20; page 6, lines 15 to 17).

Given that the documents D1, D2 and D4 all relate to cryomilled binary or ternary Al-alloys having compositions different to that given in document D3, none of these documents would provide an incentive for a person skilled in the art to select the composition of the cryomilled Al-Mg alloy defined in claim 1 and

featuring also in claim 5 of the third auxiliary request to solve the above mentioned problem.

Consequently, the subject matter of independent claim 1 and claim 5 of the third auxiliary involves an inventive step.

4.4 Dependent claims 2 to 4 and 6 to 14 are concerned with preferred embodiments of the composition of the Al-alloy defined in claim 1 or of the method set out in claim 5, respectively. Hence, these claims are also allowable.

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 grant a patent with claims 1 to 14 according to the third auxiliary request filed during the oral proceedings before the Board, a description to be adapted to these claims, and Figures 1 to 4 as filed.

The Registrar: The Chairman:

R. Schumacher T. Kriner