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Datasheet for the decision of 6 December 2011

Case Number: T 1278/10 - 3.2.07

Application Number: 03254022.1

Publication Number: 1378587

IPC: C23C 28/02, C23C 4/08, F01D 5/28

Language of the proceedings: EN

Title of invention:
High-temperature articles and method for making

Applicant:
GENERAL ELECTRIC COMPANY

Headword:
-

Relevant legal provisions:
EPC Art. 54, 116(1)
EPC R. 115(2)
RPBA Art. 12(2), 13(1), 13(3), 15(3), 15(6)

Keyword:
"Withdrawal of auxiliary request for oral proceedings four days before scheduled date and at the same time filing an auxiliary request"
"Oral proceedings continued in the absence of the appellant"
"Novelty (single request underlying the impugned decision - no)"
"Late-filed auxiliary request (not admitted)"

Decisions cited:
T 1704/06

Catchword:
-

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Case Number: T 1278/10 - 3.2.07

D E C I S I O N
of the Technical Board of Appeal 3.2.07
of 6 December 2011

Appellant: (Applicant)
GENERAL ELECTRIC COMPANY
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Schenectady
NY 12345 (US)

Representative:
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Decision under appeal:
Decision of the Examining Division of the European Patent Office posted 17 November 2009 refusing European patent application No. 03254022.1 pursuant to Article 97(2) EPC.

Composition of the Board:

Chairman: H. Meinders
Members: H. Hahn
I. Beckedorf

Summary of Facts and Submissions

I. The applicant lodged an appeal against the decision of the Examining Division to refuse the European patent application No. 03 254 022.1.

II. In this decision the following documents are cited:

D1 = US-A-6 129 991
D3 = US-A-4 198 442
D4 = US-B1-6 306 524
D7 = Thermal Barrier Coatings, Annu. Rev. Mater. Res. 2003, 33, page 397, Figure 11

III. The Examining Division held that claims 1 to 6 of the single request dated 13 May 2009 met the requirements of Rule 43(2) and of Articles 82, 84, and 123(2) EPC. It further considered, however, that the subject-matter of the independent claims 1 and 6 lacked novelty over each of the disclosures of D1, D3 and D4.

IV. With its grounds of appeal dated 26 March 2010 the appellant requested to set aside the decision and to grant a patent on the basis of claims 1-6 of the single request underlying the impugned decision, i.e. as filed with letter of 13 May 2009. In case that the Board should intend to confirm the decision to refuse, oral proceedings were requested.

V. Independent claims 1 and 6 of the request underlying the impugned decision read as follows:

"1. A coating (104) for protecting an article (100), said coating (104) comprising:

a substantially single-phase coating (104) disposed on a substrate (102), wherein said coating (104) comprises nickel (Ni) and at least 30 atomic percent aluminum (Al), wherein said coating (104) further comprises up to 0.1 atomic percent carbon, up to 0.1 atomic percent boron and a gradient in Al composition, said gradient extending from a first Al concentration level at an outer surface (106) of said coating (104) to a second Al concentration level at an interface (108) between said substantially single-phase coating (104) and said substrate (102);
wherein said first Al concentration level is greater than said second Al concentration level and said second concentration level is at least 30 atomic percent Al."

"6. An article (100) comprising:

a metallic substrate (102); and
a substantially single-phase coating (104) according to any one of claims 1 to 5 disposed on said substrate (102)."

VI. With a communication dated 8 August 2011 and annexed to the summons for oral proceedings the Board presented its preliminary opinion with respect to claims 1-6 of the single request as filed with letter of 13 May 2009.

The Board stated, after objections under Articles 84 and 123(2) EPC that the subject-matter of claims 1 and 6 lacked novelty over each of the disclosures of D1, D3 and D4:

"5. Novelty (Article 54 EPC)

The following documents cited in the examination proceedings are considered to be relevant:

D1 = EP-A-1 162 286
D3 = EP-A-1 505 176
D4 = US-A-2003/0062271

The following document is introduced by the Board (a copy is annexed to the communication):

D7 = Thermal Barrier Coatings, Annu. Rev. Mater. Res. 2003, 33, page 397, Figure 11

5.1 D4 discloses the coating of a Rene N5 superalloy substrate (containing 6.2 weight% Al) with a NiAl coating containing 32 weight% Al before an annealing treatment for 100 hours at 2200 °F (corresponding to 1204 °C) resulting - according to the microprobe analysis of Al across the interface of the Rene 5 and the NiAl coating - in a decrease of the Al content in said NiAl coating so that at the interface Rene 5/NiAl the content in the NiAl alloy was 19 weight% Al (due to the diffusion of Al into the Rene 5 substrate the Al content in the substrate increased to 9 weight% Al) which gradually increased to 32 weight% at about 900 µm from said interface (see example 1).

A binary NiAl alloy comprising about 50 atomic % Al is present in a (substantially) single phase (compare the phase diagram provided e.g. in D7 = Thermal Barrier Coatings, Annu. Rev. Mater. Res. 2003, 33, page 397, Figure 11). A binary NiAl alloy containing 32 weight%

Al corresponds to one comprising about 50.6 atomic % Al, while a value of 19 weight% Al corresponds to about 33.8 atomic % Al (this calculation is based on the binary alloy; compare also the phase diagram of the binary alloy in D7, page 397, figure 11).

Consequently, the heat treated NiAl coated Rene 5 substrate appears to meet all the requirements of claims 1 and 6 since it contains Ni and at least about 30 atomic % Al at the interface between the single phase NiAl coating and the substrate and there exists a gradient in the Al composition extending from said interface containing about 33.8 atomic % Al to the outer surface of said coating, the latter having an Al-concentration of about 50.6 atomic %. Hence the example 1 of D4 appears to be novelty destroying for the subject-matter of claims 1 and 6 of the single request. The request is thus not allowable under Article 54 EPC.

5.2 D3 discloses in its example 1 the deposition of a first coating of a first metallic layer onto a nickel-base superalloy (IN738) said first coating having a composition of (in wt.%): 6 Al, 26 Cr, 0.6 Y, 31 Ni and balance Co (36.4 Co). A second metallic layer was then deposited onto the coated substrate having a composition of (in wt.%): 26 Al, 16 Cr, 0.6 Y, 17 Ni and balance Co (i.e. 40.4 Co). The coated article was then heat treated for four hours at 1975 °F (corresponding to about 1080°C) (see example 1). After said heat treatment the (second) outer layer is essentially a single phase (Co,Ni)Al compound containing up to 20 weight % Cr and 0.6 weight% Y while the (first) inner layer is an intimate mixture of (Co,Ni)Al phase and (Co,Ni) solid solution phase (see

column 6, lines 13 to 28), i.e. due to said heat treatment for 4 hours at about 1080°C diffusion of Al from the second layer to the first layer and of Cr and Ni from the first layer into the second layer has taken place. The difference of the Al-concentrations of the (second) outer coating layer and the (first) inner coating layer is 26-6 = 20 weight%. The difference of the Al-concentrations of the NiAl coating (32 weight%) and the underlying Rene 5 alloy substrate (6.2 weight%) according to D4 before the heat treatment (see D4, example 1) is 32-6.2 = 25.8 weight%. The Al-concentration of 6 weight% Al of the said (first) inner layer according to D3 is comparable with the Al-concentration of the Rene 5 alloy of 6.2 weight% according to D4. Taking account of the fact that that the difference of the Al-concentrations according to D3 is smaller than that of D4 (i.e. 20 weight% compared to 25.8 weight%), that the heat treatment temperature is about 120°C lower than that of D4 (i.e. about 1080°C compared to 1204°C), and that the heat treatment period according to D3 is by a factor of 25 shorter than that according to D4 (i.e. 4 hours compared to 100 hours), it is evident that the Al-concentration at the interface between the two layers will be lower than the nominal value at the outer surface of the (second) outer layer but in any case will be higher than 30 atomic % as required by claims 1 and 6 of the single request.

This is due to the fact that the heat treatment according to D4 at the 120°C higher temperature for a by a factor of 25 longer period starts from a 25.8-20 = 5.8 weight% higher difference of the Al-concentrations results in a difference of 13 weight% at the interface

(32 weight% Al in the coating decreased to 19 weight% Al; see D4, example 1). Consequently, there will be considerably less diffusion in the heat treatment according to D3. Therefore it can reasonably be assumed that the decrease of the Al-concentration caused by the heat treatment according to D3 in any case cannot be more than 50% of that according to D4, i.e. 50% of 13 = 6.5 weight%. The nominal composition (in weight%) of 26 Al, 16 Cr, 0.6 Y, 17 Ni and (balance) 40.4 Co of the (second) outer coating layer of D3 is recalculated in atomic % as 13.5 Cr, 43.4 Al, 0.3 Y, 12.7 Ni and 30.1 Co. Reducing the Al content (in weight%) of 26 of this alloy by said value 6.5 results in 19.5 Al which calculates as about 34.6 atomic % Al.

Consequently, also the embodiment according to example 1 of D3 appears to be novelty destroying for the subject-matter of claims 1 and 6. The appellant's argument that the feature "substrate" should be given its normal meaning in this art and therefore would not include the MCrAIY layer cannot be followed since an article having a coating can also be considered to represent the substrate for a second, subsequently applied coating.

5.3 With respect to the MCrAlY alloy of the (second) outer layer according to example 1 of D3, which comprises (in atomic %) 13.5 Cr, 43.4 Al, 0.3 Y, 12.7 Ni and 30.1 Co, it is remarked that an essentially single phase layer is obtained although the Co content is higher than that allowed by the present application (i.e. 20 atomic %).

According to the teaching of the present application the single-phase NiAl coating (104) containing at least 30 atomic % Al can further comprise up to about 15 atomic % Cr, up to about 20 atomic % Co and it can also contain Y for which no upper limit is specified (see application as originally filed, page 4, second paragraph to page 5, second paragraph; and page 6, first paragraph; pages 10 and 11, clauses 2, 3, 7 and 8). Hence the teaching of the present application embraces MCrAlY alloys comprising up to 15 atomic % Cr. Furthermore, according to the teaching of the present application for producing a coating (104) as defined in claim 1 the second coating layer can be reacted with the first coating layer e.g. by heat treating the substrate comprising both coating layers to a temperature of from about 700°C to about 1200°C for about 0.5 hours to about 4 hours, or alternatively by in situ heating during deposition of the second layer which is predominantly aluminum (see page bridging paragraph on pages 6 and 7).

5.4 Taking account of this teaching document the Board interpreted D1 as follows:

According to the general teaching of D1 a superalloy article is first coated with a MCrAlY coating which typically consists essentially of (in weight%) 14-35 Cr, 4-30 Al, 0.1-3 Y, rare earth elements such as Ce and/or La, and/or other reactive element(s) such as Hf, Zr, Si, and the balance essentially Fe and/or Ni and/or Co (see column 2, lines 35 to 53). According to D1 the invention can be practiced "using any other CoCrAlY, NiCrAlY, CoNiCrAlY, and FeCrAlY coating alloys" although for illustration purposes a CoNiCrAlY alloy is

*used (see column 2, lines 54 to 60). Hence D1 implicitly discloses a MCrAlY alloy consisting essentially of (in weight %) 14-35 Cr, 4-30 Al, 0.1-3 Y and the balance Ni. This MCrAlY alloy can be recalculated (in atomic %) as consisting essentially of about **11.4-**36.1 **Cr**, about 8.2**-48.3 Al**, about **0.05-1.8 Y** and balance about 40.3-76.8 Ni. Onto this first MCrAlY coating an aluminide diffusion overcoating 12 is formed in situ by CVD using a low activity aluminium coating gas and substrate coating temperature of at least 1832 °F (1000°C) whereby an outwardly grown, outer single phase additive layer 12a and an inner diffusion zone 12b proximate the MCrAlY coating is formed (see column 3, lines 15 to 58).*

*Taking account of the fact that the lower Cr value of 11.4 atomic % of the NiCrAlY alloy falls within the range of "**up to 15 atomic % Cr**" according to the present application it is clear that the in situ coating with metallic Al at 1000°C should result in a single phase coating for all those NiCrAlY alloys which comprise at least 30 atomic % Al, i.e. there exists an overlap for those **NiCrAlY** alloys according to D1 which comprise **11.4-15 atomic % Cr and 30-48.3 atomic % Al** and which inherently will have the Al-gradient between the interface of the substrate and the innermost NiCrAlY coating and the outermost Al-coating layer as required by claim 1. Therefore the conclusion of the Examining Division with respect to D1 seems to be correct, as well."*

The appellant was informed that any observation to this communication should be filed well in advance, i.e. at least one month, before the date of the oral

proceedings in order to give sufficient time to the Board to prepare for the oral proceedings.

VII. With letter dated 2 December 2011 submitted by fax on the same date the appellant filed an auxiliary request comprising claims 1-5 in combination with arguments why the amendments made therein overcome the objections under Articles 84 and 123(2) EPC raised in the Board's communication annexed to the summons.

Furthermore, the appellant remarked that the arguments supplied with the grounds of appeal with respect to D1, D3 and D4 were maintained for the auxiliary request. Finally it stated "*Applicants do not intend to be represented at the oral proceedings. The applicants also withdraw their request for oral proceedings*".

VIII. Independent claims 1 and 5 of this new auxiliary request read as follows (amendments compared to claims 1 and 6 of the single request underlying the impugned decision are in bold; emphasis added by the Board):

"1. A coating (104) for protecting an article (100), said coating (104) comprising:

a substantially single-phase coating (104) disposed on a **metallic** substrate (102), wherein said coating (104) comprises nickel (Ni) and at least 30 atomic percent aluminum (Al), wherein said coating (104) further comprises up to 0.1 atomic percent carbon, up to 0.1 atomic percent boron**, at least one of chromium (Cr), zirconium (Zr), up to 20 atomic percent cobalt (Co), and up to 20 atomic percent iron (Fe),** and a gradient in Al composition, said gradient extending from a first

Al concentration level at an outer surface (106) of said coating (104) to a second Al concentration level at an interface (108) between said substantially single-phase coating (104) and said substrate (102); wherein said first Al concentration level is greater than said second Al concentration level and said second concentration level is at least 30 atomic percent Al."

"**5**. An article (100) comprising:

a metallic substrate (102); and
a substantially single-phase coating (104) according to any one of claims 1 to **4** disposed on said substrate (102)."

IX. Oral proceedings were held as scheduled on 6 December 2011. As announced with its fax dated 2 December 2011 the appellant did not appear so that the oral proceedings were continued in its absence in accordance with Rule 115(2) EPC and Article 15(3) RPBA. At the end of the oral proceedings the Board announced its decision.

Reasons for the Decision

1. *Oral Proceedings (Rule 115(2) EPC and Article 15(3) RPBA)*

With its fax dated Friday 2 December 2011 the appellant withdrew its auxiliary request for oral proceedings just four days (in which a weekend was comprised) before the date arranged for oral proceedings before

the Board and stated that it did not intend to attend the same (see point VII above).

1.1 In the present case, however, the Board considered oral proceedings to be expedient in accordance with Article 116(1) EPC so that the scheduled date for oral proceedings was maintained at which the appellant - as announced in its fax - was not present. As is consistent case law (see Case Law of the Boards of Appeal, 6th edition 2006, VI.C.2.2) the appellant in such a case is considered to rely on its written submissions.

1.2 In this context it is remarked that the principle of the right to be heard pursuant to Article 113(1) EPC is not contradicted since that Article only affords the opportunity to be heard and, by absenting itself from the oral proceedings, a party gives up that opportunity (see the explanatory note to Article 15(3) RPBA in CA/133/02 dated 12 November 2002, quoted in T 1704/06 not published in OJ EPO; see also the Case Law of the Boards of Appeal, 6th edition 2006, VI.B.3 to VI.B.3.2).

2. *Main request dated 13 May 2009*

2.1 In the communication accompanying the summons for oral proceedings the Board, taking account of the appellant's submissions as comprised in the grounds of appeal, raised amongst others objections under Article 54 EPC, explaining why in the Board's opinion the subject-matter of claims 1 and 6 of this request dated 13 May 2009 lacked novelty over each of the disclosures in D1 (interpreted in the light of D7), D3 or D4 (see point VI above).

2.2 The appellant did not reply in substance to any of these three novelty objections against claims 1 and 6 of this request underlying the impugned decision (see point VII above).

Since there has been no attempt by the appellant to refute or overcome the novelty objections raised in the above communication, the Board, having considered all facts and legal issues concerned once again, sees no reason to depart from its preliminary opinion expressed therein.

2.3 With regard to the above, the Board concludes - for the reasons set out in the communication (see point VI above) - that the subject-matter of claims 1 and 6 of the single request dated 13 May 2009 lacks novelty over each of the disclosures of D1 (in the light of the disclosure of D7), D3 and D4 (Article 54 EPC).

The Board thus principally confirms the Examining Division's decision concerning lack of novelty of claims 1 and 6 of the single request.

3. *Admissibility of the auxiliary request*

3.1 The auxiliary request was filed by faxed letter dated 2 December 2011 by the appellant, i.e. four days before the date of the oral proceedings before the Board.

3.1.1 The subject-matter of claim 1 of this auxiliary request differs from that of claim 1 of the main request in that the substantially single-phase coating, which is now on a metallic substrate, additionally requires the

presence of at least one of chromium and/or zirconium in unspecified amounts, whereas the two now also specified components cobalt and iron, due to their definition "up to 20 atomic percent" (which includes the value zero) as used in this claim 1 (see point VIII above) only represent optional and thus non-limiting components of the claimed substantially single-phase coating.

3.1.2 The letter of 2 December 2011 accompanying this request contained **neither** a justification for the late filing of this request, e.g. an explanation as to why the appellant was not in a position to file this request in due time as set out in the Board's communication annexed to the summons to oral proceedings (see point VI above), **nor** any reasoning as to why these amendments addressed the issue of novelty in view of documents D1, D3 and D4.

To the contrary, it was stated therein "*The arguments concerning novelty and the prior art of documents D1, D3 and D4 previously provided are maintained for the claims of the Auxiliary Request.*"

In this letter, however, the appellant only explained why the amendments carried out in the claims 1-5 of the auxiliary request complied with Articles 84 and 123(2) EPC (see point VII above), without addressing the issue of novelty. In this respect the auxiliary request is not substantiated with respect to this issue.

3.2 According to Article 12(2) RPBA the statement of grounds of appeal must contain the party's complete case and any amendment to a party's case may be

admitted and considered at the Board's discretion which shall be exercised in view of the complexity of the new subject-matter submitted, the current state of the proceedings and the need for procedural economy in accordance with Article 13(1) RPBA. Furthermore, according to Article 13(3) RPBA, amendments made after oral proceedings have been arranged cannot be admitted if they raise issues which the Board or other party or parties cannot reasonably be expected to address without an adjournment of the oral proceedings.

3.3 According to consistent case law (see Case Law of the Boards of Appeal, 6th edition 2006, VII.E.16.3.2) it is permissible under Article 13(3) RPBA to regard as belated auxiliary requests filed after oral proceedings have been arranged even if filed before the ultimate date set by the Board, if those requests are not substantiated, i.e. if they were not accompanied by reasons explaining why the amendments had been made and how they were intended to overcome the objections raised (by the Board) in the course of the proceedings. In such cases, neither the board nor the other parties to the proceedings could reasonable be expected to consider these points.

3.3.1 This conclusion holds all the more true, as in the present proceedings, if such an unsubstantiated request is filed only shortly before the oral proceedings. In this respect an ex-parte is not different from an inter-partes case.

3.3.2 In this context it is further remarked that it is not the Board's task to consider how the amendments made in such an unsubstantiated auxiliary request might overcome the novelty objections raised earlier.

In fact, filing such a request at this late stage is equivalent to requesting a return to written proceedings, when refusing the request on substantive grounds could run counter to observing the right to be heard. This is contrary to the principles established by Articles 13(3) and 15(6) RPBA, that oral proceedings are not necessarily adjourned and that oral proceedings should end with a decision being taken.

3.3.3 By abstaining from the oral proceedings, the appellant also made it impossible for the Board to resolve the outstanding issue and at the same time comply with said principles.

3.4 Taking account of the above the Board, in exercising its discretion in accordance with Article 13(3) RPBA, decides not to admit the auxiliary request into the proceedings.

Order

For these reasons it is decided that:

The appeal is dismissed.

The Registrar: The Chairman:

D. Meyfarth H. Meinders