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**Datasheet for the decision
of 3 March 2022**

Case Number: T 1704/18 - 3.2.03

Application Number: 05256663.5

Publication Number: 1652968

IPC: C23C30/00, C23C28/02,
C23C14/16, F01D5/28

Language of the proceedings: EN

Title of invention:

Coating systems containing beta phase and gamma-prime phase
nickel aluminide

Applicant:

GENERAL ELECTRIC COMPANY

Headword:

Relevant legal provisions:

EPC Art. 54, 56, 123(2)

Keyword:

Novelty - after amendment
Inventive step - (yes)
Amendments - added subject-matter (no)

Decisions cited:

Catchword:



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Case Number: T 1704/18 - 3.2.03

D E C I S I O N
of Technical Board of Appeal 3.2.03
of 3 March 2022

Appellant: GENERAL ELECTRIC COMPANY
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Decision under appeal: **Decision of the Examining Division of the
European Patent Office posted on 30 January 2018
refusing European patent application No.
05256663.5 pursuant to Article 97(2) EPC.**

Composition of the Board:

Chairman C. Herberhold
Members: B. Miller
N. Obrovski

Summary of Facts and Submissions

- I. The appeal lies from the decision of the examining division to refuse European patent application No. 05 256 663.5 ("the application") entitled "Coating systems containing beta phase and gamma-prime phase nickel aluminide".
- II. The following prior art was cited by the examining division:
- D1: US 4,758,480;
 - D2: M.J. Pomeroy, "Coatings for gas turbine materials and long term stability issues", *Materials and Design*, vol. 26, 2005, pages 223 to 231;
 - D3: EP 0 784 104 A1;
 - D4: US 2005/118453 A1;
 - D5: A. Taylor et al., "The constitution of nickel-rich alloys of nickel-chromium-aluminum system", *Journal of the Institute of Metals*, vol. 81, pages 451 to 464, and plates LXVII-LXXXII;
 - D6: J.H. Chen et al., "Degradation of the platinum aluminide coating on CMSX4 at 1100°C", *Surface and Coatings Technology*, vol. 92, pages 69 to 77;
 - D7: "Chapter 5: Phase and Structural Changes in Coatings during High-Temperature Tests" in: Y. Tamarin, "Protective Coatings for Turbine Blades", 2002, ASM International, pages 87 to 92;

D8: R. Tiwari et al., "Cavitation-Erosion of Plasma Sprayed Nickel Aluminides", Proceedings of the 5th National Thermal Spray Conference, 7-11 June 1993, pages 423 to 428.

- III. In its decision, the examining division held that the subject-matter of claims 1 and 3 of the request filed by a letter of 21 November 2017 lacked novelty (Article 54 EPC) over D7.
- IV. The applicant ("the appellant") filed an appeal against this decision in due form and time. It requested that the decision be set aside and that the application be granted on the basis of the claims as taken into account by the examining division (main request), or alternatively on the basis of the first or second auxiliary request, both filed with the statement setting out the grounds of appeal.
- V. With the summons to oral proceedings, the Board sent a communication pursuant to Articles 15(1) and 17(2) RPBA 2020 indicating to the appellant its preliminary opinion on the case, namely that claim 1 of the main request lacked novelty with regard to D7 but that the claims of the first auxiliary request were considered to be novel and to involve an inventive step.
- VI. By a letter received on 7 December 2021, the appellant withdrew its former main request and filed a new main request ("main request of 7 December 2021"), which was based on the claims of the first auxiliary request as filed with the statement setting out the grounds of appeal.
- VII. Wording of the claims of the "main request of 7 December 2021" underlying this decision

Claim 1 reads as follows:

"A coating system (20) on a surface region of a gas turbine engine component (22), the coating system (20) comprising an intermetallic overlay coating (24) containing beta and gamma-prime nickel aluminide intermetallic phases characterized in that the intermetallic overlay coating (24) consists essentially of 10 to 85 volume percent of the gamma-prime phase, and the balance the beta phase and in that the intermetallic overlay coating (24) comprises, by weight, at least 14% aluminum, and wherein the intermetallic overlay coating is on a substrate of said gas turbine engine component (22)."

Claim 3 relates to:

"A process of forming the coating system (20) according to any one of claims 1 or 2, the process comprising: depositing nickel and aluminum on the substrate (22) to form a preliminary coating having a preliminary aluminum content and containing the beta nickel aluminide intermetallic phase; and then heat treating the substrate (22) and the preliminary coating to sufficiently diffuse aluminum from the preliminary coating into the substrate (22) to form the intermetallic overlay coating (24) and the gamma-prime nickel aluminide intermetallic phase thereof, wherein the intermetallic overlay coating (24) has a lower aluminum content than the preliminary aluminum content of the preliminary coating and contains a greater amount of the gamma-prime nickel aluminide intermetallic phase than the preliminary coating."

Dependent claims 2 and 4 define preferred embodiments of the coating system according to claim 1 and the process according to claim 3.

VIII. The appellant's arguments with regard to the main request may be summarised as follows:

The subject-matter of claims 1 to 4 was novel over D7 because claim 1 required the intermetallic overlay coating to be on a substrate of the gas turbine engine component, whereas in D7 it was on top of a coating in the middle layer which was on top of a zone of interaction with the substrate.

The original coating disclosed in D7 contained 11 wt.% aluminum, whereas the overlay coating obtained after heat treatment contained 17 wt.% aluminum. Thus the aluminum content increased in the process according to D7, contrary to the requirement defined in claim 3.

Therefore the subject-matter of claims 1 to 4 was novel.

The subject-matter of claims 1 to 4 was also inventive over D7 because D7 provided no motivation to modify the coating such that it provided the coating as claimed in claim 1.

Reasons for the Decision

1. Main request - Article 123(2) EPC

Claim 1 is based on claims 1, 2, 6 and 8 as originally filed in combination with the first sentence of the penultimate paragraph of page 3 of the description as filed.

Claims 2 to 4 correspond to claims 7, 9 and 10 as originally filed.

Hence the amendments according to the main request do not extend beyond the application as originally filed.

2. Main request - Article 54 EPC

2.1 Claim 1 - Novelty with regard to D7

2.1.1 D7 discloses in table 5.5 on page 92 (reproduced below) the phase and chemical composition of overlay coatings for turbine blades for an

- original coating (EB)

and coatings obtained after a heat treatment

- at 1100°C for 100 h ("heat treatment a") or
- at 1100°C for 350 h ("heat treatment b") or
- at 1200°C for 1 h 15 min ("heat treatment c").

Table 5.5 Phase and chemical compositions of overlay coatings of Ni-Cr-Al system deposited on JS6U superalloy (after being tested at 1100 °C)

Test conditions	Area analyzed, phase composition	Phase	Chemical composition(a), wt%				
			Ni	Cr	Al	Co(b)	
Original coating (EB)	Averaged coating composition NiAl(60%) + Ni ₃ Al + γ	...	Base	16	11	2.6	
	Coating surface, γ	γ	Base	18	4.5	2.8	
1100 °C, 100 h (Fig. 5.12b)	Phase zone, NiAl + Ni ₃ Al + γ	NiAl	Base	8.5	16.7	0.9	
		Ni ₃ Al	Base	9.0	11.0	0.9	
	Coating middle layer, Ni ₃ Al + carbide	Ni ₃ Al	Base	9.0	11.0	1.9	
		Carbide	...	97	
	1100 °C, 350 h (Fig. 5.12c)	Zone of interaction with alloy, γ' + carbide	γ'	Base	8.2	11.0	2.8
		Coating surface, γ	γ	Base	18.5	4.5	2.8
Coating middle layer, Ni ₃ Al + carbide		Ni ₃ Al	Base	8.0	11.0	3.0	
Zone of interaction with alloy, γ' + carbide		γ'	Base	8.0	11.0	3.5	
1200 °C, 1 h 15 min	Coating surface, NiAl(80%) + Ni ₃ Al	NiAl	Base	4.5	17.0	0.5	
		Ni ₃ Al	Base	7.5	16.0	4.0	
	Coating middle layer, NiAl(40%) + Ni ₃ Al + carbide	Ni ₃ Al	Base	9.5	9.0	2.5	
		γ'	Base	10.0	9.0	7.0	

(a) Yttrium content of the coating was not analyzed. (b) Cobalt diffuses into the coating during annealing.

- 2.1.2 None of the individual coatings nor the overall coatings disclosed in D7 meet the requirements of claim 1, for the following reasons:
- 2.1.3 The original coating (EB) and each phase of the coatings obtained after a heat treatment of 100 h (treatment a) or 350 h (treatment b) at 1100°C albeit from one (the "NiAl Phase" of the "Phase zone, NiAl + Ni₃Al + γ ") all have an aluminum content below 14 wt.%, contrary to the requirement defined in claim 1.
- 2.1.4 The "phase zone, NiAl + Ni₃Al + γ " obtained after treatment a) contains NiAl (β phase) comprising 16.7 wt.% Al, Ni₃Al (γ' phase) and γ phase. However, it is not disclosed in what ratio the phases are present.

Therefore the subject-matter of claim 1 differs from the phase zone obtained by treatment a) in that the intermetallic overlay coating consists essentially of 10 to 85 volume percent of γ' phase, the balance being β phase.

2.1.5 The overlay coating obtained by heat treatment c) comprises a coating surface consisting of 80% of NiAl (β phase) and Ni₃Al (γ' phase) having an aluminum content of 17 wt.%.

The coating surface obtained by heat treatment c) as disclosed in table 5.5 of D7 is not on the substrate but on top of a coating middle layer.

Hence it is not an intermetallic overlay coating on a substrate as required by claim 1.

2.1.6 Also, the complete overlay coating obtained by heat treatment c) does not meet the requirements of claim 1, since it does not consist essentially of β and γ' phase as defined in claim 1. Moreover, it is not disclosed in D7 that a relative increase in the aluminum content from 11 wt.% (see Al content of the original coating EB) to above 14 wt.% (as required by claim 1) takes place during the heat treatment c) due to diffusion of chromium from the coating into the alloy (see D7, paragraph bridging pages 88 and 89).

2.2 Claim 3 - novelty with regard to D7

The surface coating obtained by heat treatment c) comprises 17 wt.% aluminum, whereas the original coating (EB) comprises only 11 wt.% aluminum. Hence the preliminary coating according to D7 contains less aluminum than the final surface coating.

Claim 3 on the contrary requires the intermetallic overlay coating to have a lower aluminum content than the preliminary aluminum content of the preliminary coating, as aluminum diffuses from the preliminary coating into the substrate.

The subject-matter of claim 3 is therefore novel over the process for forming the surface coating by heat treatment c) of D7. The same arguments as for claim 1 apply with regard to the processes for forming the remaining coatings summarised in table 5.5 of D7.

2.3 The examining division had not upheld any further novelty objections in regard to the claims of the main request. The Board agrees with this finding for the following reasons:

2.3.1 D1 discloses in claim 1 a NiCrAlX-type coating comprising 7.5 to 11 wt.% Al to protect the surface of a gas turbine engine component. The coating consists of at least 90 vol.% of γ and γ' phases and contains less than 10 vol.% of β NiAl, see column 3, lines 9 to 14.

The subject-matter of claim 1 (and correspondingly of claim 3) differs from the coating according to D1 at least in that the intermetallic overlay coating consists essentially of 10 to 85 vol.% of γ' phase, the balance being β -phase NiAl.

2.3.2 D2 discloses overlay coatings for gas turbine engines. The coatings comprise $\beta + \gamma'$ aluminide in a γ matrix with a typical composition of 15 - 28 wt.% Cr, 4 - 18 wt.% Al, 0.5 - 0.8 wt% Y, remainder Ni and/or Co, see page 226, left-hand column, section 4.1.

The subject-matter of claim 1 (and correspondingly of claim 3) differs from the coating according to D2 at least in that the intermetallic overlay coating consists essentially of 10 to 85 vol.% of γ' phase, the balance being β -phase NiAl.

2.3.3 D3 discloses in claim 1 a coating that has an integrated composition of from 18 to 24 wt.% Al and from 18 to 45 wt.% Pt. The balance of the composition is interdiffused components of the nickel-base alloy substrate bulk composition. It is further disclosed in column 6, lines 29 to 33 that the β NiAl is not stable if the Al content is below 18 wt.%.

The application discloses on page 8, penultimate line to page 9, line 2 that in a binary system the γ' phase is formed when the Al content is in the range of 15 - 22 wt.%. However, the coating disclosed in D3 is not a binary Al-Ni system.

Thus no direct and unambiguous further conclusions as to the amount and nature of the phases in the coating of D3 can be drawn from the aluminum content alone.

2.3.4 D4 was published after the priority dates of the application and is therefore not state of the art.

2.3.5 D5 discloses in figure 9 that β and γ' phase can be present in NiAl at the same time. However, D5 does not disclose an overlay coating on a turbine element wherein the coating consists of 10 to 85 vol.% of the γ' phase, the balance being the β phase.

2.3.6 D6 discloses in figure 5, with reference to the description on page 74, paragraph 3.2, the formation of β and γ' phase of nickel platinum aluminide. Again, it is not disclosed that the coating consists of 10 to 85 vol.% of the γ' phase, the balance being the β phase.

2.3.7 D8 discloses in table 1 a feedstock of a two-phase nickel aluminide alloy with 20% Al. This feedstock is used to apply a coating to a substrate by vacuum plasma spraying (VPS). A two-phase microstructure consisting of a β NiAl matrix and γ' Ni₃Al is formed *in situ*, see page 424, right-hand column ("Results and Discussion").

The subject-matter of claims 1 and 3 differs from the disclosure of D8 at least in that the coating is applied on the surface of a gas turbine engine element. Furthermore, it is not disclosed that the intermetallic overlay coating consists essentially of 10 to 85 vol.% of γ' phase, the balance being β -phase NiAl.

2.4 In summary, the subject-matter of claims 1 to 4 of the main request is novel.

3. Main request - Article 56 EPC

3.1 The application relates to coatings for protecting gas turbine engine components exposed to high-temperature environments (page 1, lines 1 to 3) and aims at an increased service life of the coatings (page 3, second paragraph).

D7 referred to in point II.4 of the contested decision (regarding lack of inventive step of dependent claims 2 and 4) aims at the same problem and could therefore be regarded as a suitable starting point for assessing inventive step.

3.2 The subject-matter of claims 1 and 3 differs from the coatings "**on**" the substrate disclosed in D7 (which are neither the phase zone of heat treatment a) nor the coating surface of heat treatment c), see above) in that the intermetallic overlay coating consists

essentially of 10 to 85 volume percent of the γ' phase and the balance of β -phase NiAl and in that the overlay coating comprises at least 14 wt.% aluminum.

- 3.3 According to the paragraph bridging page 4 and 5 of the application, the β and γ' -phase nickel aluminide intermetallic overlay coating has a number of advantages over overlay coatings that contain only the β phase or combined γ and γ' phases.

Reactive elements such as zirconium and hafnium have a higher solubility limit in the γ' than in the β phase, enabling significantly greater amounts of reactive elements to be incorporated into an overlay coating to further improve its environmental resistance and strength without undesirably leading to precipitation of reactive elements. These advantages are achieved while retaining advantages associated with the beta phase: superior oxidation resistance and corrosion resistance.

- 3.4 The objective technical problem to be solved in view of D7 can therefore be regarded as being to provide an improved coating system for gas turbine engine components.

- 3.5 D7 does not provide any incentive to provide a coating essentially consisting of β and γ' phase.

The remaining prior art does not provide any hint that an overlay coating as defined in claim 1 provides an improved overlay coating compared with a coating comprising essentially β -phase NiAl either.

3.6 Therefore the subject-matter of claims 1 to 4 is not rendered obvious by the cited prior art, and meets the requirements of Article 56 EPC.

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 with the order to grant a patent on the basis of the following documents:
 - description pages
 - 1, 2, 5, 6, 16 as originally filed
 - 3 as filed with letter dated 18 June 2007 and received on 21 June 2007
 - 3a filed in electronic form on 21 November 2017
 - 3b, 4, 8 filed in electronic form on 7 December 2021
 - 7 filed in electronic form on 20 July 2009
 - 9-15 filed in electronic form on 6 January 2016
 - claims
 - 1 to 4 filed in electronic form on 7 December 2021
 - drawings
 - sheets 1/4 to 4/4 as originally filed.

The Registrar:

The Chairman:



A. Voyé

C. Herberhold

Decision electronically authenticated