(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization
International Bureau

(43) International Publication Date
3 October 2013 (03.10.2013)

WIPO | PCT

(10) International Publication Number
WO 2013/143995 A1

(51) International Patent Classification:
C22F 1/10 (2006.01) *F01D 5/30* (2006.01)
F01D 5/28 (2006.01) *C23C 4/00* (2006.01)

(21) International Application Number:
PCT/EP2013/056028

(22) International Filing Date:
22 March 2013 (22.03.2013)

(25) Filing Language: English

(26) Publication Language: English

(30) Priority Data:
12161539.7 27 March 2012 (27.03.2012) EP

(71) Applicant: **ALSTOM TECHNOLOGY LTD** [CH/CH]; Brown Boveri Strasse 7, CH-5400 Baden (CH).

(72) Inventors: **ETTER, Thomas**; Kornweg 18, CH-5037 Muhen (CH). **MUECKE, Roland**; Dammstrasse 6, CH-5210 Windisch (CH).

(81) Designated States *(unless otherwise indicated, for every kind of national protection available)*: AE, AG, AL, AM, AO, AT, AU, AZ, BA, BB, BG, BH, BN, BR, BW, BY, BZ, CA, CH, CL, CN, CO, CR, CU, CZ, DE, DK, DM, DO, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, GT, HN, HR, HU, ID, IL, IN, IS, JP, KE, KG, KM, KN, KP, KR, KZ, LA, LC, LK, LR, LS, LT, LU, LY, MA, MD, ME, MG, MK, MN, MW, MX, MY, MZ, NA, NG, NI, NO, NZ, OM, PA, PE, PG, PH, PL, PT, QA, RO, RS, RU, RW, SC, SD, SE, SG, SK, SL, SM, ST, SV, SY, TH, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, ZA, ZM, ZW.

(84) Designated States *(unless otherwise indicated, for every kind of regional protection available)*: ARIPO (BW, GH, GM, KE, LR, LS, MW, MZ, NA, RW, SD, SL, SZ, TZ, UG, ZM, ZW), Eurasian (AM, AZ, BY, KG, KZ, RU, TJ, TM), European (AL, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HR, HU, IE, IS, IT, LT, LU, LV, MC, MK, MT, NL, NO, PL, PT, RO, RS, SE, SI, SK, SM, TR), OAPI (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG).

Published:

— *with international search report (Art. 21(3))*

WO 2013/143995 A1

(54) **Title**: METHOD FOR MANUFACTURING COMPONENTS MADE OF SINGLE CRYSTAL (SX) OR DIRECTIONALLY SOLIDIFIED (DS) NICKELBASE SUPERALLOYS

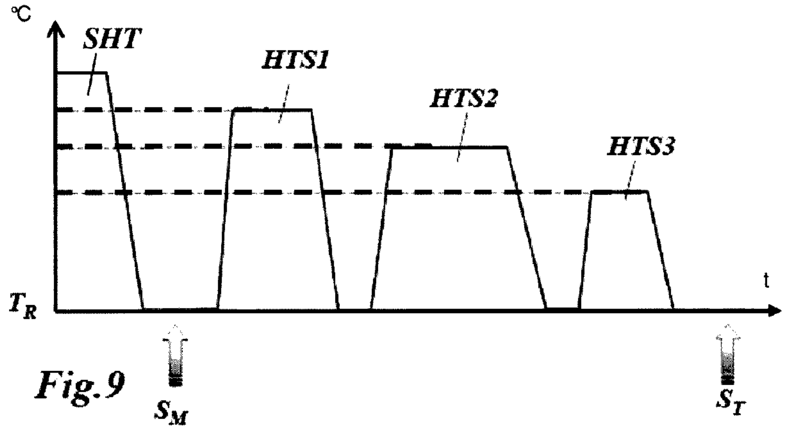

Fig.9

(57) **Abstract**: The invention relates to a method for manufacturing a component, especially of a gas turbine, made of a single crystal (SX) or directionally solidified (DS) nickelbase superalloy, comprising a heat treatment (HTS1-3) and a machining and / or mechanical treatment step (S_M). The ductility of the component is improved by doing said machining and / or mechanical treatment step (S_M) prior to said heat treatment (HTS1-3) and a solution heat treatment (SHT) of the component (11) is done prior to said machining/mechanical treatment step (SM).

DESCRIPTION

METHOD FOR MANUFACTURING COMPONENTS MADE OF SINGLE CRYSTAL (SX) OR DIRECTIONALLY SOLIDIFIED (DS) NICKELBASE SUPERALLOYS

BACKGROUND OF THE INVENTION

The present invention relates to the technology of nickelbase superalloys. It refers to a method for manufacturing a component, especially of a gas turbine, made of a single crystal (SX) or directionally solidified (DS) nickelbase superalloy, according to the preamble of claim 1.

PRIOR ART

The ductility (deformability) of single crystal (SX) and directionally solidified (DS) superalloys is lower than in conventionally cast (CC) parts. In regions of high multiaxiality, the low ductility of SX and DS materials is further reduced (see below).

On the other hand, the thermo-mechanical loading of turbine blades requires a certain degree of ductility (deformability) due to thermal strains and high mechanical loads.

The rupture strain ε_R is a material limit for describing the ductility (deformability) of the material. For a safe design, the rupture strain has to exceed the mechanical strain in the design defined by the sum of the inelastic strain ε_I and the elastic strain ε_E, as shown in Fig. 1.

The rupture strain is influenced by the multiaxiality of the material. For a uniaxial 1D state of stress (see the component 10 in Fig. 2(a)) the Poisson effect leads to a fairly high rupture strain ε_R^{1D} (Fig. 2(b)). A multiaxial 3D state of stress reduces (or even prevents) the Poisson effect, i.e. the deformability of a multiaxial stress state is only obtained by the elastic volume change, Fig. 3(a). Moreover, several damage mechanisms like the growth of creep pores are significantly affected by multiaxiality so that the rupture strain ε_R^{3D} in this case is substantially reduced (Fig. 3(b)).

In literature, the influence of multiaxiality on ductility is described by the stress ratio

(1) $$r = \frac{\sigma_H}{\sigma_{Mises}}$$

where $\sigma_H = \frac{1}{3}(\sigma_{11} + \sigma_{22} + \sigma_{33})$ is the hydrostatic stress and $\sigma_{Mises} = \sqrt{\frac{3}{2}\sigma_{ij}^{dev}\sigma_{ij}^{dev}}$ is the von Mises stress where $\sigma_{ij}^{dev} = \sigma_{ij} - \sigma_H \delta_{ij}$ denotes the stress deviator. The reduction of ductility is then described by the correction factor

(2) $$k(r) = \frac{\varepsilon_R^{3D}}{\varepsilon_R^{1D}},$$

where

(3) $$k(r) = 1.65\exp(-\frac{3}{2}r)$$

according to Rice and Tracey, and

(4) $$k(r) = \frac{\sinh\left(\frac{2}{3}\left(\frac{n-0.5}{n+0.5}\right)\right)}{\sinh\left(2r\left(\frac{n-0.5}{n+0.5}\right)\right)}$$

according to Cocks and Ashby, with $n \to \infty$ for rigid plastic deformation. Both models predict a considerable reduction of the deformability of the material due to multiaxiality (see Fig. 4).

Fig. 5 shows a central part of a gas turbine blade 11, which comprises a root 12, a platform 13 and an airfoil 14. Three different cuts 1-3 through said central part are shown in Fig. 6 with the corresponding distribution of the stress ratio r. As can be seen from Fig. 6, the multiaxiality of thick regions in turbine blades reaches values up to r=1.6. This corresponds to a reduction of the uniaxially measured ductility down to 15% using the Rice & Tracey model and 6% using the Cocks & Ashby model, respectively (Fig. 4).

Considering that the loading of turbine blades (due to pressure and centrifugal loads and non-even temperature distributions) produces mechanical strains in the order of up to 1%, a considerable ductility of the material is required.

The document US 5,451,142 describes a method to provide a layer/coating of a high strength polycrystalline superalloy bonded to the root of a nickelbase superalloy turbine blade. This layer is plasma sprayed onto the fir tree of the blade.

The document US 4,921,405 teaches a single crystal turbine blade having a portion of its attachment section (fir tree) layered with a fine grained polycrystalline alloy. According to the teaching, the layering is preferably accomplished by plasma spraying of the attachment section with a superalloy and hot isostatically compacting the sprayed superalloy to minimum porosity. The resulting turbine blade should have improved life resulting from the reduced low cycle, low temperature fatigue susceptibility of, and crack growth in, the composite attachment section.

In both cases, a special coating process has to be applied during manufacturing of the blade, which requires substantial additional time and cost efforts.

US 4,582,548 describes a single crystal casting alloy for use in a gas turbine engine. Single crystal solid blades or bars were cast and machined in the longitutinal direction. After machining they were solutioned and then pseudocoated and aged. EP 1184473 A2 discloses Nickel-base single-crystal superalloys and a method of manufacturing the same. The method is similar to the one described in US 4,582,548, the solution heat treatment of the speciemen/component and the additional heat treatment steps are done after a machining step.

SUMMARY OF THE INVENTION

It is an object of the present invention to disclose a method for manufacturing components, especially of a gas turbine, made of Single Crystal (SX) or Directionally Solidified (DS) nickelbase superalloys, which results in the necessary strength of the component without causing additional effort.

This and other objects are obtained by a method according to claim 1.

The inventive method for manufacturing a component, especially of a gas turbine, made of a single crystal (SX) or directionally solidified (DS) nickelbase superalloy, comprises a heat treatment and a machining and/or mechanical treatment step. The machining/mechanical treatment step is done prior to said heat treatment, but after a solution heat treatment of the component was done.

The machining step comprises for example a milling step or a grinding step and the mechanical treatment step could be a shot peening.

According to a first embodiment of the invention the heat treatment comprises a plurality of heat treatment steps.

Especially, the heat treatment comprises three heat treatment steps with successively reduced temperatures.

According to another embodiment of the invention said heat treatment steps take place at temperatures below the γ' (gamma prime) solvus temperature of the component material.

According to the further embodiment of the invention selected surfaces of the component are mechanically deformed / treated after the machining step and prior to said heat treatment, that a first heat treatment step at an elevated temperature, but below γ' (gamma prime) solvus temperature is done, that an additional coating is applied to said surfaces, and that a coating diffusion heat treatment step and a precipitation heat treatment step is done thereafter.

BRIEF DESCRIPTION OF THE DRAWINGS

The present invention is now to be explained more closely by means of different embodiments and with reference to the attached drawings.

Fig. 1 shows the rupture strain in a stress-strain diagram;

Fig. 2 shows the uniaxial loading of a component (a) and the corresponding stress-strain diagram (b);

Fig. 3 shows the multiaxial loading of a component (a) and the corresponding stress-strain diagram with its reduced rupture strain;

Fig. 4 shows the reduction of ductility due to multiaxial stress according to 2 different models;

Fig. 5 shows a central part of a gas turbine blade;

Fig. 6 shows the distribution of the stress ratio r in three different cut planes of the blade according to Fig. 5;

Fig. 7 shows an exemplary manufacturing procedure for a gas turbine component according to the prior art;

Fig. 8 shows a micrograph of a body manufactured according to the prior art procedure of Fig. 7;

Fig. 9 shows in a diagram similar to Fig. 7 an embodiment of the manufacturing method according to the present invention;

Fig. 10 shows a micrograph of a body manufactured according to the procedure of Fig. 9 and

Fig. 11 shows the coarse γ/γ’ microstructure with its cellular recrystallisation of the body according to Fig. 10.

DETAILED DESCRIPTION OF DIFFERENT EMBODIMENTS OF THE INVENTION

The present invention is based on investigations comprising tensile tests of specimens made of a nickelbase superalloy, which have seen different combinations of surface and heat treatments. In particular, it was successfully tried to modify the surface in a way that a subsequent heat treatment results in the formation of a ductile layer. That has been achieved by a heat treatment below the γ' (gamma prime) solvus temperature, resulting in a coarse γ/γ' (gamma/gamma prime) microstructure (cellular recrystallisation) in the outermost area.

The impact of surface layer modification on tensile ductility has been observed on SX tensile specimens.

Fig. 7 shows a (prior art) "reference" procedure where a heat treatment T(t) with 3 different heat treatment steps HTS1-3 has been done first on test bars and final machining (machining step S_M) and testing (testing step S_T) of the specimens has been done after heat treatment (specimen Z6 in Table 1).

In contrast, according to Fig. 9, plastic deformation and machining of the final specimen geometry (machining step S_M) has been done before the heat treatment (heat treatment steps HTS1-3) (specimen Z1 in Table 1), but after the solution heat treatment. Thereby, the surface near region, previously affected by plastic deformation and machining (e.g. by cold work hardening, for instance) was modified by the heat treatment.

Table 1

Specimen	Plastic Deformation	Yield strength	Tensile strength	Elongation after fracture
Z6 (acc. Fig.7)	None	966 MPa	1061 MPa	4.3%
Z1 (acc. Fig.9)	0.26%	948 MPa	1299 MPa	13.1%

According to Table 1, significant higher ductility was achieved due to previous surface treatment (plastic deformation) in specimen Z1 compared to specimen Z6. The modified surface layer 17 of specimen 15 (Z1) just below the surface 16 is shown in Figs. 10 and 11. For comparison, the un-affected surface area at the surface 16' of specimen 15' (Z6) is shown in Fig. 8.

The effect of increased ductility on SX components has also been observed on other specimens at room temperature T_R as well as at 600°C even without previous plastic deformation, only due to the specimen machining step S_M.

Table 2 shows the results for 4 different specimen with specimen 1A and 1B having been machined after a heat treatment (HTS1, HTS2, HTS3) procedure according to Fig. 7 while specimen 2A and 2B were machined before a heat treatment HTS1, HTS2, HTS3) procedure according to Fig. 9

Table 2

Specimen	Testing temperature	Yield strength	Tensile strength	Elongation after fracture
1A	23°C	832 MPa	870 MPa	9.1%
1B	600°C	805 MPa	959 MPa	6.4%
2A	23°C	805 MPa	864 MPa	20.9%
2B	600°C	751 MPa	935 MPa	16.3%

Again, significant higher ductility values were achieved in specimens 2A/2B compared to specimen 1A/1B.

A potential heat treatment sequence for increased ductility in the attachment area (fir tree) and/or areas of multiaxiality of a gas turbine blade could be as follows:

a) solution heat treatment of the blade at casting house
b) machining of fir tree
c) mechanical treatment (for example shot peening) of the fir tree and/or inner surfaces of cooling channel
d) heat treatment at elevated temperature, but below γ' (gamma prime) solvus temperature (e.g. during brazing heat treatment)
e) application of an additional coating for the airfoil;
f) coating diffusion heat treatment and precipitation heat treatment.

The characteristics of the present invention are:

- Turbine parts require a sufficient ductility of the material for carrying structural loads.
- SX (or DS) materials have typically a low ductility, which is on the limit for turbine blade applications.
- The SX (or DS) ductility can be improved by changing the sequence of machining and heat treatment.

LIST OF REFERENCE NUMERALS

10	component
11	turbine blade
12	root
13	platform
14	airfoil
15,15'	body
16,16'	surface
17	modified surface layer
HTS1-4	heat treatment step
S_M	machining/mechanical treatment step
S_T	testing step
SHT	solution heat treatment

CLAIMS

1. Method for manufacturing a component (11), especially of a gas turbine, made of a single crystal (SX) or directionally solidified (DS) nickelbase superalloy, comprising a heat treatment (HTS1-3) and a machining and/or mechanical treatment step (S_M), wherein that said machining/mechanical treatment step (S_M) is done prior to said heat treatment (HTS1-3), characterised in that a solution heat treatment (SHT) of the component (11) is done prior to said machining/mechanical treatment step (SM)..

2. The method according to claim 1, characterised in that the heat treatment comprises a plurality of heat treatment steps (HTS1-3).

3. The method according to claim 2, characterised in that said heat treatment steps take place at temperatures below the γ' (gamma prime) solvus temperature of the component material.

4. The method according to claim 2, characterised in that selected surfaces of the component (11) are mechanically deformed / treated after the machining step (S_M) and prior to said heat treatment (HTS1-3), that a first heat treatment step at an elevated temperature, but below γ' (gamma prime) solvus temperature is done, that an additional coating is applied to said surfaces, and that a coating diffusion heat treatment step and a precipitation heat treatment step is done thereafter.

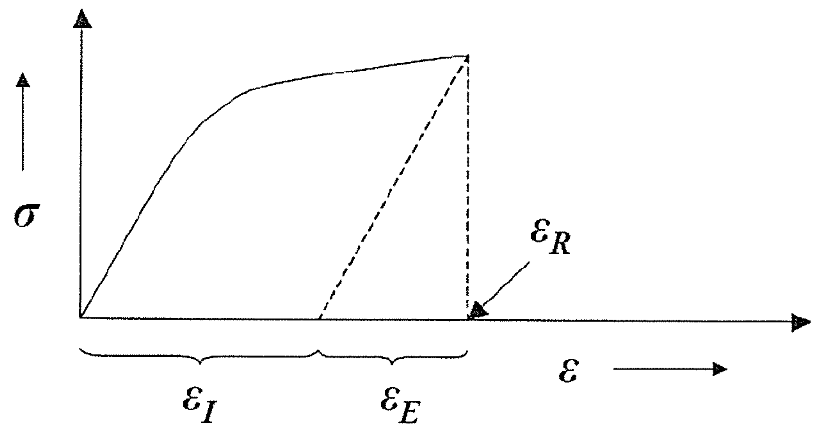

Fig.1

Uniaxial loading

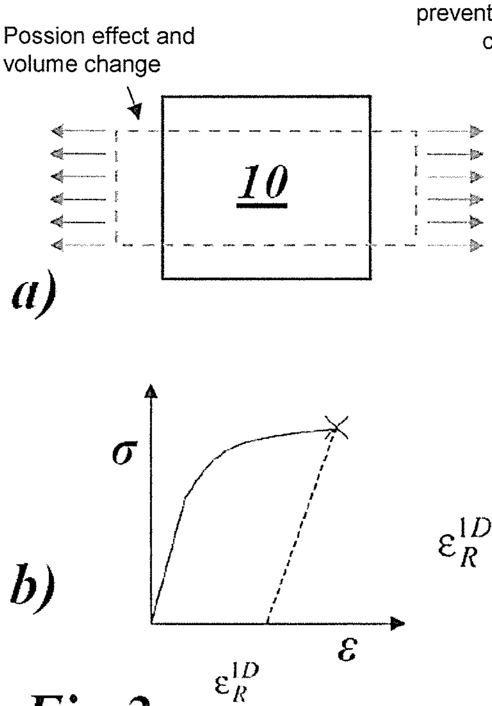

Fig.2

Multiaxial loading

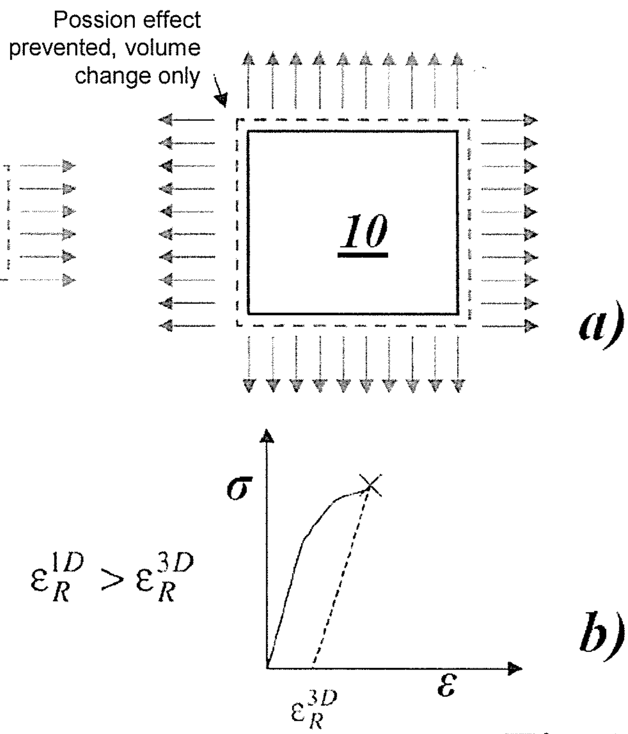

Fig.3

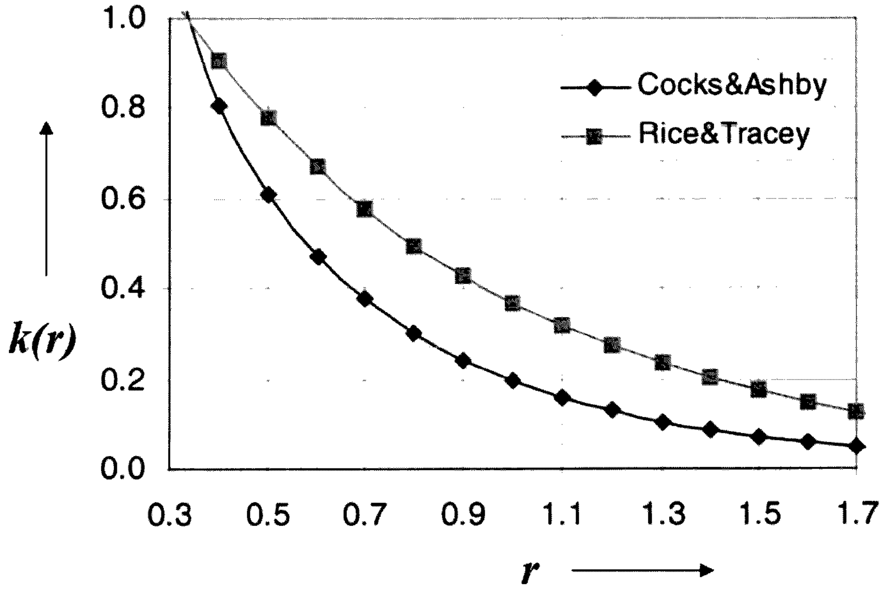

Fig.4

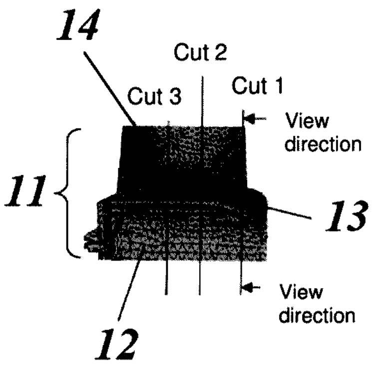

Fig.5

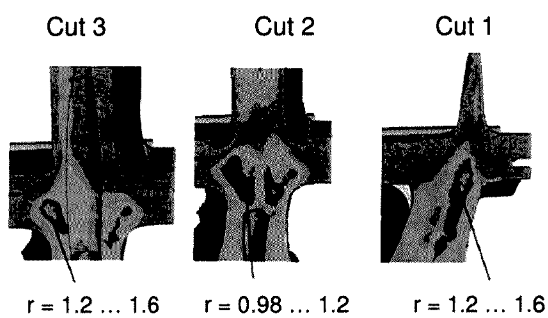

Fig.6

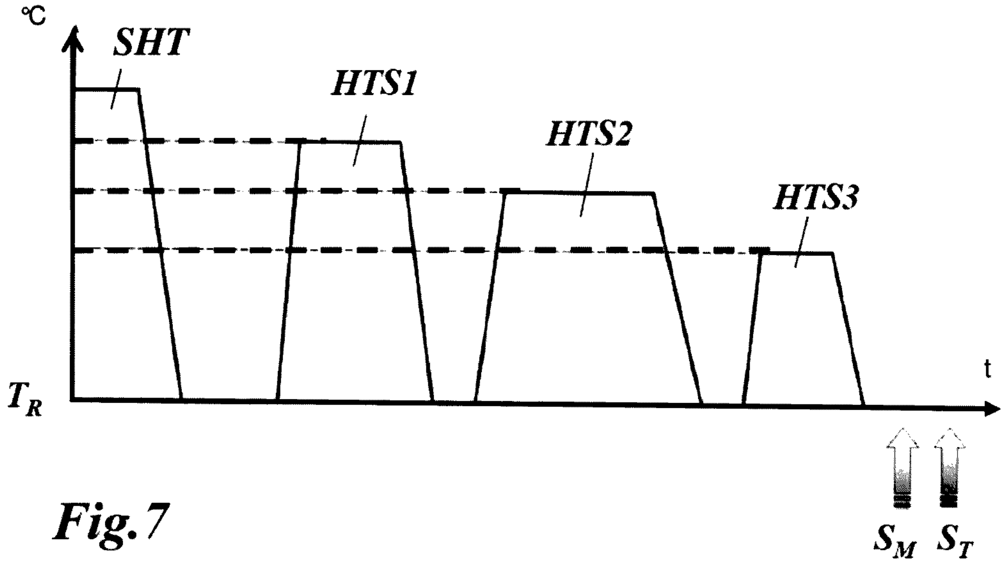

Fig.7

Prior Art

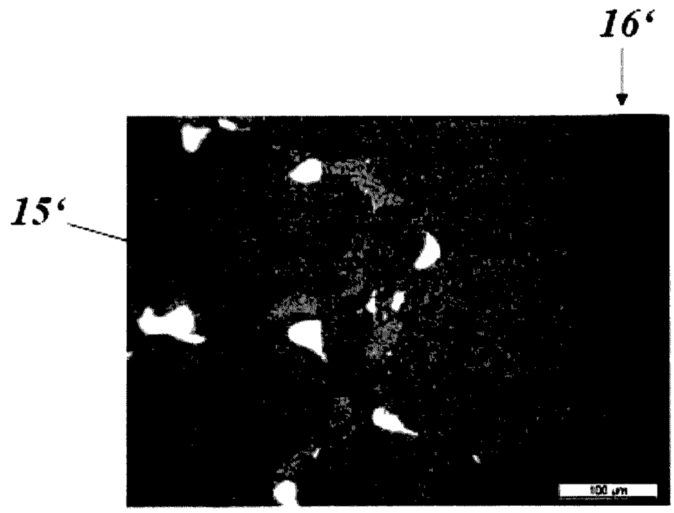

Fig.8

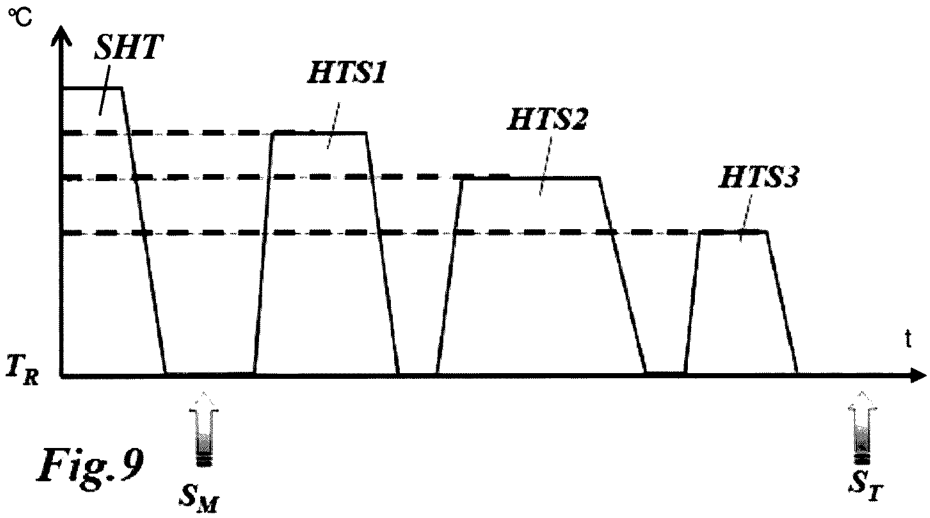

Fig.9

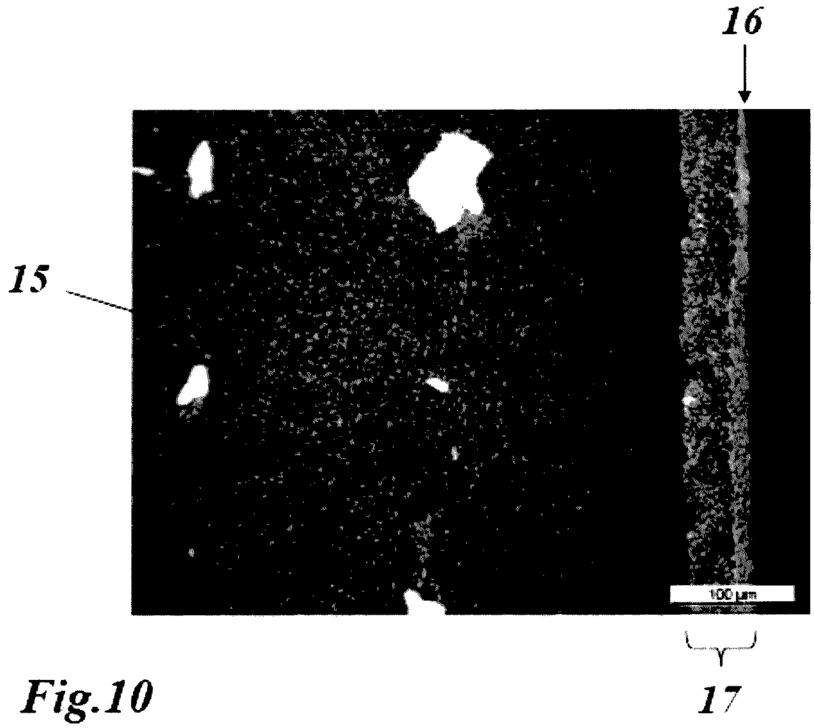

Fig.10

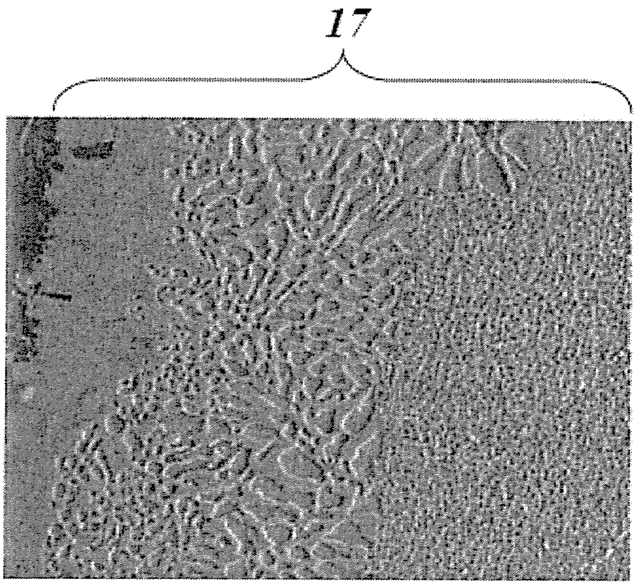

Fig.11

INTERNATIONAL SEARCH REPORT

International application No
PCT/EP2013/056028

A. CLASSIFICATION OF SUBJECT MATTER
INV. C22F1/10 F01D5/28 F01D5/30 C23C4/00
ADD.

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
C22F F01D C23C

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

EPO-Internal, WPI Data, INSPEC

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X Y	US 4 582 548 A (HARRIS KENNETH [US] ET AL) 15 April 1986 (1986-04-15) column 6 - column 7; example II -----	1-3 4
Y A	EP 1 184 473 A2 (TOSHIBA KK [JP]; INDP ADMINISTRATIVE INST NIMS [JP]) 6 March 2002 (2002-03-06) paragraphs [0038] - [0043] -----	4 1-3
A	US 5 451 142 A (CETEL ALAN [US] ET AL) 19 September 1995 (1995-09-19) cited in the application column 5 - column 8 -----	1-4
A	US 4 921 405 A (WILSON LLOYD W [US]) 1 May 1990 (1990-05-01) the whole document ----- -/--	1-4

[X] Further documents are listed in the continuation of Box C.

[X] See patent family annex.

* Special categories of cited documents :

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier application or patent but published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
15 April 2013	24/04/2013

Name and mailing address of the ISA/	Authorized officer
European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Fax: (+31-70) 340-3016	Chebeleu, Alice

INTERNATIONAL SEARCH REPORT

International application No
PCT/EP2013/056028

C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	EP 0 246 082 A1 (GARRETT CORP [US]) 19 November 1987 (1987-11-19) claims 8-10 -----	1-4

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No
PCT/EP2013/056028

Patent document cited in search report		Publication date	Patent family member(s)			Publication date
US 4582548	A	15-04-1986	NONE			
EP 1184473	A2	06-03-2002	DE	60108212	D1	10-02-2005
			DE	60108212	T2	08-12-2005
			EP	1184473	A2	06-03-2002
			US	2002062886	A1	30-05-2002
US 5451142	A	19-09-1995	NONE			
US 4921405	A	01-05-1990	CA	1298207	C	31-03-1992
			EP	0367958	A1	16-05-1990
			US	4921405	A	01-05-1990
EP 0246082	A1	19-11-1987	CA	1315572	C	06-04-1993
			DE	3767557	D1	28-02-1991
			EP	0246082	A1	19-11-1987
			JP	2782340	B2	30-07-1998
			JP	S62267440	A	20-11-1987
			US	4935072	A	19-06-1990