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ETTER et al.(10) **Pub. No.: US 2014/0053956 A1**(43) **Pub. Date: Feb. 27, 2014**(54) **METHOD FOR MANUFACTURING A
THREE-DIMENSIONAL ARTICLE****Publication Classification**(71) Applicant: **ALSTOM Technology Ltd**, Baden (CH)(72) Inventors: **Thomas ETTER**, Muhlen (CH); **Julius
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(57) **ABSTRACT**

The disclosure refers to a method for manufacturing a three-dimensional article, the method including successively building up the article from a metallic base material by means of an additive manufacturing process, thereby creating an article with a substantial anisotropy of its properties and heat treating the manufactured article at a sufficiently high temperature to reduce the anisotropy significantly by recrystallization and/or grain coarsening.

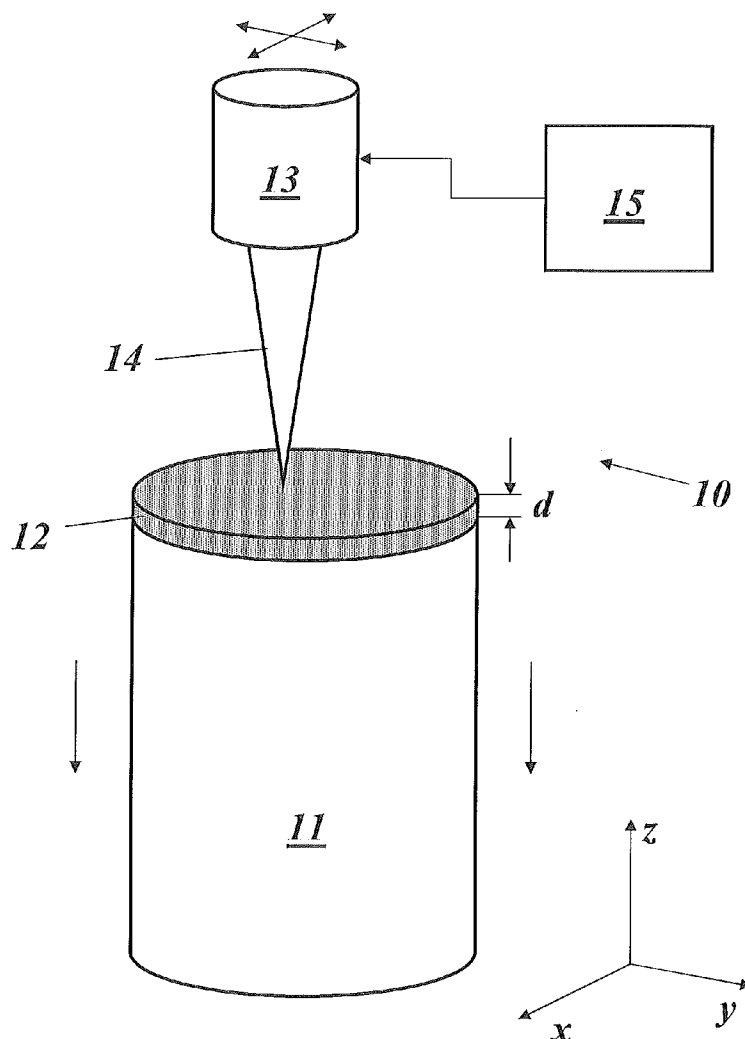


Fig.1

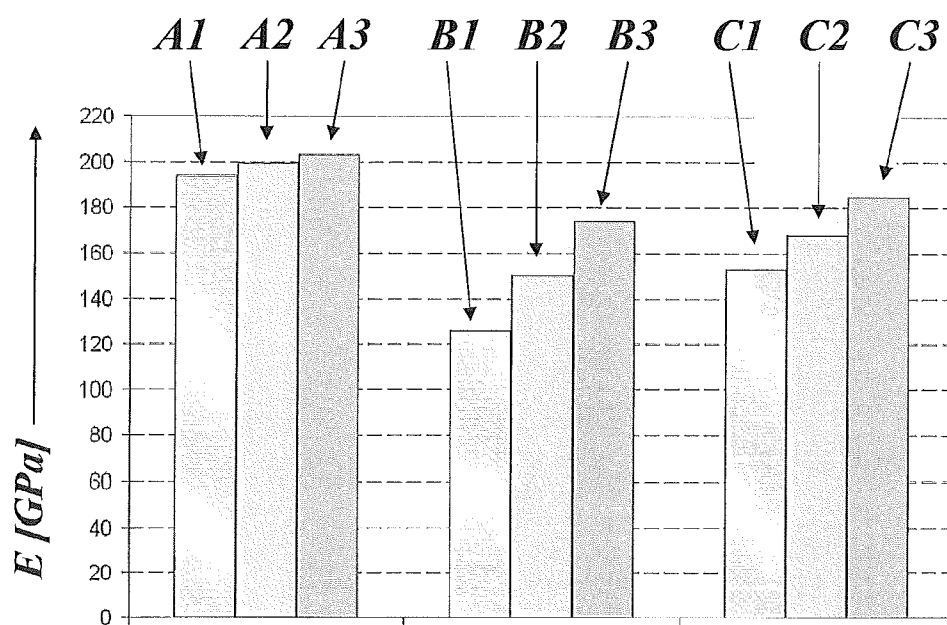
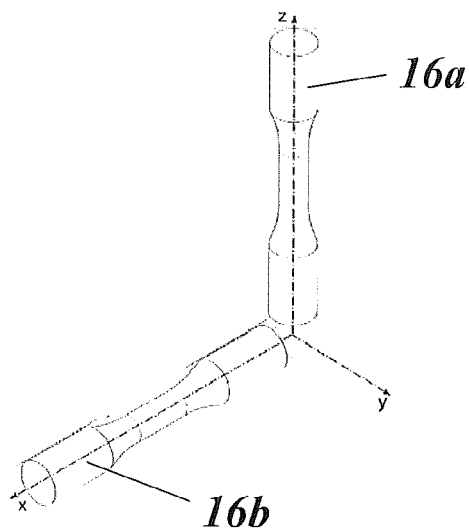


Fig.2

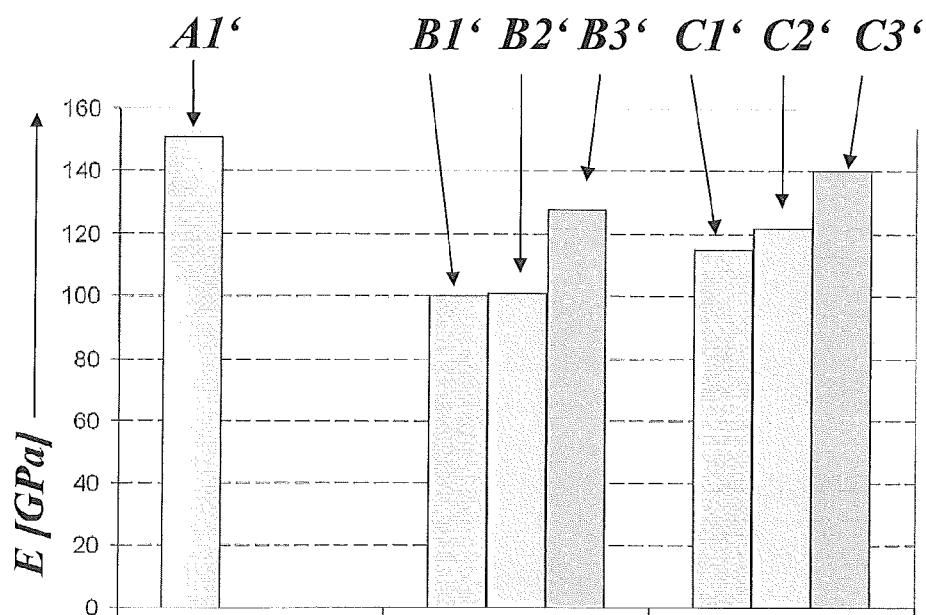
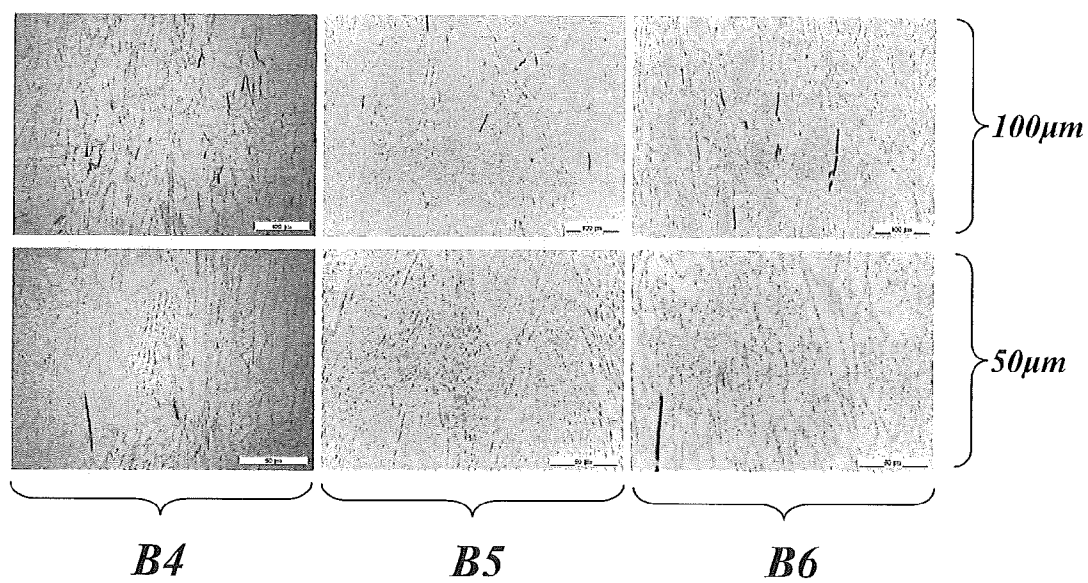


Fig.3

Fig.4



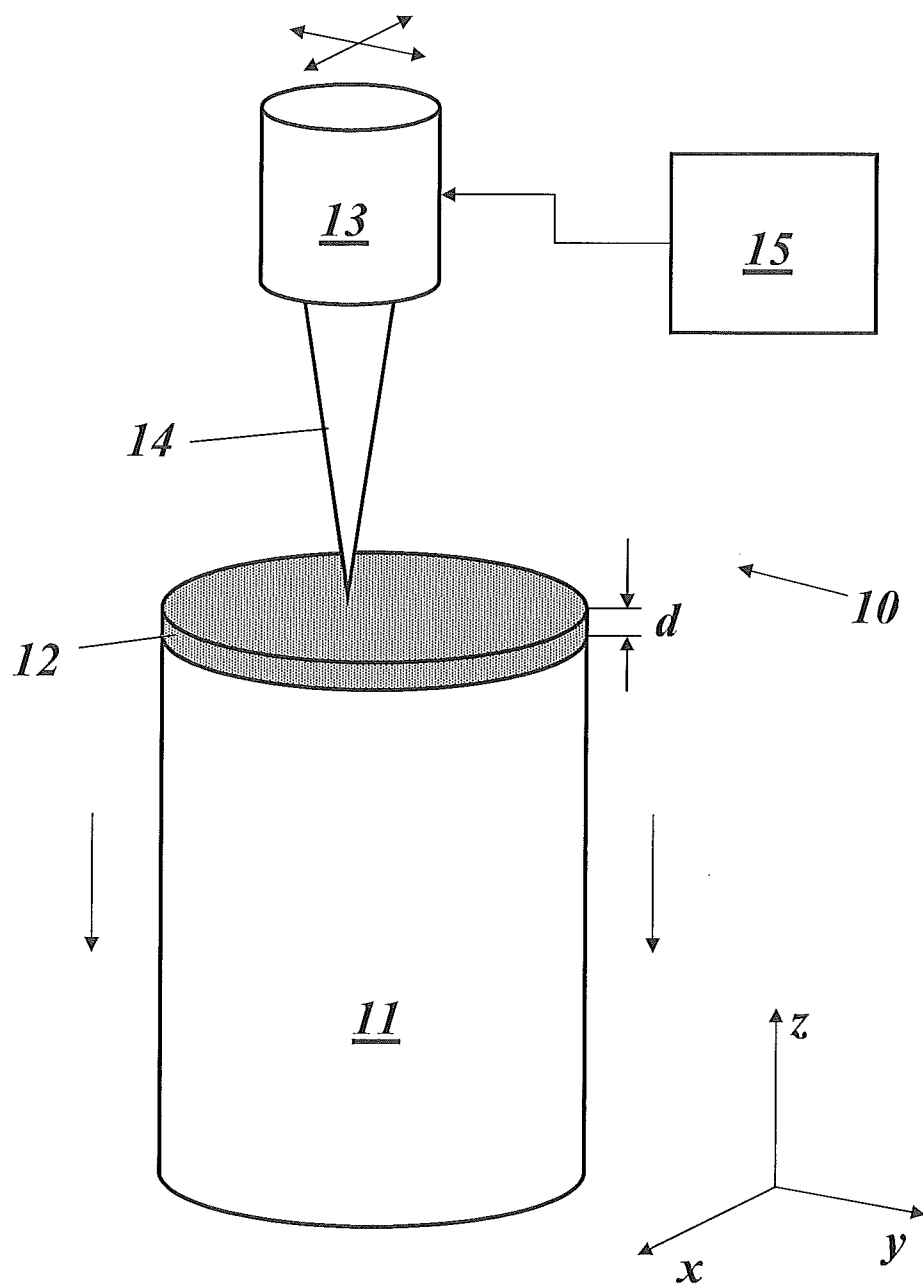


Fig. 5

METHOD FOR MANUFACTURING A THREE-DIMENSIONAL ARTICLE

CROSS-REFERENCE TO RELATED APPLICATIONS

[0001] This application claims priority to European Application 12181126.9 filed Aug. 21, 2012, the contents of which are hereby incorporated in its entirety.

TECHNICAL FIELD

[0002] The present invention relates to the technology of high-temperature resistant components, especially for gas turbines. It refers to a method for manufacturing a three-dimensional article.

BACKGROUND

[0003] The mechanical properties of articles or components, which are for example used in the high temperature environment of gas turbines, and which are manufactured by a powder-based additive manufacturing process such as selective laser melting (SLM), show in the “as-built” condition a strong anisotropic behaviour.

[0004] FIG. 5 shows a basic SLM arrangement 10, wherein a 3-dimensional article 11 is manufactured by successive addition of powder layers 12 of a predetermined layer thickness d, area and contour, which are then melted by means of a scanned laser beam 14 from a laser device 13 and controlled by a control unit 15.

[0005] When—as shown in FIG. 5—the powder layers 12 are added in z-direction, a first specimen 16a extending in z-direction (see FIG. 1) shows properties different from a second specimen 16b extending in the xy-plane.

[0006] FIG. 2 shows Young’s modulus E at room temperature for three groups of specimen of identical nominal composition, namely A1-3, B1-3 and C1-3. The first group, A1-3, is related to a reference plate made by a non-additive manufacturing process. The second group, B1-3, is related to an SLM-processed specimen extending in the z-axis (like specimen 16a in FIG. 1). The third group, C1-3, is related to an SLM-processed specimen extending in the xy-plane (like specimen 16b in FIG. 1).

[0007] Now, the first specimen of each group (A1, B1 and C1) has been manufactured according to prior art procedures.

[0008] It can be easily seen that Young’s modulus E shows a substantial anisotropy with a difference of more than 20 GPa between the z-axis specimen B1 and the xy-plane specimen C1, and is much lower (more than 50 GPa) for both specimen compared to the reference specimen A1.

[0009] The present invention assumes that an additional treatment may be necessary to reduce this anisotropy in the properties of an article made by an additive manufacturing process.

[0010] Document US 2012/000890 A1 discloses a method using laser metal forming (LMF) for repairing gas turbine blades which repairs a thickness-reduced portion of a blade tip of a gas turbine blade. The method comprises a thickness-reduced portion removing step of working a surface of the blade tip into a flat surface by removing the thickness-reduced portion of the blade tip, a build-up welding step of forming a built-up portion with a predetermined thickness by melting powder of a build-up material higher in ductility than a base material forming the gas turbine blade by a laser beam and building up the melted powder in multiple layers on the blade

tip whose surface is worked into the flat surface, a forming step of working the built-up portion into the same shape as a shape that the blade tip originally has before suffering a thickness loss; and a heat-treatment step of removing a residual strain caused by laser welding in the build-up welding step.

[0011] Document WO 2012/016836 A1 teaches a method for manufacturing a component by selective laser melting (SLM) comprising: building a heat treatment device adapted to provide a heat treatment to the component as part of the same selective laser melting for manufacturing the component; and providing a heat treatment to the component by the heat treatment device. The heat treatment is used to make the component devoid of unwanted material property, for example, like ductility instead of embrittlement.

SUMMARY

[0012] It is an object of the present invention to disclose a method for manufacturing a three-dimensional article by additive manufacturing methods without the disadvantage of a having pronounced anisotropic properties.

[0013] This and other objects are obtained by a method according to claim 1.

[0014] The method according to the invention for manufacturing a three-dimensional article comprises the steps of

[0015] a) successively building up said article from a metallic base material by means of an additive manufacturing process, thereby creating an article with a substantial anisotropy of its properties; and

[0016] b) heat treating said manufactured article at sufficient high temperature to reduce said anisotropy significantly by recrystallisation and/or grain coarsening.

[0017] According to an embodiment of the invention said additive manufacturing process is one of laser metal forming (LMF), laser engineered net shape (LENS) or direct metal deposition (DMD), and that a metallic base material of wire form is used.

[0018] According to another embodiment of the invention said additive manufacturing process is one of selective laser melting (SLM), selective laser sintering (SLS) or electron beam melting (EBM), and that a metallic base material of powder form is used.

[0019] Specifically, said method comprises the steps of:

[0020] a) generating a three-dimensional model of said article followed by a slicing process to calculate the cross sections;

[0021] b) passing said calculated cross sections to a machine control unit (15) afterwards;

[0022] c) providing a powder of said base material, which is needed for the process;

[0023] d) preparing a powder layer (12) with a regular and uniform thickness on a substrate plate or on a previously processes powder layer;

[0024] e) performing melting by scanning with a energy beam (14) an area corresponding to a cross section of said articles according to the three-dimensional model stored in the control unit (15);

[0025] f) lowering the upper surface of the previously formed cross section by one layer thickness (d);

[0026] g) repeating said steps from c) to f) until reaching the last cross section according to the three-dimensional model; and

[0027] h) heat treating said three-dimensional article (11).

[0028] More specifically, the grain size distribution of said powder is adjusted to the layer thickness of said powder layer in order to establish a good flowability, which is required for preparing powder layers with regular and uniform thickness.

[0029] According to a further embodiment of the invention the powder grains have a spherical shape.

[0030] According to just another embodiment of the invention an exact grain size distribution of the powder is obtained by sieving and/or winnowing (air separation).

[0031] According to another embodiment of the invention said powder is provided by means of a powder metallurgical process, specifically one of gas or water atomization, plasma-rotating-electrode process or mechanical milling.

[0032] According to a further embodiment of the invention said additive manufacturing process uses a suspension instead of powder.

[0033] According to another embodiment of the invention said metallic base material is a high-temperature Ni-based alloy.

[0034] According to another embodiment of the invention said metallic base material is a high-temperature Co-based alloy.

[0035] According to just another embodiment of the invention said metallic base material is a high-temperature Fe-based alloy.

[0036] Specifically, said alloy can contain finely dispersed oxides, specifically one of Y_2O_3 , AlO_3 , ThO_2 , HfO_2 , ZrO_2 .

[0037] According to a further embodiment of the invention said heat treatment is used to reduce the anisotropy of Young's modulus.

[0038] According to another embodiment of the invention said heat treatment is a combination of different individual heat treatments.

[0039] According to just another embodiment of the invention said heat treatment consists of multiple steps, each representing a specific combination of heating rate, hold temperature, hold time and cooling rate.

[0040] Specifically, at least one of said heat treatment steps is conducted at a sufficient high temperature and for a hold time long enough to partially or completely dissolve constituents in the microstructure of said manufactured article, specifically intermetallic phases, carbides or nitrides.

[0041] Specifically, at least one of said heat treatment steps is conducted at a sufficient high temperature and for a hold time long enough to precipitate metal-carbides, metal-nitrides or metal-carbonitrides, specifically one of $M(C, N)$, M_6C , M_7C_3 or $M_{23}C_6$.

[0042] Specifically, at least one of said heat treatment steps is conducted at a sufficient high temperature and for a hold time long enough to precipitate intermetallic phases, specifically one $Ni_3(Al, Ti)$ known as gamma-prime, or $Ni_3(Nb, Al, Ti)$, known as gamma-double-prime, or Ni_3Nb known as delta-phase.

[0043] Specifically, at least one of said heat treatment steps is conducted at a sufficient high temperature and for a hold time long enough to precipitate metal-borides, specifically M_3B_2 , to improve grain boundary strength.

[0044] Specifically, at least one of said heat treatment steps is conducted at a sufficient high temperature and for a hold time long enough to modify the volume fraction, size, shape and distribution of said precipitations.

[0045] Specifically, at least one of said heat treatment steps can be conducted additionally under hot isostatic pressing (HIP) conditions, to further improve the microstructure.

[0046] According to another embodiment of the invention only part of said manufactured article is subjected to said heat treatment.

[0047] According to another embodiment of the invention before and/or after said heat treatment, individual heat treatments or heat treatment steps, respectively, said manufactured articles are subjected to additional processing steps, specifically one of machining, welding or brazing.

BRIEF DESCRIPTION OF THE DRAWINGS

[0048] The present invention is now to be explained more closely by means of different embodiments and with reference to the attached drawings.

[0049] FIG. 1 shows the different orientation of two specimen made by an additive manufacturing process like SLM;

[0050] FIG. 2 shows values of Young's modulus at room temperature for three groups of specimen made of Hastelloy® X with and without heat treatment;

[0051] FIG. 3 shows values of Young's modulus at 750° C. for three groups of specimen made of Hastelloy® X with and without heat treatment;

[0052] FIG. 4 shows photographs of the microstructure in two different magnifications (100 μm and 50 μm) for three different specimen made of Hastelloy® X without and with heat treatment; and

[0053] FIG. 5 shows a basic arrangement for SLM manufacturing, which may be used in the present invention.

DETAILED DESCRIPTION

[0054] One drawback of powder-based additive manufacturing technology can be the strong anisotropy of material properties resulting from the layer-wise build-up process.

[0055] The Young's modulus (E in FIGS. 2 and 3) is significantly lower along the z-axis (z-specimen (B1-B3 in FIG. 2 and B1'-B3' in FIG. 3) compared to xy-specimens (C1-C3 in FIG. 2 and C1'-C3' in FIG. 3), for instance. The z-axis is parallel to the build direction (see FIGS. 1 and 5).

[0056] However, appropriate "post-built" heat treatments can reduce the anisotropy significantly, as it has been shown by mechanical testing of SLM specimens made of Hastelloy® X. It is important to note that the reduction of anisotropy already occurs at heat treatment temperatures typical for the corresponding wrought products made of same alloy (in FIG. 2, specimen A2, B2 and C2 have been heat treated for 0.5 h at 1125° C., and specimen A3, B3 and C3 have been heat treated for 2 h at 1190° C., while specimen A1, B1 and C1 have experienced no heat treatment at all; in FIG. 3, specimen A1', B1' and C1' are without heat treatment, while specimen B2' and C2' were held for 0.5 h at 1125° C., and specimen B3' and C3' were held for 2 h at 1190° C.).

[0057] Especially, the present invention relates to the heat treatment of articles/components made of Ni/Co/Fe-based superalloys produced by a powder-based additive manufacturing technology, such as selective laser melting SLM or laser metal forming LMF. These articles have different microstructures compared to conventionally cast material or wrought products of the same alloy (specimen A1-A3 in FIG. 2 and A1' in FIG. 3), for instance.

[0058] This is primarily due to powder-based article production and the inherent high cooling rates of the energy beam-material interaction in these processes. As a consequence, the material is very homogeneous with respect to chemical composition and principally free of segregations. In

addition, the material in the "as built" condition has a very fine microstructure (e.g. precipitates and grain size), much finer compared to conventionally cast or wrought superalloys.

[0059] As has been recognized before, Ni/Co/Fe-based superalloys produced by powder-based additive manufacturing technologies are generally free of residual eutectic contents and heat treatments can be realized at higher temperatures compared to conventionally cast components of same composition. This allows an adjustment of the microstructure over a wide range, including grain size and precipitation optimization, leading to improved material properties.

[0060] The present invention disclosure relates to specially adjusted heat treatments for Ni/Co/Fe based superalloys processed by powder-based additive manufacturing technology to reduce inherent anisotropy of this technology.

[0061] This invention disclosure is based on the discovery that anisotropic material behaviour can be reduced by appropriate heat treatments.

[0062] These specific small-grained anisotropic microstructures result primarily from powder-based and layer-wise article production as well as the characteristic high thermal gradients occurring at energy beam-material interactions. Furthermore, high thermal gradients lead to residual stresses, which can favour recrystallisation and/or grain coarsening during heat treatment.

[0063] Therefore, this invention disclosure includes the manufacturing of three-dimensional articles specifically made of a Ni/Co/Fe based superalloy by powder-based additive manufacturing technologies followed by a specially adapted heat treatment resulting in reduced anisotropic material behaviour.

[0064] Said powder-based additive manufacturing technology is especially selective laser melting (SLM), selective laser sintering (SLS), electron beam melting (EBM), laser metal forming (LMF), laser engineered net shape (LENS), direct metal deposition (DMD) or like processes.

[0065] Said powder-based additive manufacturing technology may be used to build up an article, such as a blade or vane of a gas turbine, entirely or partly, e.g. blade crown build up.

[0066] When selective laser melting SLM, selective laser sintering SLS or electron beam melting EBM is used as the additive manufacturing technology the method according to the invention manufacturing of the three-dimensional articles comprises the following steps:

[0067] a) generating a three-dimensional model followed by a slicing process to calculate the cross sections, which are passed to a machine control unit (15 in FIG. 5) afterwards;

[0068] b) preparing the powders of said Ni/Co/Fe based alloy, which are needed for the process;

[0069] c) preparing a powder layer 12 with a regular and uniform thickness d on a substrate plate or on a previously processed powder layer;

[0070] d) performing melting by scanning with a energy beam (e.g. laser beam 14) corresponding to a cross section of said articles according to the three-dimensional model stored in the control unit;

[0071] e) lowering the upper surface of the previously formed cross section by one layer thickness d (see vertical arrows in FIG. 5);

[0072] f) repeating said steps from c) to e) until reaching the last cross section according to the three-dimensional model; and

[0073] g) heat treating said three-dimensional article 11.

[0074] Preferably, the grain size distribution of the powder used in this process is adjusted to the layer thickness d to have to a good flowability, which is required for preparing powder layers with regular and uniform thickness d.

[0075] Preferably, the powder grains of the powder used in this process have a spherical shape. The exact grain size distribution of the powder may be obtained by sieving and/or winnowing (air separation). Furthermore, the powder may be obtained by gas or water atomization, plasma-rotating-electrode process, mechanical milling and like powder metallurgical processes.

[0076] When especially additive manufacturing processes like laser metal forming LMF, laser engineered net shape LENS or direct metal deposition DMD are used, material in form of a wire instead of powder can be used.

[0077] In other cases, a suspension may be used instead of powder.

[0078] When said high temperature material is a Ni-based alloy, a plurality of commercially available alloys may be used like Waspaloy®, Hastelloy® X, IN617®, IN718®, IN625®, Mar-M247®, IN100®, IN738®, IN792®, Mar-M200®, B1900®, RENE 80®, Alloy 713®, Haynes 230®, Haynes 282®, or other derivatives.

[0079] When said high temperature material is a Co-based alloy, a plurality of commercially available alloys may be used like FSX 414®, X-40®, X-45®, MAR-M 509® or MAR-M 302®.

[0080] When said high temperature material is a Fe-based alloy, a plurality of commercially available alloys may be used like A 286®, Alloy 800 H®, N 155®, S 590®, Alloy 802®, Incoloy MA 956®, Incoloy MA 957® or PM 2000®.

[0081] Especially, these alloys may contain fine dispersed oxides such as Y_2O_3 , AlO_3 , ThO_2 , HfO_2 , ZrO_2 .

[0082] The heat treatment according to the invention advantageously reduces anisotropic material behaviour, especially the Young's modulus E (see FIGS. 2 and 3). For a specimen made of Hastelloy® X, the Young's modulus E_{at} room temperature (FIG. 2) increases for a specimen extending in z-direction (B1, B2, B3 in FIG. 2) from B1 (no heat treatment HT) to B2 (0.5 h at 1125° C.) to B3 (2 h at 1190° C.). For a specimen extending in the xy-plane (C1, C2, C3 in FIG. 2) there is an analogous increase from C1 (no heat treatment) to C2 (0.5 h at 1125° C.) to C3 (2 h at 1190° C.). For each of the three procedures (no HT, 0.5 h at 1125° C. and 2 h at 1190° C.) the Young's modulus E is lowest for the z-direction specimen, higher for the xy-plane specimen, and highest for the reference specimen (A1-A3). However, the differences in E between the various specimens are smallest for the most powerful heat treatment at 1190° C. for 2 h (A3, B3, C3 in FIG. 2). The same is true for the equivalent values of Young's modulus at 750° C., as shown in FIG. 3.

[0083] The heat treatment according to the invention is done by means of independent equipment. The heat treatment improves specific material properties by optimizing the microstructure of the article. FIG. 4 shows photographs of the microstructure in two different magnifications (100 μ m and 50 μ m) for three different z-direction specimen B4, B5 and B6 made of Hastelloy® X after having been tensile-tested, with and without heat treatment, whereby specimen B4 had no heat treatment with a tensile test conducted at 750° C., specimen B5 had a heat treatment for 0.5 h at 1125° C. with a tensile test conducted at room temperature, and specimen B6

had a heat treatment for 2 h at 1190° C. with a tensile test conducted at room temperature.

[0084] In certain cases, the whole article may be subjected to said heat treatment. In other cases, only part of it may be subjected to said heat treatment.

[0085] Said heat treatment can be one-time treatment. However, it can be a combination of different individual heat treatments.

[0086] Furthermore, said heat treatment may consist of multiple steps, each representing a specific combination of heating rate, hold temperature, hold time and cooling rate. In such a case, before or after each heat treatment step the three-dimensional articles may be subject to different processing steps such as, but not limited to, machining, welding or brazing, especially in order to use the specific advantages of the specific microstructure, e.g. small grains, which are beneficial for welding.

[0087] Preferably, at least one of said heat treatment steps should be conducted at a sufficient high temperature and for a hold time long enough to partially or completely dissolve constituents in said microstructure such as intermetallic phases, carbides or nitrides. Furthermore, it is clear that at least one of said heat treatment steps is conducted at a sufficient high temperature and for a hold time long enough to recrystallize and/or coarsen the grains.

[0088] As can be seen in FIGS. 2 and 3 (comparing A3, B3 and C3 or A1', B3' and C3'), said recrystallization and/or grain coarsening induced by the described heat treatment results in microstructures comparable to wrought and conventionally cast products.

[0089] In addition, at least one of said heat treatment steps may be conducted at a sufficient high temperature and for a hold time long enough to precipitate metal-carbides, metal-nitrides or metal-carbonitrides such as but not limited to, $M(C, N)$, M_6C , M_7C_3 or $M_{23}C_6$.

[0090] In addition, at least one of said heat treatment steps may be conducted at a sufficient high temperature and for a hold time long enough to precipitate intermetallic phases such as, but not limited to, $Ni_3(Al, Ti)$ known as gamma-prime, or $Ni_3(Nb, Al, Ti)$, known as gamma-double-prime, or Ni_3Nb known as delta-phase.

[0091] In addition, at least one of said heat treatment steps is conducted at a sufficient high temperature and for a hold time long enough to precipitate metal-borides such as, but not limited to, M_3B_2 , to improve grain boundary strength.

[0092] Furthermore, at least one of said heat treatment steps is conducted at a sufficient high temperature and for a hold time long enough to modify the volume fraction, size, shape and distribution of said precipitations mentioned before.

[0093] Finally, at least one of said heat treatment steps can be conducted additionally under isostatic pressure, known as hot isostatic pressing HIP, to further improve the microstructure.

SUMMARY

[0094] Mechanical testing and microstructural assessment have shown that specimens built by the SLM process or by other additive manufacturing process have strong anisotropic behaviour. By appropriate heat treatments, the anisotropic material behaviour, such as the Young's modulus, can be reduced significantly, resulting in more isotropic material properties.

What is claimed is:

1. A method for manufacturing a three-dimensional article, comprising:
 - a) successively building up said article from a metallic base material by means of an additive manufacturing process, thereby creating an article with a substantial anisotropy of its properties; and
 - b) heat treating said manufactured article at sufficient high temperature to reduce said anisotropy significantly by recrystallisation and/or grain coarsening.
2. The method according to claim 1, wherein said additive manufacturing process is selected from the group consisting of laser metal forming, laser engineered net shape and direct metal deposition, and that a metallic base material of wire form is used.
3. The method according to claim 1, wherein said additive manufacturing process is selected from the group consisting of selective laser melting, selective laser sintering and electron beam melting, and that a metallic base material of powder form is used.
4. The method according to claim 3, said method further comprising:
 - a) generating a three-dimensional model of said article followed by a slicing process to calculate the cross sections;
 - b) passing said calculated cross sections to a machine control unit afterwards;
 - c) providing a powder of said base material, which is needed for the process;
 - d) preparing a powder layer with a regular and uniform thickness on a substrate plate or on a previously processed powder layer;
 - e) performing melting by scanning with an energy beam an area corresponding to a cross section of said articles according to the three-dimensional model stored in the control unit;
 - f) lowering the upper surface of the previously formed cross section by one layer thickness;
 - g) repeating said steps from c) to f) until reaching the last cross section according to the three-dimensional model; and
 - h) heat treating said three-dimensional article.
5. The method according to claim 4, wherein the grain size distribution of said powder is adjusted to the layer thickness of said powder layer in order to establish a good flowability, which is required for preparing powder layers with regular and uniform thickness.
6. The method according to claim 3 wherein the powder grains have a spherical shape.
7. The method according to claim 3 wherein an exact grain size distribution of the powder is obtained by sieving and/or winnowing (air separation).
8. The method according to claim 4, wherein said powder is provided by means of a powder metallurgical process, specifically one of gas or water atomization, plasma-rotating-electrode process or mechanical milling.
9. The method according to claim 3, wherein said additive manufacturing process uses a suspension instead of powder.
10. The method according to claim 1 wherein said metallic base material is a high-temperature Ni-based alloy.
11. The method according to claim 1 wherein said metallic base material is a high-temperature Co-based alloy.
12. The method according to claim 1 wherein said metallic base material is a high-temperature Fe-based alloy.

13. The method according to claim **1** wherein said alloy contain finely dispersed oxides, specifically one of Y_2O_3 , AlO_3 , ThO_2 , HfO_2 , and ZrO_2 .

14. The method according to claim **1** wherein said heat treatment is used to reduce the anisotropy of Young's modulus.

15. The method according to claim **1** wherein said heat treatment is a combination of different individual heat treatments.

16. The method according to claim **1** wherein said heat treatment consists of multiple steps, each representing a specific combination of heating rate, hold temperature, hold time and cooling rate.

17. The method according to claim **16**, wherein at least one of said heat treatment steps is conducted at a sufficient high temperature and for a hold time long enough to partially or completely dissolve constituents in the microstructure of said manufactured article, specifically intermetallic phases, carbides or nitrides.

18. The method according to claim **16** wherein at least one of said heat treatment steps is conducted at a sufficient high temperature and for a hold time long enough to precipitate metal-carbides, metal-nitrides or metal-carbonitrides, specifically one of $M(C, N)$, M_6C , M_7C_3 and $M_{23}C_6$.

19. The method according to claim **16**, wherein at least one of said heat treatment steps is conducted at a sufficient high

temperature and for a hold time long enough to precipitate intermetallic phases, specifically one of $Ni_3(Al, Ti)$, or $Ni_3(Nb, Al, Ti)$, and Ni_3Nb .

20. The method according to claim **16** wherein at least one of said heat treatment steps is conducted at a sufficient high temperature and for a hold time long enough to precipitate metal-borides, specifically M_3B_2 , to improve grain boundary strength.

21. The method according to claim **18** wherein at least one of said heat treatment steps is conducted at a sufficient high temperature and for a hold time long enough to modify the volume fraction, size, shape and distribution of said precipitations.

22. The method according to claim **16** wherein at least one of said heat treatment steps can be conducted additionally under hot isostatic pressing conditions, to further improve the microstructure.

23. The method according to claim **1** wherein only part of said manufactured article is subjected to said heat treatment.

24. The method according to claim **1** wherein before and/or after said heat treatment, individual heat treatments or heat treatment steps, respectively, said manufactured articles are subjected to additional processing steps, selected from the group consisting of machining, welding and brazing.

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