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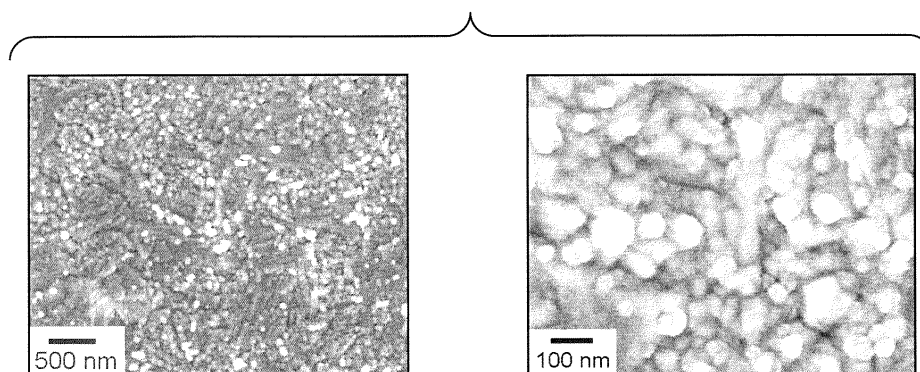
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(54) **Nanostructured superalloy structural components and methods of making**

(57) A superalloy-containing structural component includes a superalloy matrix, and a plurality of hard phase nanoparticles dispersed at grain boundaries within the superalloy matrix, wherein the plurality of hard phase nanoparticles dispersed at the grain boundaries comprise about 1 volume percent to about 30 volume percent of the structural component, and wherein the superalloy matrix and the plurality of hard phase nanoparticles dispersed at the grain boundaries within the base superalloy matrix have been thermo-mechanically processed to

form the structural component. A method for making a structural component includes introducing dislocations into a superalloy particle matrix effective to form new grain boundaries within a plurality of superalloy particles, introducing hard phase dispersoid nanoparticles at a plurality of grain boundaries of the superalloy particles effective to pin the grain boundaries, and thermo-mechanically processing the superalloy particles and hard phase dispersoid nanoparticles to form the superalloy-containing structural component.

FIG. 3



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Description

BACKGROUND

[0001] The present disclosure relates to superalloys, and more particularly to structural components comprising nanostructured superalloys.

[0002] Superalloys are metallic alloys that can be used at high temperatures, often in excess of 0.7 of the absolute melting temperature. Many structural components, such as those used in aircraft engines or power generation devices, are formed from Fe-, Co-, or Ni-base superalloys. There is a constant drive towards improving the high temperature properties of these fatigue-limited structural components in order to increase the strength or life of the aircraft engine or power generation device.

[0003] Nanostructured materials often exhibit superior mechanical properties (e.g., strength, hardness, ductility, and the like) relative to their larger-scale counterparts. Moreover, the fatigue initiation life of nanostructured materials is significantly higher than that of larger-grained materials since dislocation activity may be spread over a larger number of grains. Unfortunately, nanostructured alloys, like their larger-scale counterparts, undergo the processes of recovery, recrystallization, and/or grain growth upon heating. In fact, owing to their non-equilibrium nature, nanoscale grains are more susceptible to these processes than are micrometer scale grains. Consequently, when thermo-mechanically processing nanostructured alloys into a shaped article, the nanostructure and, consequently, the superior properties are often lost. Furthermore, during operation of the structural components comprising the nanostructured alloys, new opportunities for recovery, recrystallization, and/or grain growth arise as the working temperatures increase.

[0004] One method of inhibiting recovery, recrystallization, and/or grain growth (and therefore a method of strengthening alloys) is through Orowan strengthening, in which a fine distribution of hard phase particles is incorporated into the alloy composition matrix. The strength of such hard phase particle-reinforced alloys is inversely proportional to the spacing between the dispersoid particles, which can be controlled by controlling the size of the dispersoid particles. Thus, the use of nanoparticles as dispersoids offers the potential of substantially enhancing alloy strength.

[0005] The introduction of hard phase dispersoid nanoparticles during the processing of the alloys presents a major technical challenge. Current processes to disperse particles include powder metallurgy routes, such as mechanical alloying of micrometer-scale particles, in combination with secondary processes, which include hot-isostatic pressing and/or thermo-mechanical processing by hot-forging or extrusion. In the mechanical alloying process, nanoparticles are created by repeated fracture of the micrometer-scale dispersoid particles during milling. Unfortunately, these processes fail to produce a homogeneous distribution of nanoparticles in the alloy

matrix, especially for large components. In addition, the loading of the hard phase dispersoid particles in the alloy composites is frequently limited to less than 2 volume percent. Thus, current processes are unable to produce nanostructured alloys having a sufficiently high enough loading of nanoparticle dispersoids to provide increased strength to the alloy or article made therefrom.

[0006] There accordingly remains a need in the art for improved methods of producing nanostructured alloys that have more stable grain structures when exposed to heat. It would be particularly advantageous if nanostructured superalloys could be produced by such methods. It would be further advantageous if these nanostructured superalloys could be used in fatigue-limited structural components, resulting in increased lifetimes and/or efficiencies of the devices making use of these structural components.

BRIEF SUMMARY

[0007] A superalloy-containing structural component includes a superalloy matrix, and a plurality of hard phase nanoparticles dispersed at grain boundaries within the superalloy matrix, wherein the plurality of hard phase nanoparticles dispersed at the grain boundaries comprise about 1 volume percent to about 30 volume percent of the structural component, and wherein the superalloy matrix and the plurality of hard phase nanoparticles have been thermo-mechanically processed to form the structural component.

[0008] In another aspect, a superalloy-containing structural component includes a superalloy matrix; a gamma prime phase, wherein the gamma prime phase comprises about 10 weight percent to about 60 weight percent of the nanostructured superalloy matrix; and a plurality of hard phase nanoparticles dispersed at grain boundaries within the superalloy matrix; wherein the plurality of hard phase nanoparticles dispersed at the grain boundaries comprise about 1 volume percent to about 30 volume percent of the structural component, and wherein the superalloy matrix, gamma prime phase, and the plurality of hard phase nanoparticles dispersed at the grain boundaries within the superalloy matrix have been thermo-mechanically processed to form the structural component.

[0009] A method of manufacturing a nanostructured superalloy-containing structural component generally includes introducing dislocations into a superalloy particle matrix effective to form new grain boundaries within a plurality of superalloy particles, wherein the grains are nanostructured; introducing hard phase dispersoid nanoparticles at a plurality of grain boundaries of the superalloy particles effective to pin the grain boundaries; and thermo-mechanically processing the superalloy particle matrix and hard phase dispersoid nanoparticles to form the nanostructured superalloy-containing structural component.

[0010] The above described and other features are ex-

emplified by way of example only with reference to the following figures and detailed description.

BRIEF DESCRIPTION OF THE DRAWINGS

[0011] Referring now to the figures, which are exemplary embodiments, provided by way of example only, and wherein like elements are numbered alike:

Figure 1 is a graphical representation comparing the tensile strengths of a prior art alloy to an alloy made according to one embodiment of the present disclosure;

Figure 2 is a graphical representation of the high-cycle fatigue properties of three different Ni-20Cr alloys;

Figure 3 depicts representative scanning electron micrograph images of a nanostructured Ni-20Cr alloy, which had dispersoid nanoparticles introduced at the grain boundaries both *ex-situ* and *in-situ* according to one embodiment of the present disclosure; and

Figure 4 is a graphical representation comparing the tensile strengths of a prior art alloy to an alloy made according to another embodiment of the present disclosure.

DETAILED DESCRIPTION

[0012] Nanostructured superalloy-containing structural components and their methods of manufacture are described herein. In contrast to the prior art, the methods and structural components disclosed herein, owing to their nanoscale grain structure, allow for increased stability in the superalloy when exposed to heat. Consequently, fatigue limited structural components with increased strength can be manufactured, resulting in increased lifetimes and/or efficiencies of the devices making use of these structural components. As used herein, the term "nanostructured" refers to those materials having grains with an average longest dimension of about 1 nanometer (nm) to about 500 nm.

[0013] Also, as used herein, the terms "first", "second", and the like do not denote any order or importance, but rather are used to distinguish one element from another, and the terms "the", "a", and "an" do not denote a limitation of quantity, but rather denote the presence of at least one of the referenced item. The modifier "about" used in connection with a quantity is inclusive of the stated value and has the meaning dictated by the context (e.g., includes the degree of error associated with measurement of the particular quantity). Furthermore, all ranges disclosed herein are inclusive of the endpoints and independently combinable.

[0014] The superalloy-containing structural compo-

nent generally comprises a superalloy matrix and a plurality of hard phase nanoparticles dispersed at grain boundaries within the superalloy matrix.

[0015] Any Fe-, Co-, or Ni-base superalloy composition may be used to form the structural component. The most common solutes in Fe-, Co-, or Ni-base superalloys are aluminum and/or titanium. Generally, the aluminum and/or titanium concentrations are low (e.g., less than or equal to about 15 weight percent (wt %) each). Other optional components of Fe-, Co-, or Ni-base superalloys include chromium, molybdenum, cobalt (in Fe- or Ni-base superalloys), tungsten, nickel (in Fe- or Co-base superalloys), rhenium, iron (in Co- or Ni-base superalloys), tantalum, vanadium, hafnium, niobium, ruthenium, zirconium, boron, and carbon, each of which may independently be present in an amount of less than or equal to about 15 wt %.

[0016] An exemplary Ni-base superalloy composition, not including the hard phase nanoparticle dispersoid composition, comprises about 12 to about 20 wt % Cr, less than or equal to about 22 wt % Co, less than or equal to about 20 wt % Fe, about 2 to about 5 wt % Mo, about 0.5 to about 5 wt % Ti, about 0.5 to about 4 wt % Al, less than or equal to about 5 wt % W, less than or equal to about 3 wt % Ta, less than or equal to about 3 wt % Re, less than or equal to about 6 wt % Nb, less than or equal to about 3 wt % V, less than or equal to about 2 wt % Hf, about 0.02 to 0.2 wt % C, less than or equal to about 0.03 wt % B, less than or equal to about 0.1 wt % Zr, with the balance being essentially Ni. By "essentially Ni", it is meant that the composition may include incidental or trace levels of impurities.

[0017] In one embodiment, the superalloy matrix itself is nanostructured. In one embodiment, the grains within the superalloy matrix have an average longest dimension of about 10 nm to about 500 nm. In another embodiment, the grains within the superalloy matrix have an average longest dimension of about 10 nm to about 30 nm.

[0018] The plurality of hard phase nanoparticles may comprise an inorganic oxide, an inorganic carbide, an inorganic nitride, an inorganic carbonitride, an inorganic boride, an inorganic oxycarbide, an inorganic oxynitride, an inorganic silicide, an inorganic aluminide, an inorganic sulfide, an inorganic oxysulfide, or a combination comprising at least one of the foregoing. Exemplary inorganic oxides include yttria, alumina, zirconia, or hafnia. Exemplary inorganic carbides include carbides of hafnium, tantalum, molybdenum, zirconium, niobium, chromium, titanium, or tungsten. Exemplary inorganic sulfides and oxysulfides are cerium sulfide and cerium oxysulfide, respectively.

[0019] In contrast to the prior art, the nanostructured superalloy-containing structural components disclosed herein overcome the loading and dispersion limitations encountered in existing hard phase dispersoid strengthened alloys or superalloys. In one embodiment, the superalloy-containing structural component comprises about 1 to about 30 volume percent (vol %) hard phase

dispersoid nanoparticles. In another embodiment, the superalloy-containing structural component comprises about 10 to about 30 vol % hard phase dispersoid nanoparticles. This increased loading of the hard phase dispersoid nanoparticles results in greater grain boundary pinning and therefore greater strength in the structural component.

[0020] The plurality of hard phase dispersoid nanoparticles may be spherical, cubic, rod-like, needle-like, ellipsoidal, or like shaped. It is not necessary that each of the plurality of hard phase dispersoid nanoparticles have the same shape. In one embodiment, the plurality of hard phase dispersoid nanoparticles has an average longest dimension of about 10 nm to about 500 nm. In another embodiment, each of the plurality of hard phase dispersoid nanoparticles has an average longest dimension of about 10 nm to about 30 nm.

[0021] The structural component may further comprise the so-called "gamma prime" phase, which is an intermetallic compound generally based on the formula $Ni_3(Al/Ti)$, and serves as an additional strengthening mechanism. The gamma prime phase is particularly resistant to thermal activation, caused by increased temperatures, which can lead to recovery and therefore decreased strength. Consequently, a structural component comprising an alloy with nanostructured grains, hard phase dispersoid nanoparticles, and the gamma prime phase can experience a substantial increase in its fatigue life. Depending on the particular conditions to which the structural component is exposed, the gamma prime phase may comprise about 10 wt % to about 60 wt % of the nanostructured superalloy matrix.

[0022] The structural component may further comprise the so-called "gamma double-prime" phase, which is also an intermetallic compound generally based on the formula Ni_3Nb , and like the gamma prime phase also serves as an additional strengthening phase. The gamma double-prime, like the gamma prime phase increases in strength with temperature up to about 1200 degrees Celsius ($^{\circ}C$).

[0023] The method of manufacturing a nanostructured superalloy-containing structural component generally includes introducing dislocations into a superalloy powder particle matrix effective to form new grain boundaries within a plurality of superalloy grains, wherein the grains are nanostructured; introducing hard phase dispersoid nanoparticles at the grain boundaries effective to pin the grain boundaries; and thermo-mechanically processing the superalloy powder particle matrix and hard phase dispersoid nanoparticles to form the nanostructured superalloy-containing structural component.

[0024] Introducing the dislocations into the superalloy powder particle matrix can be accomplished by cryomilling, high pressure torsion (HPT), equal channel angular pressing (ECAP), cyclic channel die compression (CCDC), accumulative roll bonding, repetitive corrugation and straightening, twist extrusion, or a similar severe plastic deformation technique, or a combination compris-

ing at least one of the foregoing techniques.

[0025] Introducing the hard phase dispersoid nanoparticles at the grain boundaries can be done *ex-situ* and/or *in-situ*. By *ex-situ* introduction of the hard phase dispersoid nanoparticles, it is meant that the hard phase dispersoid nanoparticles are intentionally physically added to the superalloy powder particle matrix during and/or after the dislocation formation. By *in-situ* introduction of the hard phase dispersoid nanoparticles, it is meant that the hard phase dispersoid nanoparticles are created (e.g., precipitated) within the superalloy powder particle matrix, such as when cryomilling in a reactive atmosphere (e.g., in the presence of liquid nitrogen, liquid hydrocarbons, oxygen, and the like).

[0026] Thermo-mechanically processing the superalloy powder particles to form the nanostructured superalloy-containing structural component can be accomplished by forging, hot extrusion, hot rolling, and/or like techniques.

[0027] Optionally, prior to the thermo-mechanical processing, the superalloy powder particle matrix and the hard phase dispersoid nanoparticles may be consolidated into a compact. Consolidation into a compact may be performed by cold pressing, hot pressing, hot isostatic pressing, forging, extruding, and/or like consolidating techniques.

[0028] In one embodiment, a powder particle matrix of a superalloy is cryomilled in liquid nitrogen for a time effective to reduce the grain size within the powder particle matrix to the desired grain size. During the cryomilling, dispersoid nanoparticles are formed (e.g., precipitated) *in-situ*, for example by oxidizing (if any oxygen is present) or nitriding a reactive metal component of the superalloy composition. Additionally, if dispersoid nanoparticles are extrinsically added before and/or during the cryomilling, then they will be intimately mixed with the powder particle matrix such that they serve as pinning agents as well. It should be recognized that there will be a point after which no additional cold working (cryomilling) will decrease the grain size of the particle powder matrix, but instead will serve to provide an increased opportunity for the *in-situ* formation of dispersoid nanoparticles. This may be desirable depending on the specific properties targeted for the final structural component. For example, in superalloys comprising aluminum, it may be desirably to have a nitrogen content of less than or equal to about 1.0 wt % in order to avoid the increased brittleness that is accompanied by a higher nitrogen content. Once the desired grain size reduction and nanoparticle dispersoid addition has been achieved, the sample (i.e., the nanostructured powder particle matrix and hard phase dispersoid nanoparticles) are consolidated by hot isostatic pressing and subsequently forged to form the desired shape.

[0029] The nanostructured superalloy-containing structural components disclosed herein are suitable for use in at least a portion of a hot gas path assembly, such as a steam turbine, gas turbine, aircraft engine, and the like. These hot gas path assemblies can have tempera-

tures, to which the structural components are exposed, of about 800 °C, specifically about 1000 °C, and more specifically about 1200 °C. Exemplary structural components include rotating components (e.g., airfoils, discs, wheels, and the like), static components (e.g., ducts, frames, casings, buckets, vanes, and the like), combustors, and the like.

[0030] Advantageously, the nanostructured superalloy-containing structural components and methods of manufacture described herein provide for increased stability in the base superalloy when exposed to heat. Consequently, fatigue limited structural components with increased strength can be manufactured, resulting in increased lifetimes and/or efficiencies of the devices making use of these structural components. For example, the finer grains and dispersoids may make possible a doubling, or more, of tensile strength and creep resistance. Alloying of the grain boundaries can inhibit or eliminate loss in fatigue resistance from environmental exposure.

[0031] The present disclosure is illustrated by the following non-limiting examples.

Example 1:

[0032] An alloy, comprising nickel and about 20 wt % Cr (Ni-20Cr), was produced by melting and forging. The average grain diameter after heat treatment of this prior-art material is approximately 64 micrometers (μm). The same base alloy composition was produced as a powder, cryomilled in liquid nitrogen, consolidated, and heat-treated. The grain size after heat treatment of this novel material was about 64 nm. Room temperature tensile tests were conducted on both materials. Figure 1 illustrates the tensile curves for the two materials. The ultimate tensile strength of the prior art micrometer-scale material was about 87 kilopounds per square inch (ksi), or 600 MegaPascals (MPa), while the ultimate tensile strength of the nanostructured alloy was about 162 ksi (1117 MPa). This represented an 86% higher tensile strength in the alloy produced by the methods disclosed herein.

Example 2:

[0033] A nanostructured Ni-20Cr sample was prepared as described in Example 1, except that, in addition, a plurality of Al_2O_3 dispersoid nanoparticles were introduced prior to cryomilling. Figure 3 presents representative scanning electron microscope images of this superalloy composition.

[0034] The fatigue properties of 1) this nanostructured Ni-20Cr superalloy, which had dispersoid nanoparticles introduced at the grain boundaries both *ex-situ* and *in-situ* (designated "nanostructured Ni-20Cr w/ Al_2O_3 "), 2) a nanostructured Ni-20Cr superalloy prepared according to Example 1, which only had dispersoid nanoparticles introduced at the grain boundaries *in-situ* (designated "nanostructured Ni-20Cr"), and 3) a known Ni-20Cr su-

peralloy, obtained from Special Metals Corporation under the trade designation INCONEL MA754 (designated "MA754") were studied. Figure 2 displays the results of the high-cycle fatigue properties of these three samples. Data is presented for five samples of the nanostructured Ni-20Cr w/ Al_2O_3 superalloy, five samples of the nanostructured Ni-20Cr superalloy, and two samples of the MA754 superalloy. As evidenced in Figure 2, each sample of both nanostructured superalloys of the present disclosure were able to withstand significantly greater stresses than the MA754 superalloy. Furthermore, the nanostructured superalloys of the present disclosure were also able to experience increased lifetimes before failure owing to fatigue.

Example 3:

[0035] A René 104 alloy is a nickel-base superalloy having a nominal composition (in weight percent): 0.05 carbon, 3.4 aluminum, 0.05 zirconium, 3.7 titanium, 0.025 boron, 2.4 tantalum, 3.8 molybdenum, 0.9 niobium, 2.4 tantalum, 13 chromium, 20.6 cobalt, balance essentially nickel. The alloy was produced by consolidation of atomized powder, forging, and heat treatment. One sample of the powder was consolidated by hot isostatic pressing, extruded, and heat-treated to yield a micrometer-scale product. Another sample of the powder was cryomilled in liquid nitrogen and subsequently thermo-mechanically processed by hot isostatic pressing, extrusion, and heat treatment in a manner identical to the prior-art micrometer-scale product.

[0036] The two samples were examined by electron microscopy; and tensile tests were conducted. In the nanostructured René 104 alloy of the present disclosure, there is a distribution of small particles of zirconium and aluminum-rich oxides that also had been present on the prior-art powder particle surface; additionally, Ta-rich carbides and the gamma-prime phase were present. The grains of the nanostructured René 104 alloy are much finer than what was observed for the prior-art micrometer-scale product. In addition, in the nanostructured René 104 alloy of the present disclosure, there is a noteworthy distribution of fine titanium-rich particles that are not present in the prior-art micrometer-scale product. These titanium-rich particles appear to form by a reaction between the milling fluid, (i.e., liquid nitrogen) and titanium from the alloy. The titanium particles are associated with regions of much finer grain size.

[0037] Figure 4 illustrates the room temperature tensile curves for the two samples. The nanostructured René 104 alloy has higher yield (176 vs. 198 ksi) and ultimate (248 vs. 262 ksi) tensile strengths.

[0038] While the disclosure has been described with reference to exemplary embodiments, it will be understood by those skilled in the art that various changes may be made and equivalents may be substituted for elements thereof without departing from the scope of the disclosure. In addition, many modifications may be made

to adapt a particular situation or material to the teachings of the disclosure without departing from the essential scope thereof. Therefore, it is intended that the disclosure not be limited to the particular embodiment disclosed as the best mode contemplated for carrying out this disclosure, but that the disclosure will include all embodiments falling within the scope of the appended claims.

Claims

1. A structural component formed from a superalloy, the structural component comprising:

a superalloy matrix; and
 a plurality of hard phase nanoparticles dispersed at grain boundaries within the superalloy matrix; wherein the plurality of hard phase nanoparticles dispersed at the grain boundaries comprise about 1 volume percent to about 30 volume percent of the structural component, and wherein the superalloy matrix and the plurality of hard phase nanoparticles dispersed at the grain boundaries within the base superalloy matrix have been thermo-mechanically processed to form the structural component.

2. The structural component of Claim 1, further comprising a gamma prime phase, a gamma double prime phase, or both.

3. The structural component of any of the preceding Claims, wherein the structural component comprises at least a portion of a hot gas path assembly.

4. The structural component of any of the preceding Claims, wherein the superalloy matrix comprises a Ni-base superalloy, Fe-base superalloy, Co-base superalloy, or a combination comprising at least one of the foregoing superalloys.

5. A method for making a structural component comprising a superalloy, the method comprising:

introducing dislocations into a superalloy particle matrix effective to form new grain boundaries within a plurality of superalloy particles;
 introducing hard phase dispersoid nanoparticles at a plurality of grain boundaries of the superalloy particles effective to pin the grain boundaries; and
 thermo-mechanically processing the superalloy particles and hard phase dispersoid nanoparticles to form the superalloy-containing structural component.

6. The method of Claim 5, wherein introducing the dislocations comprises cryomilling, high pressure tor-

sion, equal channel angular pressing, cyclic channel die compression, accumulative roll bonding, repetitive corrugation and straightening, twist extrusion, or a combination comprising at least one of the foregoing.

7. The method of any of the preceding Claims, wherein introducing the hard phase dispersoid nanoparticles comprises extrinsically combining the hard phase dispersoid nanoparticles with the superalloy particle matrix during and/or after introducing the dislocations into the superalloy particle matrix, creating the hard phase dispersoid nanoparticles while introducing the dislocations into the superalloy particle matrix, or both.

8. The method of any of the preceding Claims, wherein thermo-mechanically processing the Ni-superalloy particles and hard phase dispersoid nanoparticles to form the nanostructured Ni-superalloy-containing structural component comprises forging, hot extrusion, hot rolling, or a combination comprising at least one of the foregoing.

9. The method of any of the preceding Claims, further comprising consolidating the superalloy particle matrix and hard phase dispersoid nanoparticles into a compact prior to the thermo-mechanically processing.

10. The method of any of the preceding Claims, further comprising introducing at least one of a gamma prime phase and a gamma double-prime phase into the superalloy particle matrix.

FIG. 1

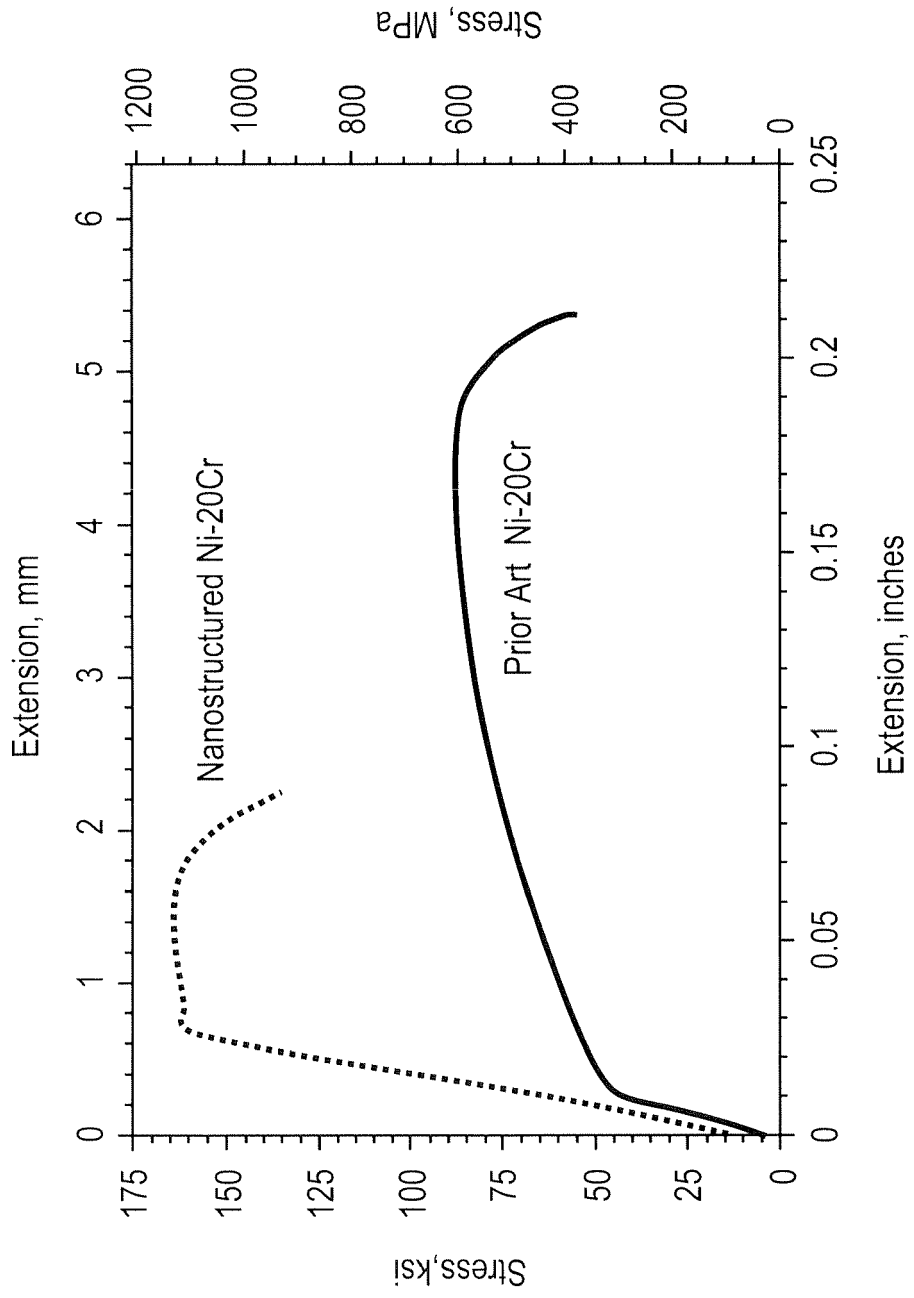


FIG. 2

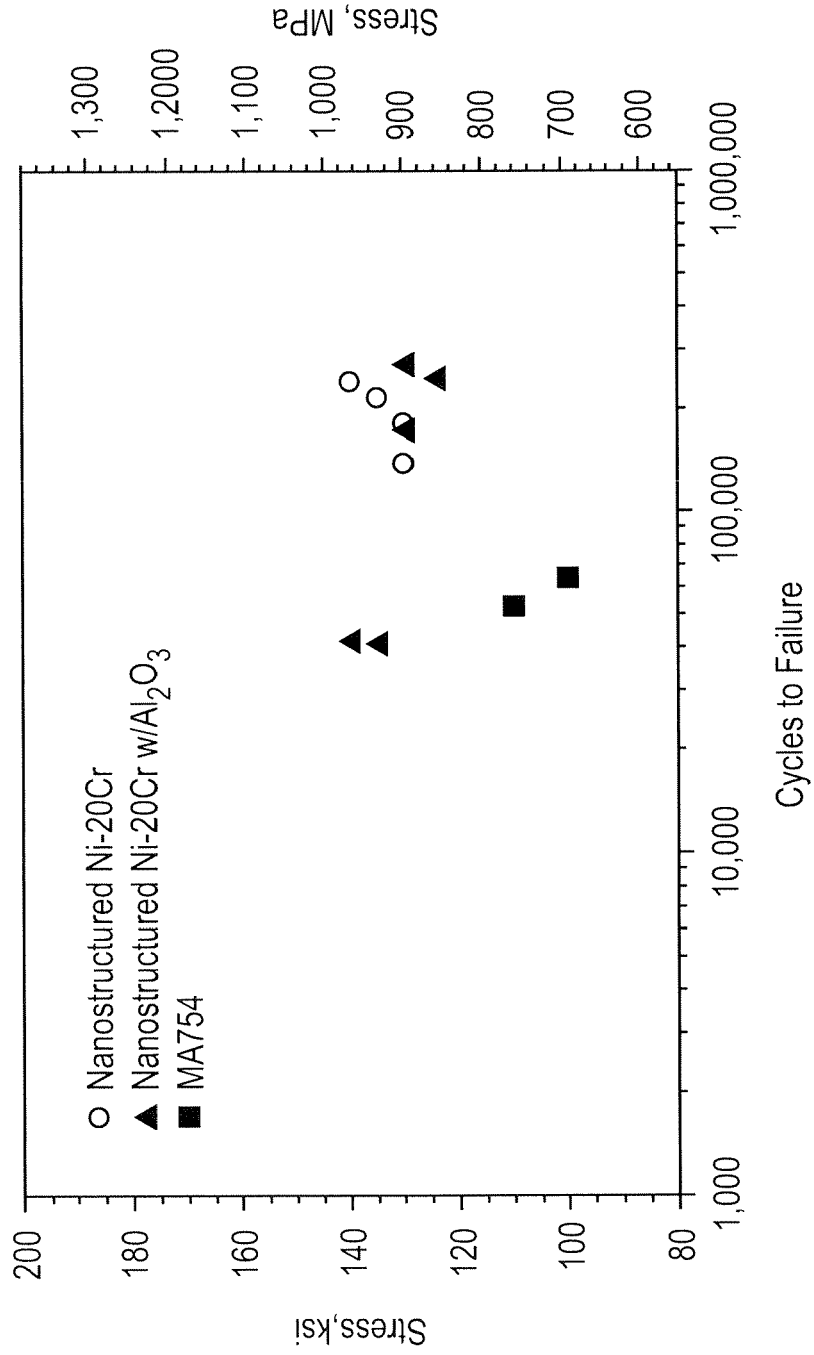


FIG. 3

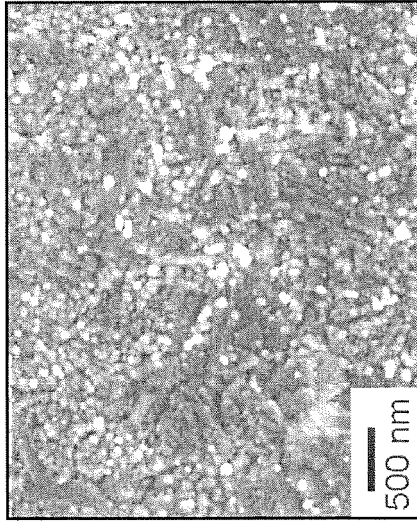
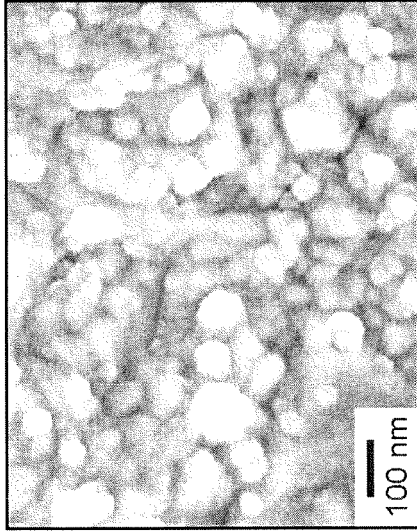
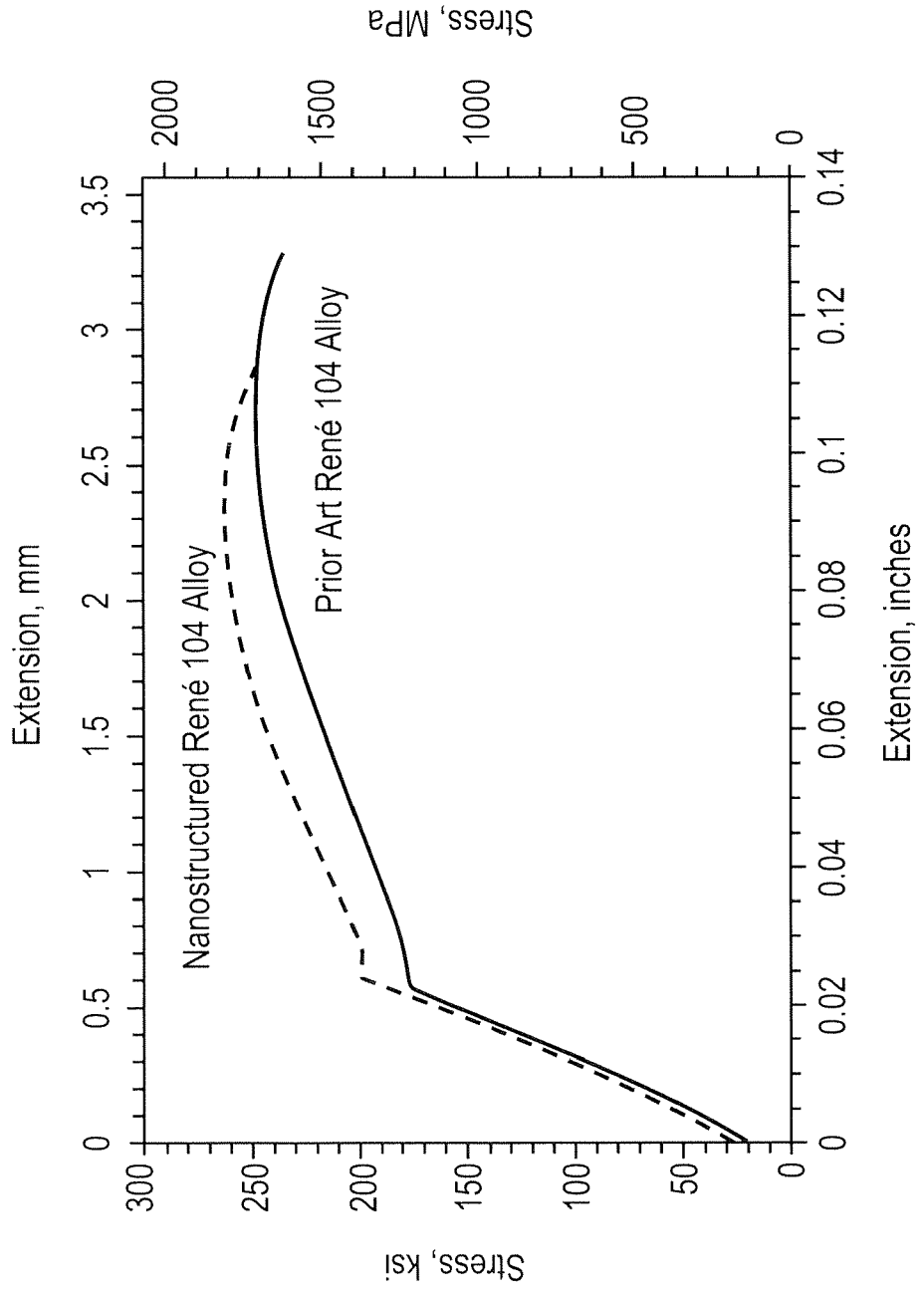


FIG. 4





DOCUMENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (IPC)
X	EP 1 548 134 A (GEN ELECTRIC [US]) 29 June 2005 (2005-06-29) * the whole document *	1-10	INV. B22F1/00 B22F9/04 C22C1/04 C22C1/05
X	RODRIGUEZ R ET AL: "Tensile and creep behavior of cryomilled Inco 625" ACTA MATER; ACTA MATERIALIA FEB 25 2003, vol. 51, no. 4, 25 February 2003 (2003-02-25), pages 911-929, XP002430292 * abstract *	1,5	
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			B22F C22C
The present search report has been drawn up for all claims			
Place of search Munich		Date of completion of the search 20 April 2007	Examiner ALVAZZI DELFRATE, M
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EPO FORM 1503 03.82 (P04C01)

**ANNEX TO THE EUROPEAN SEARCH REPORT
ON EUROPEAN PATENT APPLICATION NO.**

EP 07 10 1038

This annex lists the patent family members relating to the patent documents cited in the above-mentioned European search report. The members are as contained in the European Patent Office EDP file on
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20-04-2007

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