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(54) **Single crystal superalloy articles with reduced grain recrystallization**

(57) A single crystal casting is cast from a low carbon nickel base superalloy and heat treated in a carburizing atmosphere to introduce carbon into the casting and form carbides therein that reduce or localize grain

recrystallization in the single crystal casting during heat treating.



FIG. 1

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Description

FIELD OF THE INVENTION

[0001] The present invention relates to nickel base superalloy castings and, more particularly, to a method of heat treating single crystal superalloy castings in a manner to reduce or localize deleterious extraneous grain recrystallization during heat treatment.

BACKGROUND OF THE INVENTION

[0002] U.S. Patent 4 643 782 describes single crystal castings made from a nickel base superalloy having a composition consisting essentially of, in weight %, of 6.4% to 6.8% Cr, 9.3% to 10.0% Co, 0.5% to 0.7% Mo, 6.2% to 6.6% W, 6.3% to 6.7% Ta, 5.45% to 5.75% Al, 0.8% to 1.2% Ti, 2.8% to 3.2% Re, 0.07 to 0.12% Hf and balance essentially nickel. Carbon is held to incidental impurity levels of for example 60 ppm maximum C in the alloy.

[0003] U.S. Patent 5 759 303 describes addition of carbon to a nickel base superalloy including the alloy of the first-discussed patent above to reduce the amount of non-metallic inclusions (e.g. oxide inclusions) in the microstructure of single crystal investment castings produced therefrom.

SUMMARY OF THE INVENTION

[0004] In attempts to manufacture investment cast gas turbine engine single crystal blades from a nickel base superalloy of the first-discussed patent above, applicants discovered that such single castings were prone to develop deleterious extraneous grain recrystallization at the airfoil and/or root of the gas turbine engine blade during a subsequent conventional heat treatment to develop alloy mechanical properties wherein the castings are initially subjected to a high temperature solution heat treatment. Such grain recrystallization is to be avoided or localized to non-critical regions of a casting that are subsequently removed therefrom. Grain recrystallization is a cause for rejection of single crystal castings if present beyond a preset maximum for recrystallized grains and can result in quite low yields of acceptable heat treated single crystal castings.

[0005] The present invention provides a method of making of superalloy single crystal castings, such as gas turbine engine single crystal blades and vanes (airfoils), in a manner to address the problem of grain recrystallization during heat treatment of the single crystal castings. The invention involves the discovery that grain recrystallization can be reduced by solution heat treating the single crystal castings in the presence of gaseous species carburizing relative to the superalloy castings so as to introduce carbon into the castings in an effective amount to reduce recrystallized grains during heat treatment. For example, a carburizing atmos-

phere can be provided by introducing a mixture of carbon monoxide and an inert gas, such as argon, into the heat treatment furnace or by heat treating in a furnace having a component, such as heating elements, that inherently provides a carburizing atmosphere during heat treatment. Such a furnace to this end typically comprises heating elements, heat shields and/or other furnace components or inserts comprising graphite or other carbon-bearing material as a source of carbon for reaction with oxygen to form a carbon-bearing gas, such as carbon monoxide, in-situ in the furnace that is carburizing relative to the castings.

[0006] The carbon concentration of at least the outer surface region of the superalloy single crystal castings is locally increased during heat treatment as compared to the nominal carbon concentration of the bulk superalloy casting as evidenced, for example, by the presence of blocky carbides of one or more alloying elements, which carbides are not present when the superalloy casting is heat treated under vacuum or inert gas atmosphere only in the absence of a carburizing gas species in the furnace. The carbides form in the microstructure in a manner to pin any recrystallized grain boundaries during solution heat treatment and retard, limit and localize their growth in a manner to improve the yield of acceptable heat treated single crystal castings.

DESCRIPTION OF THE DRAWINGS

[0007]

Figures 1 and 2 are photomicrographs at 500X and 200X of single crystal nickel base superalloy castings after solution heat treatment pursuant to an embodiment of the invention.

DETAILED DESCRIPTION OF THE INVENTION

[0008] The present invention involves heat treating nickel base superalloys formulated for single crystal casting in a manner to unexpectedly and surprisingly substantially reduce or localize grain recrystallization after heat treatment of the casting at elevated temperature, such as a high temperature solution heat treatment to dissolve or solution most of the eutectic and coarse gamma prime phases present in the as-cast microstructure. Improved yields of acceptable heat treated single crystal castings are thereby achieved.

[0009] In general, the present invention can be practiced on a variety of low carbon nickel base superalloys that are formulated for single crystal casting and include W, Ta, Mo, Co, Al and Cr as important alloying elements as well as optionally Ti, Re, Y, Hf, one or more rare earth elements such as La, B, and Mg as intentional alloying elements and that suffer undesirable grain recrystallization upon heat treatment. Such grain recrystallization prone nickel base superalloys typically

have carbon concentration less than about 200 ppm by weight (about 0.02 weight % C) with some less than about 100 ppm C (about 0.01 weight % C), although the invention may be practiced with superalloys having other carbon concentrations to reduce grain recrystallization in a particular nickel base superalloy. Nickel base superalloys formulated for casting single crystal castings such as single crystal airfoils (blades and vanes), and heat treatable pursuant to the invention include, but are not limited to, those described in U.S. Patents 4 643 782 and 5 366 695 the teachings of which are incorporated herein by reference with respect to particular alloy compositions.

[0010] An illustrative nickel base superalloy casting composition heat treatable pursuant to the present invention consists essentially of, in weight % or parts per million (ppm) by weight, of about 6% to 6.8% Cr, about 8% to 10% Co, about 0.5% to 0.7% Mo, about 6.2% to 6.6% W, about 6.3% to 7% Ta, about 5.4% to 5.8% Al, about 0.6% to 1.2% Ti, about 0.10% to 0.3% Hf, up to about 200 ppm by weight B, up to about 50 ppm by weight Mg, up to about 200 ppm by weight carbon, and balance essentially Ni and castable to provide a single crystal microstructure, especially for gas turbine engine blades and vanes (i.e. airfoils).

[0011] An illustrative low carbon, high Re nickel base superalloy casting composition heat treatable pursuant to the present invention consists essentially of, in weight %, of about 1.5% to 5% Cr, about 1.5% to 10% Co, about 0.25% to 2% Mo, about 3.5% to 7.5% W, about 7% to 10% Ta, about 5% to 7% Al, up to about 1.2% Ti, about 5% to 7% Re, up to about 0.15% Hf, up to about 0.5% Nb, C less than about 0.02% or at incidental impurity level, and balance essentially Ni and castable to provide a single crystal microstructure, especially for gas turbine engine blades and vanes (i.e. airfoils). An illustrative low carbon, high Cr nickel base superalloy casting composition heat treatable pursuant to the present invention consists essentially of, in weight %, of about 11% to 16% Cr, about 2% to 8% Co, about 0.2% to 2% Mo, about 3.5% to 7.5% W, about 4% to 6% Ta, about 3% to 6% Al, about 2% to about 5% Ti, up to about 0.2% Nb, C less than about 0.02% or at incidental impurity level, and balance essentially Ni and castable to provide a single crystal microstructure, especially for gas turbine engine blades and vanes (i.e. airfoils).

[0012] The following example is offered for purposes of illustrating but not limiting the invention. Single crystal gas turbine engine blades were conventionally cast using the Bridgman withdrawal technique from commercially available CMSX-4 nickel base superalloy, described in US Patent 4 643 782, and were subjected in the as-cast condition after removal of a ceramic shell mold and a ceramic core to solution heat treatments in various atmospheres. The nominal composition, in weight %, of the single crystal blades was 6.4% Cr, 9.7% Co, 0.6% Mo, 6.4% W, 6.5% Ta, 5.6% Al, 1.0% Ti, 2.9% Re, 0.10% Hf, 30 ppm by weight C and balance

essentially Ni and impurities. After casting, the ceramic shell mold and core were removed completely from the castings in conventional manner using a mechanical knock-out procedure and chemical leaching. The single crystal blade castings then were solution heat treated in various furnaces using various atmospheres. After heat treatment, the castings were examined for the presence of recrystallized grains on the casting surfaces.

[0013] In particular, the cast single crystal blades were solution heat treated in various heat treatment furnaces. One type of furnace included graphite electrical resistance heating element and graphite sides or heat shield liners. Another type of furnace included molybdenum electrical resistance heating elements and graphite sides or heat shield liners. Different heat treatment atmospheres were provided for different heat treatment runs in the different types of furnaces.

[0014] For example, one heat treatment run involved providing a vacuum of less than 5 microns in a furnace having molybdenum heating elements and graphite heat shields or liners and then introducing a mixture of argon and 10% by volume CO at a flow rate during continued vacuum pump evacuation of the furnace to maintain 400 microns partial pressure of argon plus CO in the furnace as the atmosphere during heat treatment. The argon/10% by volume CO gas mixture was introduced from a conventional gas cylinder having a mixture of argon and 10% by volume CO therein. The mixture was introduced after the furnace temperature reached 1900 degrees F so as to reduce chromium vaporization from the castings. A total of 10 cast single crystal blades were solution heat treated in this furnace by slowly heating the castings to a solutioning temperature of 2400 degrees F plus or minus 15 degrees F over 11 hours. The solutioning temperature was held for 6 hours, and the castings were cooled to room temperature over a time of 1 hour.

[0015] The solution heat treatment dissolved most of the eutectic and coarse gamma prime phases in the as-cast microstructure. The only recrystallized grains observed were initiated proximate a core print (at a blade tip) with the recrystallized grains localized to an extent that they existed outside the finished casting dimensions for the particular blade involved; i.e. such that the localized amount of recrystallized grains on the heat treated blade would be removed by subsequent finish machining of the blade. In microstructural examination, blocky carbides rich in Ta and Ti having a lateral dimension (e.g. diameter) of less than 0.5 mil (0.0005 inch) were observed to exist throughout the airfoil and pinning the recrystallized grain boundaries as illustrated in Figure 1 by the arrow. The carbides were determined to include Ta in the approximate range of 71-77 weight % Ta, Ti in approximate range of 9-10 weight % Ti, Hf in approximate range of 2-7 weight % Hf, Ni in approximate range of 3-4 weight % Ni with other elements such as Co, W, Cr, Fe, also present in lesser amounts. The carbides were formed predominantly along cast sur-

faces, providing a high population of carbides in thin sections of the castings where grain growth is more likely to be a problematic. The carbides were attributed to the carburization of the castings, resulting in introduction of carbon into the castings during heat treatment. For example, typical carbon concentration at the airfoil surface and of the bulk airfoil was twice as high (at least 100% higher) as the as-cast carbon content at the airfoil surface. For purposes of illustration only, for a particular cast single crystal blade measured for carbon, the as-cast carbon concentration of the bulk airfoil and bulk root were increased from about 38 ppm by weight C to 113 ppm and 89 ppm by weight carbon for the airfoil and root, respectively. The carbon content at the airfoil surface was even higher, being about 171 ppm by weight C after the above heat treatment.

[0016] Similar results were achieved when the cast single crystal blades were heat treated under similar parameters in a furnace having graphite heating elements and graphite heat shields or liners and an atmosphere comprising 400 microns argon only introduced in a manner similar to the above described argon/CO mixture when the furnace reached 1900 degrees F and at a flow rate to provide the 400 microns argon partial pressure during heat treatment. Blocky carbides were observed throughout the airfoil and also throughout the thickness (35 mils) of the platform, and 25 mils inwardly from the root surface. Recrystallized grains were small and confined near the core print by the carbides (see arrows in Figure 2) pinning the recrystallized grain boundaries. For a particular cast single crystal blade measured for carbon, the as-cast carbon concentrations of the bulk airfoil and bulk root were increased to 200 and 124 ppm by weight carbon, respectively.

[0017] Similar results were achieved when the cast single crystal blades were heat treated under similar parameters in a furnace having molybdenum heating elements and graphite heat shields or liners with introduction of the above described mixture of argon/10% by volume CO when the furnace temperature reached 1900 degrees F to provide 400 microns partial pressure argon plus CO as described above during heat treatment. Blocky carbides were observed to be present interdendritically in clusters throughout the airfoil and approximately 10 mils below the root surface. Recrystallized grains were small and confined near the core print by carbide pinning the recrystallized grain boundaries.

[0018] In contrast, similar results were not achieved when the cast single crystal blades were heat treated under similar parameters in a furnace having molybdenum heating elements and graphite heat shields or liners in a vacuum of less than 1 micron or when argon gas only was introduced to the furnace after a furnace temperature of 1900 degrees F to provide 400 microns partial pressure argon only during heat treatment. Blocky carbides were not observed to be present throughout the airfoil or other regions of the castings heat treated under vacuum or under 400 microns partial

pressure argon only using the molybdenum heating elements. The castings exhibited unacceptable recrystallized grains.

[0019] The present invention provides single crystal castings having carbon concentrations increased by the heat treatment in an amount discovered to form carbides in-situ in the heat treated microstructure that pin recrystallized grain boundaries and retard, limit and localize their growth to reduce recrystallized grains that are cause for rejection of the single crystal castings and increase yield of acceptable heat treated castings. Practice of the invention as described above produced a six times increase in yield of acceptable heat treated single crystal turbine blade castings. The present invention envisions use of carburizing atmospheres or gaseous carburizing species other than carbon monoxide that are effective to introduce carbon to single crystal nickel base superalloy castings during their heat treatment in amounts effective to reduce or localize recrystallized grains.

[0020] While the invention has been described in terms of specific embodiments thereof, it is not intended to be limited thereto but rather only to the extent set forth in the following claims.

Claims

1. A method of making a single crystal casting, comprising providing a nickel base superalloy single crystal casting and heat treating the casting in presence of a gaseous species effective to introduce carbon into the casting and form carbides therein that reduce grain recrystallization in the single crystal casting during heat treating.
2. The method of claim 1 wherein said heat treating forms blocky carbides rich in Ta in said casting.
3. The method of claim 1 wherein said atmosphere includes an inert gas and carbon-bearing gas constituent in a heat treatment furnace.
4. The method of claim 3 wherein the carbon-bearing as comprises carbon monoxide.
5. The method of claim 3 wherein said carbon-bearing gas is introduced into said furnace from a source external of said furnace.
6. The method of claim 3 including generating said carbon-bearing gas in-situ in said furnace using carbon from a heating element comprising graphite.
7. A method of making a single crystal casting, comprising providing a low carbon nickel base superalloy single crystal casting including W, Ta, Mo, Co, Al and Cr as alloying elements and heat treating the casting in presence of a gaseous species effective

to introduce carbon into the casting and form carbides therein that reduce grain recrystallization in the single crystal casting during heat treating.

8. The method of claim 7 wherein the nickel base superalloy includes one or more of Ti, Re, Y, Hf, rare earth element, B, and Mg as intentional alloying elements. 5
9. The method of claim 7 wherein carbon concentration of the superalloy is less than about 200 ppm by weight. 10
10. A method of making a single crystal casting, comprising providing a nickel base superalloy single crystal casting consisting essentially of, in weight %, about 6% to 6.8% Cr, about 8% to 10% Co, about 0.5% to 0.7% Mo, about 6.2% to 6.6% W, about 6.3% to 7% Ta, about 5.4% to 5.8% Al, about 0.6% to 1.2% Ti, about 0.10% to 0.3% Hf, up to about 100 ppm by weight B, up to 50 ppm by weight Mg, up to about 200 ppm by weight C and balance essentially Ni and heat treating the casting in presence of gaseous species effective to introduce carbon into the casting and form carbides therein that reduce grain recrystallization in the single crystal casting during heat treating. 15 20 25
11. The method of claim 10 wherein said heat treating forms blocky carbides rich in Ta in said casting. 30
12. The method of claim 10 wherein said atmosphere includes an inert gas and carbon-bearing gas constituent in a heat treatment furnace. 35
13. The method of claim 12 wherein the carbon-bearing gas comprises carbon monoxide.
14. The method of claim 12 wherein said carbon-bearing gas is introduced into said furnace from a source external of said furnace. 40
15. The method of claim 12 including generating said carbon-bearing gas in-situ in said furnace using carbon from a heating element comprising graphite. 45
16. A method of making a single crystal casting, comprising providing a nickel base superalloy single crystal casting consisting essentially of, in weight %, about 1.5% to 5% Cr, about 1.5% to 10% Co, about 0.25% to 2% Mo, about 3.5% to 7.5% W, about 7% to 10% Ta, about 5% to 7% Al, up to about 1.2% Ti, about 5% to 7% Re, up to about 0.15% Hf, up to about 0.5% Nb, C less than about 0.02%, and balance essentially Ni and heat treating the casting in presence of gaseous species effective to introduce carbon into the casting and form carbides therein that reduce grain recrystallization 50 55

in the single crystal casting during heat treating.

17. A method of making a single crystal casting, comprising providing a nickel base superalloy single crystal casting consisting essentially of, in weight %, of about 11% to 16% Cr, about 2% to 8% Co, about 0.2% to 2% Mo, about 3.5% to 7.5% W, about 4% to 6% Ta, about 3% to 6% Al, about 2% to about 5% Ti, up to about 0.2% Nb, C less than about 0.02%, and balance essentially Ni and heat treating the casting in presence of gaseous species effective to introduce carbon into the casting and form carbides therein that reduce grain recrystallization in the single crystal casting during heat treating.
18. A heat treated nickel base superalloy single crystal casting, said heat treated single crystal casting having carbides formed in the microstructure at a recrystallized grain boundary.
19. The casting of claim 18 including W, Ta, Mo, Co, Al and Cr as alloying elements.
20. The casting of claim 18 further including at least one of Ti, Re, Y, Hf, rare earth element, B, and Mg as intentional alloying elements
21. A heat treated single crystal nickel base alloy casting consisting essentially of, in weight %, of about 6% to 6.8% Cr, about 8% to 10% Co, about 0.5% to 0.7% Mo, about 6.2% to 6.6% W, about 6.3% to 7% Ta, about 5.4% to 5.8% Al, about 0.6% to 1.2% Ti, about 0.1% to 0.3% Hf, up to about 100 ppm by weight B, up to 50 ppm by weight Mg, up to about 200 ppm by weight C, and balance essentially Ni, said heat treated single crystal casting having carbides at a recrystallized grain boundary.

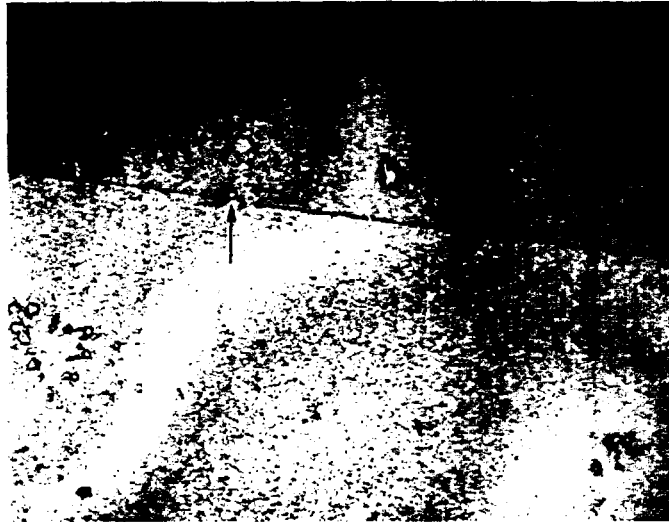


FIG. 1

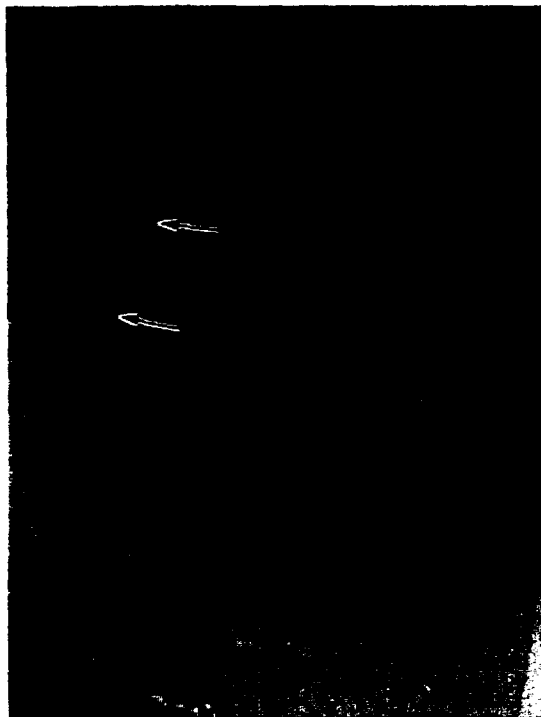


FIG. 2



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EUROPEAN SEARCH REPORT

Application Number
EP 00 10 5884

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Place of search MUNICH		Date of completion of the search 28 June 2000	Examiner Lilimpakis, E
<p>CATEGORY OF CITED DOCUMENTS</p> <p>X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document</p> <p>T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons ----- & : member of the same patent family, corresponding document</p>			

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EUROPEAN SEARCH REPORT

Application Number
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CATEGORY OF CITED DOCUMENTS X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons & : member of the same patent family, corresponding document			

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**ANNEX TO THE EUROPEAN SEARCH REPORT
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