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Buchanan

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[54] **METHOD OF MAKING SUPERALLOY BODIES**

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[52] **U.S. Cl.** ..... **148/11.5 F,** 75/226

[51] **Int. Cl.** **B22f 3/16**

[58] **Field of Search** ..... 148/11.5 F; 75/.5 BB, 214, 75/226

[56] **References Cited**

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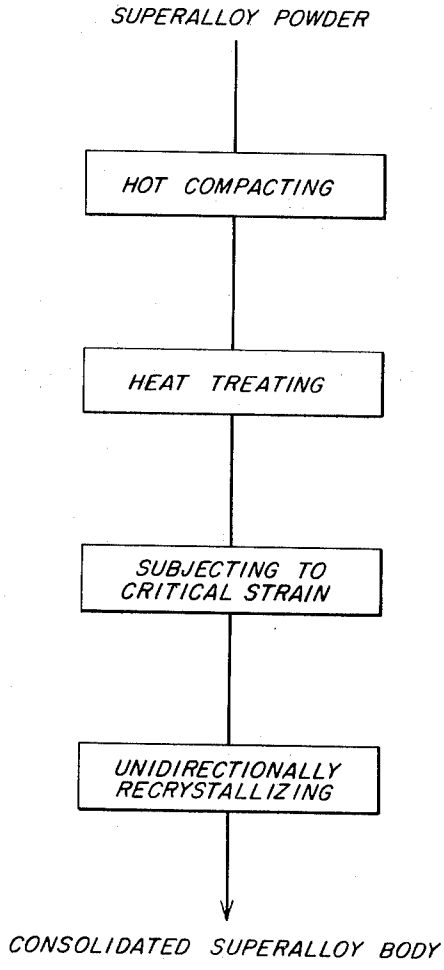
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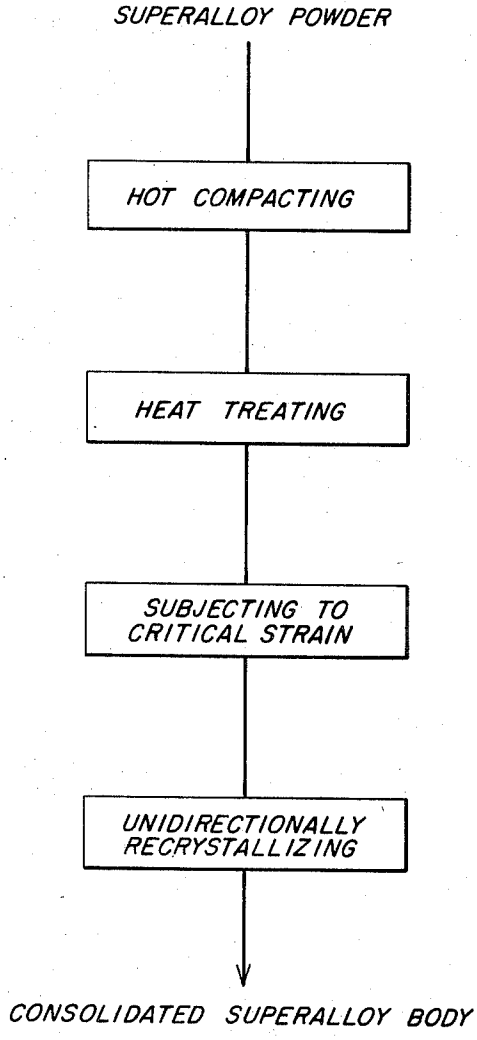
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[57] **ABSTRACT**

A method of making superalloy bodies characterized by having aligned elongated grains is provided by employing powder metallurgy procedures. The method involves hot compacting a nickel-base superalloy to a dense solid, heat treating the solid to form an essentially single phase γ structure, imparting a critical strain to the single phase material and then subjecting the material to a unidirectional recrystallization step to form an elongated columnar grained structure having grain boundaries substantially parallel to the direction of recrystallization.

8 Claims, 1 Drawing Figure




SUPERALLOY POWDER
HOT COMPACTING
HEAT TREATING
SUBJECTING TO CRITICAL STRAIN
UNIDIRECTIONALLY RECRYSTALLIZING
CONSOLIDATED SUPERALLOY BODY

METHOD OF MAKING SUPERALLOY BODIES

Superalloys are heat resistant materials having superior strength and oxidation resistance at high temperatures. Many of these alloys contain iron, nickel or cobalt alone or in combination as the principal element together with chromium to impart surface stability, and usually containing one or more minor constituents, such as molybdenum, tungsten, columbium, titanium and aluminum for the purpose of effecting strengthening. The physical properties of the superalloys make them particularly useful in the manufacture of gas turbine components.

The strength of superalloys is determined in part by their grain size. At low temperatures, fine grained equiaxed structures are preferred. At high temperatures (generally above 1600° F.), large grain size structures are usually found to be stronger than fine-grained. This is believed related to the fact that failure generally originates at grain boundaries oriented perpendicular to the direction of the induced stress. An improved technique for cast superalloys used in gas turbine engines was developed by Ver Snyder, U.S. Pat. No. 3,260,505 which discloses the preparation of a blade having an elongated columnar structure with unidirectional crystals aligned substantially parallel to the long axis of the blade. This procedure involves directional solidification whereby almost a complete elimination of grain boundaries normal to the primary stress axis occurs. A further advance was made by Piearcey, U.S. Pat. No. 3,494,709 wherein grain boundaries in superalloys were eliminated by making single crystal castings. These directionally solidified materials are not suitable for all applications. One disadvantage is that there is a substantial increase in cost over conventional castings. Further, I have found that variations in mechanical properties of a directionally solidified part occur at different distances from the chill due to differences in dendrite spacing and microstructure along the length of the casting.

An alternative processing technique for superalloys that has aroused recent interest is powder metallurgy. This has advantages for some applications from the point of view of cost reduction and improved properties and permits introduction of an inert refractory oxide for additional strengthening. Thus, U.S. Pat. No. 3,639,179 issued to Reichman et al. describes a process for making nickel-base superalloys having superior high temperature properties which employs powder metallurgy techniques. The process involves confining and densifying the powder into a billet, cold working the billet below the recrystallization temperature, recrystallizing the cold worked billet for a time sufficient to nucleate new grains and thereafter heat treating the recrystallized billet to effect growth of the grains to a desired equiaxed size. This process yielded a nickel-base superalloy characterized as being of large grain size and possessing superior tensile strength and stress rupture superalloys at elevated temperatures.

In accordance with the present invention, I have discovered a method of making superalloy bodies characterized by having aligned elongated grains or a monocrystalline grain structure by employing powder metallurgy techniques. The method involves hot compacting a nickel-base superalloy powder to form a relatively dense solid, solution heat treating the solid at a temperature below the incipient melting point, subjecting the material to a sufficient strain below the recrystallization temperature to permit subsequent recrystallization, and unidirectionally recrystallizing the strained material in a temperature gradient at a maximum temperature below the incipient melting temperature and above the γ' solvus temperature, to form a body having an elongated columnar grain or monocrystalline grain structure. The product produced by my process is substantially similar in macrostructure to the cast superalloy articles prepared by directional solidification. In addition, inhomogeneities such as eutectic modules are avoided and it is possible to prepare a material having a uniform structure throughout without this segregation characteristic of cast bodies.

The accompanying drawing, which is a flow sheet of the novel process, while not intended as a definition essentially illustrates the invention. A full discussion is set forth herein below.

Nickel-base superalloys are strong, high temperature materials which are particularly useful in gas turbine engines. A substantial listing of these materials is set forth by W. F. Simmons, *Compilation of Chemical Compositions and Rupture Strengths of Superalloys*, ASTM Data Series Publication No. DS9E, and may be represented by the nominal compositions in weight percent of the following superalloys:

TABLE I

Ingredient	René 80	René 100	René 120	IN-738	Udimet 500
C	0.17	0.18	0.17	0.17	0.08
Mn	—	—	—	0.20	0.75
Si	—	—	—	0.30	0.75
Cr	14.0	9.5	9.25	16.0	19.0
Ni	Bal.	Bal.	Bal.	Bal.	Bal.
Co	9.5	15.0	10.0	8.5	18.0
Mo	4.0	3.0	2.0	1.75	4.0
W	4.0	—	7.0	2.6	—
Cb	—	—	—	0.9	—
Ti	5.0	4.20	4.0	3.4	2.9
Al	3.0	5.50	4.25	3.4	2.9
B	0.015	0.015	0.015	0.01	0.005
Zr	0.03	0.06	0.05	0.10	—
Fe	0.2	1.0 max	—	0.50	4.0
Other	—	1.0 V	3.75 Ta	1.75 Ta	—

Initially the nickel-base superalloy is in the form of a fine metal powder which is prepared in such a way that each powder particle is substantially of the same nominal composition as the final alloy composition. A conventional technique for preparing such powders is by atomization of a melt of the alloy. The use of a powder ensures alloy homogeneity and overcomes the prob-

lems resulting from alloy segregation which occurs in large ingot sections and causes variations in physical properties within a single large part or from separate parts made from the same ingot. In addition, it is possible with powder materials to disperse a chemically inert phase, e.g., alumina or yttria, uniformly through the alloy by various milling techniques to achieve additional high temperature strength. These inert phases tend to agglomerate when added to liquid cast metal, thus preventing their utilization in cast metals.

The next step involves hot compacting the metal powder into a dense solid. The hot compaction is performed either by extrusion or by hot pressing. It is preferred that during hot compacting, a protective atmosphere or vacuum be used to prevent oxidation of some of the reactive elements in the alloy. The alloy powder may be extruded by canning it in a steel jacket and then hot extruding the billet to finished size or to stock which is machined to the desired final dimensions. At this point, in the absence of an addition of an inert phase, the dense solid consists essentially of a γ' precipitate phase with a γ matrix.

Thereafter, the dense solid is annealed to dissolve a substantial portion of the γ' phase. The reason for the anneal is related to the fact that the γ' phase appears to impede elongated grain growth. For each alloy, the annealing temperature should be above the γ' solvus and below the incipient melting temperature of the alloy. For example, René 120 has a γ' solvus temperature of about 1205° C. and an incipient melting temperature of about 1260° C. In the preferred embodiment involving René 120, the annealing temperature is about 1240° C. The annealing time is dependent on the size of the workpiece. I have found that 10–20 minutes at this temperature is preferred in a workpiece less than one-half inch thick.

The "critical strain" is defined as that amount of strain which is just sufficient to cause the growth of very large grains during subsequent recrystallization. The crux of the critical strain concept is that a certain minimum strain is required to cause recrystallization during subsequent heating. If this strain is exceeded, the recrystallized grain diameter is essentially inversely related to the amount of tensile strain. By just exceeding the critical strain in the workpiece and drawing it through a thermal gradient, a monocrystalline or elongated grain structure results. The critical strain in most of the nickel-base superalloys used in this invention are on the order of 1–3 percent at room temperature. This amount of plastic strain may be introduced in a tensile machine at a strain rate of 0.02 in./in./min. However, the desired structure may also be achieved by rolling a test piece at room temperature to 2 percent total reduction in thickness. In addition, the state of critical strain can also be achieved by straining the workpiece at any temperature below the recrystallization temperature, although larger amounts of strain are required at higher temperature due to dynamic recovery during straining. The critical strain at 1200–1400° F. is typically about 8–10 percent.

After straining, the material is unidirectionally recrystallized to provide a body having an elongated parallel grain or monocrystalline structure. This is performed by drawing the material through a gradient furnace. I have found the number of grains in the cross-section is essentially related to the efficiency of the gradient. In the preferred embodiment of the invention, the gradient is at least 1000° F./inch. The maximum temperature of the thermal gradient is above the γ' solvus and below the incipient melting temperature of the alloy in question. The rate at which the workpiece is drawn through the gradient depends on the alloy, but it has been found that speeds of ½–two inches/hour results in an elongated grain structure in most superalloys. I have found that slower speeds in this range result in monocrystalline structures, while higher speeds result in elongated parallel columnar grains.

My invention is further illustrated by the following examples:

EXAMPLE I

A billet was prepared from René 120 nickel-base superalloy powders having the composition shown in the table above, except the carbon level was 0.05 percent rather than the typical 0.17 percent. The loose powders, having mesh sizes −100+200, were encapsulated in a 3½ inches diameter stainless steel capsule having a 0.216 inch wall thickness. The capsule cavity and powder were evacuated to 10^{-6} Torr, heated to 500° C. under vacuum to remove volatile impurities, cooled to room temperature and sealed.

The entire capsule was then heated to 1175° C. for 2 hours and extruded through a die aperture of 0.6 × 1.0 inch, approximately an 18/1 reduction. Two and one-half inch lengths were cut from the billet. Four tabs, each 0.6 × 2½ × 0.072 inches were cut from the center of each length. Tabs were machined into tensile specimens having a gauge length of 0.150 × 0.072 × 1.0 inches.

The specimens were then subjected to various combinations of a prior anneal followed by being subjected to a strain at room temperature. Thereafter, the specimens were passed through a gradient furnace having a maximum temperature of 1260° C., which is slightly below the alloy incipient melt temperature but above the γ' solvus temperature. The temperature gradient was about 1093° C./inch.

To determine the speed range, if any, at which elongated grain growth would occur, variable speed anneals were used for some specimens, in which about ⅜ inch length of gauge section was passed through the hot zone of the furnace at a predetermined speed of about ¼ inch/hour, then the drive motor speed was increased to about ½ inch/hour for another ⅜ inch of gauge length, and so on. This determined for a given set of processing conditions whether elongated grain growth would or would not occur.

The results of the critical strain experiments on René 120 of specimens gradient annealed at a maximum temperature of 1260° C. are shown in Table II:

TABLE II

Sample	Initial Heat Treatment	Strain	Anneal Speed Range (in/hr)	Results
I-A	None	None	¼, ½, 1, 2, 4	No elongated grains
I-B	None	2.0%	½, 1	ditto
I-C	None	3.0%	¼, ½, ¾, 1	ditto
I-D	1240°C/15 min.	None	½, 1	ditto
I-E	1240°C/15 min.	1.0%	1	ditto
I-F	1240°C/15 min.	1.5%	1	Elongated grains
I-G	1240°C/15 min.	2.0%	½, 1	ditto
I-H	1240°C/15 min.	2.5%	1	ditto
I-I	1240°C/15 min.	3.0%	¼, ½, ¾, 1	No elongated grains

It may be concluded from the results that the necessary conditions for achieving elongated grain growth in this alloy are a high temperature heat treatment followed by a critical strain at room temperature greater than 1 percent total elongation, but less than 3 percent.

EXAMPLE II

A billet of Rene 120 was prepared using the same procedure as Example I, the only difference being the billet contained a normal carbon level, 0.17 percent. It was observed the combination of a heat treatment for 15 minutes at 1240° C. followed by a strain at room temperature of about 2 percent total strain resulted upon gradient annealing in an elongated grain structure in all attempts tried.

EXAMPLE III

A billet of Rene 120 was prepared using the same procedure as Example I, except that the powder contained 2 vol.pct. Al_2O_3 which had been milled into the metal powder by a mechanical milling process and the extrusion temperature was 1232° C.

It was found the same combination of heat treatment and strain resulted in elongated grain structure after recrystallizing in a temperature gradient. However, it was found that the room temperature strain resulting in optimum elongated grain structure was about 2.75–3.0 percent, somewhat higher than the 2 percent strain in the nondispersoid-containing alloy.

EXAMPLE IV

A billet of Rene 80 was prepared from superalloy powders having the composition shown in the table above. The loose powders, having −325 mesh size, were encapsulated in a 2 1/16 inches carbon steel extrusion can, evacuated, sealed, and extruded through a ½ inch round die aperture (approximately an 18/1 reduction) at 1093° C. Lengths of the extruded bar were solution annealed for two hours at 1210° C. and cooled to room temperature in an air blast. Microstructural examination revealed a fine-grained structure having an average diameter of approximately 20 microns.

Tensile specimens having a ¼ inch gauge diameter and 1 inch gauge length were machined from the ½ inch diameter stock. Four specimens were deformed in tension at room temperature to respective strains of 2.3, 3.0, 3.5, and 10 percent at a strain rate of 0.02 in./in./min. Following tensile deformation, the four specimens were surface ground to 0.1 inch thick flats to insure uniform heating during the gradient anneal. Specimens were then subjected to a 50 percent HCl-50 percent HNO_3 acid solution for one-half hour to remove potential grain nuclei at the specimen surface.

The specimens were then recrystallized in a temperature gradient at 1205° C. at three-eighths inch/hour. Microstructural examination revealed no evidence of elongated grain structure in the specimen deformed to 2.3 percent tensile strain, indicating the critical strain had not been reached. In the specimen strained to 10 percent strain, grains in the gauge length consisted of equiaxed grains about 20 microns in diameter. At both ends of the gauge length, where the plastic strain decreased from the nominal 10 percent to zero, a continuous region of columnar grains were observed to have been nucleated, indicating the validity of the strain anneal concept for the subject material. On the specimen strained to 3.5 percent, large (30–40 microns) equiaxed grains were observed in the gauge length, again with columnar grains at the ends of the gauge length indicating the critical strain in the gauge length had been exceeded. In the specimen strained to 3.0 percent, the grain structure in the gauge length consisted of long columnar grains believed typical of specimens strained to the critical strain.

EXAMPLE V

Following the procedure of Example I, four test specimens were prepared from Rene 120 (0.17 percent C.) extruded bar having an 0.6 × 1.0 inches cross-section. The bars were then machined to test specimens having a gauge length 1.0 inch long × 0.150 × 0.072 inch. These were strained to 2.0 percent total strain at room temperature (0.02 inch/min. crosshead speed). Thereafter the strained samples were unidirectionally recrystallized in a gradient furnace at 1254° C. at 1 inch/hour to produce elongated grains in the specimen gauge section, with fine, equi-axed grains at the gauge length ends, in the specimen shoulders, and in the specimen grip sections. The specimens were then given a heat treatment according to the following schedule: 1236° C. for 1 hour; 1093° C. for 4 hours; and 900° C. for 16 hours. Subsequently, a second gauge length having dimensions 0.50 inch long × 0.100 inch wide × 0.072 inch thick in the elongated grain portion was machined into the first gauge section of two of the four specimens.

Stress rupture tests were performed in air at the following test conditions:

Sample	Temperature	Stress
V-A	1650° F.	60 KSI
V-B	1800° F.	30 KSI

Sample V-A failed in 0.6 hours and sample V-B failed on loading. Both samples failed through the 0.150 inch width in the first gauge section in the fine-grained portion of the specimen. The stress in the 0.150 inch portion of the specimens is two-thirds that in the 0.100 inch portion. This indicates that the stress rupture strength of fine-grained Rene 120 is poor at 1650° F./40 KSI and 1800° F./20 KSI.

To evaluate the stress rupture strength of the elongated grain structure portion of the specimen, the two remaining specimens were machined. To ensure that failure occurred in the elongated portion of the specimen, the second reduced gauge sections were machined to dimensions of 0.50 inch long × 0.072 inch thick × 0.060 inch wide, so that the stress in the second gauge section was 40 percent of that in the first gauge section. Test results were:

Sample	Temperature	Stress	Time
V-C	1650° F.	60 KSI	12.7 hr. life
V-D	1800° F.	30 KSI	29.2 hr. life

The above results indicate the degree of mechanical property improvement in René 120 occurring from production of an elongated grain structure.

It will be appreciated that the invention is not limited to the specific details shown in the examples and illustrations and that various modifications may be made within the ordinary skill in the art without departing from the spirit and scope of the invention.

I claim:

1. A method of making nickel-base superalloy bodies characterized by an aligned elongated grain or a monocrystalline structure comprising the steps of:

a. hot compacting a nickel-base superalloy powder to form a relatively dense solid consisting essentially of a γ' precipitate phase within a γ matrix,

b. solution heat treating the dense solid at a temperature below the incipient melting point and high enough to dissolve a substantial portion of the γ' phase,

c. subjecting the material to a sufficient strain below the recrystallization temperature to permit subsequent recrystallization, and

d. unidirectionally recrystallizing the strained material in a temperature gradient below the incipient melting temperature and above the γ' solvus temperature to form a body having an aligned elongated grain or monocrystalline structure.

2. The method of claim **1,** wherein the strain is equivalent to an elongation at room temperature of about 1–3 percent.

3. The method of claim **1,** wherein the strain is equivalent to an elongation at a temperature of 1200°–1400° F. of 8–10 percent.

4. The method of claim **1,** wherein the nickel-base superalloy powder is compacted by canning in a steel jacket under vacuum to form a billet and the billet is hot extruded.

5. The method of claim **1,** wherein said nickel-base superalloy powder is dispersed with up to 10 percent by volume of a chemically inert phase.

6. The method of claim **5,** wherein said chemically inert phase is alumina.

7. The method of claim **1,** wherein said alloy consists essentially in weight percent of about:

Composition	Weight %
Aluminum	4.25
Chromium	9.25
Titanium	4.0
Cobalt	10.0
Molybdenum	2.0
Tungsten	7.0
Tantalum	3.75
Carbon	0.17
Boron	0.015
Zirconium	0.05
Nickel	Balance

8. The method of claim **1,** wherein the strained material is unidirectionally recrystallized by drawing the material through a gradient furnace having a temperature gradient of at least 1000° F. per inch and the material is drawn at a rate of about 0.5–2 inches per hour.

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