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[54] **METHOD OF FORMING ARTICLES OF MANUFACTURE FROM SUPERALLOY POWDERS**

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Related U.S. Application Data

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[52] **U.S. Cl.** **75/213,** 29/182, 29/420.5, 75/200, 75/214, 75/226

[51] **Int. Cl.** **B22p 1/00**

[58] **Field of Search** 75/226, 213, 214, 75/200; 29/182, 420.5

[56] **References Cited**

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[57] **ABSTRACT**

A highly alloyed superalloy material is obtained using prealloyed powders. The material is easily shaped at high temperatures when it becomes superplastic because of its particular microstructure.

9 Claims, No Drawings

METHOD OF FORMING ARTICLES OF MANUFACTURE FROM SUPERALLOY POWDERS

RELATED APPLICATION

This application is a division of copending application Ser. No. 29,917 filed Apr. 20, 1970, now U.S. Pat. No. 3,702,791.

ORIGIN OF THE INVENTION

The invention described herein was made by employees of the United States Government and may be manufactured and used by or for the Government for governmental purposes without the payment of any royalties thereon or therefor.

BACKGROUND OF THE INVENTION

This invention is concerned with shaping high strength superalloy materials made by powder metallurgy. An ultrafine grain size produced in these materials may result in superplastic behavior at high temperatures.

Conventionally cast and wrought alloys are utilized for the hot components of gas-turbine engines. Cast nickel-base alloys are used for turbine discs. In more advanced engines wrought nickel-base alloys are also used for compressor discs and blades in the latter compressor stages.

The operating cycle temperatures must be raised in advanced engines to meet the demand for increased performance. Cast nickel-base alloys that can be used at high temperatures throughout the engine have been suggested. However, most high strength nickel-base alloys are highly alloyed and metallurgically very complex. As a consequence, severe macro- and micro-segregation as well as porosity can occur in castings, such as turbine buckets and stator vanes, so that the full-strength potential of the alloy is not realized and a broad scatter of lives is obtained. Also, in ingots, the usual starting stock for breakdown operations, segregation increases the difficulty of forming the alloys in forging operations.

SUMMARY OF THE INVENTION

Fine prealloyed powders of highly alloyed superalloy compositions may be consolidated and then shaped in separate steps. Likewise these powders may be consolidated and shaped simultaneously. During the shaping operation the consolidated powders are heated to temperatures at which strength rapidly approaches zero and the material exhibits superplastic behavior. At this temperature only relatively low pressures need be applied to drastically alter the shape of the material.

At intermediate temperatures significant increases in strength over the cast or wrought counterparts of the alloyed material can be obtained by alloys consolidated or shaped in accordance with the invention. Suitable heat treatments can also improve properties over a wide temperature range compared to the cast or wrought counterparts of the alloys.

OBJECTS OF THE INVENTION

It is, therefore, an object of the present invention to produce articles of manufacture from superalloy compositions which are too highly alloyed to be formed by normal forging type operations.

A further object of the invention is to provide a method of making a superalloy article of manufacture

in which significant deformation is achieved with relatively low applied forces using a minimum number of steps.

Another object of the invention is to provide superalloys having higher strength at intermediate or at high temperatures that can be obtained by conventional cast and wrought processes.

These and other objects of the invention will be apparent from the specification which follows.

DESCRIPTION OF THE PREFERRED EMBODIMENT

The method of the present invention utilizes prealloyed powders of a highly alloyed cast superalloy composition. The powders are extremely fine and have a high purity.

The powders can be prepared by inert gas atomization or other methods, such as the rotating electrode method. When the powders are produced by atomization, remelt stock is first melted under an inert gas or in vacuum in an induction furnace, and the melt is then atomized under an inert gas. The resulting powders are screened, and only suitable powders are then used for further processing in accordance with the invention.

The prealloyed powders are then consolidated in the form of either bar stock or preforms. More particularly, the prealloyed powders can be made into bar stock by extrusion, or by a combination of hot pressing and extrusion. These powders can also be made into preforms for turbine buckets or other desired articles of manufacture. These preforms are made by extrusion, by pressing in a shaped die, or by enclosing the powder in a suitably shaped container, such as a metallic can.

An important feature of the invention is that the consolidated powders are heated to a temperature above which the material exhibits superplastic behavior and below a temperature which causes incipient melting. While a blank or preformed shape of these consolidated powders is in this temperature range pressure is applied to form the powders into the desired configuration. This pressure may be applied unidirectionally to suitably shaped dies.

Because of the superplastic behavior of the material a very low pressure is required to shape the consolidated powders. The shaping may be accomplished at pressures as low as 1,000 psi. A relatively lost cost, low strength material may be used for the dies.

After the superplastic forming operation the formed part or article of manufacture may then be heat treated to obtain suitably coarse microstructures for superior high temperature strength. If an isostatic pressure is simultaneously imposed the heat treating temperature may be above the incipient melting point or solidus of the powdered alloy material.

EXAMPLES

In order to better illustrate the invention test samples of a nickel-base superalloy described in U.S. Pat. No. 3,276,866. were prepared and tested. The nominal composition of the alloy is shown in TABLE I.

TABLE I— NOMINAL COMPOSITION OF ALLOY

Element	wt. %
Tantalum	8
Tungsten	4
Molybdenum	4
Columbium	2.5
Chromium	6
Aluminum	6

Zirconium	0.75
Carbon	0.125
Boron	0.004
Nickel	Balance

Vacuum remelt stock of this cast nickel-base superalloy was melted under vacuum in an induction furnace. The melt was atomized under argon to spheroidal powders which were screened with Tyler screens to —60 mesh. Only the —60 mesh fraction was used for further processing. The sieve analyses for the —60 mesh fraction for the alloy is shown in TABLE II.

These fine powders were sealed in evacuated mild steel cans. The canned powders were heated to 2,200° F in a furnace and transferred to an extrusion press. Here the powders were extruded into bars and the cans were reduced in size from 2 inches to approximately 9/16 inch in diameter by passing them through an extrusion die.

TABLE II — PARTICLE SIZE DISTRIBUTION OF ATOMIZED POWDER

Tyler screen size	Percent
60/100	5.0
100/500	13.5
150/270	30.0
270/325	7.0
325/400	9.0
400	35.5

The bars were first tested in the as-extruded condition. The nickel-base alloy had an elongation of more than 600 percent after testing at 1,900° F and 1,000 psi for 4.1 hours. This and similar high elongations which occurred in elevated temperature tensile and stress rupture tests indicated superplastic behavior in this temperature region.

Samples of the as-extruded powder product of the alloy were upset and formed in shapes in closed dies to show that the material can be formed in compression to take advantage of this superplastic behavior. A hydraulically operated press with an in-place graphite susceptor induction heating furnace was used. Bar specimens approximately ⅝ inch high were heated to 2,000° F and pressed. Pressure was applied to the circular ends of the specimens through high temperature alloy plates which were heated to the same temperature as the specimen. An initial load of 155 pounds was applied. The load was increased as necessary to maintain a relatively constant strain rate of between 0.03 to 0.07 inch per inch per minute. This strain rate was used to approximate the rate observed when superplasticity was encountered with the alloy in a stress rupture test. The upset specimen had a diameter of 1.1 inch and a thickness of 0.175 inch after pressing.

Standard heat treatments to effect solutioning and aging were performed in vacuum or under argon or unmachined extruded bars of the nickel-base alloy. These heat treatments did not cause appreciable grain coarsening or solutioning of the microstructure of extruded powder products. However this heat treatment did substantially improve stress rupture life for the alloy compared to the life of the as-extruded powder produced at an intermediate temperature. At 1,200° F and 105,000 psi the extruded and heat-treated powder product of the alloy had a rupture life of 975 hours, whereas the as-extruded powder product had a life of 374 hours.

The standard heat-treatments on the extruded samples of the alloy had substantially lower rupture life at

high temperatures of 1,800° to 2,000° F than as-cast samples of the same alloy. For the heat treated, extruded powder product it was 2.2 against 90 hours at 1,900° F and 15,000 psi.

By simultaneously applying pressure and heat, the incipient melting point of the as-extruded nickel-base alloy powder product was exceeded without void formation. The product was successfully heated to 2,400° F which is about 200° F above the incipient melting point under a pressure of 10,000 psi. This simultaneous application of pressure and the high temperature coarsened the microstructure to a greater degree than is possible by conventional heat treatments.

Test samples of a commercial cobalt-base alloy were also prepared and tested to illustrate the beneficial effect of the heat treatment that utilized both high temperatures above the incipient melting point and high pressures. The cobalt-base alloy identified as HS-31 was made by the aforementioned prealloyed powder process.

As-extruded bars of the cobalt-base alloy were heated treated for 1 hour at 2,400° F at atmospheric pressure. This is about 60° F above the incipient melting point.

The grain growth was accompanied by the formation of large voids. Subsequent application of isostatic pressure of 30,000 psi at 2,200° F grew the grains further and closed the voids. Operation at 13,000 psi and 1,800° F resulted in a 20 hour life, which is double that of the as-cast alloy. Operation at 61,000 psi and 1,200° F resulted in a 420 hour life compared to 10 hours for the cast alloy.

What is claimed is:

1. A method of making a superalloy article of manufacture comprising;

producing prealloyed powders of a highly alloyed superalloy composition selected from the group consisting of nickel-base super-alloys and cobalt-base superalloys,

extruding said prealloyed powders to consolidate the same to a predetermined configuration,

heating the consolidated prealloyed powders to a temperature at which the consolidated powders exhibit superplastic behavior, said temperature being substantially below the temperature which causes incipient melting, and

shaping the heat consolidated powder by superplastic deformation.

2. A method as claimed in claim **1** wherein the prealloyed powders are formed by inert gas atomization.

3. A method as claimed in claim **1** wherein the prealloyed powders are a nickel-base alloy.

4. A method as claimed in claim **3** wherein the nickel-base alloy has a nominal composition in weight percent of 8 percent tantalum, 4 percent tungsten, 4 percent molybdenum, 2.5 percent columbium, 6 percent chromium, 6 percent aluminum, 0.75 percent zirconium, 0.125 percent carbon, 0.004 percent boron, and the balance nickel.

5. A method as claimed in claim **4** wherein the prealloyed powders are shaped at a strain rate of between about 0.03 to 0.07 inch per inch per minute.

6. A method as claimed in claim **1** wherein the prealloyed powders are a cobalt base alloy.

7. A method as claimed in claim **1** wherein the prealloyed powders are consolidated by extruding the prealloyed powders into bar stock.

8. A method as claimed in claim **1** wherein the prealloyed powders are consolidated while enclosed in a shaped container.

9. A method as claimed in claim **8** including the step of enclosing the powder in a metallic can.

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