

1

3,700,434

TITANIUM-NICKEL ALLOY MANUFACTURING METHODS

Stanley Abkowitz, Lexington, Mass. (8 A St., Burlington, Mass. 01803), and John M. Siergiej, Wayland, and Ronald R. Regan, Revere, Mass.; assignors to said Abkowitz

No Drawing. Filed Apr. 21, 1969, Ser. No. 818,054

Int. Cl. B22f 1/00

U.S. Cl. 75—214

7 Claims 10

ABSTRACT OF THE DISCLOSURE

Titanium and nickel are alloyed by a unique processing procedure to form highly useful titanium-nickel alloys such as the well-known Ti-55 Ni and Ti-60 Ni alloys. The methods include blending of powders of nickel and titanium followed by pressing and sintering to form preforms or final shapes of the desired titanium-nickel alloys.

BACKGROUND OF THE INVENTION

Titanium-nickel alloys have been developed in part by the United States government as described in Pat. No. 2,174,851. Such alloys have unique properties which include unusually high hardness values, corrosion resistance, nonmagnetic properties, and high strength. Another highly desirable unique characteristic is the "memory" of titanium-nickel alloys such as Ti-55 Ni. Known applications for these titanium-nickel alloys, which are known as "Nitinol" alloys, include noise and vibration reducing components, temperature sensing devices, outer space and hydrospace erectible structures and cryogenic equipment.

In spite of the well-known advantages of Nitinol alloys, their commercial use is limited due at least in great part to the high cost of working the alloys. The alloys are conventionally made by melting to form alloy ingots which ingots are then worked by mechanical means such as milling and forging to final finished shapes. The alloys are extremely brittle and require extreme care in mechanical working often necessitating repeated heating of the material as it work hardens and as it is shaped. The expense of such working steps often makes the alloy economically prohibitive for use in otherwise desirable commercial applications.

It is an object of the present invention to provide titanium-nickel alloys having good physical characteristics which alloys can be made rapidly and efficiently.

Another object of this invention is to provide methods of forming the alloys as described above in preforms and/or final shapes which minimize conventional mechanical shaping and forming techniques.

SUMMARY OF THE INVENTION

According to the invention, titanium and nickel powdered metal particles are blended to a desired alloy composition. The blend is then pressed directly into an as pressed preform close in shape and dimension to the final shape desired. The shape is then sintered in a protective inert atmosphere at a temperature in the range of from 1650° F. to 1750° F. to permit alloying of the particles and formation of a final shape desired or a preform close in shape and dimension to the final product desired. The resultant sintered shape requires little or no forming or machining by conventional mechanical shaping procedures and has the desirable properties of titanium-nickel alloys made by conventional melting techniques.

Since final shapes or preforms are product directly, the brittle nature of the titanium-nickel alloys does not pre-

2

clude their economical use for the large variety of commercial applications already known and for additional uses where costs were heretofore considered prohibitive. Costs of forming shaped articles are maintained at commercially feasible levels since conventional mechanical steps such as forging and milling are either eliminated or greatly reduced.

BRIEF DESCRIPTION OF PREFERRED EMBODIMENTS

The powders of titanium and nickel which are used in the process of the present invention must have high purity and are of irregular shape. For example, titanium particles in the powder preferably have a purity of at least 99.5% by weight with no more than 0.12% oxygen and the nickel powder preferably has a purity of at least 99.7% by weight. Irregular shaped particles are used since they maximize mechanical interlock of the particles. Preferably, the particle size of both the nickel and titanium particles are -100 mesh A.S.T.M. which aids in producing homogeneous products, however, particle sizes can be higher. Titanium powder useful in this invention includes standard commercial pure titanium particles of irregular shape. Nickel powder useful in this invention include standard commercially pure nickel particles of irregular shape.

In a first step, the selected metal powders are mixed in a conventional powder-metal blender to form a uniform mixture of the powders. Preferably the particle sizes of the nickel and titanium particles in the powders are substantially uniform and are of irregular shape.

The composite blend is then cold pressed directly into an as pressed preform or substantially the final shape desired in either conventional mechanical or conventional isostatic pressing equipment. The pressing forms a mechanical interlock of the particles and is preferably carried out at room temperature for a few seconds to a few minutes as is conventional when pressing powdered metals. Hot pressing below sintering temperatures can be used but provides no significant advantage while increasing cost.

The shape formed by the pressing step is then sintered in a protective atmosphere at a sintering temperature in the range of from 1650° F. to 1750° F. and preferably 1700° F. for a period of time sufficient to form the alloy without causing melting. Sintering within the temperature range stated is important to obtain the desired properties in the resultant alloys. The alloys formed by sintering can be further processed by additional heat treatments at temperatures above the 1750° F. limit. For example, where the nickel content of the alloy is approximately 60% by weight or higher, the sintered preforms or shapes can be heated to temperatures of about 2250° F. to increase the homogeneous nature of the alloy and increase the density. However, it is important to first permit alloying of the metals during sintering before raising the temperature above 1750° F.

The pressing step is preferably carried out at a pressure of between 30,000 to 160,000 p.s.i. with the lower figure insuring adequate as pressed density and the upper figure preventing die damage in conventional dies used.

The sintering step can be carried out for periods of time in the range of ½ hour to 20 hours and preferably 1 to 2 hours with additional times beyond 20 hours adding no useful benefits to the material and adding to the cost of the process. The protective atmosphere used can be a vacuum such as a vacuum in the range of 1×10^4 torr or can comprise an inert gas atmosphere in the conventional sintering enclosure of the preform or final shape using helium, argon or other inert gas. In some cases, a combination of a low vacuum and flushing inert gas

3

can be used as is known in the art. Preferably the sintering step is carried out at atmospheric pressure or in vacuum although higher pressures can be used.

It is preferred to press to form a mechanical interlock of the titanium and nickel particles with an as pressed density of at least 70% after which sintering is used to cause alloying and increase the density of the sintered preform or final shape to a value of at least 80% and preferably at least 90% of the theoretical density.

The amounts of titanium and nickel in the preform or final shape are substantially identical to the amounts of powders originally incorporated. Preferably the Nitinol alloys formed by the method of this invention generally comprise from 38 to 46% by weight titanium and 62 to 54% by weight of the alloy of nickel.

In a specific example of this invention, 54.5% by weight of irregular (acicular) particles of commercially pure nickel powder (99.7% purity) are uniformly mixed with 45.5% by weight commercially pure titanium particles (99.5% purity) of irregular (acicular) shape with all the particles having a size of -100 mesh A.S.T.M. The composite blend is then pressed into the shape of a standard tensile test blank (A.S.T.M. E8-61T) at standard room temperature (72° F.) using a pressure of 60,000 p.s.i. The blank formed which has a density of approximately 86% of theoretical density, is then sintered in a furnace at a vacuum of 1×10^4 torr and at a temperature of 1700° F. for 5 hours after which it was allowed to cool to room temperature in the furnace in accordance with conventional practice. The resulting sintered test blank has a density of 91% and substantially similar physical properties as a corresponding titanium-nickel alloy made by conventional melting techniques.

For example, the resultant alloy has an ultimate tensile strength of 29,400 p.s.i., a yield strength (0.2% p.s.i.) of 22,800, an elongation of 1.8% and a percent reduction of area of 3.2 when tested by standard A.S.T.M. methods (E-8).

The resultant alloy test blank (which is considered a final shape or preform) has the known memory characteristics of 55 Nitinol. One sample test blank formed as described in the above example was bowed in its flat plane when removed from the sintering furnace. Standard test blanks are flat and have dimensions of approximately 0.225 inch width by 0.125 inch thickness by $3\frac{1}{2}$ inch length but the test blank used was uniformly bowed from a flat surface on which its ends are placed by a maximum distance (X) of 0.047. This bow existed above the transition temperature. The sample was cooled in ice water and further deformed to increase the maximum distance (X) noted to 0.058. It was then cycled between 170° C. and 0° C. to check the memory characteristic with the results noted below:

Temperature:	Distance X, inches
0° C. -----	0.057
170° C. -----	0.048-0.049
0° C. -----	0.059
170° C. -----	0.048-0.049
0° C. -----	0.058-0.059

Thus the sample exhibited the expected memory characteristic of 55 Nitinol made by conventional ingot formation.

When the above example is repeated using 60% by weight of the nickel particles and 40% by weight of the

4

titanium particles as starting materials, a titanium-nickel alloy is formed as a test bar having a yield strength (0.2%) of 25,000 p.s.i. and a density of 89% of theoretical density. Similar useful results are obtained when the amounts of titanium and nickel are varied within the ranges given above.

While specific embodiments of the present invention have been described, many variations thereof are possible within the scope of this invention.

What is claimed is:

1. A method of forming a sintered alloy consisting essentially of titanium and nickel, having the characteristics of corresponding alloys formed by melt ingot processes,

15 said method comprising,
uniformly mixing acicular particles of titanium and nickel in an amount of from 38 to 46% by weight titanium and 62 to 54% by weight nickel, consolidating said particles by pressing at a pressure in the range of from 30,000 to 160,000 p.s.i.,
20 and then sintering at a temperature in the range of from 1650° F. to 1750° F. to form an alloy in a usable shape.

2. A method in accordance with the method of claim 1 wherein said particles of titanium and nickel have a size below 100 mesh.

3. A method in accordance with the method of claim 1 wherein said particles are first pressed into a mechanically interlocked shape by said consolidating and then sintered in a protective atmosphere to form said alloy.

4. A method in accordance with the method of claim 3 wherein said atmosphere is a vacuum.

5. A method in accordance with the method of claim 1 wherein said sintering is carried out for a period of from $\frac{1}{2}$ to 20 hours.

6. A method in accordance with the method of claim 5 wherein the as pressed density is at least 70% of theoretical density and the final sintered density is at least 80% of theoretical density.

7. A method in accordance with the method of claim 3 wherein said protective atmosphere is an inert gas.

References Cited

UNITED STATES PATENTS

2,163,224	6/1939	Alexander	75-225	X
3,335,002	8/1967	Clarke	75-214	X
2,839,819	6/1958	Platte	75-225	X
2,227,176	12/1940	Berghaus et al.	75-200	
3,164,466	1/1965	Yasuda et al.	75-200	

OTHER REFERENCES

Fundamental Principles of Powder Metallurgy, W. D. Jones, p. xi, Edward Arnold (Publishers) Ltd., London, 1960.

Treatise on Powder Metallurgy, vol. 1, C. G. Goetzel, p. 1, Interscience Publishers, Inc., New York, 1949.

CARL D. QUARFORTH, Primary Examiner

R. E. SCHAFER, Assistant Examiner

U.S. Cl. X.R.

29-182; 75-170, 224, 225