

Aug. 18, 1970

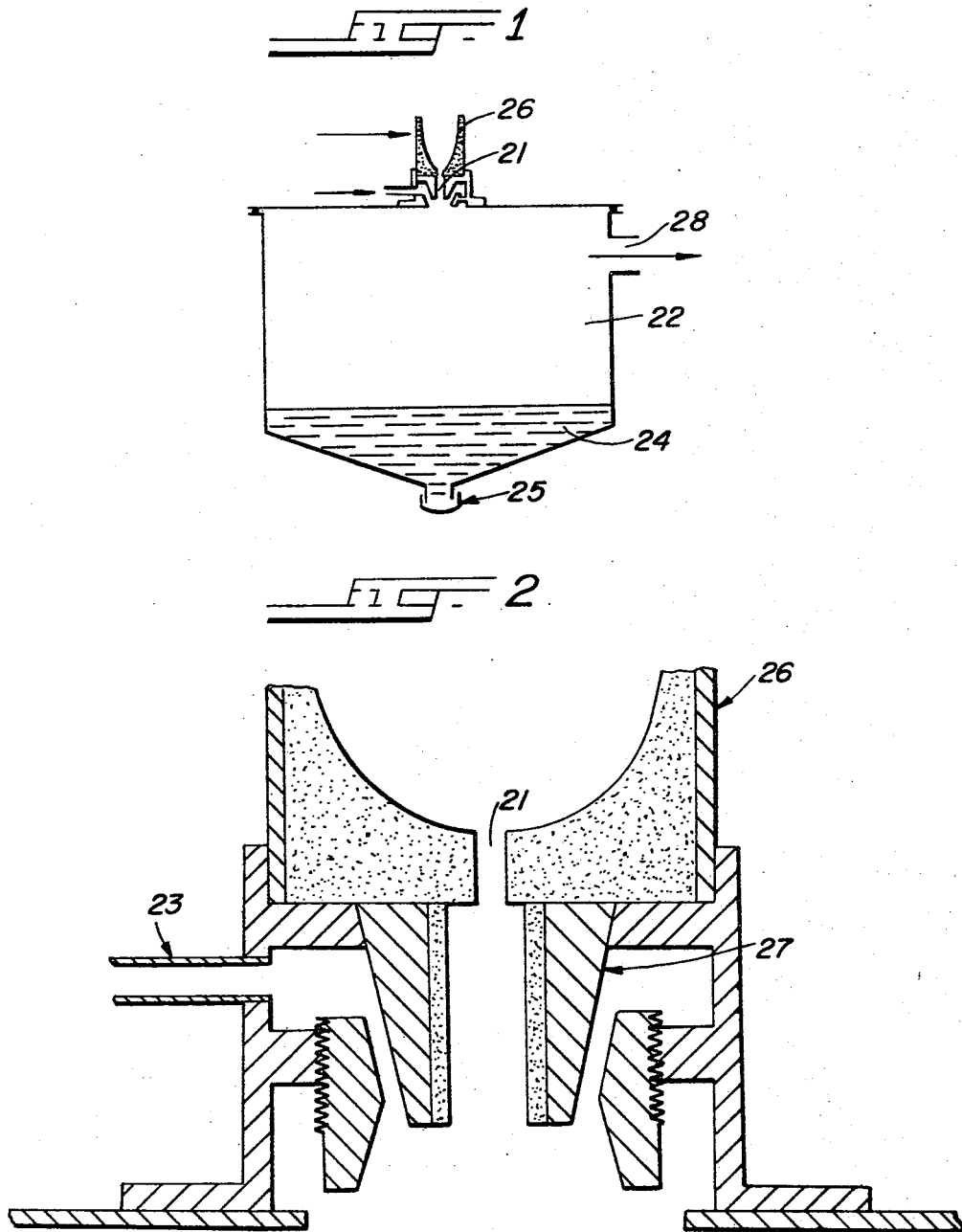
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3,524,744

NICKEL BASE ALLOYS AND PROCESS FOR THEIR MANUFACTURE

Filed Jan. 3, 1966

6 Sheets-Sheet 1



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NICKEL BASE ALLOYS AND PROCESS FOR THEIR MANUFACTURE

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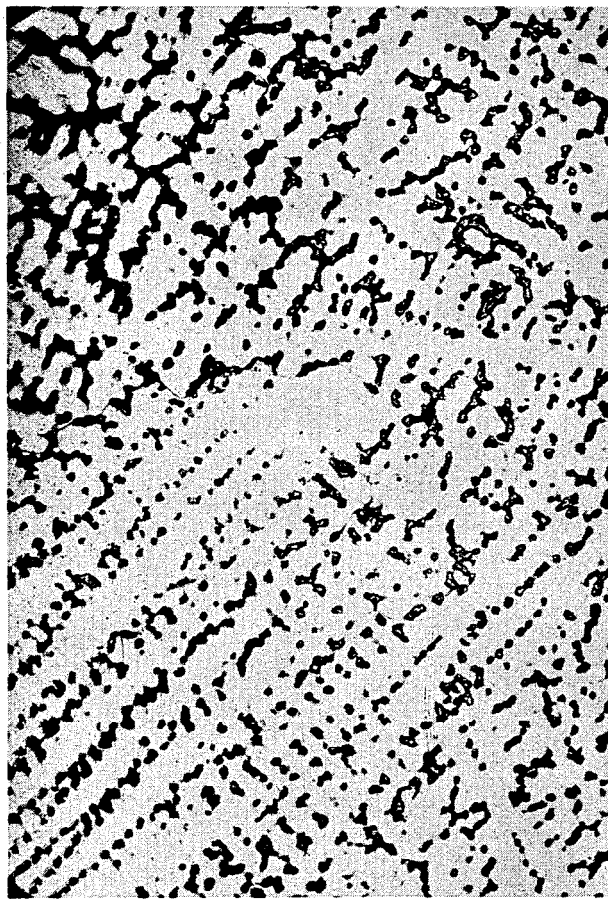


FIG. 3

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FIG. 4

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FIG. 5

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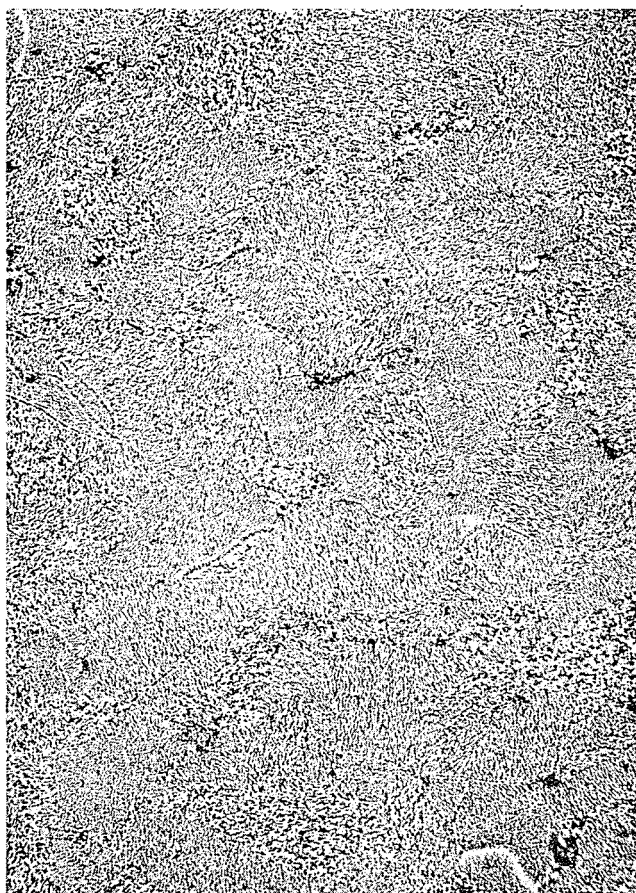


FIG. 6

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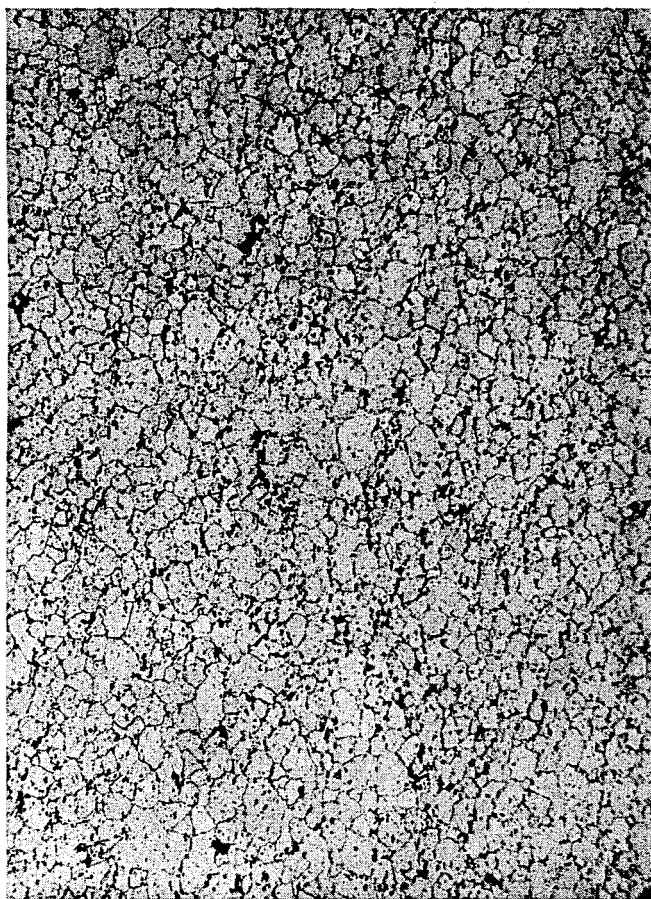


FIG. 7

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**NICKEL BASE ALLOYS AND PROCESS
FOR THEIR MANUFACTURE**
Niranjan M. Parikh, Chicago, Ill., assignor to IIT Research Institute, Chicago, Ill., a not-for-profit corporation of Illinois
Filed Jan. 3, 1966, Ser. No. 518,165
Int. Cl. B22d 23/08; B22f 7/06; C22c 19/00
U.S. Cl. 75-171 **4 Claims**

ABSTRACT OF THE DISCLOSURE

Superalloys characterized by a uniformly dispersed ultrafine hardening phase and a very fine matrix phase having a composition by weight consisting essentially of from 3.5 to 6 percent molybdenum, from 3.5 to 6.5 percent aluminum, from 12 to 17 percent chromium, iron up to 4 percent maximum, from 0.5 to 4 percent titanium, from 0 to 20 percent cobalt, carbon up to 0.20 percent maximum, boron up to 0.05 percent maximum, from 0 to 0.15 percent zirconium, manganese up to 0.25 percent maximum, silicon up to 0.50 percent maximum, 0 to 0.5 percent copper, balance nickel with incidental impurities.

The present invention relates to a novel group of nickel base "superalloys" which are characterized by a new and unique microstructure and to a process whereby such nickel base alloys may be made. More especially my invention is based upon my discovery that by the practice of a molten metal atomizing process to yield the alloy powders followed by the consolidation thereof as herein taught there results very desirable structural alloys featuring the new microstructure.

At the present time there are a number of relatively highly alloyed nickel base alloys, commonly referred to in the trade as "superalloys." These are heat-resistant, high-strength materials. One such alloy which is now commercially available has the following composition:

Carbon	0.08-0.20.
Manganese	0.25 max.
Silicon	0.50 max.
Chromium	12-14.
Molybdenum	3.8-5.2.
Aluminum	5.5-6.5.
Zirconium	0.05-0.15.
Iron	2.5 max.
Titanium	0.5-1.0.
Boron	0.0005-0.015.
Others	0.5 max. copper.
	1.8-2.8 columbium and tantalum.
Nickel	Balance.

This alloy is commonly referred to as "Inco 713C." Another of these nickel base superalloys commercially available has the following composition:

Carbon	0.15 max.
Chromium	13-17
Molybdenum	4.5-6.0
Aluminum	3.75-5.00
Iron	4.0 max.
Titanium	2.75-4.0
Boron	0.01-0.05
Cobalt	14-20
Nickel	Balance

This alloy is commonly referred to as "Udimet 700."

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To the best of my knowledge until the making of the discovery which led to the present invention the fabrication of these nickel base superalloys into structural materials has been a lengthy, expensive procedure and yet despite this one finds that the demands of modern-day technology for high strength, high temperature resistant materials is pushing the superalloys to their property extremes. On the other hand, as will be evident to those skilled in this particular art, I have discovered a relatively simpler and less expensive means of fabricating these nickel base materials and at the same time find as a result of my process there is considerable improvement in their properties. For example, the alloy compositions first given above in its cast state, i.e., following its normal method of its fabrication, has a room temperature tensile strength of around 120,000 p.s.i. When that very same alloy composition is made as taught herein to yield the desirable microstructures hereinafter more fully described, I obtain tensile strength values of around 200,000 p.s.i. Accordingly the effect and significance of my invention will be immediately apparent.

The as-cast nickel base alloys which correspond compositionally to the materials that I treat are characterized by either a very large acicular grain or relatively coarse dendritic structures. These acicular grains or dendritic structures, as the case may be, seriously detract from the physical and metallurgical properties of the materials but until the advent of my invention such materials could only be readily fabricated in their as-cast state and this resulted in these undesirable microstructures. Such acicular grains or dendritic structures also render these alloys brittle and extremely difficult to fabricate into useful shapes. When the very same alloys are made as taught herein they exhibit a fine microstructure, can be fabricated into useful shapes due to inherent high ductility of the fine microstructure and retain a large amount of the cold work induced during fabrication which phenomenon further improves the strength of the alloys.

As shown in the attached photomicrographs the present nickel base alloys are characterized by a substantially uniformly dispersed hardening phase which hardening phase is of very, very small size and it is this combination of dispersion and size which leads to the desirable properties of the nickel base alloys herein described and claimed.

Accordingly, a primary object of my invention is to provide nickel base alloys quite low in carbon content, which are characterized by a uniformly dispersed, ultra fine hardening phase and uniform, fine matrix grain size with resulting property improvement over known similar materials. The matrix grain size refers to the metallurgical grain structure.

A more specific object of my invention is to provide an alloy consisting essentially of from 3½ to 6% molybdenum; from 3½ to 6.5% aluminum, from 12 to 17% chromium, iron 4% maximum, from 0.5 to 4% titanium, from 0 to 20% cobalt, carbon up to 0.20% maximum, boron up to 0.05 maximum, from 0 to 0.15% zirconium, manganese 0.25% maximum, 0 to 0.5% copper, silicon 0.50% maximum, 1.8 to 2.8% columbium and tantalum, balance nickel with incidental impurities, which alloy is characterized by a substantially uniformly dispersed hardening phase of a size predominantly less than a few microns and a matrix grain size that is essentially less than about 50 microns.

Another important object of my invention is to provide a process of making these nickel base alloys characterized by the unique microstructure aforesaid which includes the steps of atomizing a molten alloy charge to form powders thereof and consolidating said powders into structural alloy stock.

These and other objects, features and advantages of my invention will become apparent to those skilled in this particular art from the following detailed disclosure thereof and the accompanying drawings in which:

FIG. 1 schematically illustrates an atomizing chamber for use in the practice of the present invention;

FIG. 2 schematically illustrates atomizing apparatus for use herewith;

FIG. 3 is a photomicrograph of a commercially available, as-cast nickel base superalloy, Inco 713C, at a magnification of 150 \times ;

FIG. 4 is a photomicrograph of an alloy of the same composition as shown in FIG. 3 made by the process herein taught at a magnification of 320 \times ;

FIG. 5 is a photomicrograph of another nickel base alloy, Udimet 700, in its commercial as-cast condition at a magnification of 150 \times ;

FIG. 6 is a photomicrograph at a magnification of 500 \times of an alloy of the same chemical composition as that shown in FIG. 5 but made according to the teachings of this invention; and

FIG. 7 is a photomicrograph at a magnification of 200 \times of an alloy of the same chemical composition as that shown in FIG. 5 but made according to the teachings of this invention.

In order to prepare the present alloys I commence with an atomization process of an appropriate molten alloy charge. The apparatus for this is schematically illustrated in FIGS. 1 and 2 with it of course being understood that other similar apparatus may be likewise employed. In such process one may either begin with standard alloy materials if they are available, as the initial charge or the charge may be prepared from its elemental constituents. 5 pound charges of the two superalloy compositions given above were first weighed up and then melted in an argon atmosphere in a high frequency induction unit. The heating element is not shown in the drawings and certainly other equivalent melting means can be used. It is useful to melt in the protection of inert atmosphere or vacuum in order to prevent oxidation and other undesirable contamination of the melt. The molten metal charge is then poured into a preheated (to prevent chilling) magnesia-lined pouring cup 26 placed at the top of the atomizing chamber 22. The melt passes first through a hole 21 at the bottom of the pouring cup through a refractory-lined nozzle 27. By this means the molten charge enters the atomizing chamber 22. As the charge emerges from the nozzle 27 the molten stream is first broken up into fine particles and then quickly quenched by a high pressure inert gas stream entering the chamber 22 through the gas inlet port 23. In order to assure rapid quenching of the molten atomized metal powders a water reservoir 24 is provided at the bottom of the chamber which may operate in conjunction with the atomizing stream to rapidly quench the particles. At the bottom of the chamber a capped opening 25 is provided for metal powder removal. To complete the structure there is also provided a gas exit port 28. After the alloy powders are formed they are removed from the atomizing chamber, separated from the water and dried. Drying may be most simply accomplished by limited heating to drive off the water.

The alloy powders were then canned in mild steel encapsulating material. Air was evacuated from the cans and they were then sealed. Such cans were then hot-pressed to consolidate the atomized powders. In carrying out such hot-pressing it should be noted that great care should be taken to avoid melting the particles and

at the same time contamination of the resulting sintered billets should also be avoided. The stainless steel alloys of my invention were sintered readily under an argon atmosphere to avoid the loss of volatile chromium among other things. Suitable sintering temperatures are between the range of 2550° to 2690° F.

I find it is preferable to hot-press the powders rather than merely sinter them. To accomplish this the alloy powders were sealed in 0.040 inch thick stainless steel cans, heated at 2190° F. and then hammer forged without side restraint. The height reduction was about 60%. Although the edges of the forged pieces were somewhat porous and cracked, I found that a slightly below such surfaces the forged pieces were 100% dense.

The most preferable mode of consolidating powders is by extrusion. Green compacts of the atomized powders, 1½ inch high by 1 inch diameter were sealed in close fitting mild steel cans under vacuum. These were then heated to 2200° F. in a resistance furnace, then dropped quickly into an extrusion die and rapidly loaded to 40 tons per square inch. Such load was maintained for 15 seconds, then the in plate was removed and the compact was pushed out of the die. 100% dense structures were thus obtained.

The advantages of my invention may most readily be seen by comparing FIG. 3 with FIG. 4 and FIGS. 6 and 7 with FIG. 5. The commercial materials are illustrated in FIGS. 3 and 5 and the fine-grained structure of the present alloys is readily shown in FIGS. 4, 6 and 7.

In fabricating the present alloy it should be noted that both the oxygen content and the nitrogen content should be maintained as low as possible, preferably below 1000 parts per million oxygen and below 100 parts per million nitrogen.

As seen above the alloys made according to the present invention consist of very fine matrix phase and very fine hardening phase. Such alloys are characterized by high ductility and accordingly one may readily fabricate them into useful shapes. Such fabricating step is of the utmost importance from the economic as well as a property standpoint. As a result of the fine-grained size the alloys are not only readily fabricable but have considerably better tensile strengths as opposed to the coarse cast structures of the prior art. Alloys made as taught herein are characterized by tensile strengths of around 200,000 p.s.i. as compared with values of around 120,000 p.s.i. for the commercially available materials.

It will be understood that various modifications and variations may be effected without departing from the spirit or scope of the novel concepts of my invention.

I claim as my invention:

1. An alloy consisting essentially of from 3.5 to 6% molybdenum; from 3.5 to 6.5% aluminum; from 12 to 17% chromium; iron 4% maximum; from 0.5 to 4% titanium; from 0 to 20% cobalt; carbon up to 0.20% maximum; boron up to 0.05% maximum; from 0 to 0.15% zirconium; manganese 0.25% maximum; silicon 0.50% maximum; 0 to 0.5% copper, balance nickel, said alloy being characterized by a substantially uniformly dispersed hardening phase in the major phase matrix, said hardening phase being essentially of a size less than a few microns and the said matrix phase being essentially less than about 50 microns.

2. The alloy as defined in claim 1 to which is added from 1.8 to 2.8% of a metal selected from the group consisting of columbium and tantalum and mixtures thereof.

3. The method of making an alloy characterized by a substantially uniformly dispersed hardening phase in the major phase matrix, said hardening phase having a grain size of less than 1 micron which comprises the steps of: atomizing an alloy charge of the composition defined in claim 1 in the presence of an inert environ-

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ment; rapidly quenching the atomized particles and consolidating said particles into solid stock.

4. The method of making an alloy characterized by a substantially uniformly dispersed hardening phase in the major phase matrix, said hardening phase having a grain size of less than 1 micron which comprises the steps of: atomizing an alloy charge of the composition defined in claim 2 in the presence of an inert environment; rapidly quenching the atomized particles and consolidating said particles into solid stock.

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References Cited

UNITED STATES PATENTS

3,244,506 4/1966 Reen ----- 75-0.5

5 L. DEWAYNE RUTLEDGE, Primary Examiner
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U.S. Cl. X.R.

10 75-0.5, 204, 214; 29-182.7