

July 28, 1970

N. M. PARIKH
STAINLESS STEELS

3,522,020

Filed Jan. 3, 1966

3 Sheets-Sheet 1

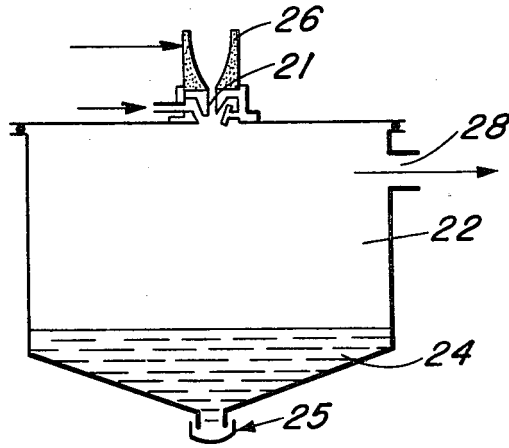


Fig. 1

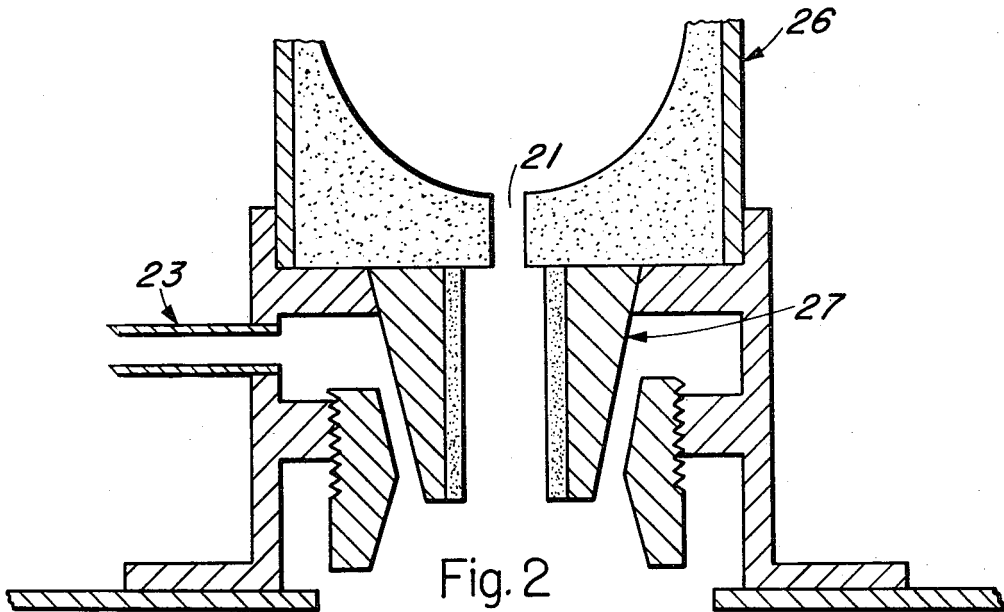


Fig. 2

INVENTOR.
NIRANJAN M. PARIKH

BY *Albert Seigel*

July 28, 1970

N. M. PARIKH

3,522,020

STAINLESS STEELS

Filed Jan. 3, 1966

3 Sheets-Sheet 2



FIG. 3

INVENTOR.
NIRANJAN M. PARIKH

July 28, 1970

N. M. PARIKH
STAINLESS STEELS

3,522,020

Filed Jan. 3, 1966

3 Sheets-Sheet 3



FIG. 4

INVENTOR.
NIRANJAN M. PARIKH

1

3,522,020

STAINLESS STEELS

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. 518,130

Int. Cl. B22f 3/12

U.S. Cl. 29—182.5

1 Claim

ABSTRACT OF THE DISCLOSURE

Fully dense stainless steel alloys characterized by having a microstructure exhibiting a substantially uniformly dispersed carbide phase of particle size essentially less than one micron, are produced from pre-alloyed powders by rapidly quenching an atomized alloy charge and subjecting the solidified particles in a suitable container to the step of hot consolidation to produce fully dense metal stock directly from prealloyed powder.

The present invention relates to a novel stainless steel of unique microstructure and to a process whereby such steel may be readily made. More especially my invention is based upon my discovery that by the use of a molten metal atomization process to yield alloy powders followed by the consolidation thereof as herein taught, there results very desirable structural alloys characterized by such microstructure.

At the present time there are commercially available cast stainless steels which are designated as "Ph 15-7 Mo" by the trade. Such alloys have the following composition:

Carbon—0.09 max. wt. percent
Manganese—1.0 max.
Silicon—1.0 max.
Chromium—14-16
Nickel—6.5-7.75
Molybdenum—2-3
Aluminum—0.75-1.5
Sulfur—0.03 max.
Iron—balance

The alloy identified as Ph 15-7 Mo is one of a number of alloys, both iron base and other base, which in present technology are referred to as "superalloys." These are heat-resistant, high strength materials which are quite well suited to meet a number of the extreme demands now placed upon modern structural materials. However, until the making of my invention the manufacture of various structures out of such superalloys has been a fairly expensive process involving the steps of casting, rolling, forging or extrusion and subsequent machining. Yet even despite the expense of the materials, the present requirements of high temperature uses is pressing the operating capabilities of the superalloys.

To meet this dual problem of expense and the need for improved superalloys I have discovered a new method of their fabrication, especially for Ph 15-7 Mo, which not only is considerably more economically feasible than the processes of fabrication of the prior art, but of even greater importance I discover that such alloys made as herein taught are characterized by a unique, fine-grained microstructure, which leads to improved physical and mechanical properties therein.

I accomplish these beneficial results by first atomizing a melt of the appropriate alloying charge to produce powders thereof followed by consolidation of the powders into solid stock. In carrying out the atomization step due care must be given in order to prevent the pick-up of embrittling impurities such as oxygen and nitrogen.

2

Powder consolidation is preferably accomplished by hot pressing the material while again protecting it from picking up undesirable embrittling impurities and in some cases following the hot pressing by an extrusion step or the like to yield the desired end-product structural shape.

The improved properties of the present stainless steels stem from the novel microstructure which is obtained by my process. Rather than the coarsened grain structure found in commercial alloys of similar compositions, I find that in the alloys made as herein taught there is an extremely fine-grained microstructure and wherein the hardening phase is of exceptionally small grain size—usually smaller than 1 micron—and furthermore that such hardening phase is substantially uniformly dispersed throughout the material.

Accordingly, a primary object of my invention is to provide a new stainless steel of the type designated Ph 15-7 Mo which is characterized by a fine-grained microstructure and wherein the hardening phase is of a size essentially less than 1 micron and wherein said hardening phase is uniformly dispersed through the major phase matrix.

Another object of my invention is to provide a process of making such stainless steels of unique microstructure which includes the steps of atomizing to form powders thereof followed by the consolidation of said powders into metal 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 Ph 15-7 Mo alloy at a magnification of 150X; and

FIG. 4 is a photomicrograph of an alloy made as herein taught at a magnification of 320X.

In order to prepare the present alloys I start 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 cast alloy materials, if they are available, as the initial charge, or the charge may be prepared from its elemental constituents. The charge of the materials was first weighed up and then melted under an argon atmosphere in a high frequency induction unit. The heating element is not shown in the drawings and obviously a number of equivalent melting means can be used. It is necessary to melt under the protection of inert atmosphere or vacuum in order to prevent oxidation and other undesirable contamination of the melt.

I weighed up and then melted 5 pound charges of the composition set out above of alloys Ph 15-7 Mo.

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 I provide a water reservoir 24 at the bottom of the chamber which may operate in conjunc-

tion with the atomizing stream to rapidly quench the particles. At the bottom of the chamber I provide a capped opening 25 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 sintering 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° and 2690° F.

I find it is preferable to hot-press the powders rather than merely sintering them. To accomplish this the alloy powders are 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 at 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 in 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 readily be seen by comparing FIGS. 3 and 4 hereof. FIG. 3 is a photomicrograph of commercially available Ph 15-7 Mo and

the coarse martensitic-type structure is immediately apparent. In distinction to this the fine-grained structure illustrated in FIG. 4 of a T-section extruded as herein taught and the advantages thereof will be immediately apparent to those skilled in this art.

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

It should 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. A fully dense consolidated powder alloy comprising: from 14 to 16% chromium; from 6.5 to 7.75% nickel; from 2 to 3% molybdenum; from 0.75 to 1.5% aluminum; carbon up to 0.09% maximum; manganese up to 1%; silicon up to 1%; sulfur maximum of 0.03%, balance iron, said alloy being characterized by a substantially uniformly dispersed hardening phase in the fine-grained major phase matrix, said hardening phase being essentially of a size less than a few microns and the said fine-grained matrix phase being less than about 50 microns.

References Cited

UNITED STATES PATENTS

2,958,618	11/1960	Allen	75—124 XR
2,958,617	11/1960	Perry	75—124 XR
3,071,463	1/1963	Hausner	75—213 XR
3,151,978	10/1964	Perry	75—124

OTHER REFERENCES

Goetzel, "Treatise on Powder Metallurgy," vol. I, pp. 1-5, 1949, Interscience Publishers, Inc., N.Y.

CARL D. QUARFORTH, Primary Examiner

A. J. STEINER, Assistant Examiner

U.S. Cl. X.R.

75—124, 201