

Jan. 5, 1971

V. C. PETERSEN ET AL

3,552,947

METHOD FOR MELTING TITANIUM-BASE ALLOY

Filed Jan. 18, 1968

2 Sheets-Sheet 1

FIG. 1.



FIG. 2.



INVENTORS.
VINCENT C. PETERSEN &
RUSSELL C. BUEHL

By *Clair L. Mullens*
Attorney

Jan. 5, 1971

V. C. PETERSEN ET AL

3,552,947

METHOD FOR MELTING TITANIUM-BASE ALLOY

Filed Jan. 18, 1968

2 Sheets-Sheet 2

FIG. 3.

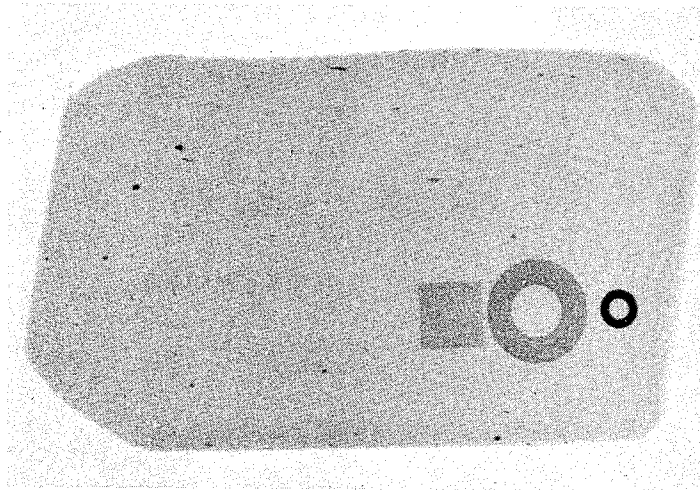
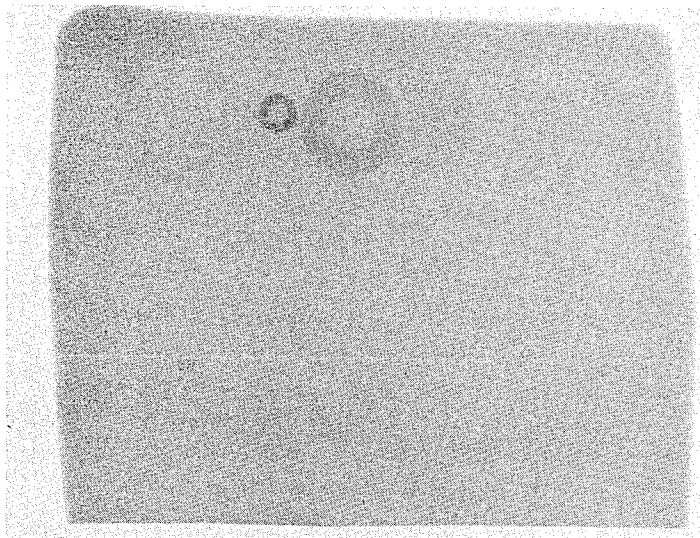


FIG. 4.



INVENTORS.
VINCENT C. PETERSEN &
RUSSELL C. BUEHL
By *Clair L. Mullen Jr.*
Attorney

1

3,552,947

METHOD FOR MELTING TITANIUM-BASE ALLOYS

Vincent C. Petersen, North Fayette Township, Allegheny County, and Russell C. Buehl, Beaver, Pa., assignors to Crucible Inc., a corporation of Delaware

Filed Jan. 18, 1968, Ser. No. 698,795

Int. Cl. C22d 7/00; C22c 15/00

U.S. Cl. 75—10

5 Claims

ABSTRACT OF THE DISCLOSURE

The present invention relates to a method for producing alloys containing effective and substantial quantities of molybdenum. More particularly, the invention relates to a method for producing molybdenum-containing, titanium-base alloys exhibiting a substantially homogeneous microstructure wherein the molybdenum is dissolved and dispersed substantially uniformly throughout the matrix metal. This is achieved by providing a charge of porous molybdenum particles, mixing said charge with a charge of particulate material, which may be titanium, zirconium or base alloys thereof, forming a consumable electrode from said mixture, and vacuum arc melting said consumable electrode to form the desired homogeneous alloy.

In the production of molybdenum-containing alloys, difficulties are encountered in achieving complete dissolution of the highly refractory molybdenum when alloyed with metals having melting points lower than molybdenum, particularly metals such as titanium and zirconium. Molybdenum has a melting point of 2610° C., whereas, zirconium has a melting point of 1852° C., and titanium has a melting point of 1668° C. Molybdenum-containing alloys of this type are conventionally produced by forming a consumable electrode of the base metal and alloying elements by compacting particles thereof. The consumable electrode is arc melted in a vacuum or inert atmosphere to form an ingot. Thereafter, the ingot is used to produce additional electrodes, which are again vacuum arc melted. This procedure is repeated until the desired purity and dissolution of the alloying elements are achieved. Although, because of its highly refractory nature, molybdenum is difficult to dissolve, nevertheless, this problem can be overcome by numerous consumable-electrode meltings. Specifically, as many as ten electrode meltings may be required for this purpose. This obviously renders the operation so expensive as not to be commercially feasible. Moreover, these numerous meltings introduce product-quality problems, such as excessively high oxygen content. In addition, since molybdenum is of a substantially greater density than the base alloy titanium, in consumable-electrode melting operations the molybdenum particles tend, during the electrode-melting operation, to drop in unmelted form from the electrode into the molten pool of the ingot and segregate to the bottom thereof before dissolution. This results in a segregation of the molybdenum in the resulting solidified ingot, even if the molybdenum is subjected to sufficient time at temperature to dissolve the metal. This segregation carries over through any remelts so that the final product is unhomogeneous. It may be seen, therefore, that this problem cannot be overcome by numerous consumable-electrode meltings, as is the case with the problem of achieving dissolution of the molybdenum particles. The problem with respect to segregation of the molybdenum during melting may be more fully appreciated when it is noted that the specific gravity of molybdenum is more than twice that of titanium; molybdenum has a specific gravity of 10.2, whereas, titanium has a specific gravity of 4.5.

2

It is, therefore, a primary object of the present invention to provide a method for producing homogeneous alloys of molybdenum and a lower melting point, lower density base metal, such as titanium or zirconium.

This, as well as other objects of the invention, as well as a complete understanding thereof, will be apparent from the following description and drawings, in which:

FIG. 1 is a photomicrograph of a molybdenum-containing titanium-base alloy produced in the conventional manner and exhibiting a characteristic dense, unmelted molybdenum inclusion;

FIG. 2 is a photomicrograph of a molybdenum-containing titanium-base alloy, substantially the same as the alloy of FIG. 1, produced in accordance with the method of the present invention and exhibiting a homogeneous microstructure free from segregated dense molybdenum inclusions;

FIG. 3 is a radiograph of the alloy of FIG. 1 showing the presence of dense, unmelted molybdenum inclusions in the alloy resulting from molybdenum segregation;

FIG. 4 is a radiograph of the alloy of FIG. 2 showing the desired homogeneous microstructure.

The problem of unhomogeneous molybdenum-containing alloys resulting from molybdenum segregation during melting is avoided in the practice of the present invention by using molybdenum particles of relatively low density in admixture with the base metal in producing the consumable electrode. For this purpose it has been found that any decrease in the density of the molybdenum particles of the resulting ingot produced during vacuum arc melting. Preferably, it is desired that the particles have a specific gravity of less than 7.4 and for optimum results within the range of 5 to 7.4. By using molybdenum particles having a specific gravity within this range, substantially homogeneous alloys may be obtained through conventional vacuum arc melting techniques. It is understood, of course, that in the conventional manner multiple meltings are employed to enhance the purity of the alloy and insure substantially complete dissolution of the alloying elements, particularly the high melting point molybdenum.

In its broadest aspects, therefore, the method of the invention comprises steps of producing a charge of molybdenum particles having a specific gravity less than about 10 and preferably within the range of 5 to 7.4, admixing said particles with a charge of a particulate material selected from the group consisting of titanium, zirconium and base alloys thereof, forming said admixture into a consumable electrode and vacuum arc melting said electrode in a vacuum or inert atmosphere.

The low density molybdenum particles consist of porous molybdenum agglomerates. The agglomerates are preferably of substantially the same size consist as the particles of the other elements making up the electrode compact. Typically, this is about minus 8 mesh (U.S. Standard Sieve). By maintaining the particles of all the elements of substantially the same size "separating-out" the particles, particularly titanium and molybdenum particles, upon agitation of the admixture during transport incidental to consumable-electrode manufacture is substantially avoided. This is necessary if segregation of the alloying elements, particularly molybdenum, is to be avoided during the subsequent manufacture and vacuum arc melting of the electrode. If the alloying elements are segregated in the electrode, they will also be segregated in the resulting ingot melted therefrom.

Although it is possible to produce the desired low density molybdenum particles by various techniques, it is presently preferred to produce the agglomerates by the following technique.

Molybdenum is typically produced by hydrogen reduction of molybdenum oxide at elevated temperatures.

3

4

At suitable temperatures, for example 1200 to 1400° C., the basically micron-sized molybdenum powder so produced will sinter into low-density agglomerates. Such agglomerates are satisfactory for the practice of this invention.

As a specific example of the practice of the invention, the following molybdenum-containing titanium-base alloy was melted both in the conventional manner and in accordance with the present invention:

	Percent
Molybdenum -----	11.5
Zirconium -----	6
Tin -----	4.5
Titanium -----	Balance

The above alloy, produced in accordance with the present invention, used minus 6 mesh particles of the alloy constituents with molybdenum agglomerates having a specific gravity of about 6.4 and produced in accordance with the technique outlined hereinabove. The alloying elements in particle form were pressed into bricks. The bricks were then welded in an inert atmosphere to form a consumable electrode, which was then melted in an evacuated furnace to form an ingot. Such operation was performed in accordance with well known vacuum arc melting techniques.

A similar consumable electrode of the above alloy was prepared in the conventional manner using dense molybdenum turnings having a specific gravity of 10.2 instead of low density agglomerates in accordance with the invention. This alloy was melted in the same manner as that described above in accordance with the present invention. The only difference between the two operations was that in one dense molybdenum turnings were used, in accordance with conventional practice, and in the other low density molybdenum agglomerates were used in place of the molybdenum turnings, in accordance with the present invention. After two melting operations, photomicrographs of the ingot produced in accordance with the conventional techniques using high density molybdenum turning and that produced in accordance with the present invention were taken; the former is identified as FIG. 1 and the latter as FIG. 2 of the drawings. In the center of FIG. 1 there is shown a typical dense molybdenum inclusion resulting from the failure of the molybdenum to dissolve in the molten metal caused by its segregation upon dropping from the electrode and into the molten pool of the ingot. Because of its high density, the molybdenum particle quickly sank within the molten pool of titanium and reached the bottom thereof without completely dissolving. Because of the relatively lower temperature prevailing at the bottom of the ingot, which in the case of a titanium-base alloy is near the solidus temperature of titanium, the molybdenum particle did not dissolve but remained as an unmelted inclusion in the microstructure of the alloy. In contrast, the photomicrograph of FIG. 2, which is of an alloy produced in accordance with the invention, shows the complete absence of undissolved molybdenum inclusions. This re-

sults from the fact that the molybdenum particles as they dropped from the electrode end and into the molten pool were of a relatively low density. Consequently, they did not sink to the bottom of the molten titanium prior to dissolving. FIG. 3, which is a radiograph of the ingot of FIG. 1, again shows, as black areas, the presence of segregated undissolved molybdenum. In contrast, FIG. 4, which is a radiograph of the ingot of FIG. 2, shows the complete absence of segregated undissolved molybdenum.

Although various embodiments of the invention have been shown and described herein, it is obvious that other adaptations and modifications may be made by those skilled in the art without departing from the scope and spirit of the appended claims.

What is claimed is:

1. A method of producing substantially homogeneous alloys containing effective quantities of molybdenum, comprising producing a charge of molybdenum particles, said particles having a specific gravity of less than 7.4, admixing said molybdenum particles with a charge of particles of at least one metal, which includes at least one metal selected from the group consisting of titanium, zirconium and base alloys thereof, forming said admixture into a consumable electrode, and vacuum arc melting said electrode, whereby the density of said molybdenum particles delays the sinking thereof in the resulting melt of lesser specific gravity.

2. A method according to claim 1 wherein said molybdenum charge constitutes porous molybdenum agglomerates.

3. A method according to claim 2 wherein said molybdenum agglomerates are about minus 6 mesh.

4. A method according to claim 2 wherein said molybdenum agglomerates and said charge are particles of substantially the same size, whereby segregation of the materials is minimized.

5. A method according to claim 2 wherein said molybdenum agglomerates have a specific gravity of about 5 to 7.4.

References Cited

UNITED STATES PATENTS

2,550,735	5/1951	Tour	75—53
2,702,239	2/1955	Gilbert	75—10
2,848,315	8/1958	Kieffer	75—10
3,005,246	10/1961	Murphy	75—10
3,101,267	8/1963	Dunn	75—10
3,269,825	8/1966	Vordahl	75—10

FOREIGN PATENTS

838,586	6/1960	United Kingdom	75—10
---------	--------	----------------	-------

WINSTON R. DOUGLAS, Primary Examiner

P. D. ROSENBERG, Assistant Examiner

U.S. Cl. X.R.

75—115.5