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M. B. VORDAHL
METHOD OF PRODUCING HOMOGENEOUS ALLOYS
CONTAINING REFRACTORY METALS

3,269,825

Filed June 18, 1963

2 Sheets-Sheet 1

FIG. 1.

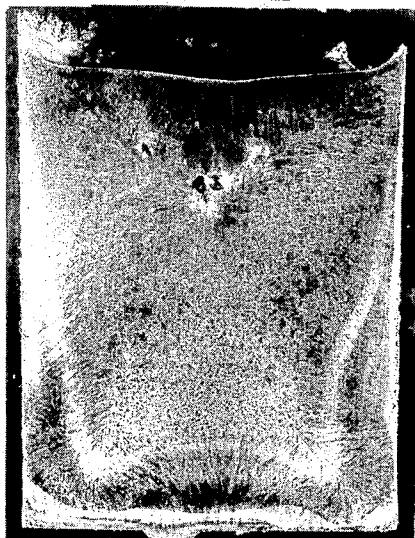
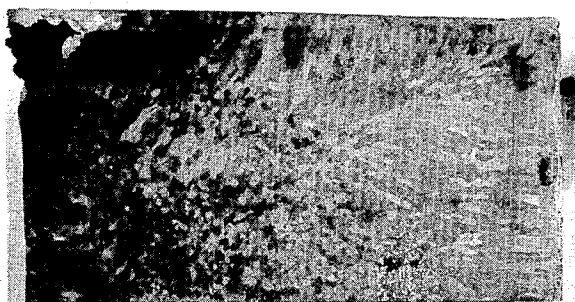


FIG. 2.



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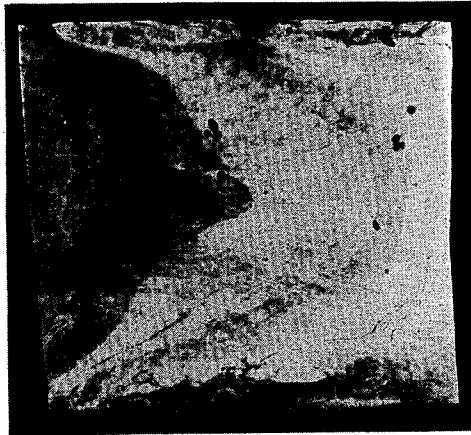
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2 Sheets-Sheet 2

FIG. 3.



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METHOD OF PRODUCING HOMOGENEOUS ALLOYS CONTAINING REFRACTORY METALS

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20 Claims. (Cl. 75-10)

This invention relates to methods of producing alloys containing substantial amounts of refractory metals, such as the element molybdenum and, particularly, to titanium base alloys containing, in addition to molybdenum, substantial amounts of tin.

In the prior art production of molybdenum-containing alloys, difficulty has been encountered in obtaining substantially complete dissolution of the high melting point element molybdenum in alloy compositions comprising a base metal having a relatively much lower melting point. Thus, molybdenum, having a melting point of 2625° C., as compared to a melting point of 1875° C., for zirconium, or 1800° C. for titanium, and 1535° C. for iron, will not readily dissolve in alloy compositions containing the latter or other relatively low melting point elements as the base metal of the alloy, except in those relatively few instances where molybdenum forms low melting point eutectic alloys with the base metal, e.g., 65 weight percent iron-35 weight percent molybdenum. However, full advantage cannot be taken even of such favorable alloy phase relationships unless it is possible to hold the same in the liquid state for extended periods of time. This, of course, is impossible, in usual vacuum arc melting procedures, utilizing water-cooled, metal molds. Difficulties are encountered principally in those situations, including, but not limited to, usual vacuum melting procedures where the high reactivity of one or more alloy components, as titanium or zirconium, prevents holding the alloy in a molten condition for appreciable periods of time. The production of alloys, as base alloys of titanium and zirconium, or other alloys, as iron-, nickel- or cobalt-base superalloys, containing major additions of the latter elements, or other reactive metals, as aluminum, falls in this category. As a consequence, additions of molybdenum to such base alloys result in nonhomogeneity of the resulting alloys as regards the molybdenum content thereof. This difficulty in producing molybdenum-containing alloys is of present and increasing importance in view of the increased need for and use of alloys containing relatively high percentages of molybdenum.

Prior art attempts to incorporate molybdenum in substantial quantities in alloys wherein the base metal has a substantially lower melting point than molybdenum have generally been unsuccessful in obtaining alloy homogeneity, despite efforts to introduce the molybdenum in various product forms which have been considered conducive to better dissolution of the molybdenum. Attempts have been made to produce alloys, for example, titanium base alloys, containing substantial quantities of molybdenum, by the consumable vacuum arc melting process, wherein a part of the consumable electrode comprises a continuous molybdenum rod. Relatively homogeneous molybdenum-containing alloys can be made by such a process but only by a series of consecutive remeltings of the alloy melted from such an initial consumable electrode. Successive remeltings, of course, add considerable expense and make the alloys so produced commercially unfeasible. However, no such procedures have been productive of substantially complete molybdenum dissolution and homogeneity upon a commercially feasible basis.

As another example of prior art attempts to produce such alloys, molybdenum powder has been incorporated with titanium sponge and the resultant mixture compacted

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into the form of a consumable electrode for vacuum arc melting. However, it has been found that the molybdenum powder has a pronounced tendency to stratify in such an electrode with the resultant production of a melt containing dense layers of unmelted molybdenum. Still further attempts have been made by utilizing a mixture of titanium powder plus molybdenum powder and, although molybdenum distribution in the resulting melts have been found to be generally improved, titanium powder with the required low oxygen content is commercially unavailable. Recognizing the necessity for introducing molybdenum in a physical form having relatively small cross sectional areas, prior art attempts have also included the addition of molybdenum to the electrode in the form of foil or wire. However, such highly finished forms of molybdenum are quite expensive, making the use of such physical forms economically impractical.

Therefore, it is an object of the present invention to provide a method of obtaining substantially complete dissolution of refractory metal additions, as molybdenum, in relatively lower melting point base alloy compositions.

It is a further object of the invention to provide a method for producing alloy compositions having uniformly distributed therethrough substantial quantities of molybdenum.

It is a still further object of the invention to provide alloys and products thereof comprising a base metal having a melting point substantially lower than that of molybdenum and containing substantial quantities of the elements molybdenum and tin, wherein the molybdenum component is substantially completely dissolved therein and uniformly distributed therethrough.

It is a particular object of the invention to provide products comprising titanium base alloys containing molybdenum and tin, and methods for making such alloys and products.

The foregoing and other objects and advantages of the invention will be more readily apparent by an inspection of the following specification and drawings, wherein

FIG. 1 is a photographic illustration of a section, in elevation, of an ingot of a titanium-molybdenum-tin alloy produced by a single vacuum arc melting, in accordance with the invention;

FIG. 2 is a similar view of an ingot, of substantially the same composition as that shown in FIG. 1, but produced in accordance with prior art practice, and

FIG. 3 is a similar view of an ingot of a titanium-molybdenum alloy produced in accordance with prior art practice.

It is, of course, essential to the production of alloys exhibiting the maximum benefit of the intended molybdenum content thereof, and to the uniformity of the consequent alloy properties throughout the mass of the alloy product, that the molybdenum content be completely dissolved in the metal matrix. Complete dissolution of the molybdenum, or other high melting point alloy component, is essential, inasmuch as a single, undissolved, sizeable inclusion of the refractory metal in an alloy ingot or other mill stock unit makes the same unfit for its intended purpose. Any such inclusion, incorporated in a finished article subject to mechanical stress, as for example, a turbine blade or a structural component of an aerospace vehicle, might well, because of its stress-raising character, cause the part to crack or rupture catastrophically. Thus, the aim of the present invention is not production of uniformity of dispersion of the refractory metal alloy component, but, instead, is the virtually complete dissolution of such components in the base alloy and elimination of unmelted refractory inclusions.

It is evident that the nature of the benefit conferred by an alloying addition of the refractory metal and, consequently, the full realization of the benefits due to

the dissolution thereof in the base alloy, is dependent upon the alloy class involved or, indeed, upon the particular alloy under consideration. Therefore, it is further evident, that the present invention, in the inventive method thereof, being directed to the attainment of substantially complete dissolution of molybdenum and other refractory metals, is not limited to a single, restricted class of alloys nor to any particular alloy composition.

However, exemplary of alloys and products thereof deriving substantial benefits from the practice of the invention, and in accordance with the foregoing objects, the present invention provides structurally uniform, molybdenum-containing alloys and products, particularly those comprising titanium base alloys, specifically, alloys comprising from about 6 to about 15%, preferably 10 to 12 or 14% molybdenum, from about 4 to about 15%, preferably 4 to 8% tin, balance substantially titanium, wherein molybdenum, in powder form, is mixed with powdered tin, the powder mixture roll pressed into the form of thin flakes, the composite flakes are admixed with granules of a desired base metal, having a melting point substantially lower than that of molybdenum, and the resultant admixture is then compacted into the form of electrodes for consumable electrode vacuum arc melting. The molybdenum homogeneity so obtained is of an order heretofore unknown in commercially producible products.

The method of the invention is broadly useful in the production of nearly all alloys containing appreciable quantities of refractory metal, as molybdenum, in a base metal having a melting point substantially lower than that of the refractory metal. It is especially useful in the production of molybdenum-containing alloys of the more refractory and reactive metals, or of other metals or alloys which, for a variety of reasons, e.g., reactivity with atmospheric constituents, need for a high degree of freedom from non-metallic inclusions, etc., are normally melted by the consumable electrode vacuum arc melting process. It is not intended that the invention be applied to the production of such alloys containing substantial quantities of aluminum, e.g., quantities of the same order of magnitude as the molybdenum content thereof, in view of the ready availability of economical molybdenum-aluminum master alloys which facilitate the production of alloys of substantially homogeneous molybdenum distribution.

Although certain alloys and products thereof, i.e., ternary titanium-molybdenum-tin alloys, are particularly contemplated herein, other alloys and products also amenable to the practice of the invention, include, inter alia, the aforesaid titanium base alloys containing, in addition to the mentioned elements, up to about 10% in total of one or more of the elements zirconium, tantalum, columbium and vanadium, up to about 4% in total of one or more of the elements iron, manganese or chromium, up to about 2% in total of the elements copper, nickel and cobalt, up to about 2% aluminum and up to about 0.5% silicon. Titanium-molybdenum-tin alloys additionally containing the element zirconium are especially contemplated. Still other alloys, as, for example, superalloys, stainless steels or tool steels, containing the aforesaid quantities of zirconium, tantalum, columbium or vanadium, are also contemplated, especially in those instances wherein it is required that the metal be of the greatest possible structural homogeneity and cleanliness. In many such additional alloy systems, metals other than tin may be used as the binding and lubricating material to enable production of high metal point metal flakes from powders thereof. Exemplary of such other metals, useable in amounts effective to lubricate and bind the higher melting point metal powder particles during rolling, are copper, silver and high purity iron, nickel or cobalt. The invention also contemplates similar alloys and products thereof wherein the base metal is zirconium.

The preferred class of alloys and products, as hereinabove described, comprise titanium base alloys having

a beta microstructure. Such titanium base alloys are highly useful in a variety of applications, for example, in high structural uses, in view of their superior formability high strength structural uses, in view of their superior formability and strengths as compared to alpha and alpha-beta titanium alloys. However, such superiority of certain beta titanium alloys is offset to a great extent by reason of their relatively poor stability, particularly upon exposure to elevated temperatures. For example, the single currently commercially significant beta titanium alloy, Ti-13% V-11% Cr-3% Al, possesses the highest strength-weight ratio of any metallic sheet, but that alloy is strengthened by the formation therein of intermetallic compounds which, upon exposure of the alloy to a temperature of 600° F. for 100 hours, make the alloy almost glass-brittle.

Therefore, a beta titanium alloy, strengthened by other means, e.g., by solid-solution strengthening, and hence less susceptible to such thermal instability, would be of great utility. Such alloy compositions are known, for example, as disclosed in United States Patents Nos. 2,769,707 and 2,797,996. However, such alloy compositions have not heretofore gained commercial importance because it has been impossible to produce them economically on a commercial scale due to the difficulty in melting and solutioning the molybdenum contents. By means of the present invention, however these, and other molybdenum-containing alloys are readily and economically producible.

The workability of the preferred titanium base alloy products, i.e., mill products, as wide, thin sheet and strip, foil, wire, bars, billets and forgings, of the aforesaid titanium-molybdenum-tin alloys, is excellent. Although such alloys, produced as herein described, can be heat treated to a strength from two to three times that of commercially pure titanium, they hot work at least as easily as titanium and, because of their beta crystal structure, they cold roll more easily than the unalloyed element. These solid-solution beta titanium alloys are also considerably more readily workable than the aforementioned presently commercially important beta titanium alloys. Thus, quenched, relatively thin-section (e.g., up to about 1-inch thickness) products, comprising an alloy consisting essentially of, by weight percent, 12% molybdenum, 5% tin, balance substantially titanium, have typical annealed, room temperature properties as follows: 80,000 pounds per square inch (80K s.i.) 0.2% offset yield strength, 120K s.i. ultimate tensile strength, 50% reduction of area and 1T bend. On the other hand, the Ti-13% V-11% Cr-3% Al alloy is two to three times more difficult to hot and cold work. In the newer beta alloy, as aforesaid, the element tin stabilizes the beta structure with considerably less alloy content than is necessary to other, compound-strengthened beta alloys and yet stabilizes the strong, ductile beta condition at least 200° F. higher than the maximum service temperature of prior beta alloys (about 600° F.).

I have found that the use of tin in conjunction with molybdenum enables the ready reduction of molybdenum to the aforesaid flake form, and it is believed that tin, when incorporated, as aforesaid, in powdered form, with the powdered molybdenum, in amounts upwardly of about 10% by weight of the molybdenum, acts as a binder and lubricant, thereby enabling conversion of the molybdenum powder into the form of the desired, relatively thin, molybdenum flakes when the powder mixture is fed between horizontally opposed rolls.

The flake dimensions may be varied to a considerable degree without detriment to the utility of the flakes for the intended purpose. Thus, powdered mixtures, having an average particle size of about minus 300 mesh, of molybdenum and tin were fed into a set of 8-inch diameter polished steel rolls to produce flakes of about 1/2 inch in diameter and of thicknesses varying between about 5 and 30 mils. Such flakes, which are preferably held to thicknesses under about 10 mils, were found to have a

density in excess of 90% of the theoretical value. The flakes were relatively friable but were sufficiently firm to be handled for subsequent mixing with other alloying components. Such flakes were then added to titanium sponge comprising approximately ¼-inch diameter particles in proportions to produce titanium base alloys containing about 8 to 14% by weight of molybdenum and about 4 to 14% by weight of tin. Exemplary of these alloys, produced in accordance with the invention, are those set forth in Table I hereinbelow.

Table I

Alloy Designation	Composition, Weight Percent		
	Mo	Sn	Ti
1.-----	12	4	Bal.
2.-----	12	6	Bal.
3.-----	12	8	Bal.
4.-----	12	10	Bal.
5.-----	12	12	Bal.
6.-----	14	14	Bal.

After production, in accordance with the invention, the alloys of Table I were tested, in accordance with standard testing procedures, and the physical properties thereof were determined. For example, an alloy, ingot No. 98,707, was produced to a nominal analysis of 12% molybdenum, 6% tin, the balance titanium (actual analysis was 11.4–12.2% molybdenum, 6.4–6.5% tin, the balance titanium) by blending titanium sponge and molybdenum-tin flakes made by rolling 200–300 mesh powders of molybdenum and tin (in a weight ratio of 2 molybdenum to 1 tin), compacting the blended materials to briquettes and vacuum arc melting the briquettes. This ingot was consumable electrode vacuum arc remelted to form a further ingot, No. 98,725, which was then forged to the form of 4-inch square billet, at a temperature of 1400° F., reheated for 1 hour at 1400° F., and water quenched. Pieces cut from this billet were machined to the form of standard, ¼-inch diameter, 1-inch length, test specimens which were then evaluated by standard test procedures, with results as shown in Table II.

Table II

Specimen	Ingot Position	Direction	U.T.S., K s.i.	0.2% Offset Y.S., K s.i.	Percent Elong.	Percent Red. Area
BL-1.-----	Outside corner..	Longitudinal..	122.2	107.7	17.0	35.4
BL-13.-----	Outside face.....	do.....	128.3	115.3	17.0	42.9
BL-14.-----	Mid radius.....	do.....	130.4	122.1	15.0	33.7
BL-16.-----	Center.....	do.....	131.8	121.9	17.0	36.5

Similar specimens were also evaluated after quenching followed by heating for 24 hours at 900° F., with results as shown in Table III.

Table III

Specimen	Ingot Position	Direction	U.T.S., K s.i.	0.2% Offset Y.S., K s.i.	Percent Elong.	Percent Red. Area
BL-6.-----	Outside corner..	Longitudinal..	195.8	187.9	5.0	-----
BL-19.-----	Outside face.....	do.....	194.9	184.5	8.0	11.6
BL-20.-----	Mid radius.....	do.....	195.4	182.1	10.0	17.6
BL-21.-----	Center.....	do.....	194.1	181.3	10.0	17.5
BT-1.-----	Outside face.....	Transverse....	189.4	182.6	4.0	6.3
BT-3.-----	Center.....	do.....	200.0	186.1	4.0	-----

It will be seen from the data of Tables II and III that alloys produced in accordance with the invention exhibit highly useful mechanical properties, of great uniformity throughout the ingots so produced.

The alloys contemplated for production in accord-

ance with the invention also show exceptional stability and strength at temperatures up to at least 800° F. For example, specimens, as above, comprising 12% molybdenum, 4% tin, the balance titanium were machined from bar stock, rolled at 1300° F., heated at 1400° F. for 1 hour, quenched, then reheated at 900° F. for 24 hours. These specimens were tensile tested, at a standard strain rate of ½% per minute, at 800° F. Such specimens showed the following property characteristics: 130K s.i. ultimate tensile strength, 110K s.i. 0.2% offset yield strength, 4% elongation and 20% reduction in area. In comparison, the ultimate tensile strength of the alloy 5% Al–2.5% Sn–balance Ti, commonly currently used for elevated temperature applications, has an ultimate tensile strength of about 82K s.i. at 800° F.

Sheet specimens of an alloy, comprising 12% molybdenum, 4% tin, the balance titanium, were heated to 1400° F., quenched, reheated to 900° F. for 24 hours, and subjected to standard room temperature tensile tests, with the following results: 179K s.i. ultimate tensile strength, 174K s.i. 0.2% offset yield strength, 2% elongation and 8% reduction in area. After creep exposure of such sheet specimens at 600° F., under a load of 80K s.i. for 100 hours, during which 0.03% creep occurred, further tests at room temperature showed the following property characteristics: 176K s.i. ultimate tensile strength, 174K s.i. 0.2% offset yield strength, 2% elongation and 14% reduction in area.

Still further sheet specimens of the same alloy, heated to 1400° F., quenched, aged for 24 hours at 950° F., and tensile tested at room temperature, showed the following results: 174K s.i. ultimate tensile strength, 164K s.i. 0.2% offset yield strength, 13% elongation and 34% reduction in area. After creep exposure at 800° F., under a load of 65K s.i., for 100 hours, and an observed creep of 0.93%, the following tensile properties were determined when tested at room temperature: 181K s.i. ultimate tensile strength, 174K s.i. 0.2% offset yield strength, 12% elongation and 32% reduction in area.

The foregoing property data clearly illustrates the

manifest utility of the alloys contemplated for production in accordance with the invention. As aforesaid, however, the application of such alloys to the uses for

which their properties, as above illustrated, admirably suit them, has been precluded by the inability, prior to the present invention, to successfully produce the alloys free of undissolved molybdenum inclusions.

The aforesaid disability of the prior art, however, has

now been overcome by the invention. Thus, metallurgical examination of alloys produced in accordance with the invention showed substantially complete and uniform molybdenum dissolution in the metal matrices.

Thus, in one example of an alloy produced in accordance with the invention, one part by weight of tin, in the form of 200 mesh powder, was mixed with two parts by weight of 300 mesh molybdenum powder. Both powders were relatively free of oxygen, i.e., they contained less than about 0.25% of that element. The powders were mixed in a tumbling barrel for about ten minutes and then poured into a chemical feeding device and thereby fed between the rolls of a horizontal rolling mill. The powders were rolled at room temperature with a tight setting on the rolls. In this manner, there were produced flakes of molybdenum powder bonded with tin and having dimensions between $\frac{1}{4}$ inch and 2 inches in diameter and between 2 and 6 mils in thickness. The flakes were then screened to eliminate the fine flakes, e.g., those below about $\frac{1}{4}$ -inch diameter and the unflaked powder. The flakes, together with titanium sponge, which had previously been sized to eliminate coarse chunks, e.g., pieces greater than about $\frac{1}{4}$ -inch diameter, were then placed in a tumbling barrel and mixed by rotating the barrel several times. The mixture was then poured into compacting dies and compacted under about 1,000 tons pressure, at room temperature, into the form of cylindrical bricks of about 6 inches diameter and 5 inches height. A number of such bricks were then welded together to form a consumable electrode which was then melted, in a vacuum arc furnace, into a water-cooled copper crucible having a diameter of 10 inches. The melting was carried out at between 20 and 40 volts and 2,000 to 6,000 amperes to form a 12-inch high ingot which, upon analysis, was shown to contain 6% by weight tin, 12% by weight molybdenum, balance substantially titanium. Upon removal of the ingot from the crucible, the ingot was sectioned lengthwise, polished and etched. Examination of the prepared, sectioned ingot showed that the same was free of segregated, i.e., unmelted, molybdenum, as illustrated in FIG. 1.

In contrast, FIG. 2 illustrates an ingot of substantially the same composition as the ingot illustrated in FIG. 1, but wherein similar bricks, consisting of compacted titanium sponge, were drilled through their centers, a $\frac{3}{4}$ -inch diameter molybdenum rod inserted therein, and the composite welded together for subsequent vacuum arc melting as in the case of the FIG. 1 ingot. It will be noted from FIG. 2, depicting the sectioned, polished and etched ingot, that considerable quantities of unmelted molybdenum are present.

By way of further example, a binary alloy was prepared containing 30% by weight of molybdenum, balance substantially titanium, by mixing titanium sponge with chopped, high purity molybdenum wire, and compacting the mixture in dies to form bricks as aforesaid, which bricks were then welded together and vacuum arc melted to form a 10-pound ingot. The same was sectioned, polished and etched, whereupon considerable numbers of large, undissolved molybdenum particles were observed. This ingot is illustrated in FIG. 3. The etchant utilized in preparation of the ingots of FIGS. 2 and 3 was an aqueous solution of 30% nitric acid, 3% hydrofluoric acid, and etching was done at room temperature.

Depending upon the length of time exposure of the sample to the etching medium, the undissolved molybdenum particles appear either black, as in FIG. 3, or white, as in FIG. 2. In the later fig., the undissolved molybdenum makes its grossest appearance as the cluster of white-etching spots indicated by the arrow. The appearance of FIG. 2 is the result of a relatively extended etching time, whereas the black-etching molybdenum in-

clusions are produced by a shorter, controlled etching time.

Experience has shown that remelting ingots such as those illustrated in FIGS. 2 and 3 up to five or six times fails to dissolve the molybdenum. Thus, attempts to produce titanium-molybdenum and titanium-molybdenum-tin alloys by utilizing the molybdenum addition in the form of machined chips consistently failed to produce homogeneous alloys. Thus, compacted bricks, consisting of titanium sponge and molybdenum chips, were vacuum arc melted to ingot form. The ingot was then machined to chips, compacted and remelted. This entire cycle was repeated up to six times but unmelted molybdenum was still present in considerable quantities after the final melting.

Similar attempts, utilizing loosely sintered agglomerates of molybdenum powder and titanium sponge have also been unsuccessful in producing homogeneous final alloys. Thus, after repeated meltings, the initial agglomerated molybdenum "rondels" were still recognizable.

Unalloyed molybdenum is quite non-ductile and alloys containing undissolved particles of molybdenum, as in the ingots illustrated in FIGS. 2 and 3, are susceptible to fracture when subjected to mechanical stress. Fracture occurs adjacent one or more particles of undissolved molybdenum. This undesirable characteristic of non-homogeneous molybdenum-containing alloys obviously makes such alloys unsuitable for the production of fabricated articles and particularly those which are subjected to even moderate extremes of mechanical stress during use. Thus, although alloys containing unmelted molybdenum do not have property characteristics, as measured by ordinary mechanical property tests, significantly different from the same alloys, as contemplated herein, wherein the molybdenum is completely dissolved, fabricators and users of alloys have experienced catastrophic failures, under use conditions, traceable to the presence of dense inclusions, as undissolved molybdenum particles. Consequently, alloys containing even one sizeable such inclusion cannot be used to best advantage and are, indeed, unsaleable for applications as jet engine rotating parts, etc., Consequently, the high valuable mechanical properties of the alloys, as above illustrated, cannot be utilized.

The homogeneous alloys, made in accordance with this invention, exhibit the desirable mechanical properties, as uniform, high tensile strength, elongation and area reduction values, and also exhibit a high spread between yield and ultimate strengths. These properties are, of course, highly advantageous in forming the alloys by various mechanical means. Additionally, these alloys, having strain-transformable beta microstructure, can be overaged to a ductile, stable condition in contrast to the compound-strengthened beta-titanium alloys containing large quantities of chromium, manganese, or equivalent alloying elements. The aforesaid alloys, produced in accordance with the invention, can be hot rolled at temperatures upwardly of 1200° F., for example, up to about 1800° F., at much lower roll pressures than other beta titanium alloys. In small thicknesses, e.g., foil gages, the alloys produced as aforesaid can be cold rolled as readily or even more easily than unalloyed titanium and, hence, are especially useful in the production of thin sheet or strips. Moreover, and most importantly, the substantially complete elimination of undissolved dense inclusions frees the alloys for a variety of applications, as aforesaid, which were previously precluded.

Heat treatment of alloys produced in accordance with the invention may take one or another of several alternative forms. For example, such alloys, melted as aforesaid, and reduced to a desired final form by hot and/or cold working, may, in the ductile, overaged condition, then be solution-treated at temperatures of, for example, from 1275 to 1700° F., followed by aging at 700 to 1100° F. The alloys may be solution-treated as

aforesaid, followed by cold-working below the recrystallization temperature, followed by aging at a temperature somewhat lower than that above-described. Alternatively, the alloys may be cold worked followed by solution treatment at a combination of time and temperature below the recrystallization temperature, followed by aging as above-described.

The foregoing specific embodiments and examples are merely illustrative of the invention and it is to be understood that various modifications and additions may be made by those skilled in the art without departing from the spirit and scope of the invention.

I claim:

1. A method of producing substantially homogeneous alloys containing effective quantities of molybdenum, comprising admixing the molybdenum in powdered form with powdered tin in an amount effective as a binder for the molybdenum, rolling the mixture to the form of flakes, mixing the flakes with a particulate material selected from the group consisting of titanium, zirconium and base alloys thereof, forming the flake-particulate mixture into a compacted consumable electrode, and vacuum arc melting the electrode.

2. A method of producing substantially homogeneous alloys comprising an effective amount of a first alloying addition selected from the group consisting of at least one of the elements molybdenum, zirconium, columbium, tantalum and vanadium, a second alloying addition comprising an effective amount of tin and the balance substantially titanium, comprising admixing the first and second alloying additions in powdered form, rolling the powdered mixture to the form of flakes, mixing the flakes with granulated titanium, forming the flake-granule mixture into the form of a consumable electrode, and vacuum arc melting the electrode.

3. A method of producing substantially homogeneous alloys comprising an effective amount of molybdenum as an alloying element and a matrix metal having a melting point substantially lower than that of molybdenum, comprising reducing the molybdenum addition to powdered form, mixing with the powdered molybdenum a quantity of powdered tin in an amount of at least about 10% by weight of the molybdenum, rolling the molybdenum-tin powder mixture to the form of flakes, mixing the flakes with granules of the matrix metal, forming the flake-granule mixture into a consumable electrode, and melting the electrode.

4. A method of producing substantially homogeneous alloys comprising an effective amount of molybdenum as an alloying element and a matrix metal having a melting point substantially lower than that of molybdenum, comprising reducing the molybdenum to powdered form, mixing with the powdered molybdenum a quantity of powdered tin in an amount effective to bind together the molybdenum powder particles upon application thereto of cold-rolling pressure, rolling the molybdenum-tin powder mixture to the form of flakes, mixing the flakes with granules of the matrix metal, forming the flake-granule mixture into a consumable electrode, and melting the electrode.

5. A method of producing substantially homogeneous titanium base alloys containing from about 6 to about 15% by weight of molybdenum and from about 4 to about 15% by weight of tin, comprising admixing the molybdenum and tin in powdered form, rolling the powdered mixture to the form of flakes, mixing the flakes with granulated titanium, forming the flake-granule mixture into the form of a consumable electrode, and vacuum arc melting the electrode.

6. A method of producing substantially homogeneous ingots of an alloy comprising about 10 to about 12% molybdenum, from about 4 to about 8% tin and the balance substantially titanium, comprising mixing the molybdenum and tin in powdered form, rolling the powdered mixture to the form of flakes, mixing the flakes with gran-

ules of titanium, forming the flake-granule mixture into a consumable electrode, vacuum arc melting the electrode, and casting the melted metal into ingot form.

7. A method of producing a substantially homogeneous ingot comprising from an effective amount up to about 15% molybdenum, up to about 10% in total of at least one of the elements zirconium, tantalum, columbium and vanadium, up to about 4% in total of at least one of the elements iron, manganese and chromium, up to about 10% in total of at least one of the elements copper, nickel and cobalt, up to about 2% aluminum, up to about 0.5% silicon, from an effective amount up to about 15% tin, and the balance substantially titanium, which method comprises mixing together, in powdered form, the elements selected from the group consisting of molybdenum, zirconium, tantalum, columbium, vanadium and tin, rolling the powdered mixture to the form of flakes, forming a granulated mixture of the remaining alloying elements, mixing the flakes and granules, compacting the flake-granule mixture into the form of a consumable electrode, melting the electrode, and casting the melted metal into ingot form.

8. A method of enhancing the solubility of molybdenum alloying additions to base alloys of a metal selected from the group consisting of titanium and zirconium comprising mixing the molybdenum, in powdered form, with powdered tin in a quantity of at least about 10% by weight of the molybdenum, rolling the molybdenum-tin mixture to flake form, adding the flakes to granules of the base metal, mixing the flake-granule mixture, compacting the latter mixture to electrode form, and melting the electrode.

9. A method of enhancing the solution of a metal alloying addition to alloys of a base metal having a substantially lower melting point than said alloying addition, comprising mixing the alloying addition in powdered form with an effective amount of a powdered lubricating and binding metal, rolling the powder mixture to the form of flakes, mixing the flakes with granules of the base metal, compacting the flake-granule mixture, and melting the compacted mixture.

10. An article produced in accordance with the process of claim 9.

11. A method in accordance with claim 9, wherein the lubricating and binding metal is tin.

12. A method of enhancing the solution of a refractory metal alloying addition to alloys of a base metal having a substantially lower melting point than said alloying addition, comprising mixing the alloying addition in powdered form with an effective amount of a powdered metal effective as a binder and lubricant for the refractory metal powder particles upon application thereto of cold rolling pressure, rolling the powder mixture to the form of flakes, mixing the flakes with granules of the base metal, compacting the flake-granule mixture, and melting the compacted mixture.

13. An article produced in accordance with the method of claim 12.

14. A master alloy for addition to a matrix metal having a melting point substantially below that of molybdenum, said alloy consisting essentially of molybdenum, and at least about 10% by weight thereof of tin, said master alloy being in the form of relatively thin, friable flakes.

15. A consumable electrode for producing base alloys of a metal selected from the group consisting of titanium and zirconium, comprising a compacted mixture of base metal granules and flakes of an alloying composition comprising molybdenum and at least about 10% by weight thereof of tin.

16. An electrode in accordance with claim 15 wherein the base metal is titanium.

17. An electrode in accordance with claim 15 wherein the base metal is zirconium.

18. A consumable electrode comprising a compacted mixture of a flaked composition consisting essentially of

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molybdenum and tin, and granules of a matrix metal having a melting point substantially lower than that of molybdenum.

19. As an alloying addition to a base metal having a melting point lower than molybdenum, a composition of matter consisting essentially of molybdenum and at least about 10% by weight of tin. 5

20. A method of producing relatively thin friable flakes of molybdenum, comprising reducing the molybdenum to powdered form, admixing with the molybdenum pow- 10

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der at least about 10% by weight thereof of powdered tin as a binder and lubricant, and rolling the powder mixture into the form of flakes.

No references cited.

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UNITED STATES PATENT OFFICE CERTIFICATE OF CORRECTION

Patent No. 3,269,825

August 30, 1966

Milton B. Vordahl

It is hereby certified that error appears in the above numbered patent requiring correction and that the said Letters Patent should read as corrected below.

Column 1, line 53, after "obtaining" insert -- complete --; column 4, line 3, strike out "high structural uses, in view of their superior formability"; line 53, for "to" read -- in --; column 7, line 71, for "later" read -- latter --; column 8, line 20, for "molbdenum" read -- molybdenum --, line 42, for "high" read -- highly --; column 11, line 3, for "moylbdenum" read -- molybdenum --.

Signed and sealed this 1st day of August 1967.

(SEAL)

Attest:

EDWARD M. FLETCHER, JR.
Attesting Officer

EDWARD J. BRENNER
Commissioner of Patents

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