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UNITED STATES PATENT OFFICE

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ALLOY AND ITS MANUFACTURE

No Drawing. Continuation of application Serial No. 702,799, filed March 29, 1924. This application filed October 22, 1926. Serial No. 143,515.

This invention relates to the production of refractory metals and more particularly to an alloy of metals of the class which includes tungsten and tantalum and to the method of producing the same.

This invention is a continuation of application Serial No. 702,799, filed March 29, 1924 and entitled An alloy and its manufacture.

Tungsten is one of the most refractory of the metals which are producible in ductile, coherent form. It has been found, however, that the melting point of tungsten may be increased to an appreciable extent by the addition thereto of varying quantities of other metals. For instance, when tantalum is alloyed or mixed with tungsten in the proportion of from ½ to 5% of tantalum by weight, that the fusion point of the tungsten is raised to such an extent as to require an increase of around 10% in the current required to fuse the same. This increase in the melting point of tungsten is extremely advantageous in certain applications.

Owing to the high temperatures necessary to produce metals of the tungsten-tantalum class in ductile form and to the susceptibility of such metals to oxidation and contamination from impurities, it has heretofore been impossible, so far as I am aware, to successfully prepare an alloy of tungsten and tantalum, although many futile attempts have been made with this end in view.

One of the objects of this invention is to provide an alloy or mixture of refractory metals having a higher melting point than pure tungsten.

Another object is to provide an alloy of tungsten and tantalum.

A further object is to provide a process of producing such alloys and for thoroughly diffusing the constituent metals through one another.

Other objects and advantages will hereinafter appear.

In accordance with the present invention, the constituent metals are purified to a high degree and thoroughly intermixed, pressed into slugs of convenient size, baked or fired to render the same more coherent and sintered at a high temperature into a compact coherent mass. The sintered slug is then preferably heat treated as will more fully appear, to cause a thorough diffusion of one of the metals to the other and to anneal the treated bars. The bars may then be rolled, swaged and drawn into the desired shape or otherwise fabricated.

In place of mixing the purified metals, the purified oxides thereof may be mixed and subsequently reduced after which the resulting metal mixture may be pressed in the slugs.

In the preferred embodiment of the invention the pure tantalum metal powder may be prepared so as to eliminate impurities therefrom such as columbium, silica, carbon, etc., by the well known reduction of the double fluoride (K_2TaF_7) with sodium metal. Briefly, this process comprises dissolving tantalum oxide in hydrofluoric acid, purifying, adding potassium fluoride and recrystallizing for purity. After drying, the salt thus formed is mixed with a suitable quantity of sodium cuttings, sealing in an iron bomb and heated to a red heat. The charge, after cooling, is removed from the bomb, disintegrated with alcohol and water, purified with dilute acid and finally washed with water and alcohol. The tungsten metal should also be thoroughly purified in any desired manner as by the well known process of reducing purified tungsten oxide with hydrogen.

After purification, the metals are mixed in the proportion of about .05% to 5.0% of tantalum by weight. The mixing should be extremely thorough, preferably being carried out by ball milling for several hours. The intimate mixture is then pressed into slugs or bars without a binder in the manner well known in the manufacture of tungsten for wire drawing purposes, the pressures used being in the neighborhood of 20 tons per square inch. The pressed slugs or bars which may be of the size of ¼″ square and 6 to 8″ long is carefuly placed onto a strip or boat of molybdenum or nickel and the same introduced into a vacuum furnace and fired at from 1000 to 1200° C. for from ten minutes to one hour or sufficiently long to permit the slugs or bars to become sufficiently strong so that they may be readily handled.

The fired slugs are then placed in a vacuum furnace and sintered into coherent compact bars. A vacuum furnace suitable for this purpose is described in Patent 1,596,306 of Rentschler and Marden. This sintering operation will be carried out in substantially the same manner as pure tungsten is sintered and for substantially the same period of time. The sintering temperature should be about 90% of the fusing temperature of the bar, this temperature being readily determined by heating a test bar to its fusing point. In the case of a tantalum-tungsten alloy, however, this sintered temperature will be somewhat higher than in the case of pure tungsten, due to the higher melting point of the alloy. The current required to sinter the bar, of course, varies with the size of the bar and the composition of the alloy but in general, is about 10% higher than that required to sinter a bar of pure tungsten of corresponding size. Thus, for instance, with a ¼" bar of 6" length, the sintering current will be around 2000 amperes. The time required to sinter the bar varies from a few minutes to several hours depending on the conditions and circumstances so that I do not desire to be limited to any particular treating period. In firing and sintering the slug, care should be exercised to prevent the slug from coming into contact with hydrogen, inasmuch as this gas appears to exert a deleterious influence on the bar, rendering the tantalum brittle and causing the slug to crack and break up.

In place of mixing the purified metal powders it may be desirable to add the tantalum in oxide form to the tungsten metal since the tantalum oxide is reduced in the presence of tungsten yielding pure metallic tantalum. During this process a small quantity of tungsten oxide may be formed which is readily volatilized out of the slug during a high temperature treatment.

After the bar has been sintered it may be swaged, rolled and drawn to the desired form by any of the well known methods used in producing tungsten, but in order that a homogeneous and ductile alloy of the best quality may be produced I find that it is desirable to anneal the slug for about two hours at a temperature of around 2000 to 2100° C., although both time and temperature may be varied over a considerable range. This annealing appears to distribute the tantalum more thoroughly through the tungsten. Chemical analysis has shown that the tantalum remains in the tungsten during the treating operations, either intimately admixed or in the form of a solid solution with tungsten and that it does not volatilize out of the tungsten on treatment at the temperature which it is necessary to heat the tungsten in order to render the same ductile. This is contrary to the findings with such metals as thorium and zirconium. The exact form of the alloy is extremely difficult to determine and the term alloy is used throughout the claims in its broad sense as including the intimate fusion of the metals together.

It will be understood, of course, that the invention is susceptible to many modifications without departing from the spirit thereof and that the different phases thereof may be performed in different manners within the scope of the appended claims.

What is claimed is:

1. The method of making a tungsten-tantalum alloy comprising, mixing powdered tungsten metal with tantalum oxide, compressing and sintering the mixture in vacuo to consolidate it and reduce the tantalum oxide and heat treating at about 2000° C. to diffuse the metals, one through the other.

2. The method of making alloys of tungsten-tantalum which comprises intimately mixing tantalum oxide with tungsten metal powder in the proportion to give from .5 to 5.0 per cent tantalum, compacting the mixture into bars, sintering the bars in vacuo to effect consolidation of the metal and substantial reduction of the tantalum oxide content thereof, and subsequently annealing the treated bars at temperatures approximately 2000° C. for a sufficient period of time to effect substantial diffusion of the reduced tantalum metal throughout the body of the bar.

3. The method of making a tungsten-tantalum alloy comprising intimately admixing the metal powders in the desired proportions, compacting, sintering the compacted slug in vacuo to approximately 1000° C. to 1500° C. to consolidate, heat-treating the sintered slug to temperatures approximately but below the fusion temperature in vacuo, and continuing the heat-treatment for an interval of time at temperatures approximately 2000° C.

4. The method of making a tungsten-tantalum alloy comprising intimately admixing the substantially pure metal powders in the proportions of from .5 to 5.0% tantalum to 99.5 to 95% tungsten, compacting, sintering the compacted slug in vacuo to approximately 1000° C. to 1500° C. to consolidate, heat-treating the sintered slug to temperatures approximately but below the fusion temperature in vacuo, and continuing the heat-treatment for an interval of time at temperatures approximately 2000° C.

In testimony whereof, I have hereunto subscribed my name this 21st day of October, 1926.

JOHN WESLEY MARDEN.