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REFRACTORY ALLOY FOR WIRES AND RODS.

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To all whom it may concern:

Be it known that I, FREDERICK W. ZONS, a citizen of the United States, and a resident of New York, county of New York, and State of New York, have invented certain new and useful Improvements in Refractory Alloys for Wires and Rods, of which the following is a specification.

This invention relates to a refractory alloy for making wires or rods and relates especially to an alloy composed of the metals tantalum and tungsten preferably in conjunction with certain other additions as will be hereinafter described.

Highly refractory and highly infusible metals such for example as tungsten have been extensively used in the form of rods or wires for contact points, incandescent lamp filaments and the like. Tungsten of itself possesses the disadvantage that it is an extremely intractable metal to convert into a ductile form suitable for being drawn into wires etc. In the present invention it is an object to so combine the metal tungsten with a metal of slightly lower melting point, preferably tantalum, to form an alloy which is somewhat more easily fusible than tungsten and which therefore may be more readily converted into a molten state and subjected to heat treatment or other process to thereby alter the structure of the alloy and in some cases form various eutectic mixtures having advantageous properties in certain applications.

In order to secure eutectic formation as above mentioned it is preferable to have present from one to two chemical equivalents of tantalum metal to ten equivalents of tungsten (say about 181 to 362 parts by weight of tantalum to 1840 of tungsten) but the proportion may be varied according to the specific eutectic desired, degree of ductility and melting point.

For making fine wires or filaments it is preferable to have present a few per cent of an oxide of the rare earth such as thoria or ceria or in some cases calcium oxide or beryllium. Ordinarily between one and five per cent of such oxides may be used in order to retard crystallization or other structural changes liable in some cases to take place. A mixture of oxides such as equal parts of thoria and ceria, or of one part of calcium oxide to two parts of thoria, or of two parts of ceria to one part of uranium oxide, may be used.

As stated above tungsten as ordinarily prepared forms a very brittle metal which requires much mechanical treatment to render it suitable for the purpose aforesaid. On the other hand tantalum does not have this brittle quality to the same degree and when incorporated with the tungsten metal affords a very useful alloy.

For resistance wire or filament material too high a proportion of tantalum is not desirable as it has greater conductivity than tungsten. In the present invention it is an object to combine and proportion tungsten and tantalum to secure a desirable degree of ductility or workability without using an inordinate amount of tantalum to thereby increase the conductivity in an objectionable degree for manufacture of incandescent lamp filaments. On the other hand for rods to be used for contact points in internal combustion engines, distributing devices etc. a higher percentage of tantalum may be used. In the latter case four or five equivalents of tantalum may be used to ten equivalents of tungsten. The thoria or other oxide employed in the filament to prevent or control crystallization may be omitted from the contact point or rod material.

The introduction of the oxide into the alloy may take place prior to formation of the metals themselves. For example thorium nitrate may be admixed with tungstic oxide in proportion to afford two or three per cent of the thorium oxide in the finished metal. A paste may be thus formed which is dried and ground and then reduced at about 1800° F. in a current of hydrogen. The reduction at this stage affects only the tungstic oxide practically no reduction to metallic thorium occurring, the nitrate being converted to the oxide.

The powdered metal obtained in this manner is charged into a ball mill with the requisite amount of powdered tantalum metal as for example one equivalent of the latter to 10 or 20 equivalents of tungsten powder (i. e. 181 of tantalum to 1840 or 3680 of tungsten) and the metal powders well mixed.

This mixing may be carried out by simply turning the ball mill without having any grinding balls present or in some cases protracted grinding may be carried out to secure intimate incorporation of the metals.

The formation of a rod or wire may be carried out in several ways. In one case the powder may be exposed to a pressure of

nine or ten tons in a mold to form a rod-shaped mass which may be baked for a short time at about 1800° C. to bring about slight sintering so as to make a firmer bar of material. The bar may then be suspended vertically into a sintering vessel and subjected to a current of about 2000 amperes more or less, the amperage being sufficient to maintain the temperature of the bar slightly below the melting point. If too high a temperature is reached the bar will fuse and fall apart. This sintering operation is carried out in an atmosphere of an inert gas or a reducing gas such for example as hydrogen. The temperature is preferably maintained between 100 and 200° C. below the melting point of the alloy. After fifteen minutes to a half hour the sintering operation is complete and the bar is allowed to cool in an atmosphere of hydrogen. It then may be mechanically worked in various ways as by swaging or other similar treatment.

Another method of producing the alloy is that of placing the powdered metallic mixture obtained from treatment in the ball mill as above described in a mold of refractory material, the mold being shaped in the form of a bar or rod and the powder being packed into the mold with as much pressure as the mold walls will stand. The mold is placed in a treating vessel where it is exposed to an inert or reducing atmosphere while a current of electricity is passed from end to end of the powdered mass thereby heating it first to a sintering temperature and finally to a molten state.

The degree of sintering up to the point when the mass becomes molten may be controlled by checking the operation when desired thus producing bars which are more or less sintered according to the treatment received. On the other hand by converting the alloy throughout into a molten condition and then allowing cooling to go on in a regulated manner definite structural changes or formation of eutectic bodies may be engendered. This is not possible at least to anything like the same degree when the particles of metal are merely sintered or fritted together.

The molds used for carrying out this heat treatment may be placed horizontally being open at the top so that the entire bar is exposed along one longitudinal surface or again the mold may be set vertically so that the bar is formed in a vertical position. In this case it is possible to use a cylindrical tube of carbon or graphite which may be lined with refractory material forming a cylinder which serves as the mold. The current in this case is passed through the carbon tube which acts as a resistor and enables the sintering or melting of the material to take place without actual passage of the electric current through it. It is ordinarily preferable to completely separate the metallic powder from the carbon resistor and by refractory material as aforesaid to prevent the formation of the carbides. In other cases where for example the material is to be used for contact points and where the presence of carbides is not of consequence the casting may be made in the carbon resistor itself the metal thus coming into direct contact with carbon and a shell or coating of carbide or alloy containing carbide tending to form at the surface of the molten material.

In another case the mold of refractory material may be spaced away from the resistor as for example by being placed in a carbon tube of larger dimensions than the mold and allowing the heating to take place by radiation from the electrically heated carbon surfaces. This arrangement allows the ready introduction of a reducing gas such as hydrogen.

By the proper choice of molds of desired dimensions the molten alloy may be formed to a rod of any desired dimension thus precluding the necessity of working an otherwise larger bar or rod to the required dimension. In this way rods suitable for contact points etc. may be made direct by casting without resorting to the expensive mechanical treatment in which losses due to the handling of the material in this way as well as the labor costs, oxidation of metal etc. are substantially decreased. By carrying out the method of casting as aforesaid it is possible to carry the temperature substantially higher than when the sintering method of compacting the metal is employed. Thus metal of greater density suited to better advantage in certain applications than the merely sintered metal may be obtained. Also it is possible to bring about a slight reduction of the rare earth oxide or other oxide employed to prevent objectionable crystallization. It is my belief that a partial reduction of for example thorium oxide taking place in this manner yields particles of the thoria slightly eroded through such reduction and forming more substantial cementing surfaces for adhesion to the alloy thereby assisting in the case of filament material in producing a more stable structure.

In place of tantalum I may use in some cases osmium to form the molten and cast product. For contact points molybdenum may be used in place of or with tantalum and tungsten. Nickel is a very desirable addition to such contact point alloy, a product composed of molybdenum, nickel and tungsten being recommended. Not over one or two per cent of nickel is necessary to modify the properties of a molybdenum tungsten alloy in marked degree. It is characteristic of such high melting point

alloys that the introduction of a third metal oftentimes has a marked influence on the product. Tungsten alone may be modified for some purposes by the addition of one or two per cent of nickel.

In one modification of the invention the mixture of metals to form the alloy as above described, is subjected to high pressure as for example to ten or fifteen tons per square inch to form a molded mass. The latter is then baked to a sufficiently high temperature to give it enough strength to make it sufficiently coherent to maintain its own weight while being suspended in a heating chamber without passage of an electric current through the bar itself. One means of accomplishing this is the use of a cylinder of carbon or graphite placed vertically and having at either end suitable connections for the passage of electric current. The bar is suspended in this chamber and the temperature of the latter raised to the sintering point through heat radiated from the walls of the carbon treating vessel or resistor. In this way no clamping or connections are needed for the passage of electric current through the bar itself and hence losses due to breakage of the bars in handling this way are eliminated. Furthermore the direct electrical heating method leaves the metal unsintered at the clamped ends entailing a waste to that extent as it is necessary to cut off these unsintered ends in any subsequent mechanical treatment of the rod.

What I claim is:—

1. An alloy consisting principally of tungsten and tantalum, the former being present in amount about two to about twenty times the quantity of the latter.

2. A rod shaped casting of an alloy composed of not more than two equivalents of tantalum to ten of tungsten.

3. An alloy composed principally of tungsten and tantalum the former being present in amount about two to about twenty times the quantity of the latter, and containing a few per cent of a refractory earthy oxid distributed through the mass.

4. A wire comprising an alloy of tungsten and another high melting point metal having distributed through it particles of a non-metallic refractory substance capable of retarding crystallization.

5. The process of making an alloy which comprises melting tungsten and tantalum together in the presence of a reducing gas.

6. The process of making an alloy which comprises sintering and melting a mixture comprising tungsten and tantalum powders in the presence of a reducing gas.

7. The process of making an alloy of tungsten and tantalum which comprises mixing the two metals in powdered form, packing into a mold of refractory material and heating to the melting point whereby such alloy is formed.

8. The process of making a sintered mass comprising an alloy of tungsten and tantalum which comprises mixing the powdered metals, packing into a mold of refractory material and heating to a temperature at least reaching the sintering point.

9. The process of making a rod comprising an alloy of tungsten and another high melting point metal which comprises packing the powdered metals into a mold of refractory material and applying heat essentially to the mold whereby the latter is raised at least to the sintering point of the metallic material and a rod of alloy material is obtained.

10. The process of forming a rod of high melting point metal which comprises packing the metallic material in the form of a powder into a carbon tube and passing an electric current through the carbon contained and powdered metal to compact and sinter the latter.

11. The sintering of a rod of high melting point alloy by heating without directly passing the electric current through the metallic body.

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