UNITED STATES PATENT OFFICE.

CLEMENS A. LAISE, OF WEEHAWKEN, NEW JERSEY, ASSIGNOR, BY MESNE ASSIGNMENTS, TO GENERAL ELECTRIC COMPANY, OF SCHENECTADY, NEW YORK, A CORPORATION OF NEW YORK.

ALLOY AND THE METHOD OF PRODUCING SAME.

1,423,338. Specification of Letters Patent. Patented July 18, 1922.

No Drawing. Application filed September 13, 1920. Serial No. 409,964.

To all whom it may concern:

Be it known that I, CLEMENS A. LAISE, a citizen of the United States, and a resident of Weehawken, county of Hudson, State of New Jersey, have invented certain new and useful Improvements in an Alloy and the Method of Producing the Same, of which the following is a specification.

This invention relates to a new and useful alloy and to the method of producing the same. It relates more particularly to the production of an alloy that shall possess desirable properties which make it especially suitable for uses as electrical contacts, magneto points, targets and sheet metal for wireless equipment, and wire for heating and lighting elements.

One of the objects of my invention is to produce an alloy that will resist oxidation and will in fact be less readily oxidized than the metal tungsten when used for the purposes as above indicated.

Another object of my invention is to produce an alloy that will resist the tendency to become pitted when used as an electrical contact point, etc., and will continue to present a smooth surface even after long use.

Another object of my invention is to produce an alloy that may be readily welded to steel and will be less apt than tungsten would be to become separated or detached from a steel or similar backing or holder.

Another object of my invention is to produce an alloy having the desirable properties enumerated above but which at the same time shall be cheaper to manufacture than platinum-iridium.

Heretofore contact points have been made of tungsten but these are unsatisfactory especially for magneto purposes because the tungsten becomes oxidized and its surface becomes roughened or pitted when in use and difficulties also arise because the tungsten discs often come loose from the steel backing or support to which they are attached as by welding. Platinum points or contacts and also those made of a platinum-iridium alloy have been found to be more satisfactory than tungsten in these respects, but such points or contacts are very expensive.

By the present invention I am enabled to produce an alloy or mixture that fulfills the requirements of a good contact and at the same time it is very much cheaper than platinum or platinum-iridium. For the purpose of producing a suitable product in accordance with this invention, I add to the metal tungsten a small quantity of gold. This produces a mass that will not readily oxidize as the gold appears to provide a non-oxidizable surface film that effectively projects the tungsten particles from oxidation.

For the purpose of producing a product still more advantageous than the tungsten-gold mixture, I add a small amount of vanadium compound thereby obtaining an alloy or mixture which has proven more satisfactory in the manufacture of contact and magneto points than the materials heretofore used for this purpose. Such an alloy or mixture does not readily oxidize, does not readily crystallize or become brittle, can be welded to steel, and possesses considerable malleability and ductility. The presence of the gold and vanadium in the tungsten enables the production of a weld with steel which is superior to that produced with substantially pure tungsten.

I have also found it advantageous to introduce into the mixture a small quantity of compounds of other materials such as calcium or thorium. The following specific examples are given as being illustrative of the invention, but it is to be understood that the invention is not intended to be restricted to the exact materials or proportions given:

No. 1.

Calcium vanadate	0.1%	to	0.5%	by weight
Gold	0.5%	to	.5%	" "
Tungsten	99.4%	to	94.5%	" "

No. 2.

Calcium vanadate	0.1%	to	0.5%	by weight
Gold	10.0%	to	15.0%	" "
Tungsten	89.9%	to	84.5%	" "

No. 3.

Thorium oxide	0.5 %	to	1 %	by weight
Vanadium	0.06%	to	0.1%	" "
Gold	0.5 %	to	5 %	" "
Tungsten	99.0 %	to	94 %	" "

It has been found in practice that the gold and vanadium exert additional advantageous

effects in that the defective material produced during the manufacturing process is so much less than with substantially pure tungsten that the extra cost of the gold is more than compensated for. Also the presence of the gold and vanadium enables a sintered slug to be produced which is quite ductile and malleable before it is mechanically worked. The slugs may therefore be swaged, hammered or rolled and thus worked into sheets, plates or rods, the latter being subsequently drawn into wire. For convenience it is preferable to work the material hot, as in the case of working metal of high melting point. Wire so produced is suitable for filaments for incandescent lamps, especially those known as daylight lamps or those which use a colored or non-transparent glass bulb in which the deposition or distillation of a small amount of gold out of the wire is not objectionable. The wire is also useful as heating units for electric furnace work.

Instead of using pure gold I may use palo metal or a gold-palladium mixture with certain advantageous results. Also, a gold-platinum mixture may be used instead of pure gold.

A process of making the material will now be described in further illustration of this invention. I first prepare substantially pure tungsten trioxide, WO_3. This may be done by taking crude tungstic acid containing such impurities as alkaline earths, silica, iron, arsenic, etc., dissolving the same in a dilute ammonia or alkaline hydrate solution, and filtering. The filtrate consisting for the most part of ammonium tungstate, is then heated to a temperature of about 70 degrees to 80 degrees C. and slowly introduced into a vessel containing a boiling mixture of about 3 parts nitric acid, 6 parts hydrochloric acid and 4 parts water. A current of steam is injected into the mixture while the filtrate is being introduced and a precipitate of WO_3 is obtained which is removed and washed with hot distilled water.

The oxide of tungsten obtained as described may then be dissolved in ammonia by treating it with a mixture of NH_4OH and distilled water. The mixture should be heated for about an hour and then filtered, thereby obtaining a substantially pure filtrate of ammonium tungstate in solution from which it may be crystallized out by evaporation.

To one kilogram of the ammonium tungstate crystals I add 7.5 to 10 grams of calcium vanadate and 20 to 25 gms. of gold chloride. The ingredients are thoroughly mixed, then dried, and ball-milled for several hours.

Instead of introducing calcium vanadate into the ammonium tungstate I sometimes prefer to introduce a solution of calcium nitrate and ammonium vanadate in such amounts as to give approximately the same proportions of tungsten, calcium and vanadium as above mentioned. When the material is to be used for wire manufacture I prefer to use thorium nitrate instead of calcium nitrate.

It is also practicable to introduce the calcium vanadate and the gold chloride into the tungsten trioxide instead of the ammonium tungstate, but I prefer the latter procedure.

The mixture produced as above specified containing the tungsten, gold, vanadium and calcium (or thorium) may be reduced in any convenient way as by means of hydrogen in a gas furnace or electric furnace. For this purpose a charge of the mixture may be placed in a nickel boat for example, which may be placed inside of a tube made of heat resisting material such as steel, porcelain, alundum or silica which is heated from 2 to 3 hours to a temperature of about 600 degrees to 650 degrees C. and then for about 5 or 6 hours to a temperature of 900 to 950 degrees C. while hydrogen is being simultaneously passed through the tube. This reduces the material and leaves it in a very fluffy, sticky state, having a weight of about 40 to 50 grams per cubic inch. The material may be reduced in other ways, as for example, by means of carbon, but I prefer to use hydrogen.

The above mixture possesses properties that render it adaptable for being pressed as the metallic powder is quite sticky. This powder is placed in a mold and pressed into ingots at a pressure of about 15 to 30 tons per square inch, the size and shape of the mold being determined by the purpose for which the ingots are to be used.

The ingots may then be sintered together by heating them in a chamber containing a reducing atmosphere, such as hydrogen, or a mixture of hydrogen and nitrogen, at a temperature of 1200 degrees C. for half an hour, and then heating them to a high temperature about 90 per cent to 95 per cent of their melting point, which is to be previously determined, for another half hour. This high temperature may be produced by sending a current of electricity through the pressed up ingots or they may be placed upon a tungsten slab that may be heated to a temperature of about 2800 degrees C. by means of an electric current. In the latter case I prefer to use a tungsten slab that has a small percentage of silica, magnesia, thoria or lime incorporated therein to raise its melting point. The presence of the gold in the ingot to be sintered lowers its melting point so that it can be melted if desired on such a tungsten slab used as a resistor element.

When producing an alloy containing a high percentage of gold such as 15 per cent

to 20 per cent, I first met the gold in a sand or carbon crucible, and then introduce into this molten gold enough of the above described metal powder or discs to amount to 80 to 85 per cent by weight of the alloy. The gold alloys with the powder, and the final melt is either permitted to cool or first poured into a mold of any desired shape.

In either case the mixture or alloy produced as above described is ductile and may therefore be taken to a power hammer and mechanically worked while hot and may be rolled or drawn into the desired shapes using gradually decreasing temperatures as ordinarily employed in the art. Discs or sheets of small size may be punched out of the main or large sheet in the usual ways that are well known in this art, or the ingots may be swaged into rods and the discs cut off from the rod in the well known way.

It is to be understood that my invention is not limited by the exact figures mentioned above as to weights and temperatures, etc., but may vary therefrom and therefore the scope of the invention is to be determined only by the claims appended hereto.

Having now described my invention what I claim as new and desire to secure by Letters Patent is:

1. An alloy comprising tungsten, gold and vanadium, the tungsten predominating and the vanadium constituting about 0.1 per cent of the weight of said alloy.

2. An alloy comprising tungsten, gold and vanadium in substantially the proportions respectively of 9 to 1 to 0.1 by weight.

3. An alloy comprising tungsten, gold, vanadium and a constituent to retard crystallization, the tungsten predominating.

4. An alloy comprising tungsten, gold, vanadium and calcium, the tungsten predominating and the calcium constituting about 0.5 per cent of the weight of said alloy.

5. An alloy comprising tungsten, gold, vanadium and calcium, the tungsten being from approximately 9 to 150 times the weight of the other ingredients.

6. An alloy comprising tungsten, gold, vanadium and calcium, the tungsten predominating and the gold being approximately ten times the weight of the vanadium and calcium.

7. An alloy comprising tungsten, gold, vanadium and calcium, the gold being approximately ten times the weight of the vanadium and calcium, the tungsten being from approximately 9 to 150 times the weight of the other ingredients.

8. The herein described process which comprises mixing an oxide of tungsten with gold and vanadium salts and reducing the mixture at a high temperature.

9. The herein described process which comprises mixing an oxide of tungsten with gold and vanadium salts and reducing the mixture at a temperature between 600 degrees C. and 950 degrees C.

10. The herein described process which comprises mixing an oxide of tungsten with gold and vanadium salts and reducing the mixture at a temperature between 600 degrees C. and 950 degrees C. in the presence of hydrogen.

11. The herein described process which comprises mixing an oxide of tungsten with gold and vanadium salts and reducing the mixture at a high temperature, and then subjecting the mixture to a temperature of about 1200 degrees C.

12. The herein described process which comprises mixing an oxide of tungsten with gold and vanadium salts and reducing the mixture at a high temperature, and then subjecting the mixture to a temperature of about 95 per cent of its melting point.

13. The herein described process which comprises mixing an oxide of tungsten with gold and vanadium salts and reducing the mixture at a temperature between 600 degrees C. and 950 degrees C., for about eight hours and subjecting said mixture to a temperature of about 1200 degrees C. for approximately half an hour.

14. The herein described process which comprises mixing an oxide of tungsten with gold and vanadium salts and reducing the mixture at a temperature between 600 degrees C. and 950 degrees C., pressing said mixture under a pressure from 15 to 30 tons per square inch and sintering the same.

15. The herein described process which comprises mixing an oxide of tungsten with gold and vanadium salts and reducing the mixture at a temperature between 600 degrees C. and 950 degrees C., pressing said mixture under a pressure from 15 to 30 tons per square inch and sintering the same at a temperature above 90 per cent of its melting point.

16. The herein described process which comprises mixing an oxide of tungsten with gold and vanadium salts and reducing the mixture at a temperature between 600 degrees C. and 950 degrees C., pressing said mixture under a pressure from 15 to 30 tons per square inch and sintering the same at a temperature of approximately 1200 degrees C.

17. The herein described process which comprises mixing an oxide of tungsten, gold chloride and calcium vanadate and reducing said mixture.

18. The herein described process which comprises mixing an oxide of tungsten, gold chloride and calcium vanadate in substantially the proportions of 9 to 1 to 0.1 and reducing said mixture.

19. The herein described process which comprises mixing an oxide of tungsten, gold

chloride and calcium vanadate in substantially the proportions of 9 to 1 to 0.1 and reducing said mixture at a temperature between 600 degrees C. and 950 degrees C.

20. The herein described process which comprises mixing an oxide of tungsten, gold chloride and calcium vanadate in substantially the proportions of 9 to 1 to 0.1 and reducing said mixture, at a temperature between 600 degrees C. and 950 degrees C., and pressing the resulting product at a pressure between 15 and 30 tons per square inch.

21. The herein described process which comprises mixing an oxide of tungsten, gold chloride and calcium vanadate in substantially the proportions of 9 to 1 to 0.1 and reducing said mixture, at a temperature between 600 degrees C. and 950 degrees C., pressing the resulting product at a pressure between 15 and 30 tons per square inch, and sintering the same.

22. A body consisting of tungsten and gold, and small admixtures of vanadium and calcium tungsten predominating.

23. A body of gold and tungsten, containing an admixture to retard crystallization tungsten predominating.

In witness whereof I have hereunto set my hand at the borough of Manhattan, city and State of New York, this 11th day of September, 1920.

CLEMENS A. LAISE.

In presence of:

ISABEL R. FLETCHER.