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ALLOY AND PREPARATION OF THE SAME FOR CONTACT BODIES AND IGNITION POINTS.

APPLICATION FILED MAY 2, 1921.

1,418,081. Patented May 30, 1922.

Fig. 1.

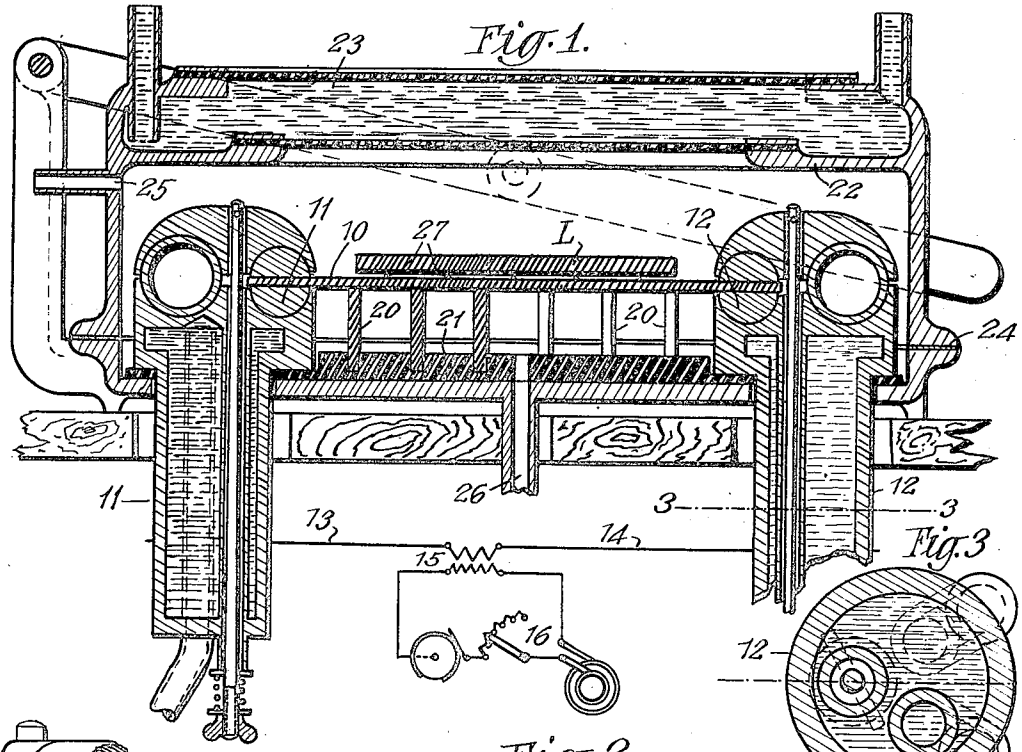
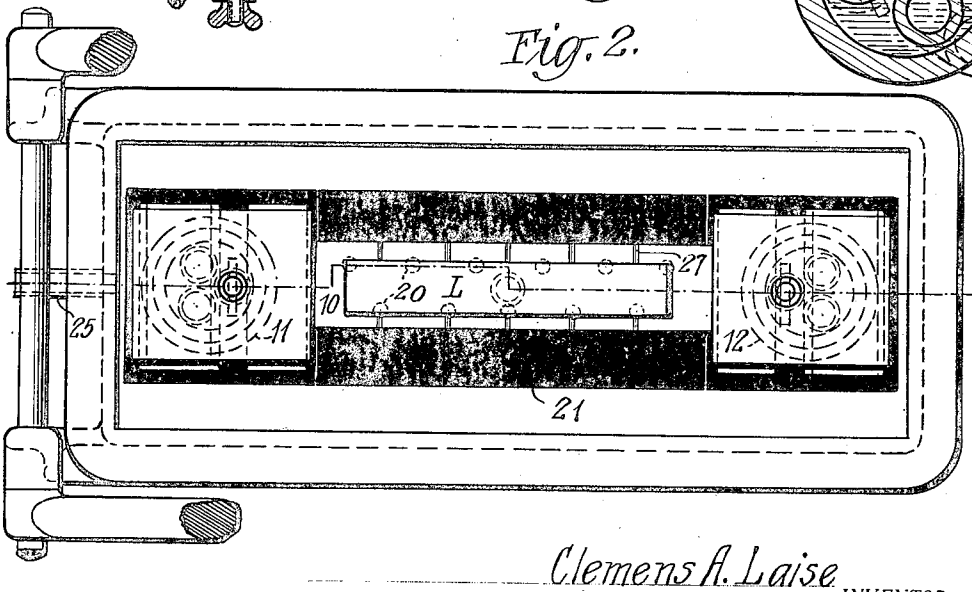


Fig. 3

Fig. 2.



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ALLOY AND PREPARATION OF THE SAME FOR CONTACT BODIES AND IGNITION POINTS.

1,418,081. Specification of Letters Patent. Patented May 30, 1922.

Application filed May 2, 1921. Serial No. 466,337.

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 Alloys and Preparation of the Same for Contact Bodies and Ignition Points, of which the following is a specification.

This invention relates to improvements in alloys and the object is to provide a new and superior alloy of very compact and fine grained structure for use as face plates or contact points for electric make and break contacts and magnetos and ignition points or other electrical devices.

The particular object of the invention is to make a special alloy for such purposes as those mentioned above which will be practical in use and which will supplant the use of either pure platinum or tungsten, and which will be economical to manufacture.

In the drawings forming part of the specification:

Fig. 1 is a vertical section of a furnace which I find very efficient for the treatment of my improved alloy in preparing the same for use as face plates in the manufacture of contact points.

Fig. 2 is a plan view of the same with the cover removed.

Fig. 3 is a section on line 3—3 of Fig. 1.

The particular alloy made in accordance with the present invention is malleable and consists of a compact and very fine grain structure of tungsten, molybdenum, vanadium and gold in the following proportions:

Alloy #1.	By weight.	By volume.
	Per cent.	*Per cent.*
Tungsten	96.7 to 87.5	93.65 to 77.2
Molybdenum	3.0 to 10.0	6.0 to 20
Vanadium	0.05 to 0.5	.10 to .8
Gold	.25 to 2.0	.25 to 2.0

I find that the small percentage of gold results in a considerable increase of electrical conductivity of the contact material. Consequently, the following alloy is also embodied in my present invention.

Alloy #2.	By weight.	By volume.
	Per cent.	*Per cent.*
Tungsten	97 to 90	93.5 to 80
Molybdenum	3 to 10	6.0 to 20
Vanadium	.05 to .5	.1 to .8

The alloys are prepared in the following manner:

The starting material is a tungstic acid which has been purified so that it is chemically pure WO_3 containing not more than .25% mineral impurities. This tungstic acid is dissolved by slowly introducing 1,000 grams of the same into a mixture of 1,000 cc. of C. P. ammonium hydrate sp. gr. .900, and 2500 cc. distilled water. After stirring for half an hour most of the WO_3 is dissolved and the ammonium tungstate solution is filtered. The solution should then have a clear water colored appearance with just a tinge of yellow color.

This ammonium tungstate solution is then placed into a porcelain evaporating dish. About 500 cc. of C. P. HCl is permitted to run into the same from fine constrictions with constant stirring, until the solution begins to get turbid and very fine crystals begin to float around the same. Silica tube immersion heaters are then introduced into the dish and the solution is evaporated to $\frac{2}{3}$ of its original bulk. The heaters are then taken out and the liquid is allowed to cool to room temperature. The resultant crystals produced by the combined action of salting out and evaporation, have a very fine needle like structure particularly suited for the production of my materials. These crystals are then allowed to settle; the mother liquor is poured off; the crystals are washed several times with distilled water then filtered through a Buchner vacuum funnel and dried in a nickel pan in a gas furnace heated to about 300 to 400 degrees C. long enough so that most of the ammonia is driven off but so that it still contains some undecomposed ammonium tungstate and the color has a

tinge of green rather than pure yellow, and contains about 98 per cent to 99 per cent WO_3 and 2 per cent to 1 per cent NH_3.

To prepare the alloys I add to each 1,000 grams of WO_3 powder a distilled water solution containing 80 grams of C. P. ammonium molybdate and 5 grams of ammonium vanadate. These are thoroughly mixed in a porcelain evaporating dish and then dried and ignited at a temperature of 200 to 350 degrees C. To prepare alloy #1 I then add to each 1,000 grams of the above alloy mixture, a slightly acid solution containing 15 grams of gold chloride. This is thoroughly incorporated and mixed and after drying is milled for one hour or so that the ingredients are all uniformly distributed throughout the same.

Now having the oxides of my starting materials which have a brownish-green color, the step of reducing the same is performed. This is preferably carried out in a reduction furnace so constructed that hydrogen passes through the oxides during the reduction and produces a very fine sticky powder. I find it is desirable to effect this reduction in three steps: First, to heat the mixture in a steady flow of hydrogen at about 400 to 500 degrees C. for approximately two hours, thereby driving off the ammonia and reducing the ingredients to a purple oxide; second, to subject this purple oxide to a temperature of about 600 to 700 degrees C for about four hours and then to about 900 degrees C. for approximately two hours. The material thus produced becomes gray or brownish gray and is allowed to cool in hydrogen and is taken out and sifted through a 100 mesh sieve. The third step is to then reduce this material at 800 to 900 degrees C. for approximately two hours and finally at 1100 to 1200 degrees C. for another two hours.

For ordinary purposes a flow of about 10 to 15 cu. ft. per hour of dry hydrogen or of hydrogen bubbled through aqua ammonia is maintained through a tube approximately 2″ in diameter, which is electrically or otherwise heated, said tube being made of steel, alundum or porcelain. The tube contains nickel boats each containing about 500 grams of the above mixture of oxides.

The final reduced product should consist of a fine sticky gray metal having a weight of about 39 to 45 grams per cubic inch. The metal after standing in sunlight for a few days has a tendency to assume a yellowish cast apparently due to a slight oxidation of the vanadium.

This metal is then packed into nickel-chromium alloy molds, being finally forced into the same by applying pressure by means of a plunger and arbor press, the molds having previously been heated in air so that they are coated with a coating of chromium oxide not reducible in hydrogen.

These molds are held together by means of clamps and containing the metal powders are placed into an electric or gas furnace through which hydrogen is continuously flowing and which are subjected to a temperature of about 1100 to 1300 degrees C. Each mold remains therein for a half an hour and is then shoved into a cooling chamber for five minutes and then taken out. On opening the mold it will be found that owing to the combined pressure and heat treatment the powdered metal has sintered together into a compact very fine grained sintered ingot having a metallic ring.

These sintered rods or bodies are subjected to a further heat treatment, so that the body will shrink to about 80 per cent of its original volume and will be correspondingly increased in density, to obtain a compact and very fine grained structure.

This result is accomplished by passing a current through the bodies under treatment until they are heated to a temperature amounting to 85 per cent to 92 per cent of their fusing current for a period of thirty minutes or by subjecting them to an external fusing operation after each of which they are ready for mechanical working.

As an effective means for subjecting these bodies to the above stated external heat treatment before working, I have found it desirable to place these rods into a heat treating furnace, as illustrated in Figs. 1 and 2. This furnace consists essentially of a refractory metal slab 10, such as tungsten, which may either be flat or in the shape of a semi-circular tube, connected at both ends to water cooled copper electrodes 11 and 12 so that the slab has sufficient freedom of motion to allow for expansion. The leads 13 and 14 are connected to these copper electrodes which are in turn connected up with a transformer 15 and induction regulator 16 by means of which the current through the slab is regulated. The slab or semi-circular tube 10 is supported on refractory metal or tungsten rods 20, which in turn are held in place by means of non-conducting refractory lining 21. The cover 22 of the furnace is water cooled as at 23 and is lined with rubber at 24 to make the apparatus gas tight. Hydrogen or a reducing gas is introduced through inlet 25 and passes out at 26. Fine tungsten wires or tungsten projections 27 are placed on the slab 10 so that the major portion of the current passes through the slab rather than through the material to be heated, the heat being transferred to the alloy by means of radiation and conduction rather than being generated by the current.

The size of the heating element or refractory metal slab depends upon the size of the ingots or rods to be fused. I usually use a slab which is about 15″ long x 35″ x .045″ thick and it takes about 1600 amperes at 20

volts to bring it to 90 per cent of its fusing current or to a temperature of about 2800 degrees to 3000 degrees C.

The alloy metal rods or slabs L are placed upon the heating element 10 and since the melting point of the alloy is lower than that of the tungsten slab, it is brought to a state of fusion. After the rod has been fused in this manner for about thirty minutes which results in a contraction to about 80 per cent of its original volume, I find that it is then sufficiently fused to permit of ready mechanical working. When fusing larger size slabs it is desirable to turn the same so as to get a uniform sintering effect on both sides.

As I find that the denser metal gives the best results for contacts, I endeavor to submit the ingots to the highest possible temperature. As above stated, after the original volume of the slug has been reduced by 18 per cent to 25 per cent, or after its density has been increased by 18 per cent to 25 per cent, it is in a suitable condition for mechanical working by hammering, rolling, drawing or swaging into sheets or rods suitable for contact purposes by methods well known in the art.

In order to reduce the oxidation of these alloys to a minimum during the mechanical working operations, I find it desirable to coat the same with a thin layer of chromium oxide, since the latter is non-volatile and non-reducible in hydrogen.

As in the case of all metal working operations it is preferable to first work these alloys hot and then gradually reduce the temperature and finally work them cold with intermediate annealing.

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

1. An alloy containing about 87 to 97 per cent by weight of tungsten, about 10 to 3 per cent of molybdenum, and not more than one per cent yet a substantial amount of vanadium.

2. An alloy containing about 87 to 97 per cent by weight of tungsten, about 10 to 3 per cent of molybdenum, about 0.05 to 0.5 per cent of vanadium, and the remainder of gold.

3. An alloy consisting of about ten to thirty times as much tungsten by weight as molybdenum and about twenty to sixty times as much molybdenum as vanadium.

4. An alloy containing about 87 to 97 per cent by weight of tungsten and containing molybdenum and vanadium in the proportions ranging from about 20 to 60 of molybdenum to one of vanadium.

5. An alloy containing about 3 to 10 per cent by weight of molybdenum, the remainder being tungsten, vanadium and gold, the tungsten predominating and the vanadium least.

6. An alloy containing about 3 to 10 per cent by weight of molybdenum, about 96 to 87 per cent of tungsten, vanadium and gold, the gold predominating.

7. The herein described process which comprises reducing a mixture containing oxides of tungsten, molybdenum and vanadium, and heating the reduced product under pressure.

8. The herein described process which comprises reducing a mixture containing oxides of tungsten, molybdenum and vanadium, and heating the reduced product under pressure in a nickel chromium alloy mold.

9. The herein described process which comprises reducing a mixture containing oxides of tungsten, molybdenum and vanadium, and heating the reduced product under pressure in a nickel chromium mold to a temperature of about 1100 degrees to 1300 degrees C., removing the ingot and subjecting it to a heat treatment until its volume is reduced by fusion by approximately 20 per cent.

In witness whereof I have hereunto set my hand at the borough of Manhattan, city and State of New York, this 29 day of April, 1921.

CLEMENS A. LAISE.