

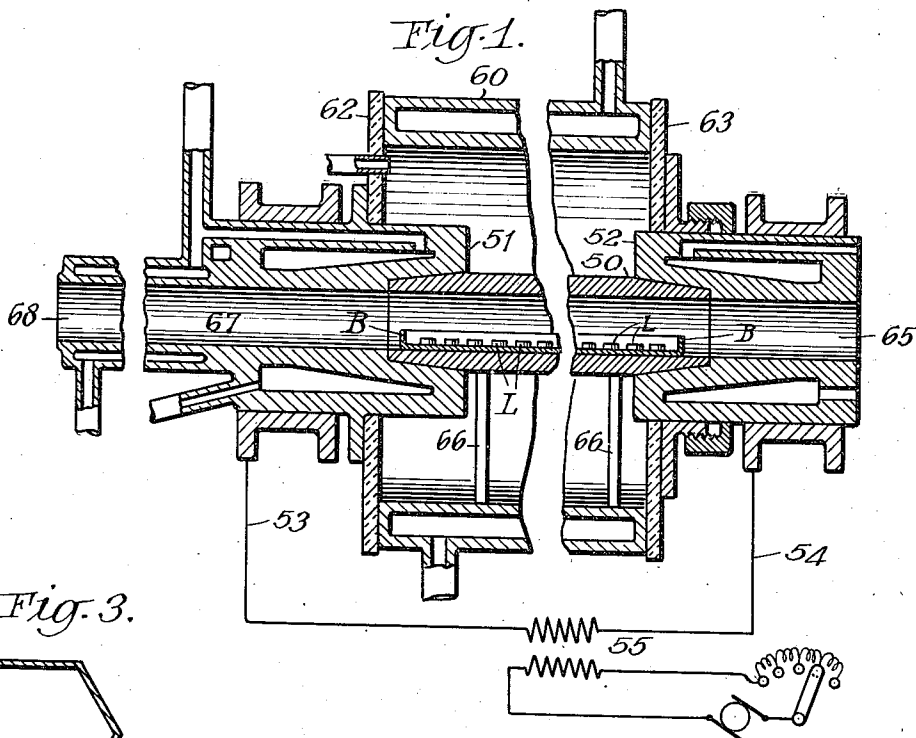
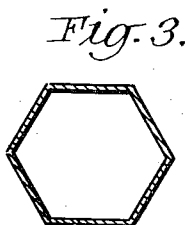
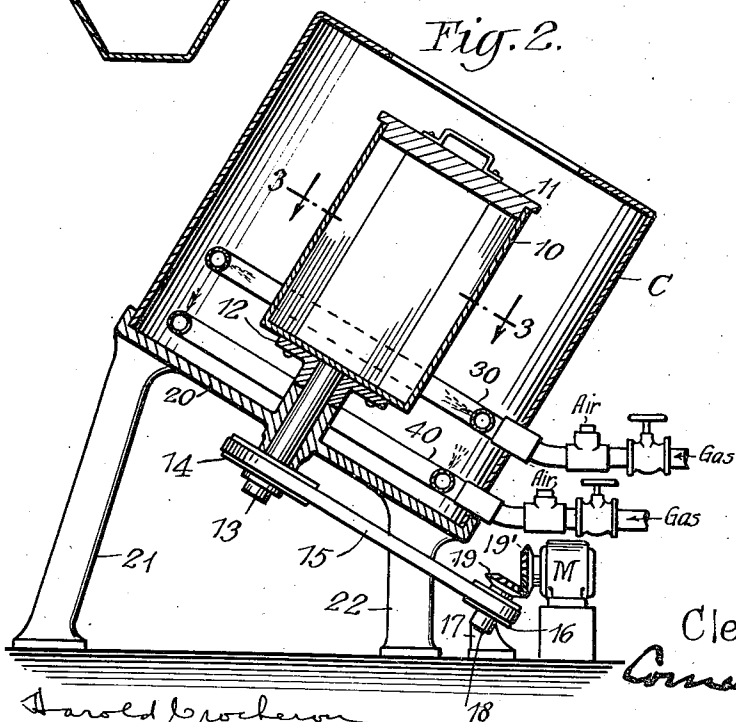
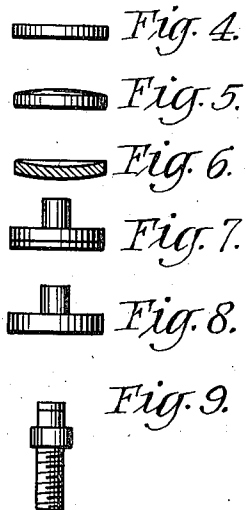
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REFRACTORY METALLIC BODY OF HIGH DENSITY AND PROCESS FOR MAKING THE SAME

Filed June 12, 1922



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# UNITED STATES PATENT OFFICE.

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REFRACTORY METALLIC BODY OF HIGH DENSITY AND PROCESS FOR MAKING THE SAME

Application filed June 12, 1922. Serial No. 567,619.

*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 Refractory Metallic Bodies of High Density and Processes for Making the Same, of which the following is a specification.

This invention relates to the production of metallic bodies of refractory metals of a density or specific gravity above 18, such bodies being of small dimensions usually not exceeding 5 to 10 grams in weight.

I have discovered that instead of mechanically working metallic bars of refractory metals by hammering, swaging, rolling or drawing the said metals and then shaping them into small bodies by cutting or punching, I can press up the refractory metallic powders into any desired shape, then fuse or sinter the pressed up bodies into solid coherent forms and then finish the said bodies for commercial use by my improved rotary impact finishing process.

I have found that by carrying out these steps, not only a superior product is obtained, but a product can be manufactured much more economically than by means of processes now known.

The refractory metallic bodies which are ordinarily desired for use in the arts may be of any suitable shape, but are usually in the form of cylindrical discs having a thickness not exceeding  $\frac{1}{8}$ " to  $\frac{1}{4}$ " and a diameter ranging between  $\frac{1}{16}$ " to  $\frac{3}{4}$ ". As it is well known, the dimensions and shapes may, however, vary according to the purposes for which the products are to be used. The desired shapes or bodies are commonly used as face plates for contacts or ignition points in gas engine installations for magneto points, contacts for telephony or telegraphy, for X-ray targets, circuit controllers, rectifiers, and for many other purposes which will readily suggest themselves.

My invention especially appertains to the use of refractory metals having a normal specific gravity of approximately 18 and above, and especially those metals which do not readily corrode or are not acted upon by the standard acids and alkalis. The metallic bodies which are particularly desirable to produce are bodies of tungsten, platinum, iridium, gold thorium, tantalum,

uranium and alloys or mixtures of these metals. Some of the precious and refractory metallic bodies have been produced heretofore either by bringing the metal into a molten state and pouring it into a form of a rod or slug, or by pressing the metallic powder into an ingot or slug and then sintering the ingot into a coherent body. These coherent bodies were then repeatedly hammered, rolled or swaged while hot, the temperature being reduced at stated times as the bodies became smaller, and then either drawn down into a rod by cold drawings, with intermediate annealings, or else by hot drawing, the temperature being likewise reduced at intervals as the cross section of the rod becomes smaller. The discs were then cut from the rods or else punched from the rolled plates.

One of these processes is in practice today by the General Electric Company and it is set forth at great length in Patent No. 1,082,933 of December 30th, 1913, to William D. Coolidge, assignor to General Electric Company.

In order to overcome the great difficulties and to save the enormous expense resulting from the mechanical working of refractory metallic bodies having a normal specific gravity above 18, and furthermore, to overcome the great difficulties of shaping these bodies into definite forms to make them suitable as face plates for various purposes I have invented a new process and product which will be hereinafter set forth. Since the bodies to be produced are usually very small and thin, this process can be effectively carried out on a very large scale.

I have discovered a process by which bodies made from any one of the metals specified or alloys of these metals, including tungsten and alloys of tungsten, in which, instead of mechanically working the metal by hammering, rolling or drawing into the shape desired, I am enabled to eliminate this long expensive proceeding. I have found that commercial bodies of tungsten metals can be produced by a process differing essentially from the special processes of the prior art in all which the forms required for industrial use were produced by a special mechanical working. In my process the metallic bodies are first shaped into the desired forms by pressing the metallic powder into that form and fusing or sintering it.

subject the sintered body to a finishing impact process for a considerable length of time under a constant temperature by placing the sintered shapes of tungsten with impact balls, such as tungsten steel in a tumbling barrel, and subjecting them during the tumbling to a heat treatment. The temperature during this heat treatment is not only constant but is maintained at a temperature much below the melting point of the metal. I have found that a dull red heat is an operative temperature. When the precious metals are used the tumbling and impact step may be carried out at room temperature.

In the accompanying drawings I have illustrated mechanisms which I have found very suitable for use in aiding in the performance of the several steps of my process.

Fig. 1 is a sectional elevation partly broken away illustrating a furnace suitable for sintering the pressed up bodies from powdered metal.

Fig. 2 is a cross section of a tumbling apparatus within which sintered bodies or shapes are placed together with the impact balls.

Fig. 3 is a cross section of the tumbling barrel shown on line 3—3 of Fig. 2.

Figs. 4 to 9 illustrate various products in which Fig. 4 is a horizontal view of one form of disc.

Fig. 5 is a similar view of another form having a convex surface.

Fig. 6 is a cross section of a disc, one surface of which is concave and the other convex.

Fig. 7 is a contact having a face plate of tungsten and a backing of iron or steel.

Fig. 8 is a contact consisting wholly of tungsten.

Fig. 9 is a screw contact having a tungsten face plate.

Briefly referring to the drawings:

The sintering furnace illustrated in Fig. 1 consists essentially of a refractory metal tube or slab 50 of tungsten or tungsten alloy, which has a stationary water cooled copper electrode 51 attached to one end, and a movable water cooled copper electrode 52 attached to the other end so as to allow for the expansion or contraction of the metal tube or slab 50 during the heating operation. The ends of the tungsten tube 50 fit into the water cooled electrodes 51 and 52, as shown, to which the main leads 53 and 54 are attached, these leads being connected in circuit to a transformer 55, conventionally shown. The entire chamber is enclosed by a water cooled cylindrical casing 60 which rests on supports of insulating material 62 and 63. The pressed up forms or shapes L to be sintered are placed upon a boat B of suitable material and are then introduced into the tube or

on to a slab through the opening 65. The refractory metal slab or tube is supported by refractory metal rods 66 of the same material as the heating element. The shapes to be sintered are allowed to remain in the heating element after it has reached its maximum temperature approximately 2700 degrees C., for about ten minutes. The current passing through the heating element is then interrupted, the boat is shoved into the water cooled chamber 67 by means of a tungsten rod and then removed through the opening 68. The operation is carried out in an atmosphere of hydrogen or in a low pressure hydrogen vacuum. In the latter case long rubber stoppers are placed into each end and the chamber is evacuated through a glass tube in one of these rubber stoppers. The forms or shapes being heated by external means no blow holes are formed in the center and a much more compact mass is therefore obtained.

In Fig. 2 is shown a tumbling barrel 10, the hexagonal sides being to more effectually throw the contacts about during its rotation, as readily understood. The barrel 10 is provided with a cover 11 and a fixed bearing 12 on its underside through which the shaft 13 extends and to which it is secured. The supporting plate 20 is provided with legs 21 and 22 of different length in order that the support may be held at an angle to the horizontal. On the shaft 13 is a pulley 14 which operates a belt 15 which engages a pulley 16 on a support 17 to which is secured a shaft 18 carrying a gear 19 meshing with a gear 19' secured to the shaft of the motor M. The rotating drum is mounted within a casing C which rests upon the supporting plate 20, as shown. Within the casing are provided air and gas burners 30 and 40 arranged with apertures, as shown, the burner 40 being especially arranged so that the flames may impinge against the lower portion of the tumbling barrel.

The products shown in Figs. 4 to 9 are clearly illustrated, having the shapes above specified.

The essential steps of my invention are as follows:

First: The production of a very fine or fine grained metallic powder of high density the great fineness of the powder tending to produce a body of high specific gravity.

Second: The pressing of this dense powder into coherent bodies in the desired shape at high pressures, thus forming the body into its desired shape and at the same time increasing the density.

Third: The fusing or sintering of these compressed bodies in an inert or reducing atmosphere or even in vacuum or under pressure at a temperature near their melting

point, thereby also distilling out any volatile constituents which may purposely have been introduced to draw the particles of refractory metal together. This step further increases the density.

Fourth: The mechanical tumbling of the above metallic bodies either at room temperature or at a higher temperature, which higher temperature, however, is constant but which is much below the melting point of the metal being acted upon so that by means of this impact with hard bodies, such as tungsten or vanadium steel balls, their density is still further increased.

While my process is applicable to the production of bodies or shapes of the kind adapted for use in the arts, it is particularly applicable for the production of dense face plates of tungsten, platinum, iridium and their alloys.

In order to produce face plates of tungsten or platinum or their alloys I first produce as nearly a chemically pure tungsten or platinum powder as possible, preferably by the reduction of the pure tungsten oxide with hydrogen, if tungsten powder is desired, or by the ignition of ammonium chloroplatinate if platinum is desired. These metallic powders should be as void of crystalline structure as possible, though in some cases I may add a certain percentage of crystalline powder in order to have the metal flow better at the tablet machine. To the chemically pure metallic powders I then add a small percentage of a fluxing metal which alloys with the main constituent at low temperatures but which is volatilized out at the higher temperatures and thereby has a tendency to draw particles of the main constituents together. For tungsten, I prefer to use about 1 to 2 per cent of copper, iron, gold or nickel.

To the chemically pure tungsten powder I prefer to add 1 per cent to 1.5 per cent of chemically pure nickel powder and the mixture of tungsten and nickel powder is then ball-milled from 10 hours to 24 hours. The ball milling may be done in nickel containers using balls or discs of coherent tungsten so that there is no contamination of impurities. After this metallic powder has attained a weight of about 80 to 100 grams per cubic inch it is ready for pressing.

The pressing step is preferably carried out on a Stokes tablet machine #N or #O, the shape of the disc depending upon the purpose for which the body is to be used. As above stated, the metal powder should be of such a structure that it will readily flow from the hopper and is compressed at a pressure of about 50 tons to 100 tons per square inch.

The bodies of pressed tungsten powder are placed into nickel boats and heated in hydrogen at a temperature of 1000 degrees C. to 1200 degrees C. for about a quarter to

half an hour. This imparts sufficient strength to them so that they can be handled and still retain their sharp edges. If the metal has the proper physical structure so as to produce a strong disc this step is not necessary.

These small bodies are then placed into the tungsten boats B and subjected to a heat treatment in a tungsten tube furnace such as shown in Fig. 1. This treatment is preferably carried out in an atmosphere of hydrogen at a temperature of about 2700 degrees C. if tungsten bodies are produced, and at a temperature of about 1600 degrees C. to 1700 degrees C. if platinum or platinum alloy bodies are produced. This treatment consists of a combination sintering, volatilization and fusing action; and at the above temperatures a treatment of 5 to 10 minutes duration is sufficient. During this treatment the volatile constituents which draw the metal together are distilled out and the particles of refractory metals are fused together to dense coherent bodies. After cooling in hydrogen they are removed from the furnace.

They are then subjected to my impact finishing process whereby they are tumbled in a revolving barrel such as shown in Figs. 2 and 3 with hardened balls or impact bodies, which may be round or sharp edged depending upon the purpose for which they are to be used. The balls as used in bearings which are made of vanadium or tungsten-steel or of other tool steel or alloys such as stellite, etc., which will not readily soften under heat, are commonly used. The tumbling may be done at room temperature or at elevated temperatures. In the latter case the tumbling barrel may be heated by means of gas as shown in Fig. 2, a metal casing C being placed around the barrel A which encloses the heat and keeps it at a uniform temperature approximately at 400 degrees C. The heating of the barrel may also be done electrically. When treating substantially pure tungsten discs, a dull red heat is preferable whereas when the platinum metals are subjected to this treatment the operation may be carried out at room temperature. During this impact finishing process the pores of the discs are closed, the surface of the discs are compacted and made denser, the surface of the metallic bodies is polished and the density of the metallic bodies themselves is considerably increased.

The tumbling is usually carried out over a period of from four hours to thirty hours depending upon the character of the metals that are being tumbled or until the metallic bodies attain the desired density. The tumbling may also be carried out in an inert reducing or hydrogen atmosphere.

After tumbling operation is completed the metallic bodies are treated with weak

acids or alkalies to cleanse the surface and are then tumbled in saw-dust after which they are ready for welding or for the market.

- 5 The impact finishing treatment is carried out at one temperature and not at a gradation of temperatures. Furthermore, at no stage of manufacture have the metallic bodies had a fibrous or flaky structure imparted to them.

10 The essential characteristics of a successful face plate of a refractory metal for contacts or X-ray work is that the same be of the highest density obtainable and that it be of fine grain structure. Both of these features are attained through my process. Furthermore, the face-plates of tungsten or similar metals as made to-day vary decidedly in structure owing to the non-uniform heating process to which they are subjected during the process of manufacture whereas by my process all discs are uniformly heated from an external source and therefore are uniform in structure.

25 Thus for instance in the prior art bars of pressed tungsten are sintered by sending a current through the same equivalent to about 90 per cent of the fusing current in an atmosphere of hydrogen. By this means a temperature gradient is produced between the interior of the slug and the outside from which the heat is carried by the hydrogen. This produces non-uniformity across the slug and furthermore since the ends are held at water cooled electrodes there is a decided difference in structure between the metal near the water-cooled electrodes and the metal near the center of the slug. All of these factors cause a non-uniformity in the structure of the discs made from rods or plates of such material so that it has given the manufacturer of ignition systems considerable trouble. Since my process subjects the metallic bodies to a more uniform heat treatment most of these undesirable features are eliminated.

Furthermore, any pores that may be in the metallic bodies made according to the old process remain open which causes pitting whereas any pores in the metallic bodies produced by my process are closed during the impact treatment.

Another advantage of my process is also manifested in that I find that instead of merely making a disc I can make a complete rivet by my process such as shown in Fig. 8 thereby avoiding the extra operation of welding the disc on to the steel or nickel blank. This is especially desirable when working with tungsten or tungsten alloys or mixtures where the cost of the material of which the contact is made is low enough so as to permit of this procedure.

My process also enables me to make discs or rivets of alloys or mixtures of the pre-

ious metals and metals of the tungsten group thereby enabling me to produce a contact of these metals which may be substituted for platinum or platinum-iridium and which is equally as good and decidedly cheaper.

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

1. A method of forming articles of the kind described which comprises pressing fine powders of metal into a desired form, sintering the formed article at a high temperature and compacting the articles at a substantially uniform but lower temperature by an impact, tumbling action.

2. A method of forming articles of the kind described which comprises compacting a metal powder containing a small quantity of a comparatively volatile substance under pressure to a desired form, volatilizing said substance at a high temperature and compacting the article at a lower temperature by an impact, tumbling action.

3. A method of forming articles of the kind described which comprises compacting a metal powder into a desired form, sintering the formed article at a high temperature and compacting said article by an impact, tumbling action at a temperature of approximately 400 degrees C.

4. A method of forming articles of the kind described which comprises compacting a metal powder containing a small quantity of a comparatively volatile substance under pressure to a desired form, volatilizing said substance from said article at a high temperature and compacting said article by an impact, tumbling action at a temperature of approximately 400 degrees C. in a reducing atmosphere.

5. A method of forming metallic articles of the kind described which comprises compacting a powder of metals of a density of over 18 to a desired form, sintering said metals and compacting said sintered article at a substantially uniform lower temperature by an impact, tumbling action.

6. A method of forming metallic articles of the kind described which comprises compacting a powder of metals of a density of over 18 with a small amount of a more volatile metal, heating said compacted article to a temperature sufficient to volatilize said more volatile metal, and compacting said article by an impact, tumbling action at a substantially dull red heat.

7. A method of forming articles of the kind described which comprises compacting a powder of metals of a density of over 18 with a small amount of a more volatile metal to a desired form, and heating said articles to a temperature sufficient to volatilize said more volatile metal and then compact said article by an impact, tumbling

action at a substantially uniform temperature.

8. A method of forming articles of the kind described which comprises compacting tungsten powder containing a small amount of nickel powder to a desired form, heating said article to a temperature sufficient to volatilize said nickel and compacting said article at a lower temperature by an impact, tumbling action.

9. An article of manufacture comprising a body of tungsten of high density which is free from a fibrous structure, the surface of which has been still further densified by an impact, tumbling action.

10. An article of manufacture comprising

a refractory metal of high specific gravity, the density of the surface of which is increased over that on the inside of the body by an impact, tumbling action at a temperature approximating a dull red heat.

11. An article of manufacture comprising a contact of tungsten of high density which is free from a fibrous structure, the surface of which being denser than the inside, the said surface being formed by an impact, tumbling action.

In witness whereof I have hereunto set my hand at the borough of Manhattan, city and State of New York, this 6th day of 30 June, 1922.

CLEMENS A. LAISE.