Oct. 2, 1923. 1,469,761

J. A. YUNCK

INGOT OF REFRACTORY METALS AND METHOD OF MAKING SAME

Filed April 28, 1921

Fig. 1.

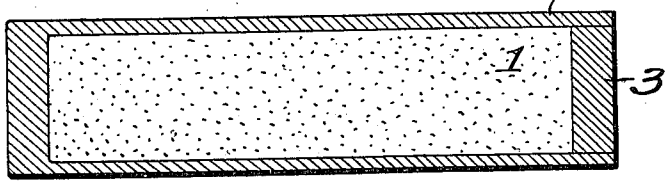


Fig. 2.

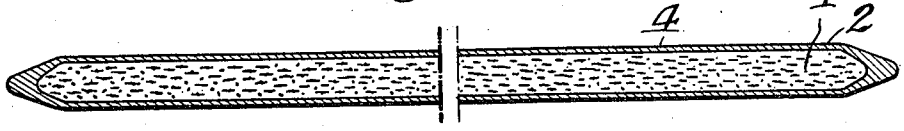
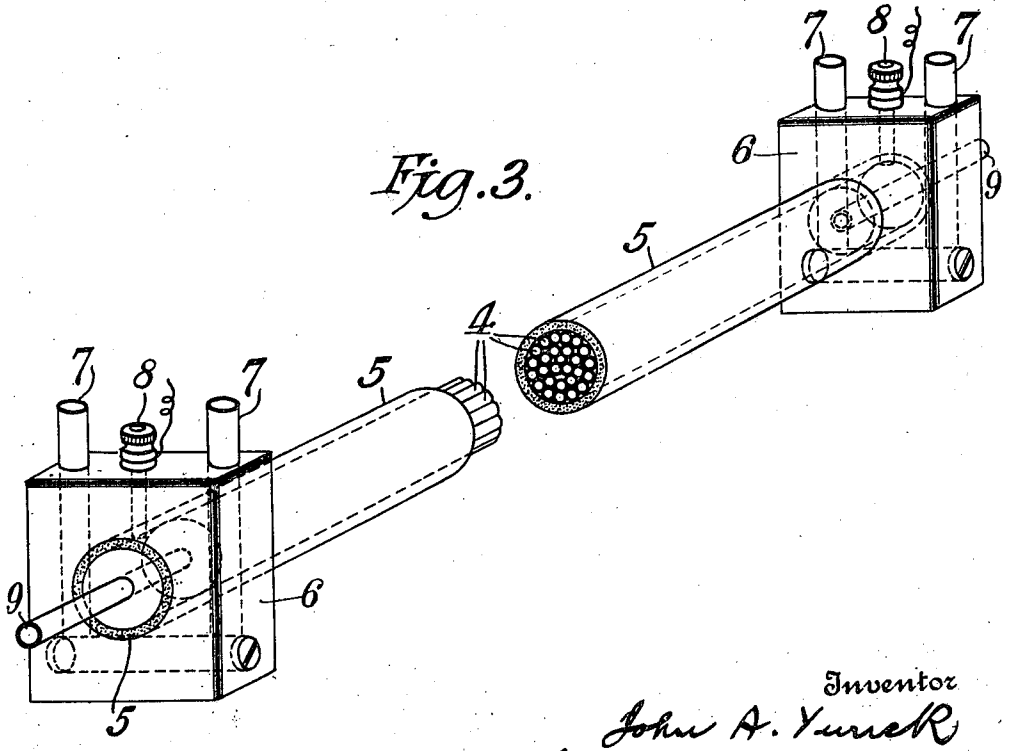


Fig. 3.



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By his Attorney

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INGOT OF REFRACTORY METALS AND METHOD OF MAKING SAME.

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To all whom it may concern:

Be it known that I, JOHN A. YUNCK, a citizen of the United States of America, residing at South Orange, county of Essex, State of New Jersey, with a post-office address at 85 Nassau Street, Orange, N. J., have invented certain new and useful Improvements in Ingots of Refractory Metals and Methods of Making Same, of which the following is a specification.

My invention relates to the production of ductile ingots composed of tungsten or mainly composed of tungsten or similar refractory metal, and consists of an improved process for carrying out such purpose, and of the product of such process.

Heretofore the usual method of producing such ingots has been the formation of an agglomerated body of tungsten powder by high pressure in a mold or by extrusion through a die, baking the same and then sintering it by passing an electric current through it sufficient to raise it to a temperature of about 3300 degrees centigrade, or more. This sintering process is apt to produce a structure of equi-axed crystals in the resulting ingot which renders it very brittle and increases the difficulty of the subsequent swaging and wire drawing process usually applied to it. Also the original agglomerated mass is very fragile and difficult to handle without breaking, and it is apt to be not uniform in density, which adds to the danger of breakage in handling. Moreover the size of ingot which can be produced by these prior processes is limited. My invention avoids these difficulties, simplifies the process so that much less skill and experience is required for its successful operation, and renders the production of a much larger ingot possible.

The following is an example of the best method of carrying out my process, referring to the accompanying sheet of drawings in which:

Fig. 1 is an axial section of the initial cartridge composed of finely powdered metal in a suitable shell or container,

Fig. 2 shows the same after having been swaged cold into the form of a rod, parts being broken away, and

Fig. 3 is a perspective view of the preferred form of electric furnace in which the rods are subsequently treated, parts being broken away.

Throughout the drawings like reference characters indicate like parts.

1 is a body of finely powdered tungsten or similar refractory metal produced in any of the well-known ways, and may be either pure tungsten, or a mixture of tungsten with a minor proportion of thorium or other beneficial additions, or an alloy of tungsten and thorium such as is described and claimed in my prior application, Serial No. 742,711, filed Jan. 17, 1913. This is tightly rammed and packed into a cylinder 2 of some ductile metal of much lower melting point, such as copper. This cylinder may conveniently be formed with an interior diameter of one inch and an exterior diameter of an inch and a quarter. After the cylinder is filled its open end is closed with a plug 3 of the same metal which is held in position by hard solder, or may be brazed or screwed in so as to press firmly on the contained powder.

The cartridge so formed, shown in Fig. 1, is then swaged down while cold, or moderately heated, to a diameter of about three-eighths of an inch. Preferably the reductions in diameter at each passage through the swaging machine is about .02 of an inch. This produces the elongated, composite rod 4 shown in Fig. 2. This swaging gradually compresses together the particles of tungsten and tends to give each particle an elongated form in the direction of the major axis of the ingot so that the resulting mass is more fibrous in structure.

A number of these rods 4, 4, are then placed in a carbon or graphite tube 5, each end of which is held in a water cooled electrode 6. The connections through which the cooling water is circulated through the electrodes are indicated at 7. 8, 8 represent binding posts to which electrical connections may be made for passing a heavy current of electricity through tube 5. A suitable current of hydrogen, or other non-oxydizing gas may be passed through the tube from end to end by means of connections not shown in detail but indicated at 9, 9, as tubes passing through ordinary stoppers.

The carbon tube acts as a resistance to the current and is heated thereby to a temperature sufficient to melt and then vaporize the copper envelope 2 of each rod. This is usually accomplished by slowly raising the temperature to about 1000 degrees centigrade in a period of about 15 minutes. Af-

ter the copper has been vaporized the temperature is further raised to about 2750 degrees centigrade during a period of about 30 minutes and maintained at that temperature for about 10 minutes, after which the tube is allowed to cool for about 30 minutes, thereby annealing the enclosed ingots. Said ingots, being of tungsten, which, according to recent determinations, has a melting point of about 3400 degrees centigrade, are not fused during this heating but merely have their particles fritted together thereby. During the entire operation a steady flow of hydrogen is preferably maintained through tube 5, though other non-oxydizing or reducing gas may be used.

The result is a ductile ingot, or group of ingots, of tungsten, or of tungsten-thorium alloy, or similar composition, each consisting of particles firmly fritted together and of a homogeneous and somewhat fibrous structure (to the extent previously stated) which may be easily hot swaged, and wire-drawn down to filamentary form for use in incandescent electric lamps, or for other purposes.

The carbon tubes are cheap and while they do not last indefinitely, each one may be used for a number of operations before being finally discarded.

The crystallizing and equi-axing action of sintering, on the ingots, resulting in the former processes in which such sintering is performed at a much higher temperature than any I employed in carrying out my present invention, is avoided and the ingot produced according to my invention is much tougher and more ductile than those made by the old processes. Moreover, the size of ingots was limited in the pressing or extruding processes while with my process rods of five feet in length can be made and subsequently treated in the form of tube furnace here shown, each such rod producing about 50000 feet of wire when later drawn down to lamp size.

Having described my invention, I claim:

1. The herein described process of forming an ingot containing tungsten or alloy thereof which comprises the following steps: first, packing fine powder mainly composed of tungsten into a cylinder of soft and ductile metal such as copper, and closing the open end of such cylinder with a plug of the same metal; second, swaging down the cartridge so formed to approximately one-third of its original diameter; third, heating the composite rod so formed in a carbon tube in a non-oxydizing atmosphere until the soft metal envelope has been removed by vaporization and the tungsten particles have been firmly fritted together.

2. The herein described process of forming an ingot containing tungsten or alloy thereof which comprises the following steps: first, packing fine powder mainly composed of tungsten into a cylinder of soft and ductile metal such as copper, and closing the open end of such cylinder with a plug of the same metal; second, swaging down the cartridge so formed to approximately one-third of its original diameter; third, heating the composite rod so formed in a carbon tube in a non-oxydizing atmosphere until the soft metal envelope has been removed by vaporization and the tungsten particles have been firmly fritted together by passing a current of electricity through said carbon tube of sufficient intensity to raise said tube and contents to a temperature of approximately 2750 degrees centigrade, and then allowing the same to cool slowly.

3. The herein described process of forming an ingot containing tungsten or alloy thereof which comprises the following steps: first, packing a fine powder mainly composed of metallic tungsten into a cylinder of ductile metal and closing said cylinder; second, swaging said cylinder and contents down to a reduced diameter at a temperature below its fusing point and to an extent sufficient to materially compact its contents; third, heating the composite rod so formed in a non-oxidizing atmosphere until the metal envelope has been removed by vaporization and its contents have been firmly fritted together.

JOHN A. YUNCK.