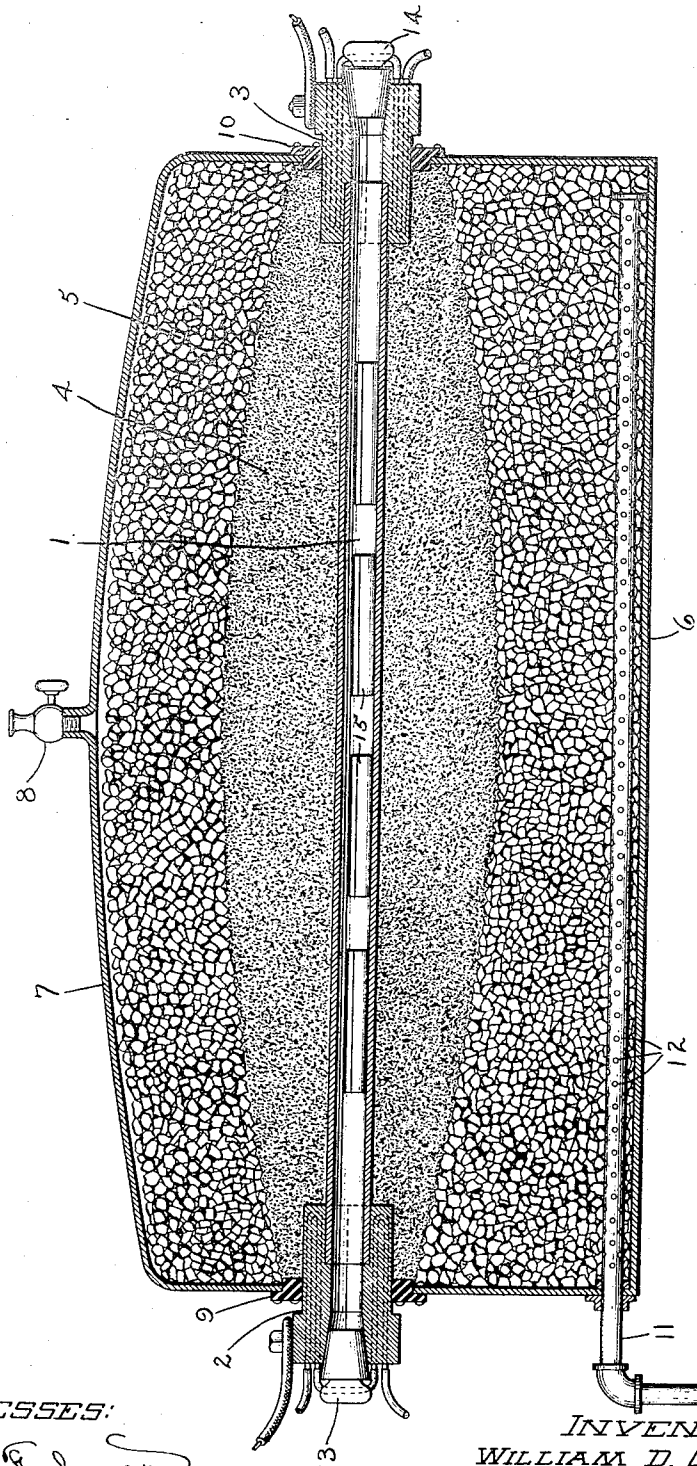


W. D. COOLIDGE.
RESISTANCE FURNACE.
APPLICATION FILED DEC. 3, 1907.

1,004,557.

Patented Oct. 3, 1911.



WITNESSES:

Lester H. Palmer
J. Ellis Allen

INVENTOR
WILLIAM D. COOLIDGE.

BY *Albert S. Davis*
ATTY.

UNITED STATES PATENT OFFICE.

WILLIAM D. COOLIDGE, OF SCHENECTADY, NEW YORK, ASSIGNOR TO GENERAL ELECTRIC COMPANY, A CORPORATION OF NEW YORK.

RESISTANCE-FURNACE.

1,004,557.

Specification of Letters Patent.

Patented Oct. 3, 1911.

Application filed December 3, 1907. Serial No. 404,892.

To all whom it may concern:

Be it known that I, WILLIAM D. COOLIDGE, a citizen of the United States, residing at Schenectady, county of Schenectady, State of New York, have invented certain new and useful Improvements in Resistance-Furnaces, of which the following is a specification.

It is the object of my present invention to provide an electric furnace of the resistance tube type capable of use in heating refractory materials to exceedingly high temperatures, temperatures even as high as that at which carbon begins to vaporize, without danger that the material under treatment will be contaminated with carbon. To effect this result I use as the resistance material of the furnace, a refractory metal, either with or without the admixture of other suitable material. As a metal suitable for this purpose, the refractory element tungsten is suggested.

The novel features of my invention are particularly pointed out in the appended claims, for a better understanding of which reference may be had to the following description taken in connection with the accompanying drawing which is a sectional elevation of the furnace.

The resistance member of the furnace consists of a refractory metal tube 1 supported and supplied with current by copper terminals 2 and 3. These terminals are preferably water cooled and are of sufficient mass to prevent destructive heating at the ends of the resistance tube 1.

To protect the resistance tube 1 from oxidation, it is surrounded or packed with some non-carbonaceous heat insulator 4 such as calcium oxid, alumina, thoria, magnesia, zirconia, etc. Farther away from the resistance tube, outside of the very hot zone, carbonaceous material 5, such as coke, may be used.

The resistance tube and its refractory packings above described, are all inclosed in a metal box 6 having a dome-shaped top 7 in which is a gas outlet 8. The water-cooled terminals of the resistance tube pass through the annular walls of the metal box 6 but are insulated therefrom by refractory bushings 9 and 10. Projecting into the metal box, near the bottom, is a metal tube 11 having perforations 12 through which hydrogen or other inert gas may be introduced.

This gas can move upward through the box and out through the opening in the dome and serves to prevent oxidation of the outside of the resistance tube 1. The inside of the tube can be similarly protected from oxidation by the passage of a stream of hydrogen or other inert gas through the tube. For most purposes, however, it is only necessary to close the ends of the tube with stoppers 13 and 14. Whatever oxygen enters the tube when the charge 15 is being introduced or removed, is very quickly consumed and is so small in quantity as not seriously to affect the resistance material.

I have indicated above that tungsten is a suitable material for the construction of the refractory heater tube 1 and I will hereinafter describe in some detail how such tubes can be built up out of tungsten powder such as is obtainable by reduction of tungsten trioxid with hydrogen. However, this present application is limited to the furnace and its various features, and no claim is herein made to the procedure by which the tube is produced or any of the steps comprised therein, nor to any of the various matters necessarily referred to or described in the description of such procedure. This procedure and other matters not claimed herein are claimed in certain of my pending applications, among which I may particularly mention No. 316,006, filed May 9, 1906, and No. 377,781, filed June 7, 1907.

In general, the process of constructing tungsten tubes from tungsten powder consists in mixing the powder with a binding material free from carbon, shaping the mixture into tubes, and subsequently removing the binder and heating the residue to shrink or sinter it into a dense homogeneous body. As a binding material, I prefer to use an alloy containing bismuth, cadmium and mercury. The alloy may have the following proportions: cadmium 42 per cent., mercury 53 per cent., and bismuth 5 per cent. This alloy is smooth and uniform and, when heated somewhat, becomes so plastic that it may readily be impregnated with tungsten powder, and in fact with almost any powder, by simply rubbing the two together in a chemist's mortar. Tungsten powder may be added to the alloy to the extent of 30 to 50 per cent. by weight of the final mixture. I call the product a mixture because I do not at present believe that

the tungsten alloys with the material of the binder. The mixture is ductile and pliable and can readily be rolled or worked by any of the well known metal working processes.

To obtain tubes suitable for the construction of the tube furnace, I squirt the plastic mixture above mentioned through a tubular orifice at a temperature of 90 to 150 degrees C. The method of extrusion may be quite like that commonly employed for the manufacture of lead pipe. The material comes out as a smooth, silver-white tube, somewhat pliable and tough.

The next step in the process of manufacture consists in removing the easily vaporizable metals of the binder. I prefer to effect this in two stages. As the first stage, I proceed by hanging an unfinished tube in a vertical glass tube which I then evacuate and heat externally either electrically or with a gas flame to drive out most of the mercury and some of the cadmium from the unfinished furnace tube. Most of the bismuth and probably some of the cadmium stay behind with the tungsten. The baked tube is then removed and subjected to the final or firing treatment. This firing operation can be effected in either one of two ways. According to one method of firing, the baked tube is heated in a vacuum furnace until freed from all vaporizable components and until sufficiently shrunk and sintered to permit mounting in the furnace shown in the drawing. According to a modified method of treatment, the tube is baked in an open tube furnace under protection of hydrogen or other inert gas until the same purification and shrinkage are effected.

United States Patent #785,535, Arsem, March 21, 1905, describes a furnace quite suitable as a means for firing the tube in a vacuum. Inasmuch as the changes in the tube are due solely to vaporization of the binder and sintering or fritting of the refractory residue, and are not due to reduction or other changes produced by the surrounding atmosphere, a very good vacuum may be maintained in the furnace during the heat treatment. This insures a final product free from oxid, nitrid, etc.

The firing operation in the vacuum can be effected in a few hours. The maximum temperature of treatment should be about 1500 to 1600 degrees C. The tube comes out strong and well sintered. It is comparable to tubes produced in the open tube furnace as hereinafter described at length. Inasmuch as the graphite heater of the Arsem furnace is liable to vaporize or disintegrate somewhat at a temperature of 1600 to 1800 degrees C., I consider that the tube, unless specially protected, cannot advantageously be heated to the highest temperature attainable in that furnace.

Apparatus suitable for carrying out the firing operation at atmospheric pressure may be of the general type illustrated in the drawing accompanying this application, with the exception that the resistance tube, which may be of tungsten, carbon or graphite, should have a diameter sufficient to take in an unfinished tungsten tube. When this method of manufacture is used, a liberal supply of hydrogen is advantageous to prevent oxidation of the tungsten during treatment.

I find that complete shrinkage and sintering of the tube can be produced at a temperature of 1800 to 2000 degrees C., but I also find that substantially complete shrinkage may be attained at temperatures as low as 1500 degrees. In case the firing is carried out in a carbon tube furnace, I prefer to use the lowest possible temperature, or even to leave the shrinkage somewhat incomplete, rather than endanger the tungsten by the liberation of carbon vapors from the graphite tube. If the tungsten tube happened to be only partly shrunk when mounted in the furnace, it can be given a preliminary heat treatment and thus finished while in position in its own furnace. Such tungsten tubes, when completely shrunk and sintered, are permanent in their electrical conductivity and are strong and of uniform size. They can be heated to an enormously high temperature and are serviceable for the production of metal lamp filaments, for the treatment of refractory compounds, and for researches of various kinds requiring high temperatures and a carbon-free atmosphere.

As a modification of the furnace just described, I will hereinafter describe a resistance tube consisting of refractory metal such as tungsten mixed with an inert material capable of giving the tube a high resistance and otherwise improving it as a furnace member.

As an inert material I prefer to use a refractory oxid such as thoria. This may be incorporated with the tungsten when the latter is introduced into the ductile alloy of cadmium, mercury and bismuth. It remains with the tungsten throughout the successive treatments of baking and firing. The proportion of thorium oxid may be varied between wide limits, but when used in the following proportions: 20 per cent. thoria, 20 per cent. tungsten, 60 per cent. cadmium-mercury-bismuth alloy, the resulting product is altogether satisfactory.

The thorium oxid serves in several ways to increase the efficiency of the tube as a furnace heater. It gives the tube greater specific resistance than that of pure tungsten, thus permitting the use of higher voltage and consequently simplifying somewhat the electrical problems of furnace operation. In case a certain percentage of thoria is not

exceeded, the refractory oxid also serves to stiffen the tube, giving it greater power to resist distortion when heated to the temperatures at which tungsten becomes somewhat

5 soft and plastic. In addition to these functions, it furnishes ready means for so changing the refractory tube that no tungsten is exposed to the oxidizing action of air. To attain this result it is only necessary to heat
10 the tube in air and thereby oxidize the tungsten lying near the surface, subsequently vaporizing the oxid so produced. This leaves a porous shell of thoria completely enveloping the homogeneous inner portion
15 of mixed tungsten and thoria. Such a tube is relatively inert and offers the further advantage that at high temperatures it does not tend to stick or weld to "boats" or other objects which may be placed in the tube
20 for treatment.

What I claim as new and desire to secure by Letters Patent of the United States, is,—

1. In an electric furnace, the combination of a refractory metal heater tube, a refractory packing inclosing said tube, and means
25 for slowly passing an inert gas through said packing to protect said tube from oxidation.

2. In an electric furnace, the combination of a tubular heater consisting essentially of
30 oxidizable metal, a carbon-free packing surrounding said heater, and means for slowly passing an inert gas through said packing to protect said tube from oxidation.

3. In an electric furnace, the combination
35 of a resistance tube consisting essentially of tungsten, means for protecting the outside of said tube from carbon, and means for circulating an inert gas about said tube to protect it from oxidation.

40 4. In an electric furnace, a resistance tube

consisting of tungsten with an admixture of refractory oxid, and a refractory packing surrounding said tube and protecting it from carbon vapors.

5. In an electric furnace, the combination
45 of a heater tube consisting essentially of tungsten with an admixture of thoria, said tube having a surface coating consisting principally of thoria, means for protecting said tube from carbon vapor, and means for
50 circulating an inert gas about said tube.

6. In an electric furnace, the combination of a tubular heater, a refractory packing surrounding said heater, a substantially gas-tight envelop inclosing said heater and pack-
55 ings, and means for slowly passing an inert gas through said packing to protect said heater from the deteriorating effect of air.

7. In an electric furnace, the combination of a tubular heater consisting essentially of
60 tungsten, a refractory oxid packing for said heater, a porous packing inclosing said refractory oxid, a gas-tight envelop inclosing said heater and packings, means for circulating an inert gas within said envelop, and
65 means for cooling the ends of said tubular heater.

8. In an electric furnace, the combination of a resistance heater consisting essentially of tungsten and having a chamber opening
70 into the air, means for supplying current to said heater, and means for protecting said heater from oxidation.

In witness whereof, I have hereunto set my hand this 30th day of November, 1907.

WILLIAM D. COOLIDGE.

Witnesses:

BENJAMIN B. HULL,

HELEN ORFORD.