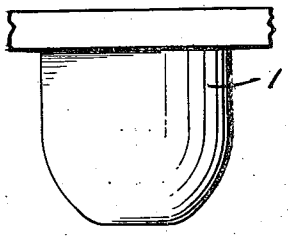
June 26, 1928. 1,675,119

J. W. MARDEN

MANUFACTURE OF ARTICLES OF REFRACTORY METAL OR METALLIC MIXTURES

Filed April 10, 1922

FIG. 1.



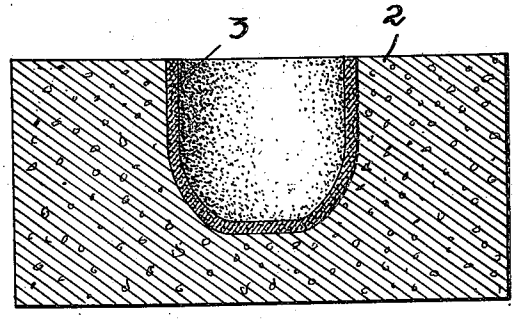


FIG. 2.

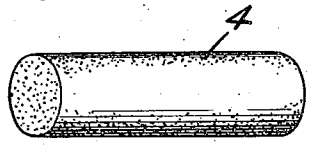


FIG. 3.

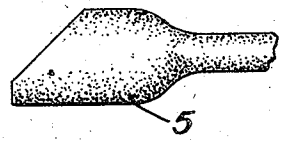


FIG. 4.

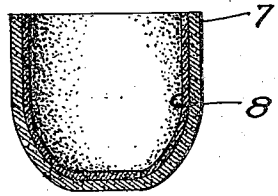
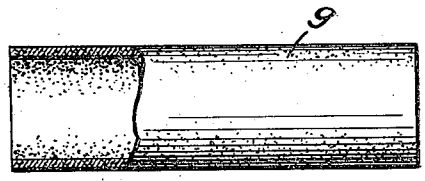


FIG. 5.



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UNITED STATES PATENT OFFICE.

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MANUFACTURE OF ARTICLES OF REFRACTORY METAL OR METALLIC MIXTURES.

Application filed April 10, 1922. Serial No. 551,042.

This invention relates to the manufacture of articles of refractory material and more particularly to the production of crucibles, tubes, X-ray targets and the like from highly refractory metal.

An object of my invention is the provision of a method for forming articles directly from powdered molybdenum, tungsten or the like by casting.

Another object of my invention is the formation of crucibles or the like of molybdenum and other refractory metals provided with a lining of thoria or other inert earth.

A further object of my invention is the production of a composition of matter and articles made therefrom comprising a refractory metal with which is mixed thoria to increase the resistance thereof and make it more refractory.

Other objects will be apparent as the description proceeds.

It is very difficult, by the usual method, to form refractory metals into various shapes, such as those suitable for X-ray targets, crucibles and the like. That is, to form an X-ray target, for instance, by the usual method of working tungsten, a billet square in cross section would be formed and the same worked at a high temperature by means of a swaging machine until approximately the size of the target is secured. Then, because of the impossibility of machining tungsten, the target would have to be ground to shape, thereby entailing a large amount of labor and expense. In practicing many metallurgical processes, it is desirable to have, but difficult to make, crucibles and the like composed of refractory metals, such as molybdenum, with an inert highly refractory lining such as thoria. In the formation of tube furnaces for heating billets of tungsten preparatory to swaging, the same are heated by passing current through a wire wound around a tube of alundum. Difficulty has been experienced with furnaces of this type because of the interaction between the tungsten and the alundum.

This invention avoids the difficulties before enumerated by forming X-ray targets, crucibles, furnace tubes and other articles for high temperature work directly in final form from the powdered refractory metal by casting.

In practicing this process, the refractory metal powder is mixed with water, a small quantity of another material, preferably a double fluoride salt to serve as a binder, and a small quantity of a suitable acid. Enough water is used in making this mixture to give the proper consistency for casting. This suspension, which is termed the "slip," is poured into a plaster of Paris mold to make the required shape. The mold is kept full for a short space of time, depending upon its dryness, the proportion of water in the slip and the thickness of the article to be formed. The mold takes water from the outer portions of the slip, thereby forming a deposit of the metal powder on the inside of the mold of a thickness which may be varied according to the length of time it is allowed to deposit. If a hollow article is being formed, the excess slip is then poured out and the article, after a few minutes, is removed in the ordinary way. The article is then thoroughly dried and later ignited in hydrogen or other non-oxidizing atmosphere where it loses its non-metallic additions, and the metal powder sinters and becomes hard, the degree of sintering depending upon the temperature and the duration of heating.

By this method, a conducting crucible or the like may be formed of a metal, such as molybdenum, tungsten or the like. On the inside of a crucible of the character designated, a coating of thoria or other infusible inert substance may be cast in a similar manner and the two shells sintered together. Such crucibles should be of value for high-frequency induction furnace work.

Solid bars can be cast in this manner, as well as hollow-ware. Although there seems to be no advantage at present, it might be pointed out that solid cylinders of tungsten or molybdenum might be cast for sintering and treating by the usual method for forming filament wire instead of refractory metal billets, square in section, being formed by the usual pressing method.

Any sort or shape of vessel may be made as indicated. A tungsten tube, for example, may thus be made and substituted for the usual carbon tube in an electric furnace, thus being able to attain high temperatures without the presence of hydro-carbon vapors.

Referring to the drawing,

Fig. 1 shows the shaping and receiving

molds for casting a metallic crucible; which is shown in section in the receiving mold;

Fig. 2 shows a cylindrical billet formed in a similar manner;

Fig. 3 shows an X-ray target formed by casting according to this method;

Fig. 4 shows a refractory metal crucible lined with thoria or other refractory inert substance; and

Fig. 5 illustrates a hollow cylinder adapted for use in a tube furnace.

In casting a crucible by this method, the shaping part 1, (Fig. 1) of a plaster of Paris mold is formed as follows: A solid block of plaster of Paris is turned at one end on a lathe to obtain the shape of the exterior of the article to be cast. A soft soap solution may then be applied to the shaped block and allowed to dry. The block is then placed on a piece of board and surrounded with sheet metal or cardboard. A mixture of plaster of Paris and water, preferably four parts of plaster of Paris and three parts of water, is poured into the cup thus formed and allowed to dry, thus forming the cup shaped part 2 of the mold. When hard, the two pieces of plaster of Paris are separated. The part 2 having the depression is retained and serves as a receiving mold in which is cast a metallic crucible 3. Into the depression is poured the metallic slip and the crucible formed as hereinafter more fully described.

When it is desired to form a billet for swaging and drawing, this billet may be formed as a solid cylinder 4, (Fig. 2), instead of the square section now used and may be cast from tungsten powder, thus eliminating the necessity of using a hydraulic press.

In the making of various electrical devices, the shaping of parts made of refractory materials, for example, the target 5 (Fig. 3) of an X-ray tube is very much simplified by casting in the finished form by the method herein described.

The process of casting shapes, such as shown in Figs. 1, 2 and 3 in plaster of Paris molds made up in a manner similar to that previously described, is as follows:

The process will be described for making a molybdenum crucible but it is to be understood that crucibles or the like of tantalum, tungsten metals of the tungsten class and other refractory metals or mixtures thereof are made in a similar manner. Molybdenum powder is mixed with a suitable amount of water and a small amount, for example, 10% of cryolite (Na_3AlF_6), together with a little acid such as phosphoric acid, to make a good cream or slip. It is then poured into a plaster of Paris mold formed as described with reference to Fig. 1, that is, the slip is poured into the part 2 of the mold, allowed to deposit to a suitable thickness therein and the excess poured out. The crucible thus formed will dry enough to set and is soon readily removed from the mold, as it will shrink away from the same due to loss of moisture, and can be separated therefrom by gently tapping the mold.

The crucible 3 formed as above described is then thoroughly dried and later ignited in hydrogen where it loses the cryolite, the acid and the remaining moisture and the metal shrinks and sinters together, becoming quite hard. The degree of sintering depends on the temperature and the duration of the heating. A crucible formed by this method may afterwards be turned or smoothed on a lathe or similar machine when such operation is needed. It may also be hammered or subjected to other treatment to eliminate porosity and further strengthen it.

A conducting crucible of a refractory metal formed in this way may be provided with a thoria crucible cast within it, thus forming a compound crucible 6 (Fig. 4) comprising an outer shell of molybdenum 7, for example, and an inner shell 8 of thoria. The inner shell is preferably cast in place before the outer shell is sintered. The two shells may then be sintered together by high temperature in a reducing or inert atmosphere. In actually making crucibles in this way, the thoria shrinks away from the molybdenum but very slightly, so that there is formed a metal conducting crucible lined with a thin crucible of the chemically inert thoria. Such a crucible should be very valuable for induction furnace work.

A tube 9 (Fig. 5), suitable for use as the heating element in a tube furnace such as that used for heating tungsten billets preparatory to swaging the same, may be advantageously cast in a plaster of Paris mold in a way similar to that in which crucibles are cast. It may have a composition of tungsten powder with the addition of thoria or other refractory earth varying from a small amount to 50% or more thereof to increase the electrical resistance of the tungsten so that the tube can serve the double function of a heating tube and a secondary for inductive heating.

By the term "cold-cast" used in the claims, applicant means cast without fusion, for example cast from a slip or with a vehicle to hold the powdered material in suspension and allow the mixture to flow like a liquid or semi-solid into a mold or form.

It is to be understood that many changes may be effected in the herein described means of carrying out my invention and that the same is limited only by the scope of the appended claims.

What is claimed is:

1. A cold-cast refractory metal crucible with a lining of inert refractory material cold-cast therein.

2. A cold-cast crucible composed of metal of the tungsten class with a thoria lining cold-cast therein.

3. A cold-cast refractory metal crucible lined with thoria.

4. A cold-cast tungsten crucible lined with an inert refractory material.

5. A cold-cast and sintered refractory metal crucible with a lining of inert refractory material cold-cast therein.

6. A cold-cast and sintered crucible composed of metal of the tungsten class with a thoria lining cold-cast therein.

7. A cold-cast and sintered refractory metal crucible lined with thoria.

8. A cold-cast and sintered tungsten crucible lined with an inert refractory material.

In testimony whereof, I have hereunto subscribed my name this 7th day of April 1922.

JOHN WESLEY MARDEN.