

UNITED STATES PATENT OFFICE.

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REFRACTORY ARTICLE.

No Drawing.

Application filed April 24, 1922. Serial No. 556,334.

This invention relates to a method of producing refractory ware and to the articles resulting from practicing such method.

An object of the invention is to provide a method of producing a reinforced refractory body.

Another object of the invention is to produce a refractory body having comparatively thin walls but possessing a relatively high degree of strength.

A still further object of the invention is to produce a hollow body, such as a crucible, having the necessary tenacity to withstand handling incident to its use.

Other objects will be apparent upon a reading of the following description.

It is well known that certain refractory bodies, especially hollow bodies composed of homogeneous metallic powder such as tungsten or the like, are of a fragile character and must be handled accordingly to prevent breakage.

The common method of making these bodies is first to mold a metallic powder into a hollow member and then heat the molded body to a temperature at which the powder is sintered to produce a coherent mass. Refractory bodies of this character, commonly used as crucibles, lack the necessary amount of toughness or tensile strength to avoid rupture which often occurs through comparatively light impact with other bodies.

By the present method, a refractory body may be produced which is sufficiently tenacious to avoid rupture through handling when employed as a crucible or for any purpose for which it may be intended.

To produce a refractory body in accordance with my invention, I employ a tangled or entwined mass of wire which may be of tungsten, molybdenum or other metal. It is possible, with my method, to employ any discarded or scrap wire, such as the residue commonly resulting in manufacture of certain electrical devices. A quantity of this wire, which is often termed metallic wool, may be given any desired shape and positioned in a mold. Metallic powder, such as tungsten, is deposited in the mold until the interstices within and on the sides of the shaped wool are substantially filled. A suitable plunger is inserted into the mold and pressure applied until the intermingled or

interlaced wire and metallic powder are compressed to form a compact mass.

Refractory bodies made according to my process may, for certain purposes, be found strong enough, after pressing, by reason of the embedded wire, to be handled without the necessity of sintering, inasmuch as the entwined wire gives considerable strength to the body. A body may be produced without sintering and may be heated during a pressing operation to a degree sufficient to set the wire which otherwise might tend to oppose being compacted. A more intimate union between the wire and the powdered metal may, however, be obtained by sintering the mass after the pressing operation has been performed.

The present method is particularly adapted for making a crucible which may have its walls composed of a layer or layers of metallic wool, such as tungsten and a metallic powder.

A crucible may be produced by depositing one or more layers of tungsten or other suitable wool upon the inner surface of a mold and a given quantity of a suitable metallic powder, such as tungsten, may be deposited within the mold. A die or male member may then be inserted in the mold to press the powdered substance into intimate contact with the wool or intermingled wire. The die is then caused to exert sufficient pressure upon the powder and wool to compress them into an adherent mass. The hollow body thus produced will have the wall thereof reinforced by the wool and may be removed from the mold and placed in a sintering furnace, whereupon it attains greater strength by reason of the resultant more intimate association between the powdered substance and the wool through incipient fusion.

Heretofore, when crucibles were produced by compressing a powdered metal, great difficulty was encountered in transferring the partially finished crucible from the mold to the sintering furnace, because the crucible was extremely fragile.

A crucible made according to my process may be more safely and, consequently, more conveniently removed from a mold, inasmuch as the entwining reinforcing strands of wire practically constitute an internal fabric within the wall of the crucible and hold the metallic powder in place.

It will be appreciated that, by reason of this reinforcing net work of wrought material within the wall of a crucible, the crucible will, after sintering, possess, by virtue of the inherent tenacity of the metallic wire or a wool, sufficient strength to resist, to a great extent, the impact due to careless handling.

A wall of a refractory substance having substantially the equivalent of a fabricated interior and consequent relatively high tensile strength is of advantage for other purposes than in the making of a crucible. For instance, an electrode of spherical or hemispherical shape may be desired. I have found that an electrode having a thin wall is desirable to meet certain conditions but was very difficult to obtain before recourse was had to the present method of embodying a net work of wire within the wall to give it strength to support its own weight.

In practicing my method, I have found that, by placing a quantity of tungsten wool in a mold together with the tungsten powder and applying pressure during the application of sufficient heat to set wire, good results are obtained. A body is thus formed which may then be sintered in any desirable manner. I have also found that a convenient manner of temporarily associating the wire and tungsten or other powder previously to pressing, is to mix any suitable volatile binder, such as cryolite of camphor, with the powder to form a pasty or plastic substance which will then flow into the crevices or interstices of the mass or wire under pressure.

Heat being applied during the pressing operation, the cryolite or other binder is volatilized, leaving the metallic elements in a compressed state. The molds for compressing the mass during a heat treatment must necessarily be of a metal which will withstand the heat without losing its strength or hardness at a temperature at which tungsten wire will set.

My process may also be practiced for the production of slugs used in the manufacture of tungsten wire, in which case, a quantity of tungsten powder and tungsten wool or scrap filament wire is united by the application of pressure in a suitable mold. The resultant slug has considerable strength and may be more readily handled for the sintering, swaging and drawing operation than slugs composed entirely of tungsten powder. In the manufacture of these slugs, it may be desirable to obtain a slug for the production of a filament wire containing a given amount of thorium to increase the resistance of the wire; in which case, a quantity of thorium powder may be added to the tungsten powder during the production of the slug.

It will also be evident that, by the present method, a refractory body may be shaped to

any desirable form. This is an advantage, inasmuch as bodies of various configuration are required for electrodes. For instance, electrodes for use in arc lamps or in X-ray tubes may be produced.

In the manufacture of targets for X-ray tubes, I may combine a suitable powdered material, other than tungsten, with a metallic mass of wire or wool; for instance, it may be desirable to produce a target having a relatively high heat-conductive material incorporated therewith.

It has been found difficult heretofore, to produce an intimate union between certain substances such, for instance, as copper, having high heat conductivity and refractory bodies such as tungsten. The reason being that a layer of oxide forms upon the tungsten and this interposed layer of tungstic oxide prevents the copper from adhering in a degree sufficient to provide a practical target.

By the use of tungsten wool, a target may be made by compressing a quantity of copper powder and a layer or layers of tungsten wool in a mold until the powder has been forced around the wire in the manner above explained when tungsten powder is employed. In this case, however, it is preferable to provide a layer of tungsten powder, a layer of tungsten wool and a layer of copper powder; the tungsten wool being interposed between the layers of powder. Pressure may then be applied to force the powders disposed upon each side of the wire or wool into the pockets or interstices between the wire.

The compressed body may then be placed in an inert environment and an electrical current of sufficient strength may be passed therethrough. The tungsten portion of the body will offer high resistance and become heated to a point at which it will be sintered, whereas, the copper portion of the body will rapidly conduct heat away at a sufficient rate to avoid its being heated excessively. If desirable, the layer of copper may be of sufficiently greater mass than the tungsten wool, and the conductivity of the copper will be relatively greater than that of the tungsten, with a corresponding increase in the dissipation of heat. It may be preferable to provide a cooling element, such as a water jacket, in association with the copper portion of the body and thereby carry away the heat to avoid the vaporization of the copper to any great extent during the time the tungsten is being sintered.

Although the temperature of the copper may be raised to a point at which the copper melts, a mold may be provided to confine the molten copper in contact with the tungsten wire so that, after the tungsten has been sintered and the entire body cooled, the copper will be bound fast by the integral portions

thereof which lie between the strands of tungsten wire. Thus, the copper and tungsten are consolidated and produce a composite body for use as a target in X-ray tubes or for other
5 uses. It is obvious that, in place of the tungsten powder, a layer of other refractory powder may be used, or a layer of tungsten or nickel wire and a layer of copper or other material of relatively high heat conductivity
10 may be found desirable. Thus, a composite body composed of elements of different atomic weights may be produced.

By means of my process, I may produce a composite body of a given coefficient of expansion by utilizing a metallic wool of a
15 given coefficient of expansion and uniting it by means of my method with a metal of a different coefficient of expansion to obtain a slug or other body. A slug of the above character may readily be swaged and drawn with
20 the result that a wire will be obtained of a predetermined coefficient of expansion. The production of a body which will have a given coefficient of expansion is particularly desirable in the electrical art as, for example,

where it is desired to seal metal in glass to form a hermetic seal.

I have described my invention as principally employing tungsten powder and tungsten wire, but it is obvious that any other
30 refractory wire may be intermingled with other metals or their oxides to produce a concrete body and it is to be further understood that, although I have given certain specific
35 methods embodying my invention and examples of articles produced thereby, it is to be understood that many variations may be made therein without departing from the spirit and scope of the invention.

What is claimed is:

1. A crucible composed of tungsten powder sintered to tungsten wool. 40

2. A vessel composed of a metallic refractory powder sintered to a mass of metallic wool. 45

3. A vessel composed of tungsten powder sintered to metal wool.

In testimony whereof, I have hereunto subscribed my name this 22nd day of April, 1922.

CHARLES FREDERICK LORENZ.