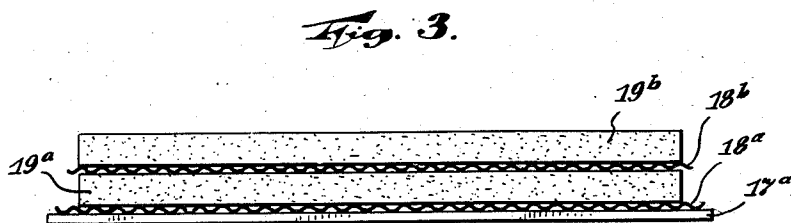
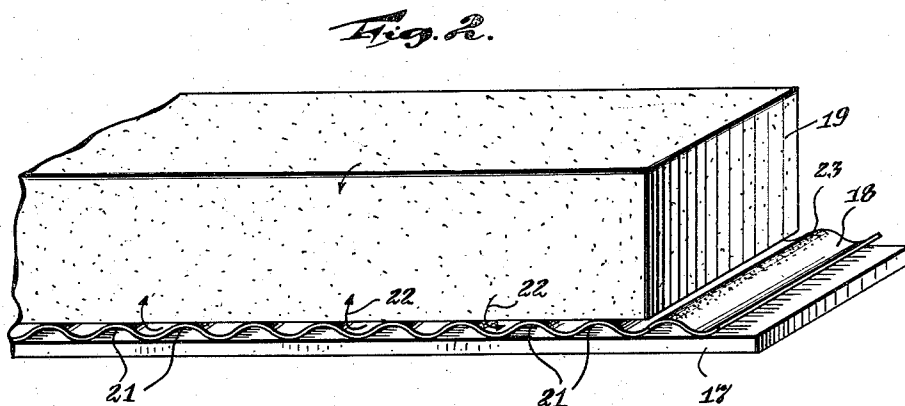
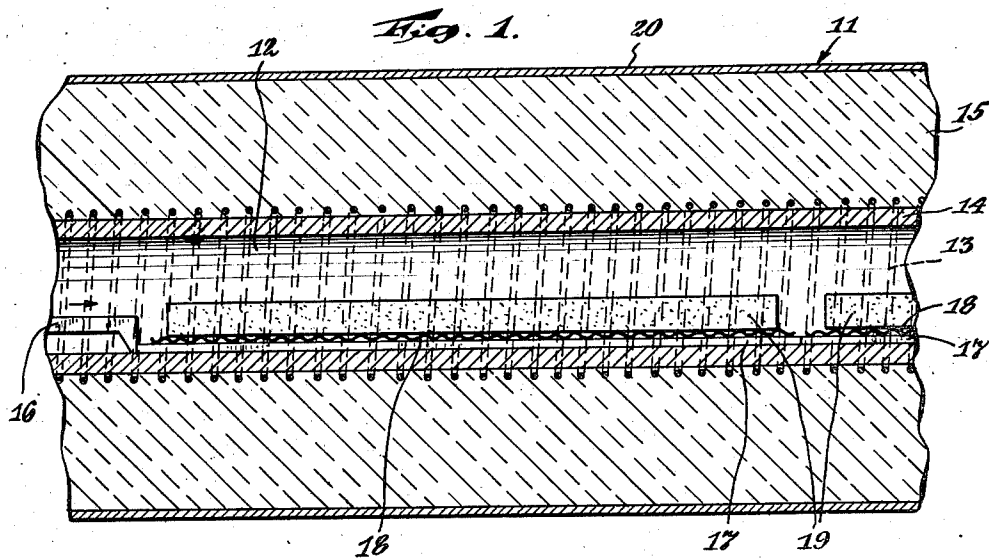


Dec. 2, 1947.

W. J. NEWMAN
METHOD AND APPARATUS FOR CONSOLIDATING REFRACTORY
METAL POWDER TO DENSE COHERENT FORM
Filed March 13, 1944

2,431,691



INVENTOR
W. J. NEWMAN.
BY *W. J. Newman*
ATTORNEY.

UNITED STATES PATENT OFFICE

2,431,691

METHOD AND APPARATUS FOR CONSOLIDATING REFRACTORY METAL POWDER TO DENSE COHERENT FORM

William J. Newman, East Orange, N. J., assignor to Westinghouse Electric Corporation, East Pittsburgh, Pa., a corporation of Pennsylvania

Application March 13, 1944, Serial No. 526,153

14 Claims. (Cl. 75-22)

1

This invention relates to the consolidation of metal powder, and more particularly, to an improvement in the method of and apparatus for consolidating metal in a convenient and economical manner.

The principal object of my invention, generally considered, is an improvement in powder metallurgy, and more particularly in connection with the manufacture of refractory metals such as molybdenum and tungsten in coherent form.

Another object of my invention is to prevent sticking to the supporting means during the sintering operation, of refractory metal powder after having been pressed into shape.

A further object of my invention is to provide a support for pressed ingots of refractory metal powder, such as molybdenum and tungsten, during the sintering operation, whereby sticking and the formation of cracks during such sintering treatment is obviated.

A still further object of my invention is the provision of a corrugated support of refractory metal for use during sintering to hold pressed metal powder of corresponding character for uniform treatment by the ambient atmosphere in the sintering furnace and to avoid sticking to the supporting slab or boat.

Other objects and advantages of the invention will become apparent as the description proceeds.

Referring to the scale drawing:

Fig. 1 is an axial sectional view of a furnace containing pressed ingots or slugs of refractory metal powder, during the process of sintering in accordance with my invention.

Fig. 2 is a fragmentary perspective approximately full size view of an ingot, a supporting corrugated metal plate, and an underlying slab or boat, without the furnace shown in Fig. 1.

Fig. 3 is a view corresponding to Fig. 1, but showing only the supporting slab or boat, the corrugated underlying supporting strip, and the employment of such a strip on top of the lower ingot in order to allow for double or multiple decking of such ingots.

In the final heat treatment of refractory metal bars, and particularly molybdenum in a moist hydrogen-bearing reducing atmosphere, or one preferably saturated at between about 20° C. and 40° C. with water vapor, at approximately 1600° C., or between about 50% and 70% of its temperature of melting as measured in degrees C., described and claimed as a "reversible reaction" treatment in the Hall et al. application Serial No. 486,812, filed May 13, 1943, and the Hall et al. application, Serial No. 511,392, filed November 23,

2

1943, both cases now replaced by the Hall et al. continuation-in-part application, Ser. No. 579,080, filed February 21, 1945, it is found desirable to use relatively long ingots, for example those of the order of 20" to 30" in length, or such lengths as can be conveniently pressed in molds available. If, then, these ingots are large in cross section, the weight per running inch is high and where slabs or boats of refractory metal, such as tungsten or molybdenum, are used to carry these through a horizontal furnace, the pressed ingot, especially on account of being rough, has a tendency to weld onto the slab or boat, such welding resulting from the same reaction and at the same temperature that brings about the sintering of the bar.

As this occurs before the shrinkage of the bar is ended, such shrinkage being of the order of 15% to 17%, there is a tendency of the bar to pull apart, developing transverse cracks at intervals.

I propose to avoid the foregoing difficulty by using directly under the bar of molybdenum or tungsten a preferably transversely corrugated, desirably resilient strip of thin molybdenum, tungsten, or other similar refractory metal, thereby not only supporting the latter at a plurality of closely-spaced positions along its length, and because of the resiliency of the strip avoiding concentration or substantial variation of pressure at the support positions, but at the same time holding the ingot above the floor of the furnace and allowing for free circulation of the ambient treating atmosphere, such as wet hydrogen, around the ingot. I thereby provide for the manufacture of metal of uniform strength and density, avoid the local welding or fusion of the ingot to the support, and at the same time make it possible to double or multiple deck the ingots.

In accordance with the Hall et al. applications, previously referred to, refractory metal such as molybdenum and tungsten are desirably manufactured in dense coherent form by pressing in molds and then either sintering to final density or firing at a temperature not high enough to cause actual sintering to the finished condition then forming to shape, as by machining, and afterwards sintering to final density, the sintering temperature being about 1600° C., if molybdenum is the metal. It is the final sintering of such material that I am particularly concerned with, and especially when said material is molybdenum, although the same principle applies to other similar materials such as tungsten.

Referring to the drawing in detail, and first considering the embodiment of my invention il-

illustrated in Figs. 1 and 2, there is shown a furnace 11 comprising a middle or intermediate hot zone 12 provided by any suitable heating means, such as resistance wire 13 around a heating tube 14 and the whole enclosed in suitable insulating means such as alumina or magnesia 15 held in an outer casing 20. At the front end (not shown) there is a relatively cool vestibule portion. At the rear or exit end there is a relatively cold tunnel (not shown) the length of which is sufficient for effecting the desired cooling of the treated material and may be shortened by surrounding, at least a part thereof, with a water cooling jacket (not shown).

The furnace is provided with a pusher element 16 as well as means for introducing and exhausting wet hydrogen. The pusher element 16 acts on a slab or boat 17 of suitable refractory material, such as molybdenum or tungsten, upon which rests a thin transversely or slightly diagonally corrugated preferably resilient strip 18 of molybdenum, if the ingots being treated in said furnace are molybdenum as is assumed in the embodiment illustrated. A similar strip formed of tungsten may be used for supporting molybdenum or tungsten ingots.

On the corrugated strip 18, the upper portions of the corrugations of which are desirably coated with a film of refractory inert finely-divided or powdered material 23, such as alumina, thoria, or zirconia, is supported a relatively long ingot 19 of molybdenum, or other selected refractory metal, pressed from the powder in accordance with the said last-mentioned Hall et al. application. The powder is to avoid the possibility of sticking during the sintering operation. It may be sprinkled on dry or made into an aqueous, for example, suspension and painted on the top surface of the top portions of the corrugations. The upwardly opening grooves between the corrugations should, however, not be appreciably restricted by said powder. It will be understood that although I have shown an elongated ingot rectangular in cross section, my invention is not limited to treating such, as ingots elongated or otherwise, and of various shapes may be supported in a similar manner, and the dangers, previously referred to, especially noticed in treating long heavy ingots, are avoided by using my invention.

It will, however, be understood that the corrugations in the supporting strip are desirably transverse to the length thereof, so that the supporting lines are longitudinally spaced along the length of the supported ingot or pressed shape rather than transversely spaced. The corrugated strip 18 is thin enough so that the same distributes the weight of the ingot, as by necessary flexure or partial flattening, so that substantially equal portions of the weight are supported along the crest of each corrugation 21, rather than having concentrated points of pressure.

It will be seen that by virtue of the transverse corrugations, free circulation of the wet hydrogen or other treating atmosphere under the ingot, as indicated by the arrows 22, is permitted, thereby allowing for uniform sintering action resulting in a uniform contraction at all parts of the ingot or pressed shape, at the same time thereby avoiding not only the transverse cracks previously encountered, but also the possibility of warping due to quicker treating along one face than along another, as it will be understood that shrinkage occurs during the sintering operation.

The supporting corrugated strip 18 may also

be considered as an anti-friction device under the ingot, allowing free sliding of the same therealong during the shrinking operation, thereby not only avoiding any severe pressure and consequent welding or sticking but allowing for slight changes in the lines of support as the ingot shrinks, and avoiding any possible tendency to stick. The upper surfaces of the supporting members 17 desirably have applied thereto a film of refractory inert powder, like the tops of the corrugations in the strip 18, to avoid sticking tendencies during sintering treatments.

Referring now to the embodiment of my invention illustrated in Fig. 3, there is shown a supporting slab 17^a, which may correspond with the slab 17 of the preceding embodiment shown, said slab being used in a furnace as in said preceding embodiment. On the slab 17^a is a corrugated strip of molybdenum or the like 18^a, corresponding with the strip 18 of the preceding embodiment and likewise supporting a refractory metal ingot 19^a, assumed to be molybdenum and formed of pressed powder like the ingot 19. On the ingot 19^a is a corrugated strip 19^b corresponding with the strip 18^a and in turn supporting a similar ingot 19^b. The tops of the corrugations in the strips 18^a and 19^b, as well as the surfaces supporting said strips, desirably have applied thereto films of refractory inert powder, like the tops of the corrugations in the strip 18.

It will, therefore, be seen that I have made possible what was heretofore thought impossible, and that is, the treatment of ingots superimposed or multiple decked with respect to one another. Although I have only shown two ingots so superimposed, it will be understood that I am not limited and if the ingots are sufficiently small or the furnace sufficiently large I may superimpose several, one upon another, but separated therefrom by transversely or diagonally corrugated strips of similar refractory metal.

A satisfactory embodiment of the apparatus used in sintering slugs of molybdenum 24" long, 1½" width and 1" thick, is a plate of molybdenum .015 to .02" thick and corresponding in length and width with that of the base of the slug to be treated, in this case about 24" long and 1½" wide, and with corrugations ⅛" deep or somewhat more, spaced a distance of ⅜" or somewhat more between crests. Of course it will be understood that substantial variations may be made in the thickness of the supporting plate, the depth of and the distances between corrugations, and that the heavier the slug the thicker the corrugated plate.

From the foregoing it will be seen that I have provided an improvement in connection with the manufacture of refractory metal, and particularly molybdenum, whereby the tendency to stick or weld to the support during sintering is overcome, while at the same time provision is made for a more uniform treatment of the pressed slug or shape, while allowing for free circulation under as well as above and around the pressed ingot during the final sintering operation.

Although preferred embodiments of my invention have been disclosed, it will be understood that modifications may be made within the spirit and scope of the appended claims.

I claim:

1. The method of consolidating molybdenum powder to dense coherent form, comprising pressing said powder to a shape about 24" long, 1½" wide, and 1" thick, placing said pressed shape on a transversely corrugated resilient plate

of molybdenum, and heating for a relatively long period of time said pressed shape in a hydrogen-bearing reducing atmosphere, saturated between about 20° C. and 40° C. with water vapor, to a temperature, not higher than about 70% of that of fusion as measured in degrees C., at which a reversible reaction between such materials occurs.

2. The method of consolidating molybdenum powder to dense coherent form, comprising pressing said powder to a shape not lighter than one about 12" long, $\frac{3}{4}$ " wide and $\frac{3}{4}$ " thick, placing said pressed shape on a transversely corrugated resilient plate of molybdenum, and heating in a hydrogen-bearing reducing atmosphere saturated at a temperature between about 20° C. and 40° C. with water vapor to a temperature of between about 50% and 70% of that of fusion as measured in degrees C., for a relatively long period of time.

3. The method of consolidating molybdenum powder to form dense coherent metal, comprising pressing said powder to an elongated shape heavy per longitudinal inch, placing the pressed shape on a transversely corrugated resilient plate of molybdenum, and heating in an atmosphere of hydrogen saturated at about 30° C. with water vapor to a temperature of about 1600° C. for about two hours.

4. The method of consolidating a metal powder selected from the group consisting of molybdenum and tungsten to form dense coherent metal, comprising pressing the selected powder to an elongated body of heavy weight per running inch, placing the body on a transversely corrugated resilient plate formed of the same metal to support it at a plurality of spaced positions from end to end and allow for circulation of the treating atmosphere thereunder, and heating said pressed body in a hydrogen-bearing reducing atmosphere containing a large enough proportion of water vapor to provide for a reversible oxidation-reduction reaction, to a temperature not higher than about 70% of that of fusion as measured in degrees C. but sufficiently high to cause the reaction to occur rapidly, for a relatively long period of time until a strong coherent article is produced.

5. The method of consolidating a metal powder selected from the group consisting of molybdenum and tungsten to form dense coherent metal, comprising pressing the selected powder to an elongated shape heavy per longitudinal inch, placing the pressed shape on a transversely corrugated resilient plate of the same metal, and heating said pressed shape in a hydrogen-bearing reducing atmosphere saturated, at a temperature not higher than 40° C. but high enough to provide for a reversible oxidation-reduction reaction, with water vapor, to a temperature not higher than about 70% of that of fusion as measured in degrees C., for a relatively long period of time until a strong coherent article is produced.

6. The method of consolidating a metal powder selected from the group consisting of molybdenum and tungsten to form dense coherent metal, comprising pressing the selected powder to an elongated shape at least as large as 12" x $\frac{3}{4}$ " x $\frac{3}{4}$ ", placing the pressed shape on a transversely corrugated resilient plate of the same metal, and heating said pressed shape in a hydrogen-bearing reducing atmosphere containing water vapor, to a temperature between about 50% and 70% of that of fusion as measured

in degrees C., for a relatively long period of time.

7. The method of consolidating a metal powder selected from the group consisting of molybdenum and tungsten to form dense coherent metal, comprising pressing the selected powder to an elongated shape at least as large as 12" x $\frac{3}{4}$ " x $\frac{3}{4}$ ", placing the pressed shape on a transversely corrugated resilient plate of the same metal, and heating said pressed shape in a hydrogen-bearing reducing atmosphere saturated at a temperature between about 20° C. and 40° C. with water vapor, to a temperature between about 50% and 70% of that of fusion as measured in degrees C., for a relatively long period of time.

8. The method of consolidating molybdenum powder to dense coherent form, comprising pressing said powder to an elongated shape at least as heavy as one about 12" x $\frac{3}{4}$ " x $\frac{3}{4}$ ", placing said pressed shape on a transversely corrugated resilient plate of metal selected from the group consisting of molybdenum and tungsten, and heating said pressed shape in a hydrogen-bearing reducing atmosphere saturated at a temperature between about 20° C. and 40° C. with water vapor, to a temperature between about 50% and 70% of that of fusion as measured in degrees C., for a relatively long period of time.

9. The method of consolidating tungsten powder to dense coherent form, comprising pressing said powder to an elongated shape at least as heavy as one about 24" x 1 $\frac{1}{2}$ " x 1", placing said pressed shape on a transversely corrugated resilient plate of tungsten, and heating said pressed shape in a hydrogen-bearing reducing atmosphere saturated between about 20° C. and 40° C. with water vapor, for a relatively long period of time to a temperature not higher than about 70% of that of fusion as measured in degrees C., at which a reversible reactance between such materials takes place rapidly.

10. The method of consolidating tungsten powder to dense coherent form, comprising pressing said powder to a shape having a length of about 24", a width of about 1 $\frac{1}{2}$ ", and a thickness of about 1", placing said pressed shape on a transversely corrugated resilient plate of tungsten, and heating in a hydrogen-bearing reducing atmosphere saturated at a temperature between about 20° C. and 40° C. with water vapor to a temperature of between about 50% and 70% of that of fusion as measured in degrees C., for a relatively long period of time.

11. The method of consolidating tungsten powder to form dense coherent metal comprising pressing said powder to an elongated shape of heavy weight per running inch, placing the pressed shape on a transversely corrugated resilient plate of tungsten, and heating in an atmosphere of hydrogen saturated between about 20° C. and 40° C. with water vapor, to a temperature between about 1600° C. and 2000° C. for a relatively long period of time.

12. The method of consolidating a metal powder selected from the group consisting of molybdenum and tungsten to form dense coherent metal, comprising pressing the selected powder to consolidate it into an elongated ingot heavy per longitudinal inch, placing said ingot on a transversely corrugated resilient plate of the same metal, placing a similar resilient plate of metal on top of said ingot, forming another ingot in the same manner, placing said second ingot on top of the ingot-supported resilient plate of metal, and heating said ingots in a hydrogen-

7

bearing reducing atmosphere saturated at a temperature between about 20° C. and 40° C. with water vapor to a temperature between about 50% and 70% of that of fusion as measured in degrees C. for a relatively long period of time.

13. The method of consolidating a metal powder selected from the group consisting of molybdenum and tungsten to form dense coherent metal, comprising pressing the selected powder to an elongated shape heavy per longitudinal inch, applying a film of inert refractory powder to the upper surface of a transversely corrugated resilient plate of the same metal, placing the pressed shape on said powder covered plate, and heating said pressed shape in a hydrogen-bearing reducing atmosphere saturated at a temperature between about 20° C. and 40° C. with water vapor, to a temperature between about 50% and 70% of that of fusion as measured in degrees C. for a relatively long period of time.

14. In apparatus for consolidating to dense coherent form a refractory metal powder selected from the group consisting of molybdenum and tungsten, comprising a furnace with heating means, a tube of refractory material adapted to stand a desired high temperature obtained by activation of said heating means, insulating means around said tube and heating means,

8

whereby the tube may be raised in temperature sufficiently to heat enclosed selected metal powder to a sintering temperature, and means for introducing a hydrogen-bearing reducing atmosphere into said tube, the combination therewith of a transversely corrugated resilient plate of the metal selected for consolidation disposed in said tube, in order to support an elongated pressed shape, heavy per longitudinal inch, of the selected metal powder, at a plurality of uniformly-spaced positions along its length, to prevent sticking and crack formation, and allow for uniform sintering and treatment of said shape in the hydrogen-bearing atmosphere.

WILLIAM J. NEWMAN,

REFERENCES CITED

The following references are of record in the file of this patent:

UNITED STATES PATENTS

Number	Name	Date
534,207	Edison	Feb. 12, 1895
1,536,438	Koehnline	May 5, 1925
1,731,255	Marden	Oct. 15, 1922
1,768,719	Stewart	July 1, 1930
1,808,314	Nisbet	June 2, 1931
2,300,048	Koehring	Oct. 27, 1942