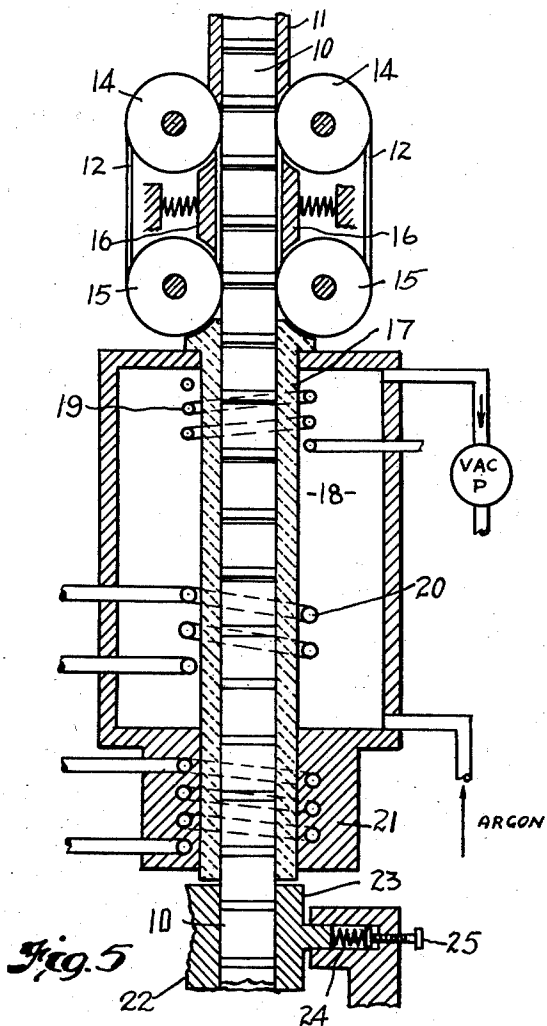
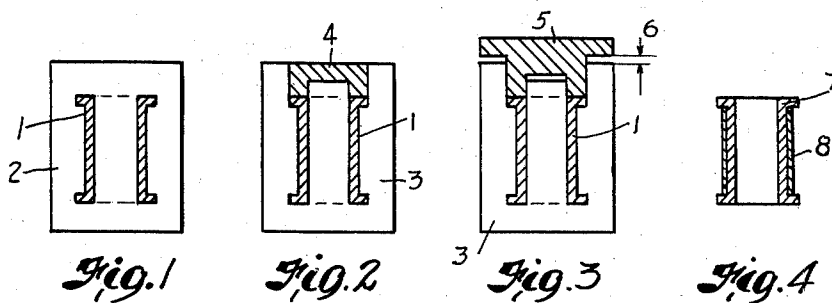


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METHOD OF AND APPARATUS FOR MAKING ARTICLES
FROM POWDERED METAL BRIQUETS
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METHOD OF AND APPARATUS FOR MAKING ARTICLES FROM POWDERED METAL BRIQUETS

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The present invention relates generally as indicated to a method of and apparatus for making articles from powdered metal briquets and more particularly to certain improvements in the methods and processes disclosed in my co-pending applications Serial Nos. 104,369, 161,344, and 202,707, all now abandoned. This invention also is a continuation-in-part of my co-pending application Serial No. 303,317, filed August 8, 1952, now Patent No. 2,803,046, and Ser. No. 252,938, filed Oct. 24, 1951, now Patent No. 2,787,817.

It is one object of this invention to provide a method and apparatus by which accurate articles of desired denseness (porous or non-porous) may be formed from powdered metal briquets.

It is another object of this invention to provide a method and apparatus by which and with which a powdered metal briquet may be partially or completely fused while contained within a non-distorting heat-resistant enclosure, which enclosure may, in some instances, be wholly or partly of a material which imparts desired physical or chemical properties to the briquet therein or which bonds to the briquet.

It is another object of this invention to provide a method and apparatus by which and with which a briquet of powdered material, contained in a mold, is pre-heated, preferably in vacuum, to render the briquet gas-free and is further heated under vacuum to sinter the briquet while it is mechanically compacted so as to densify the same and to make it accurately conform to the mold.

It is another object of this invention to provide a method and apparatus incorporating independent heat control at the briquet pre-heat, degassing and briquet sintering or fusing zones.

It is another object of this invention to provide a method and apparatus which involves a two-stage treatment of powdered metal in a mold viz. a first stage degassing and sintering operation and a second stage further degassing and fusing operation.

Other objects and advantages of my invention will appear as the description proceeds.

To the accomplishment of the foregoing and related ends, said invention then comprises the features herein-after fully described and particularly pointed out in the claims, the following description and the annexed drawings setting forth in detail certain illustrative embodiments of the invention, these being indicative, however, of but a few of the various ways in which the principles of the invention may be employed.

In said annexed drawings:

Fig. 1 is a cross-section view of a powdered metal briquet and surrounding enclosure;

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Fig. 2 is a cross-section view similar to Fig. 1 except that a false piston is included in the enclosure in contact with the powdered metal briquet;

Fig. 3 is a cross-section view showing the false piston of Fig. 2 replaced by a piston which is adapted to exert pressure on the powdered metal briquet when the latter is melted;

Fig. 4 is a cross-section view of a special form of powdered metal briquet with a surrounding solid metal band to which the powdered metal is to be bonded or alloyed; and

Fig. 5 is a cross-section view of one form of apparatus which may be employed for the practice of the present method.

Broadly stated, the present method involves the steps of providing a powdered metal briquet, enclosing the briquet in a body of heat-resistant material, pre-heating the enclosure and briquet therein in a vacuum chamber to expel and to evacuate substantially all of the gases therefrom, partially or completely fusing the briquet in vacuum progressively from the bottom to the top thereof, and applying mechanical pressure on the briquet while in fused condition and exposed to vacuum whereby to form an article of desired density within the enclosure. The enclosure and fused briquet are subsequently cooled to solidify the metal and then the enclosure may be removed or broken away from the finished article. Similarly, the present apparatus is such as to efficiently perform the foregoing method.

Referring now more particularly to the drawing and first to Fig. 1 thereof, the powdered metal briquet 1 is formed in well-known manner by the mechanical consolidation of the metal powder in suitable dies provided with cavities of the desired shape and size. The pressure required to form the briquet 1 will, of course, vary according to the particular metal which is used; and, except in the case of low-melting point metals, the pressed compact or briquet is mechanically weak and must be handled with care. Any of a variety of different metals and alloys may be employed for forming the powdered metal briquet 1. Examples of some of the metals and alloys from which powdered metal briquets may be formed are: RC 130 B or Ti Alloy 150A, whose compositions are as follows: Mn 4%, Al 4%, O 0.2%, N .1% max., and Ti balance; and Fe 1.3%, Cr 2.7%, O .25%, N .02% max., C .02% max., and Ti balance.

After the briquet 1 has been formed, it is enclosed in a porous body 2 of heat-resistant material, usually a ceramic material including carbon, mica, plaster of Paris, asbestos, stabilized zirconium oxide, and other heat-resistant and preferably, but not necessarily, non-metallic materials which can be heated to at least the melting point of the briquet without melting, burning, or otherwise rendered inoperative by the melting of the briquet therein. Enclosing of the briquet 1 within the body of a heat-resistant material 2 may be done as is disclosed in my co-pending applications Serial Nos. 161,344 and 202,707.

In the case that the enclosure 2 is in the form of a mold made of two or more pieces, the same may be pre-fired or fused so that said mold will have a very close fit in the guide portion of the apparatus which will be presently described.

In Figs. 2 and 3 the briquet 1 is enclosed within a

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composite enclosure which comprises a body 3 of heat-resistant material and a plug or piston 4 of any desired material such as metal, said piston being positioned adjacent one end of the briquet 1 so as to form therewith and with the body 3 a cylinder for a piston 5. It can be seen from Fig. 3 that when the briquet 1 is fused, pressure on the top of the piston 5 will cause the end of the piston to exert pressure on the top of the fused briquet. The degree of compacting of the melted briquet 1 is determined by the distance 6 which is additionally selected so that the article will be of the desired dimensions in the direction of pressure application thereon. Thus, where the briquet is of a tubular form as shown in Figs. 1 to 3, the axial dimension thereof will be somewhat greater than that of the finished article because of the reduction in bulk owing to the fusing thereof and mechanical consolidation in addition, when the piston 5 is employed as shown in Fig. 3. The diameters of the briquet can, of course, be made to correspond with the desired final diameters of the article except to take into account shrinkage owing to thermal contraction.

In Fig. 4, the powdered metal briquet 7 is formed inside a continuous solid metal band 8 and the composite briquet 7 and band 8 will be enclosed in heat-resistant material as shown in Fig. 1 or 2 and upon fusing of the briquet 7, the same will become bonded to or alloyed with the band 8 according to the particular selection of materials for the briquet and for the band 8.

In Fig. 5, there is shown a preferred form of apparatus through which the briquets and surrounding enclosures of Figs. 1 and 3, for example, are adapted to pass. In Fig. 5, the composite enclosures and briquets therewithin are represented by the reference numeral 10 and for purposes of illustration, these composite enclosures and briquets are of the form illustrated in Fig. 3.

At the top of the apparatus, there is provided a guide tube 11 in which the units 10 are superimposed one upon the other with the pistons 5 at the top of each unit. These units 10 are fed downwardly as by means of power-driven chains or belts 12 which run over sprocket wheels or pulleys 14 and 15. The opposed parallel portions of the belts 12 may be pressed into frictional engagement with the units 10 therebetween as by means of spring loaded blocks 16. It is to be understood that positive feed means may be employed if desired and in such case, the exterior of the units 10 may be provided with gear teeth or like formations for meshing with teeth on the wheels 14 or with spaces in the opposed chains 12. One pair of the wheels 14 or 15 will be power-driven and as evident, downward feeding of the units 10 may be accomplished at a constant speed.

The units 10 and pistons 5 may be successively pushed down through tube 11 by a reciprocating hydraulic ram.

The apparatus below the just-described feeding device comprises a porous or perforate guide tube 17 which may be formed of graphite or like material having a non-oxidizable coating on its inner surface. Such coating may be of stabilized zirconium oxide or similar refractory material.

The guide tube 17 thus interiorly coated serves to seal the units 10 from the surrounding atmosphere and also to provide a precision guide. Said guide tube 17 also alleviates gas contamination, but, at the same time the porosity of said guide 17 enables evacuation of gases from within the briquets and molds.

The upper portion of the guide tube 17 has a heating chamber 18 therearound for pre-heating the units 10 and briquets 1 therein as they pass downwardly through the guide tube 17. Said heating chamber may be heated by the frequency heating element 19.

As the units 10 pass downwardly through the heating chamber 18, substantially all of the gases are expelled and evacuated therefrom.

Below said pre-heating chamber 18 or as shown in the

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lower portion of said chamber, there is a high frequency heating coil 20 which encircles the guide tube 17. Thus, as the units 10 move downwardly at constant speed, the pre-heated briquets 1 will be further heated and melted or fused from the bottom thereof upwardly; and, because the gases have been expelled by the preheating of said briquet 1 and the enclosure 3 therearound and by the vacuum in chamber 18, the melted metal will completely conform with the cavities in the enclosures 3 without any trapped gas pockets. Where the pistons 5 are employed in association with the enclosures 3, the resistance to downward movement of the units 10 as will be hereinafter explained will cause the end of the piston 5 to exert pressure on the fused briquet 1 for densifying the same and further causing the melted briquet to completely fill the cavity within the enclosure 3 including sharp corners, etc.

The chamber 18 is preferably operated under a vacuum or partial vacuum with an inert gas such as argon therein when desired by means of a vacuum pump and exhaust pipe system attached thereto.

In passing downwardly through the high frequency heating coil 20, the briquets 1 within the units 10 are successively melted and compacted and thereafter the units 10 pass through a cooling unit 21 which surrounds the guide tube 17 and through which a suitable cooling medium is adapted to be circulated. Therefore, as the units 10 pass downwardly through the portion of the guide tube 17 located within the cooling unit 21, the enclosures 3 and melted briquets 1 therewithin are cooled to effect solidification of the compacted, sintered briquet 1.

Thus, when the units 10 are discharged from the lower end of the guide tube 17, the finished accurate articles of desired density are removed from within the enclosures 3 by breaking away the enclosures 3 where the latter are of one-piece construction or by opening same for removal of the articles therewithin in the case that said enclosures are formed of two or more mating pieces.

Where the porous enclosure 3 is formed of graphite or of a ceramic material having carbon therein a certain amount of carbon pick-up may be obtained in this type of fusion casting whereby at least the outer skin of the melted and subsequently solidified briquet 1 is made more abrasive resistant. For example, in casting titanium alloys, the outside skin is partially converted to titanium carbide.

In order that a desired resistance may be built up against the downward movement of the units 10 through the guide tube 17, there is disposed adjacent the lower end of said guide tube a friction mechanism which includes for example, a fixed shoe 22 engaging one side of superimposed units 10 and a movable spring load shoe 23 which resiliently presses against the opposite side of said units 10. The pressure which the shoe 23 exerts against the units 10 may be varied by adjusting the compression of the spring 24 by means of a suitable adjusting screw 25. Instead of the particular resistance unit shown at the bottom of Fig. 5, other well-known expedients may be employed. For example, a mechanism such as used for downward feeding may be positioned adjacent the lower end of the guide tube 17 and instead of positively driving one pair of the wheels 14 or 15, adjustable brakes may be associated therewith for building up the desired resistance against downward movement of the units 10, or a reciprocating hydraulic ram may be used to assist and/or control the resistance for compression desired.

When the briquet is of the form illustrated in Fig. 4, it is possible to bond the solid metal 8 to the powdered briquet 7 by using the apparatus illustrated in Fig. 5. It should be further noted that the briquets 1 may be made of spherical particles of metal or loose spherical particles may be loaded into a mold cavity and by fusing the spherical particles only partly, the final article will be

partially permeable and will be uniformly orificed since in that case, only the contacting portions of the spherical particles will bond together.

As one specific example of the present method nickel powder was pressed at a pressure of five tons per square inch to form a briquet that was enclosed in a porous graphite mold 3 and pushed down through a porous graphite guide tube 17 having high frequency coils 19 and 20 therearound operated at 9600 cycles for preheating the mold and melting the nickel briquet. The pressure in the vacuum chamber 18 was about 8 to 10 microns of mercury for degassing the mold and the briquet during heating and fusing of the latter. As the nickel compact was pushed downwardly it was melted as observed by an optical pyrometer. The pressure used to push the graphite mold 3 down through the graphite guide tube 17 was approximately 100 pounds per square inch and this closed the mold and when the cooled mold came out from the lower end of the apparatus the powdered nickel briquet had been transformed so as to be as dense as a solid rolled bar of nickel within about .02% and equally as dense as a cast nickel article. The graphite mold 3 and the guide tube 17 were of about 70% density so as to be porous to extract or evacuate gases there-through from the nickel briquet prior to fusing and during fusing.

As another example of the present method, a briquet of commercial pure titanium $\frac{1}{2}$ " in diameter and 1" in length was degassed in a vacuum of from 5 to 20 microns of mercury and was heated in the upper zone by the coil 19 operated at 30 kw. 450,000 cycles to a temperature of about 1800° F., again a graphite guide tube 17 and graphite mold 3 having been employed. A titanium rod was used as a pusher actuated by a hydraulic cylinder to move the mold 3 and enclosed titanium briquet into the fusion zone which was located about 4" below the upper zone, the lower coil 20 being operated at 30 kw. and 450,000 cycles for a period of 30 seconds which resulted in heating to approximately 2450° F. as read by a comparative radiant pyrometer. At the bottom heating zone, the briquet was squeezed in the mold 3 with a pressure of 10 tons per square inch applied through the upper hydraulically actuated rod and while the mold was supported by a lower hydraulic rod under the same pressure during the aforesaid 30 second period.

It has been found that there is a great advantage in preheating the briquet in a high vacuum to de-gas the same and at this stage the pushing means need not have any appreciable pressure applied thereto.

In the last-mentioned example the pressure in the lower hydraulic cylinder actuated rod was bled by a needle valve so as to induce downward movement while the pressure was kept at about 10 tons per square inch. The heat was applied intermittently to the mold and titanium briquet, and as aforesaid, in the upper zone, the heating may be conducted at 1250° F. to de-gas and sinter the titanium briquet. By following this process the final density of the briquet was about 97% of that of the published density of the metal titanium. Furthermore, the metal briquets thus formed were practically identical within $\frac{1}{100}$ of a gram and the compression and de-gassing and fusion compressed them to a final weight within $\frac{1}{100}$ of a gram.

The main feature of this invention is the two-stage operation, that is, vacuum de-gassing and pre-heating of the briquet so that it is shape retaining and thereafter further heating the de-gassed briquet and densifying the metal. The highest temperature needed in pressing and consolidating titanium powder of 200 mesh at 18 microns Hg vacuum is 2880° F., this being just under the reaction temperature of graphite and titanium. Fusion can also be obtained at 2450° F. at 18 microns Hg vacuum of 200 mesh titanium, without apparent reaction. Sintering may be accomplished at about 1800° F. on 200 mesh titanium powder and degassing at 18 microns Hg.

Such sintering can be accomplished without compacting pressure, but it takes about one hour to make a self-sustaining briquet in vacuum.

In any event, in the present method the first stage is a degassing and sintering operation and the second stage is a further degassing and fusing operation.

The present invention may be employed for the making of sintered electrodes for electrolytic condensers from titanium tantalum or other film forming metals. By the present method the degree of porosity and hence the area of the electrode exposed to the electrolyte can be regulated.

Other modes of applying the principle of the invention may be employed, change being made as regards the details described, provided the features stated in any of the following claims, or the equivalent of such, be employed.

I therefore particularly point out and distinctly claim as my invention:

1. A method of continuously producing articles from powdered metal comprising placing such powdered metal in a plurality of enclosures, moving such enclosures seriatim through dual heating zones, preheating each enclosure in a first heating zone to degas the powdered metal, heating each enclosure in a second heating zone, releasably opposing the movement of the enclosures through such second zone to force adjacent enclosures together, and then using such force to compress the metal while so heated to a desired density to form such articles.

2. A method of continuously producing articles from powdered metal comprising placing such powdered metal in a plurality of enclosures, moving such enclosures seriatim through dual heating zones, preheating each enclosure in a first heating zone to degas and sinter the powdered metal therein to a shape-retaining form, simultaneously removing the gases expelled from such heating zone, heating each enclosure in a second heating zone to a temperature higher than the temperature of such first zone, releasably opposing the movement of the enclosures through such second zone to force adjacent enclosures together, using such force mechanically to compress the metal while so heated to a desired density, and then sequentially cooling each enclosure and compressed metal therein to form such articles.

3. A method of continuously producing articles from powdered metal comprising placing such powdered metal in a plurality of enclosures, moving such enclosures successively through a hollow guide member, preheating each enclosure along one zone of such member to degas and sinter the powdered metal to a shape-retaining form, simultaneously evacuating the area defined by such one zone to remove the expelled gases and thereby adapt the sintered powdered metal for subsequent compression, heating each enclosure along a second zone of such member to a temperature higher than the temperature of such first zone, releasably opposing the movement of such enclosures through the member to force adjacent enclosures together, and then using such force to compress the metal while so heated to a desired density.

4. A method of continuously producing articles from powdered metal comprising placing such powdered metal in a plurality of porous molds, positively driving such molds in superimposed relation successively through a hollow guide member by abutting each mold against a preceding mold, preheating each mold along one zone of such member to degas and sinter the powdered metal to a shape-retaining form, simultaneously evacuating the area defined by such one zone to remove the expelled gases from such metal and at least in part through each porous mold and thereby adapt the sintered powdered metal for subsequent compression, heating each mold along a second zone of such member to a temperature higher than the temperature of such first zone while simultaneously evacuating such second zone also, releasably opposing the movement of the molds through the hollow member to force adjacent molds together, using

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such force to compress the metal in each mold while so heated, and then cooling the compressed metal before removing each mold from such hollow member.

5. The method of claim 4 wherein heating each mold along such second zone includes fusing the metal.

6. The method of claim 4 wherein heating each mold along such second zone includes further sintering of the metal.

7. The method of claim 5 further including the step of making at least one of such molds from a carbon-containing ceramic to fuse carbon into the outer surface of the articles formed as defined.

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8. The method of claim 4 further including the steps of including a metal band within a mold and bonding such moldable metal thereto.

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