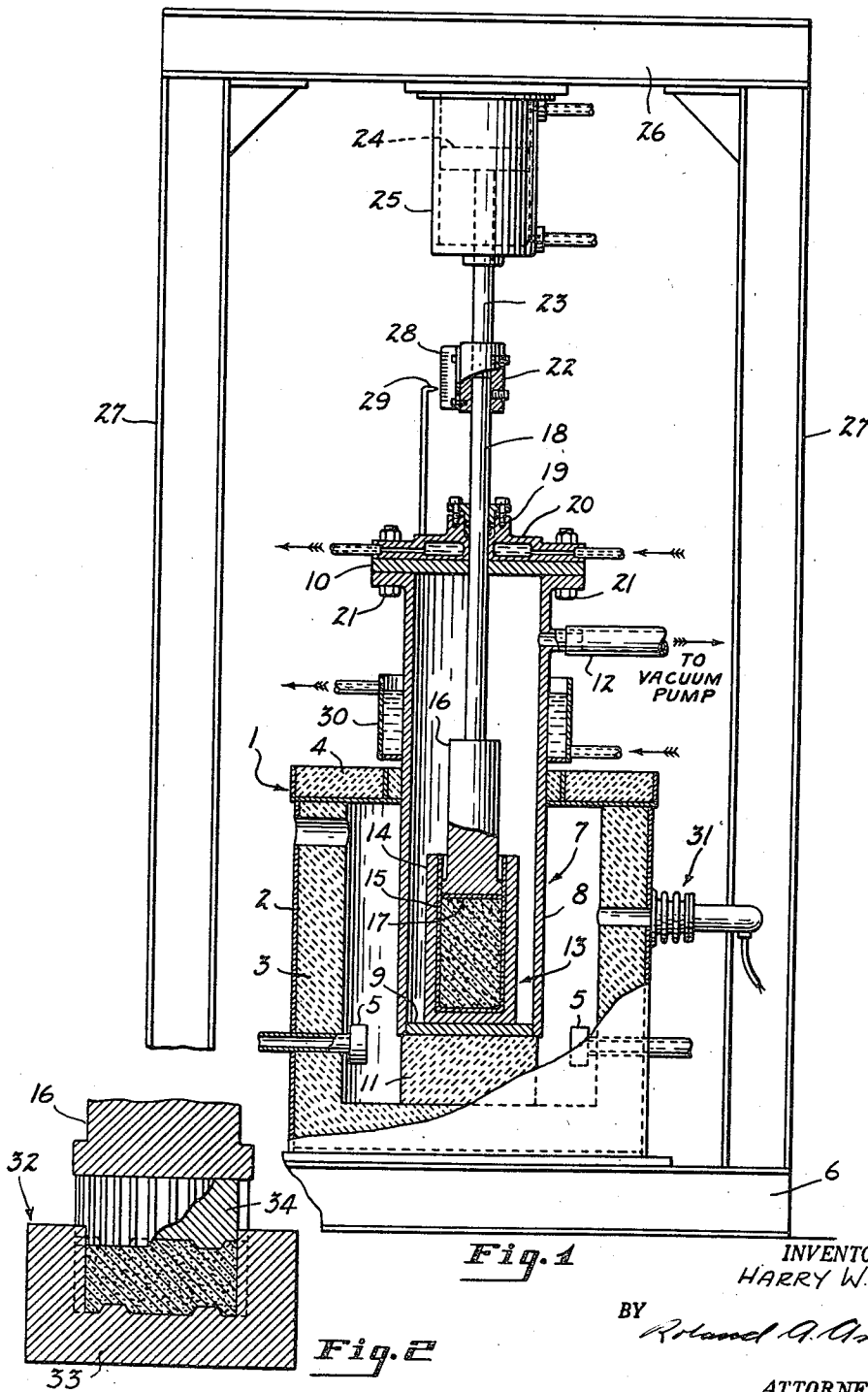


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METHOD FOR PRODUCING MALLEABLE AND
DUCTILE BERYLLIUM BODIES
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METHOD FOR PRODUCING MALLEABLE AND DUCTILE BERYLLIUM BODIES

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Continuation of abandoned application Serial No. 770,082, August 22, 1947. This application February 28, 1955, Serial No. 491,103

8 Claims. (Cl. 75-225)

The invention relates to a new and improved method of producing bodies of metallic beryllium having desirable physical properties which it has not been commercially feasible to secure heretofore. This application is a continuation of my earlier filed application Serial No. 770,082, filed August 22, 1947, now abandoned.

It is an object of the invention to provide an improved method of producing bodies of beryllium which is capable of effecting purification of the constituent metal, of securing excellent physical properties including a fine-grained heterogeneous structure, marked malleability, considerable ductility, machinability and insensitiveness to thermal shock, of providing control of the density of the metal up to its maximum theoretical density, and of producing very large bodies having the stated properties throughout.

Other objects of the invention incidental or ancillary to those indicated above will be apparent from the description which follows.

With the noted objects in view the invention, in its most important aspect, consists in charging beryllium in a powdered state suitable for powder metallurgy purposes into a container or die having a cavity of the form of the body to be produced and subjecting the powdered material to a prolonged treatment in which it is subject simultaneously to vacuum, heating at a sintering temperature low enough to prevent substantial grain growth in the metallic mass and mechanical pressure great enough to maintain the sintering charge in contact with the container walls, with the effect of removal of impurities present, sintering and condensation to desired density of the powdered mass and conformation thereof to the container cavity. Preferably the container or die of suitable strength is fitted with a thin-walled metal liner which facilitates the mechanical condensing of the charge as well as its separation from the die.

In another aspect, the invention may include controlling the density of the sintered body produced in the above stated treatment by controlling the weight of the charge of powdered material introduced into the treating container, measuring reduction in volume of the treated charge from time to time while it is subject to the above-noted vacuum, heat and pressure treatment and terminating the pressure and heating when the volume of the charge is reduced to a predetermined value calculated in relation to the weight of the charge.

In order that the invention in its various aspects may be clearly and fully understood the procedure and the special apparatus preferred for the practice of the invention will be described with reference to the accompanying drawing.

In the drawing,

Fig. 1 is a view, partly in side elevation and partly in central vertical section, of a preferred form of apparatus.

Fig. 2 is a view on a larger scale of a sintering container in the form of a die suitable for use in the furnace of Fig. 1 to produce a beryllium gear wheel.

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The process of the present invention may start with powdered metallic beryllium of technical grade from any convenient source. However, ordinary technical grade beryllium powder contains a considerable amount of impurities, including flux material, slag, oxides and metals other than beryllium, and since it is desirable that the powder have a minimum of such impurities it is desirable to take advantage of any economically feasible purification of the metallic beryllium to be used. In one such method, of a mechanical nature, the unpurified metallic beryllium is treated for removal of friable material, such as slag, by a series of crushing and screening operations which leave the metal reduced to a particle size suitable for further reduction by grinding and screening.

Beryllium powder, whether produced by such a method of crushing, grinding and screening or otherwise, may still contain some impurities and the present invention is designed to effect further purification of the metal, utilizing the furnace apparatus shown in the drawing.

This apparatus comprises a furnace having an open-topped structure designated generally by the numeral 1 and comprising an outer metal shell 2 with a lining 3 of refractory material. 4 is a cover of similar metal and refractory construction. Oil or gas-fired burners 5 mounted in the side walls of the furnace serve to heat the interior chamber thereof. The furnace rests upon a suitable foundation comprising channel or I beams 6.

The treating chamber of the furnace generally designated by the numeral 7 is formed by a steel tube 8 closed at its lower end by disc 9 hermetically welded to the tube. The tube is flanged at its upper end and closed by cover plate 10. Chamber 7 is supported somewhat above the bottom of the furnace 1 on a refractory block 11 of heat insulating material. At its upper end the furnace tube 8 is fitted with an outlet conduit 12 which is adapted to be connected to the intake of a vacuum pump (not shown) which may be of a conventional diffusion type to produce a high vacuum.

A container 13 for the powdered material to be treated is shown in the treating chamber 7 of the furnace. Container 13 comprises an open-topped steel pot 14 and a thin walled liner 15 of stainless steel which is removably fitted in the cavity of the pot. The heavy walls of the pot reinforce the liner so that it is adapted to sustain higher internal pressures than it otherwise could. Co-operating with container 13 is a heavy plunger 16 with its lower end formed to slidably fit the liner 15. A facing plate 17 of stainless steel is attached (as by spot welding) to the end of the plunger. Parts 15 and 17 are preferably made of 309 stainless steel (22-24% chromium, 12-15% nickel, plus small amounts of carbon, manganese and silicon). Plunger 16 has a rod 18 which passes through a hole in closure plate 10 and through a stuffing box 19 carried by a water jacket 20 which rests on plate 10 and serves to cool the stuffing box. The stuffing box 19 is packed with asbestos packing well saturated with high vacuum grease. Bolts 21 serve to secure plate 10 and water jacket 20 to the top of the furnace tube 8, with air-tight joints between the tube and the plate and the plate and water jacket.

The plunger rod 18 has its upper end connected by coupling 22 to the lower end of rod 23 of piston 24 which is operatively mounted in fluid power cylinder 25. The cylinder is carried by a superstructure comprising beam 26 and uprights 27 which connect the beam 26 to base members 6.

A graduated scale 28 is adjustably mounted on coupling 22 to move with plunger 16 and is arranged to co-operate with fixed pointer 29 supported by the furnace tube 8. Scale 28 thus arranged is adapted to indicate the

position of plunger facing plate 17 in liner 15 and is preferably calibrated to indicate directly the volume of the liner for any position of the plate.

The portion of the furnace tube 8 extending above cover 4 of the furnace is fitted with a water jacket 30 to prevent the upper part of the tube from reaching unduly high temperatures. To indicate and control the temperature of the treating tube within the furnace chamber the furnace is fitted with a radiation pyrometer 31. As a check on the temperature the furnace may also be fitted with an optical pyrometer (not shown).

In the use of the heat treating apparatus for carrying out the method of the present invention, the procedure may be varied in a number of respects, depending especially upon the degree of purity specified for the beryllium bodies to be produced, the density required for such bodies and cost considerations. For purposes of explanation a description will first be given of the procedure suitable for the production of beryllium bodies of predetermined form and size, of a specified density and a high degree of purity.

Preliminarily it may be noted that the invention allows of wide latitude as to the size of the beryllium bodies produced. In fact the invention has made it possible to produce metallic beryllium bodies of maximum density and excellent physical properties far larger in size than anything heretofore reported. Also, in the practice of the invention, the forms of bodies produced may be varied, subject only to the obvious conditions that the container or die in which the body is sintered must have its wall parts which cooperate with the pressure-applying plunger formed with inner surfaces parallel to the plunger movement. The bodies may be solid or tubular or otherwise recessed. For simplicity of illustration the accompanying drawing shows apparatus for producing bodies of cylindrical form.

Assuming that bodies of beryllium to be produced are to be of a high degree of purity and a density of 1.85, which is approximately the density of cast beryllium, that the container 13 and plunger 16 have been provided of desired form and size, and that the powdered beryllium for use has been subjected to gross purification, as by the mechanical crushing and screening method above referred to, a charge of such powder is accurately weighed out corresponding to the density specified for the body to be produced of known or computable volume, and is introduced into the liner 15 and the latter lowered into container 14 resting in the open furnace tube 8. With the power piston 24 held in its uppermost position, the plunger 16 with parts 10, 19 and 20 assembled on rod 18, is lowered into the furnace tube and secured by coupling 22 to the piston rod 23. This supports the plunger with its lower end above the top opening of container 13.

The vacuum pump is now started and operated until the pressure in the furnace tube is reduced to 10 microns of mercury, or lower, and it is evident (from low rate of change in pressure) that the air is correspondingly exhausted from the powder charge. The evacuation of the heating tube should, of course, be at a sufficiently slow rate to avoid drawing powder from container 13. Exhausting to the vacuum pressure noted may take from one to five hours, depending upon the initial degree of purity of the metallic powder and the size and shape of the body to be produced.

Following the exhausting stage described the furnace is heated, while the operation of the vacuum pump continues, until the temperature of the treating tube reaches about 900° C. During this stage gas and vaporizable impurities present in the charge are evolved and removed by the pump. Such evolution of vapor may be accompanied, for a time, by increased pressure in the treating tube. When the pressure is again fairly stable at 10 microns of mercury or lower, which may be after heating from one to forty hours, pressure fluid is admitted

to the top of the power cylinder to lower the plunger 16 and apply a low mechanical pressure to the mass of powder, the operation of the vacuum pump meanwhile continuing.

The mechanical pressure applied should be great enough to prevent contraction of the sintering mass away from the container walls but not great enough to distort the container, which may be weakened at the relatively high sintering temperature of beryllium. A pressure of 70 pounds per square inch on the top of the charge has been found suitable for this purpose. While higher pressures than this may be used, unduly high pressures during sintering are not only unnecessary but also objectionable as they tend, by distortion of the container, to cause formation of cracks in the sintered body and difficulty in separating it from the container. As the sintering temperature of the beryllium varies with its purity, the latter and also the character of the constituent material of the container may affect the maximum mechanical pressure suitable for use. In any case the suitable pressure will be low in comparison with compacting pressures commonly used in powder metallurgy.

At the same time that mechanical pressure is applied to the charge heating of the charge is increased until the container 13 is at a temperature sufficiently high to insure thorough sintering throughout the mass of the charge without unreasonably prolonging the treatment. This temperature will vary with the purity of the beryllium making up the charge. With high purity assumed for the procedure being described the temperature should be carried to about 1125° C. Heating very far above this temperature may cause pronounced grain growth and should be avoided because such growth would prevent attainment of the malleability and ductility which is one of the objects of the invention. With lower degrees of purity the temperature at the sintering stage in question may be as low as 1000° C.

The charge should be held under the specified mechanical pressure at the higher sintering temperature until sintering is accomplished throughout the mass. During the heating of the charge under mechanical pressure the operator observes the volume gauge 28 and when the gauge indicates a container volume which, according to prior computation, corresponds to the density specified for the product, heat is turned off the furnace and mechanical pressure on the charge is terminated by raising the power piston 24.

The operation of the vacuum pump is continued throughout the heating and preferably, after the furnace burners are turned off, during the cooling of furnace and charge.

When the container 13 has cooled to a temperature of blackness the furnace tube is opened, the container lifted out and the charge dumped out with the liner 15 possibly adhering to it, though making the liner of stainless steel minimizes such adherence. The liner is then peeled from the sintered body, being cut into several pieces if necessary, to free it from the sintered mass. It will be understood that the use of the relatively thin-walled liner facilitates the carrying out of the treatment because it prevents adherence of the sintering charge to the main walls of the container and facilitates compacting of the charge and its separation from the container.

By the use of sintering containers of suitable character a wide variety of beryllium articles can be produced. For example, a toothed spur gear wheel of beryllium can be produced by using in place of container 13 of Fig. 1 a die such as is shown in Fig. 2. Here the die, indicated generally by the numeral 32, comprises a female body 33 and a cooperating male body 34, both parts preferably formed of stainless steel. In carrying out the process the beryllium powder is charged into die body 33, the male member 34 inserted into the top of the die cavity, the assembly lowered into the furnace tube 8 and the latter closed with plunger 16 resting on top of die mem-

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ber 34. The further procedure may then be as above described.

The die 33 may be so designed as to depth of its chamber that when a suitable weight of powder is charged into the die, compression of the charge during the treatment will produce the nominal maximum density of metallic beryllium when the bottom of plunger 16 engages the top of die body 33. Such compacting of the charge usually involves a reduction in volume of about fifty percent. If a lesser density is desired for the body to be produced, the operator can readily achieve this by following the indication on scale 28 and stopping the application of pressure and heat to the charge at a suitable point to produce a predetermined density below the maximum.

In the last described procedure the stainless steel metal of the die in addition to reducing adherence to the die of the sintered metal, has the further advantage that, up to the sintering temperatures of beryllium, the coefficients of thermal expansion of both metals are nearly identical; so that, on cooling, separation of the sintered body from the die is facilitated and cracking of the sintered body by differential contraction of the die and sintered body is avoided. This feature of similar coefficients of expansion is especially important in forming beryllium bodies having recessed surface contours with beryllium embracing or surrounding portions of the die, because most die material, other than stainless steel, suitable for use at the temperatures and pressures involved in the present process have coefficients of expansion at such temperatures considerably less than that of beryllium, so that parts of the beryllium structure embracing the die material are subjected to shrinkage stresses caused by the greater contraction of the beryllium against the more slowly contracting die material.

Metallic beryllium bodies produced in the manner described are not only characterized by a fine-grained heterogeneous microscopic structure, predetermined density, high malleability, ease of machining and insensitiveness to thermal shock, but also are marked by extremely high purity of the constituent metallic beryllium. It is believed that the high purity attained is to an important extent due to the evacuation of the container while the charge therein is in an uncompacted state. This applies to the evacuation both before and during heating of the charge. With the charge in this state the evacuation with the pump before heating and in the early stages of the heating more effectively removes water vapor and oxidizing gases from the charge so that oxidation of the charge in the later heating stage is largely prevented. The uncompacted state of the charge during vaporization of impurities also favors a more complete removal of such impurities by the vacuum pump. Also, it should not be overlooked that minimizing of oxide formation in the charge correspondingly favors sintering and high strength of the integrated body. In addition, the removal from the finely divided charge of gases and impurities in vapor form is an important factor securing freedom from porosity and permitting accurate and reliable determination of density of the sintered body.

The applications of vacuum, sintering heat and low mechanical pressure which characterize the present method has the further great advantage, already briefly noted in the foregoing description, that it makes possible the production of a metal such as beryllium in extremely large bodies having excellent physical properties including fine grain structure, good malleability, ductility, and machinability, good resistance to temperature shock and full density. It is believed that these physical properties are at least in part essentially due to the prolonged treatment by evacuation and heating which strips from the interstices and pores of the metal mass all vaporizable material so that it is possible for the moderate mechanical pressure applied to the softening mass to more effectively consolidate the metal to its full density. It will be ap-

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preciated that in the production even of small bodies the stripping action referred to in its very nature requires a considerable amount of time and, furthermore, that the time must increase with the size of the metal mass under treatment. It will also be appreciated that the prolonged time of treatment is an essential feature of the method regardless of the degree of purity of the powdered metal to which the treatment is applied, since it is practically impossible in the handling of the metallic powder to avoid adsorption of oxidizing gases and vapors on the surfaces of the powder particles, and as the powder material is consolidated under pressure these adsorbed materials line the resulting pores in the mass and, together with any other vaporizable impurities that may be present in the material of the charge, must be removed in order to attain the desired density under the moderate mechanical pressures that characterize the method.

As already noted, considerable variation of the procedure followed and of the construction of the apparatus used is possible in practice of the present improvements. For example, if the density of the product is not critical and the highest degree of purity unnecessary a less complete evacuation of the charge container is permissible. This will shorten the time of the evacuation treatment prior to heating or during heating or both.

Other variations permissible in particular cases will be apparent from the nature of the treatment and the considerations indicated by the foregoing description.

Accordingly, both as to the method of procedure and the apparatus employed, it will be understood that the invention is not limited to the specific disclosure but may be varied within the bounds of equivalency and the terms of the appended claims.

What is claimed is:

1. In a method of producing malleable bodies of metallic beryllium, the steps of charging a mass of the metal in a finely divided state suitable for powder metallurgy purposes into a container having an interior having the form of the body to be produced; subjecting the interior of the container and the charge therein to the combined action of evacuation and heating at a temperature below about 900° C. until a large part of any gas and vapor sorbed in the charge has been removed therefrom as evidenced by the pressure in the container; thereafter, while maintaining the evacuation of the container, subjecting it and the charge to sintering heat within the temperature range from about 1000° C. upward to a temperature below that at which pronounced grain growth of the beryllium treated would occur and prolonging the heating at such sintering temperature until the charge is sintered throughout; during the prolonged combined evacuating and heating treatment in the stated sintering temperature range subjecting the charge to mechanical pressure great enough to maintain the sintering charge in contact with the container walls but both insufficient to cause distortion of the heated container and much lower than would be required, in the absence of said evacuation, to produce the same density of the charge; and separating the container from the sintered body so formed.

2. The method of claim 1 in which the charge is subjected to a mechanical pressure of about 70 pounds per square inch during the evacuating and heating treatment thereof at the sintering temperature.

3. The method of claim 1 in which the finely divided metal is charged into a container having a thick-walled outer structure and a separable thin-walled liner fitting the inner surfaces of the outer structure and the container is separated from the sintered charge by first removing the liner and sintered charge from the outer structure and then stripping the liner from the sintered charge.

4. In a method of producing malleable bodies of metallic beryllium, the steps of charging a mass of the metal in a finely divided state suitable for powder metallurgy purposes into a container with an interior having the

form of the body to be produced; subjecting the interior of the container and the charge therein to the combined action of evacuation and heating at a temperature below about 900° C. until a large part of any gas and vapor sorbed in the charge has been removed therefrom as evidenced by the pressure in the container; thereafter, while maintaining the evacuation of the container, heating it and the charge to a sintering temperature within the range from about 1000° C. to about 1125° C. and prolonging the heating at such sintering temperature until the charge is sintered throughout; during the prolonged combined evacuating and heating treatment in the stated sintering temperature range subjecting the charge to mechanical pressure great enough to maintain the sintering charge in contact with the container walls but both insufficient to cause distortion of the heated container and much lower than would be required, in the absence of said evacuation, to produce the same density of the charge; and separating the container from the sintered body so formed.

5. The method of claim 4 in which the charge is subjected to a mechanical pressure of about 70 pounds per square inch during the evacuating and heating treatment thereof at the sintering temperature.

6. The method of claim 4 in which the finely divided metal is charged into a container having a thick-walled outer structure and a separable thin-walled liner fitting the inner surfaces of the outer structure and the container is separated from the sintered charge by first removing the liner and sintered charge from the outer structure and then stripping the liner from the sintered charge.

7. In a method of producing malleable bodies of metallic beryllium having recessed surface contours the steps of charging a mass of the metal in a finely divided state suitable for powder metallurgy purposes into a chambered die of stainless steel having its chamber walls shaped to form the recessed surfaces of the beryllium body to be produced; subjecting the chambered die and the charge therein to the combined action of evacuation and heating at a temperature below about 900° C. until a large part of any gas and vapor adsorbed in the charge has been removed therefrom as evidenced by the pressure in the die chamber; thereafter, while maintaining the evacuation of the die chamber, subjecting the die and the charge to sintering heat within the temperature range from about 1000° C. upward to a temperature below that at which pronounced grain growth of the beryllium treated would occur and prolonging the heating at such sintering

temperature until the charge is sintered throughout; during the prolonged combined evacuating and heating treatment in the stated sintering temperature range subjecting the charge to mechanical pressure great enough to maintain the sintering charge in contact with the chamber walls of the die but both insufficient to cause distortion of the said walls and much lower than would be required, in the absence of said evacuation, to produce the same density of the charge; and separating the die from the sintered body so formed.

8. In a method of producing malleable bodies of metallic beryllium in which a mass of the metal in a finely divided state suitable for powder metallurgy purposes is charged into a container with an interior having the form of the body to be produced, the steps of simultaneously subjecting the container and the charge therein to prolonged and effective evacuation and sintering heat within the temperature range from about 1000° C. upward to a temperature below that at which pronounced grain growth of the beryllium treated would occur and prolonging the heating at such sintering temperature until the charge is sintered throughout; and during the prolonged combined evacuating and heating treatment in the stated sintering temperature range subjecting the charge to mechanical pressure great enough to maintain the sintering charge in contact with the container walls but insufficient to cause distortion of the heated container.

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