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3,845,185

METHOD FOR PRESSURE SINTERING A REFRACTORY POWDER

Filed March 30, 1973

3 Sheets-Sheet 1

FIG. 1.

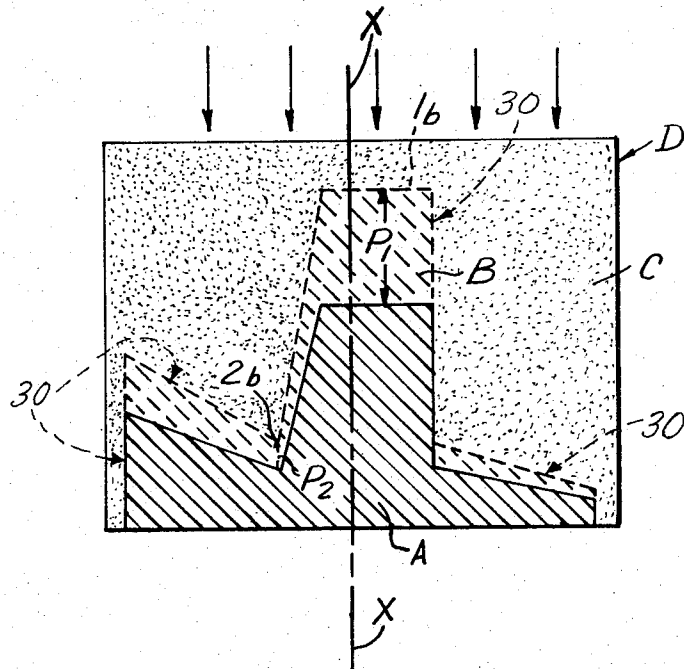


FIG. 2.

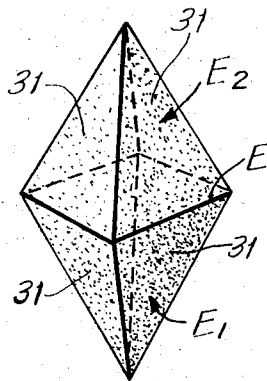
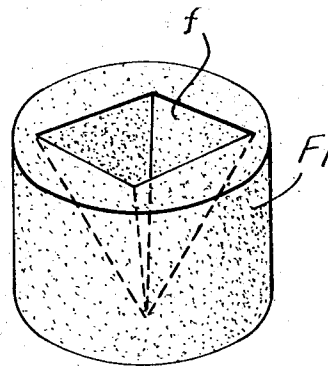


FIG. 3.



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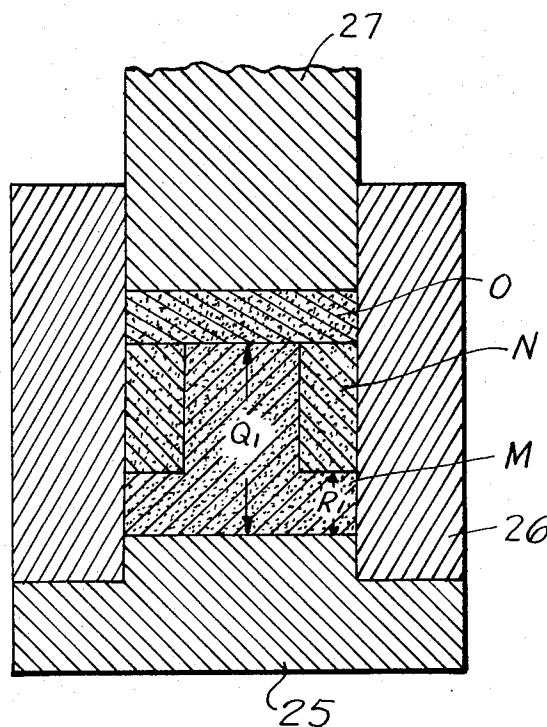
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METHOD FOR PRESSURE SINTERING A REFRACTORY POWDER

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FIG. 7.



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3,845,185 METHOD FOR PRESSURE SINTERING A REFRACTORY POWDER

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Continuation-in-part of abandoned application Ser. No.
161,060, July 9, 1971. This application Mar. 30, 1973,
Ser. No. 346,624

Claims priority, application Japan, July 18, 1970,
45/62,921

Int. Cl. F27b 9/14

U.S. Cl. 264—86

6 Claims

ABSTRACT OF THE DISCLOSURE

A method for producing a precisely dimensioned sintered product by pressing a refractory powder along one direction, wherein a refractory powder is formed into a first body having sections parallel to said pressing direction which are elongated at the same fixed ratio for all sections of the desired product parallel to the pressing direction, and at least one other, or second, body, which fits in and/or around said first body to form a composite body having a uniform thickness in the pressing direction is made from the same refractory powder, said first and second bodies being bound together to form said composite body with a thin layer of a parting agent interposed between them, and said composite body is placed in a die and is then pressed in said pressing direction, while being subjected to the sintering temperature of the refractory powder, whereby upon removal of the second body a sintered product comprising the first body of desired form and uniform density is obtained.

BACKGROUND OF THE INVENTION

This is a continuation-in-part of application Ser. No. 161,060, filed July 9, 1971, now abandoned.

The present invention relates to an improved method for sintering a refractory powder by subjecting the powder to high, sintering temperature while applying uni-axial pressure to a body formed of said powder (this method will be called hot-pressing hereinafter).

DESCRIPTION OF THE PRIOR ART

Conventionally it has been considered that a sintered body of complicated form could not be obtained by hot-pressing since the powder to be sintered is packed into a die of suitable form and is pressed uni-axially by means of a punch, or the like, when the sintered body is to have a cylindrical form, and is pressed tri-axially when the sintered body is to be a hexahedron having mutually perpendicular surfaces. In the conventional hot-pressing method, the absolute value of the shrinkage at any portion in one surface is equal to the stroke of the punch, and therefore, every part undergoes compression of the same absolute amount regardless of the form of the punch. For this reason, a body having a simple form such as a circular plate or a parallelepiped can be obtained, but when the body is to have varying thickness (height), a sintered body of uniform density cannot be obtained.

In general, since the mobility of the sintering powder is not always high during hot-pressing, the powder does not move very well from the thick part to the thin part, or vice-versa. Therefore, in order to obtain a sintered body of uniform density, each part of the body must have been pressed at the same fixed ratio along the pressing direction, or directions, compared with the same part of the body before being pressed. This will be further explained with reference to the drawing hereinafter.

Conventionally, an isostatic hot-pressing method is employed for pressure sintering of a body having a com-

plicated form. In this method, the powder to be sintered is packed into a metal, or rubber container and sealed under vacuum, or diminished pressure. The powder is then sintered at high temperature while pressing with high pressure gas, or oil. The powder is usually pressed uniformly from all directions, so that a sintered body having the desired form and size is obtained starting with a body which in size is several times larger in all directions than the desired product.

Since in the isostatic hot-pressing method, the powder is pressure-sintered in a sealed container, gas, moisture, and the like, absorbed into the powder and in the sealed container is released and reacts with the powder at high temperature so that a sintered body having the desired form cannot be obtained. For example, when magnesia (MgO) is sintered, blucite ($Mg(OH)_2$) is produced by reaction of the moisture content with the magnesia, and the mechanical strength of the magnesia sintered body is considerably lowered, or lost entirely. Even when the moisture does not react with the powder and it remains magnesia, the close adherence between the surface of the sealed container and the powder inside the container is prevented by the moisture, and the pressure cannot be uniformly applied to the powder. Therefore, the powder cannot be sufficiently sintered to have the desired form. Furthermore, the container must be made of rubber, metal, and the like, having high deformability, but such materials have insufficient refractory property, so that it is very difficult to sinter a powder having a high melting point such as nitride, a boride, or the like. When a nitride powder is sintered, it is necessary to prevent its decomposition, or to promote its nitrification. However, it is impossible to prepare the requisite atmosphere in the described hot-pressing method, so that this method cannot be applied. For example, when the powder that is to be sintered is beta-alumina ($Na_2O \cdot 11Al_2O_3$) which decomposes into its component elements ($Na_2O \cdot Al_2O_3$) in air at high temperature, it is necessary to prevent the decomposition of beta-alumina by feeding a material such as sodium carbonate (Na_2CO_3) in excess into said beta-alumina beforehand, to elevate the vapor pressure of sodium oxide (Na_2O) at high temperature. Alternatively, it is necessary to supply sodium carbonate which is decomposed and volatilized, but this cannot be done during the sintering because the gas generation within the sealed container is undesirable and prevents the formation of a sintered body of desired shape.

SUMMARY OF THE INVENTION

The present invention relates to an improved hot-pressing method substantially as follows. The body (hereinafter called a first body) is formed of a part of a refractory powder to be sintered and which body is uni-axially enlarged compared with the desired sintered product. Another part of the refractory powder is employed to form at least one other, or second, body which fits in the first body and the two bodies are combined together to form a composite body. The composite body is formed to have equal length (thickness) in all of its portions lying in planes parallel to the elongated axis (see X, FIG. 1) of the first body, and by hot-pressing the composite body along the said axis at sintering temperature of the first powder, upon removal of the second body a sintered body of desired form and size remains.

It will be apparent from the preceding, that a primary object of the invention is to overcome the defects and disadvantages briefly outlined in connection with conventional hot-pressing processes including the isostatic hot-pressing process.

Another important object of the invention is to provide an improved hot-pressing method which enables the for-

mation of sintered bodies of complicated form and uniform density.

Yet a further object of the invention is to provide an improved hot-pressing method, having the above-described characteristics, which is easy and inexpensive to perform.

BRIEF DESCRIPTION OF THE DRAWINGS

The novel features which are considered characteristic of the invention are set forth with particularity in the appended claims. The invention, itself, however, both as to its organization and its method of operation, together with additional objects and advantages thereof, will best be understood from the following description of specific embodiments when read in connection with the accompanying drawings, wherein like reference characters indicate like parts in the several figures, and in which:

FIG. 1 diagrammatically illustrates in cross-section a method of forming a sintered body according to the present invention,

FIG. 2 is a perspective view of a different body preparatory to being formed into a sintered product by the method of the present invention,

FIG. 3 is a diagrammatic perspective view of a portion of a second body suitable for use with the body of FIG. 2 in following the inventive method,

FIG. 4 is a perspective view of a complete composite body including the body of FIG. 2 as formed for applying the hot-press method of the present invention,

FIG. 5 is a perspective view, partially broken away, showing another composite body suitable for forming a sintered product different from the products illustrated in FIGS. 1 and 2,

FIG. 6 is a sectional diagram showing apparatus usable in forming still another embodiment of the sintered product in accordance with the method of the present invention, and

FIG. 7 diagrammatically illustrates in cross-section apparatus which is used to show the accuracy in size of the sintered products in accordance with the method of the present invention.

Referring now more particularly to the drawings, FIG. 1 illustrates a composite body suitable for forming one sintered product by the method of the present invention. This Figure will also illustrate the disadvantages and the inability of the conventional hot-pressing method referred to above. For example, the sintered body A of uniform density and having unequal thickness vertically in various parts, cannot be obtained using the conventional hot-pressing method because the absolute value of shrinkage is equal in each of said parts. Thus, if the stroke of a punch is adjusted to the shrinkage of P_1 at the top part of 1_b , the hollowed part of the punch, corresponding to 2_b , would reach the bottom of body A when a pressure is applied. Thus, the desired shrinkage of P_2 would be far exceeded and the desired configuration would not be obtained.

However, with the present invention, a body B of the sintering powder shown by the broken lines is formed larger than the desired sintered body A shown in full lines with all vertical sections, parallel to one pressing direction shown by the arrows to be reduced in the same proportion. Accordingly, when pressure is applied to the covering body C in said pressing direction, the body B is reduced to the desired size and shape, i.e., body A.

By way of further explanation, the first body B is enlarged with respect to the desired sintered body A only in the pressing direction shown by the arrows at the top of FIG. 1, and this enlargement is at a fixed ratio for each vertical section of the body. The second body C is formed of another part of the same refractory powder forming the first body B. Also, the densities of the first and second bodies should be the same. The first and second bodies are then combined together and form the composite body D as a cylindrical column with uniform thickness in the direction of the axis X while a thin layer 30 of a parting

agent is placed between the first and second bodies B, C. Upon placement of the composite body in a die and upon applying pressure along the axial direction while, at the same time, subjecting the composite body to high sintering temperature of the powder forming the composite body D, the body B is reduced to the size and shape of the desired product A.

The composite body described in the preceding paragraph may be made in any one of the following ways:

- (1) Compressing a part of a refractory powder into a first body having enlarged dimensions parallel to an axis of the body as compared to the desired product; forming a thin layer of a parting agent on the surfaces of said first body; compressing another part of the same refractory powder into a second body having a cavity of correct size to receive the first body and the same density as that of the first body, and inserting the first body in the cavity of the second body to form a composite body,
- (2) Preparing a slurry of a refractory powder with a liquid, pouring a part of the slurry into a cavity of a porous mold capable of absorbing the liquid and thereby forming a first body of the refractory powder; forming a second body from another part of the same slurry by the same method as that of forming the first body; drying the first and second bodies, forming a thin layer of a parting agent on the surfaces of the first body, and combining the first and second bodies to form a composite body.
- (3) Forming a block of a refractory powder by applying an isostatic pressure, machining first and second bodies from the block, combining the first and second bodies to form a composite body.

The important points of the invention are that the first and second bodies are made of the same refractory powder, that the first and second bodies have the same density and/or porosity as each other and that the composite body has a uniform thickness in the pressing direction. The above three points are essential to make a sintered refractory product having desired shape and uniform density throughout all parts. Also, if these three points are satisfied, the first and second bodies have the same sintering temperature and compressibility under any sintering condition. Therefore, the most suitable sintering temperature and/or pressure can be employed for the desired product.

As the parting agent, a refractory powder whose melting point is higher than that of the powder to be sintered, and which does not react with the powder to be sintered, is employed. Preferably, the parting agent is formed as a thin plate by mixing said refractory powder with an organic binder, pouring the mixture on a flat plate such as a glass plate after degassing the mixture to solidify the mixture. When the first powder, for forming the sintered product, is alumina, the refractory powder of the parting agent is zirconia or boron nitride. When mica is the powder for forming the sintered product, zirconia, boron nitride, alumina, silica or magnesia is used to form the parting agent. When silicon nitride is the powder to be sintered, boron nitride is used to form the parting agent. As the organic binder of the parting agent, poly-methyl-methacrylate, polystyrene, cellulose, dextrin, or the like, may be employed.

When the parting agent is formed as a thin plate, the sintered body and the second, or covering body can be easily separated, and the surfaces of the sintered product are very smooth because the organic binder is decomposed and volatilized during the sintering operation and the parting agent remains as a thin layer.

When the composite body is compressed in the inventive process, at sintering temperature, the first body is sintered and reduced in size to form a product having uniform density and desired shape, being compressed by the same ratio at all parts in the enlarged direction thereof. During the hot-pressing step, the powders of the first body and the second body do not mix together, nor does

the resultant sintered body change to any more complicated shape, or bend in directions perpendicular to the pressing direction. It has been proved that the first body is subjected to uniform reduction in size by displacement in directions parallel to axis of pressure and proportional to the thickness thereof.

According to the present invention, a sintered product having uniform and high density and of simple form such as a column, a cone and a pyramid, as well as very complicated forms such as that of a turbine blade, can be made. Should the desired product having a certain curvature, as for example a turbine blade, this curvature can be produced easily by selecting a suitable pressing direction, forming a body to be sintered having very small curvature, and pressure sintering this body from the direction for increasing the curvature by means of the hot-press method.

Illustrative of several different forms of embodiments of the products which can be formed by the present invention and as will be explained in detail with reference to the drawings, are the following embodiments:

EMBODIMENT 1

(Hereinafter all statements of percentage are intended to cover percentage by weight.)

98% alpha-alumina powder passable through a 200-mesh screen and 2% magnesia powder also passable through a 200-mesh screen, were thoroughly mixed. Then 1% polyvinyl alcohol (P.V.A.) aqueous liquid, which was diluted to 1% by water, was added to the mixture and the resultant material was pressed (with 300 kg./cm.² pressure) in a mold whose inside diameter was 40 mm. to form a column-shaped body. This column-shaped body was 40 mm. in diameter and 40 mm. in height. From this body, an octahedron E, FIG. 2, was machined. Body E, referred to hereinafter as the first body, it will be noted included two pyramids E₁ and E₂, having square bottom surfaces in contact with each other and pressed together. Each of these pyramids was 20 mm. in height and the length of a side of the said bottom surface was 20 mm.

Next from the same powder composition as described in the preceding paragraph, another columnar body was compacted in the same pressing mold. From this columnar body two columns F₁, F₂ (FIGS. 3 and 4) each being 40 mm. in diameter and 25 mm. in height were machined. As shown in FIGS. 3 and 4, each of the F₁, F₂ columns was formed with a pyramid-shaped cavity *f* having a height of 20.2 mm. and whose axis was parallel with that of the column. The pyramidal cavity *f* had a square upper opening, as viewed in FIG. 3 whose side was 21.6 mm. in length. The two columns F₁ and F₂, together formed the covering body, or second body G, FIG. 4.

Next, each surface of the octahedron E, FIG. 2, was covered with a separate sheet of film 31 0.3 mm. thick to act as a parting agent. This film was made by pouring poly-methyl-methacrylate including 40% boron-nitride on a glass plate and then drying it. Then the pyramid E₁ was inserted into the cavity *f* of column F₁ and the pyramid E₂ of the octahedron projecting therefrom was inserted into the cavity *f* of the other column F₂ so as to form the column-shaped composite body G of about 50 mm. in height shown in FIG. 4. During the assembly of composite body G, a second sheet of the same parting agent film 31 of 0.3 mm. thickness, made in the same manner as described, was inserted between the contact surfaces of the two columns F₁ and F₂ to surround the octahedron E.

The composite body G was disposed in a tube-shaped graphite die whose inside diameter was 40 mm. and the body was heated with high frequency electric means and then hot-pressed for 15 minutes, at 1650° C. with 300 kg./cm.² pressure, directed along the central axis X₁ of the composite body and exerted by a punch having a plate-shaped pressing surface whose external diam-

eter was 40 mm. The gas generated during sintering was dispersed outwardly through a gap between the die and the punch.

After the hot-pressing step, the die was cooled gradually and the composite body G was removed from the graphite die. The compressed body G had an external diameter of 40 mm. and a height of about 35 mm. By striking the body G lightly and pulling the upper and lower parts in opposite directions, with slight force, the composite body was easily separated into three parts corresponding to E, F₁ and F₂ and thus an octahedron-shaped alumina sintered product corresponding to E but of reduced height was obtained. The pyramid portions of the sintered product each had a square bottom surface whose sides measured about 19.3 mm. The height of each pyramid was about 14 mm. Each surface of the octahedron was plain and smooth, and the whole open porosity was less than 0.2% when measured by means of a water absorbing method. Therefore, it was found that the sintered product formed as above described was sufficiently homogeneous.

EMBODIMENT 2

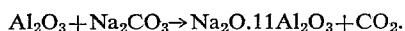
The method of the invention in this embodiment was applied to the making of a cylinder-shaped beta-alumina crucible having an internal diameter of 18 mm. and depth of 30 mm.

8% sodium carbonate (Na₂CO₃) was added to alpha-alumina (Al₂O₃), and thoroughly mixed. Then 2% aqueous solution of 0.5% polyvinyl alcohol was added to the mixture. The mixture was then pressed at room temperature under 200 kg./cm.² pressure so as to be solidified into a cylinder. This cylinder was machined into a crucible H forming the first body. The cylinder had a main body portion H₁ and an outwardly projecting rim H₂ surrounding the open upper end thereof as shown in FIG. 5. The external diameter of the main body H₁ was 20 mm. while its internal diameter was 18 mm. The external diameter of the rim H₂ was 30 mm. and its thickness was 7.5 mm. The height of the crucible (the distance between the upper surface of the rim and the bottom surface of the main body H₁) was 45 mm. Then, boron nitride as a parting agent was sprayed to a thickness of about 0.2 mm. over the entire internal and external surfaces of the crucible H. A cylinder J₂ and a cup J₁ were shaped to fit, respectively, inside and about the crucible under its rim and thereby forming a second body. J₁ and J₂ were made of the same sintering powder as described above in the making of the crucible H. The cup J₁ had an external diameter of 30 mm. a height of 60 mm. and a cavity for receiving the cylindrical portion of the crucible. The cylinder J₂ was formed to an external diameter of 18 mm. and a height, or length of 45 mm. for insertion into the crucible. When J₁ and J₂ were assembled in and about the crucible H, a column-shaped composite body shown in FIG. 5 was formed having an external diameter of 30 mm. and a height of 67 mm.

This composite body was put into a graphite die having an internal diameter of 30 mm. and was hot-pressed under 300 kg./cm.² pressure by means of a punch along the axial direction of the composite body for 15 minutes at a temperature of 1500° C. As a result a crucible product of beta-alumina as sintered was formed from crucible H to a reduced size of 30 mm. in height or depth, the internal diameter remaining 18 mm. During sintering, graphite powder which had been placed around the inside of the graphite die and the outside of the punch was displaced because gas was generated within the die, thus proving that carbonic acid gas (CO₂) was generated as a result of the decomposition of the sodium carbonate used in forming the first body H. In the conventional method, previously described, sintering powders which produce gas during sintering cannot be used.

After the hot-pressing step, the apparatus was cooled, and the sintered crucible formed from body H was removed from the composite body. The resultant crucible

was nearly of the desired size having an internal diameter of 18.1 mm. and a depth of 30.5 mm. When the rim was examined by X-rays, the primary material composing the crucible was found to be beta-alumina ($\text{Na}_2\text{O} \cdot 11\text{Al}_2\text{O}_3$). A slight amount of alpha-alumina was detected and a small amount of sodium aluminate, but very little. During hot-pressing, the reaction was as follows:



EMBODIMENT 3

Silicon nitride powder was mixed thoroughly with 8% silicon and then a 2% aqueous solution of polyvinyl alcohol was added. After drying the material was compressed under 500 kg./cm.² in a rubber press. The first body K FIG. 6, in the form of a bolt was machined from this material. The screw portion of the bolt had an external diameter of 10 mm. and a length of 30 mm. A nut L, forming a covering, or second body, was machined in the same manner and from the same material as the bolt. The clearance between the bolt and the nut was 0.15 mm.

A parting agent, boron, nitride powder dispersed in ethyl ether was sprayed onto the surfaces of the bolt K and the nut L. Discs 11 and 12 were formed in the same manner and from the same material as the bolt and nut. The discs were laid in contact with the upper and lower surfaces of the body formed by the bolt K and nut L. Each disc was 20 mm. in external diameter and 15 mm. in thickness.

Two sheets of parting agents were formed of boron nitride powder and polymethylmethacrylate as binder. These sheets each had a thickness of 0.2 mm. and they were laid between each of the discs and the bolt K. Then, all of the mentioned parts were assembled within a tube-shaped graphite die 2 having an internal diameter of 20 mm. The spaces formed between the internal surface of the die 2 and the head of the bolt K were packed with boron nitride powder. Parts 11, 12, K and L together with the boron nitride packing formed a composite body.

Die 2 supported on a lower punch 22 extending into the die and a pedestal 23 formed of adiabatic brick were together placed inside a silica cylinder 3 having an external diameter of 200 mm. and an internal diameter of 180 mm. the die 2, base 22 and pedestal 23 as well as the upper punch 21 being separated from the silica cylinder 3 by graphite fiber 4, occupying all of the space within the cylinder and surrounding the die and punch. The described assembly was then inserted within another silica cylinder 5, and nitrogen gas was introduced into cylinder 5 through a duct 71 provided in a lower supporting table 7. Air within the cylinder 5 was exhausted through an upper vent 6 by means of a vacuum pump. When the air was completely replaced by nitrogen gas, the vacuum pump was stopped. Nitrogen gas was continuously fed at the rate of 500 cc./min. The described composite body in the die was pressed by the punch 21 at 300 kg./cm.² against the lower punch 22 supported on the pedestal 23 made of an adiabatic brick and was heated gradually and then kept at a temperature of 1800° C. for 30 minutes. After being cooled, the resultant sintered products, bolt K and nut L, were removed and the bolt was easily separated from the nut by unthreading. The external diameter of the screw portion of the bolt was 10 mm. its pitch was 1 mm. and its length was 19.5 mm. The bolt and nut thus obtained closely fitted each other. Under test it was determined that they had sufficient strength to be used at a temperature of 1540° C.

By X-ray diffraction measurement of the bolt and nut, it was determined that nitrogenization of the sintered body had been sufficiently accomplished because little silicon was found to remain in the body. Ordinarily, under conventional treatment, when nitrogen is not introduced, almost all the silicon remains, and the bolt and nut cannot

not be sufficiently sintered. However, under the described inventive method, during the hot-pressing operation, nitrogen gas being introduced in the die, a silicon nitride sintered body having sufficiently high density is obtained. Furthermore, it was determined that the decomposition of silicon nitride was prevented while the silicon nitride was hot-pressed.

From the above it will be seen that in the method of the present invention, gas, moisture, and the like, generated in the sintering powders during the hot-pressing operation can be easily removed, and any gas necessary for sintering can be fed into the powder during the hot-pressing operation. In addition, there is no restriction or limitation of the sintering temperature as in the conventional method due to the latter's use of non-refractory metal, rubber, or the like containers. Therefore, the method of the present invention is highly efficient.

EXPERIMENTS VERIFYING UNIFORM DENSITY AND ACCURACY OF PRODUCTS

The following explains, as illustrated in FIG. 7, the results of experiments made to determine density of products as well as preciseness of dimensions when the inventive method is followed as compared with deviations from the inventive method.

Silicon nitride powder was mixed thoroughly with 5% magnesia powder and then a 2% aqueous solution of polyvinyl alcohol was added. After drying, the material was compressed under 500 kg./cm.² in an isostatic press. The density of the resulting bulk was 1.87 gr./cm.³. A pin-shaped body M, a ring-shaped body N, and a round plate O were machined from the bulk. The core of the pin-shaped body M had an initial length Q_1 of 33.90 mm. a diameter of 20.00 mm. and a flange with an initial thickness R_1 of 11.20 mm. and a diameter of 40 mm. The ring-shaped body N had an outer diameter of 40.00 mm. an inner diameter of 20.10 mm. and a length of 22.70 mm. The round plate O had a diameter of 40.0 mm. and a thickness of 10.00 mm.

A parting agent, boron nitride powder, dispersed in ethyl ether was sprayed onto the surfaces of the pin-shaped body M, ring-shaped body N and round plate O. Said bodies M and N and plate O were combined to form a composite body. Then, the composite body was inserted in a tube-shaped graphite die composed of a cylindrical die 26 having an inner diameter of 40.00 mm. a bottom die 25 and a punch 27. The composite body was sintered at 1770° C. for 30 minutes under a pressure of 100 kg./cm.² in the same manner as described in Embodiment 3. After the sintering, the resulting sintered pin-shaped product was taken out and measured to determine its core length and flange thickness. The measurements were 23.30 mm. as the final length Q_2 and 7.75 mm. as the final thickness R_2 . Then the reduction ratios

$$\frac{Q_1 - Q_2}{Q_1} \times 100, \frac{R_1 - R_2}{R_1} \times 100$$

of said length and thickness were calculated to be 31.2% and 31.0% respectively. These data show that the pin formed as a sintered product has a uniform density throughout all parts.

A second and third pin were formed in the same manner described above, but were sintered under 200 kg./cm.² and 300 kg./cm.², respectively. The resulting second pin had a core length Q_2 of 20.40 mm. and a flange thickness R_2 of 6.70 mm. which similarly were calculated to represent core and flange reduction ratios of 39.7% and 40.1%. The third pin had a measured core length Q_2 of 18.45 mm. and flange thickness R_2 of 6.10 mm. which were calculated to represent core and flange reduction ratios of 45.6% and 45.3%. Thus, it can be seen that the core and flange reduction ratios of each of the pins produced under different conditions are almost identical and, therefore, it is known that each pin produced had a uniform density throughout.

For density comparison with the three pins described above which were formed by the method of the present invention, additional pins were formed from the same green compacts M as the pins mentioned above and in the same manner described above except that their second bodies, ring and round plate O, although of the same size and shape were formed of a different material. Namely, these second bodies were made of a bulk refractory powder consisting of carbon powder having a density of 1.75 gr./cm.³. Thus, in these cases, the pin-shaped bodies M were formed of silicon nitride and the ring N and plate O were formed of carbon. The resultant product pin, formed under 100 kg./cm.² was measured to have a core length Q₂ of 26.90 mm. and a flange thickness R₂ of 7.50 mm., so that the core and flange reduction ratios were calculated to be 20.7% and 33.3%. These greatly different reduction ratios show that the product pin had quite a different density in its core than in its flange.

Similarly, pins made in the same manner as the comparison pins of the preceding paragraph, but formed under 200 kg./cm.² and 300 kg./cm.², respectively, were measured to have a core length Q₂ of 24.60 mm. and a R₂ of 6.60 mm., and a Q₂ of 23.10 mm. and a R₂ of 6.00 mm. respectively. Thus, for the pin formed under the 200 kg./cm.² pressure the core and flange reduction ratios were 27.4% and 41.0%, while for the pin formed under 300 kg./cm.² the core and flange reduction ratios were 31.9% and 46.3%.

These comparisons show clearly that when a different refractory powder is used in the second body from that in the first body the resultant product does not have uniform density. Therefore, it is very difficult and probably not possible to obtain a product having a desired shape and size from a green compact made of one powder and a second body made from a different powder.

Although certain specific embodiments of the invention have been shown and described, it is obvious that many modifications thereof are possible. The invention, therefore, is not intended to be restricted to the exact showing of the drawings and descriptions thereof, but is considered to include reasonable and obvious equivalents.

We claim:

1. A method of producing a precisely dimensioned sintered product by pressing a refractory powder along one direction, comprising,

- (1) forming a refractory powder into a first body of non-uniform dimension along the said one pressing direction and similar to the desired product in shape but elongated at the same fixed ratio for all sections of said desired product parallel to said pressing direction,
- (2) covering said first body with a layer of a parting agent having a higher sintering temperature than that of said refractory powder,
- (3) forming another portion of the said refractory powder into at least one second body which has the same density as that of said first body and which interfits with said first body to form a composite body

having a uniform thickness in said pressing direction,

(4) assembling said first and second bodies into said composite body,

(5) placing said composite body within a die,

(6) compressing said composite body within said die by applying pressing force along said pressing direction of said first body to produce a reduction of thickness amounting to said fixed ratio for all sections of said first body parallel to said pressing direction while subjecting the composite body to the sintering temperature of said refractory powder,

(7) removing the composite body from said die, and

(8) removing the resultant sintered second body from the resultant sintered first body of reduced thickness in said pressing direction to yield said precisely dimensioned sintered product as said resultant sintered first body.

2. A method according to Claim 1, wherein said refractory powder is isostatically pressed to form a bulk having a uniform density throughout all the parts thereof, and said first and second bodies are machined from said bulk.

3. A method according to Claim 2, wherein said composite body is in a shape of a column.

4. A method according to Claim 2, wherein said refractory powder is selected from the group consisting of alumina, magnesia and silicon nitride and said parting agent is selected from the group consisting of boron nitride and graphite.

5. A method according to Claim 4, wherein said parting agent is a solid film composed of a mixture of refractory powder, selected from the group consisting of boron nitride and graphite, and an organic binder.

6. A method according to Claim 1, wherein said refractory powder is dispersed in a slurry and the steps of forming said first and second bodies comprise pouring said slurry into porous moulds capable of absorbing liquid and thereby forming the first and second bodies respectively, removing the resulting first and second bodies out of said moulds, and drying said first and second bodies prior to assembling them into said composite body.

References Cited

UNITED STATES PATENTS

3,717,694	2/1973	Edison	264—332
3,467,745	9/1969	Lambertson et al.	264—332
3,589,880	6/1971	Clark	264—332
2,990,602	7/1961	Brandmayr et al.	264—332
3,230,286	1/1966	Bobrowsky	264—332
3,279,917	10/1966	Ballard et al.	264—332

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