Aug. 5, 1941. B. A. JEFFERY 2,251,454

PREPARING AND MOLDING MATERIAL

Filed April 16, 1935

Fig. 1

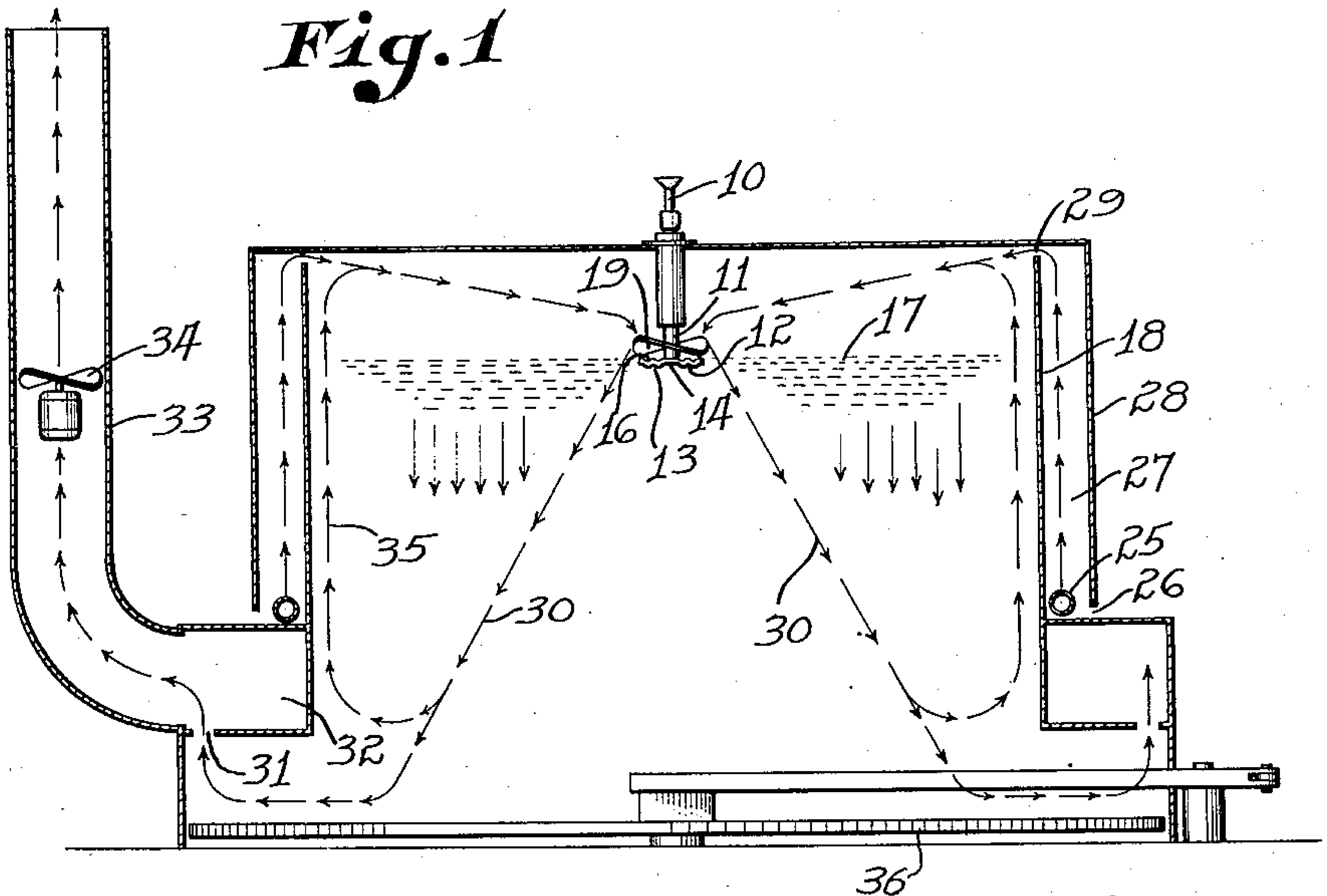


Fig. 2

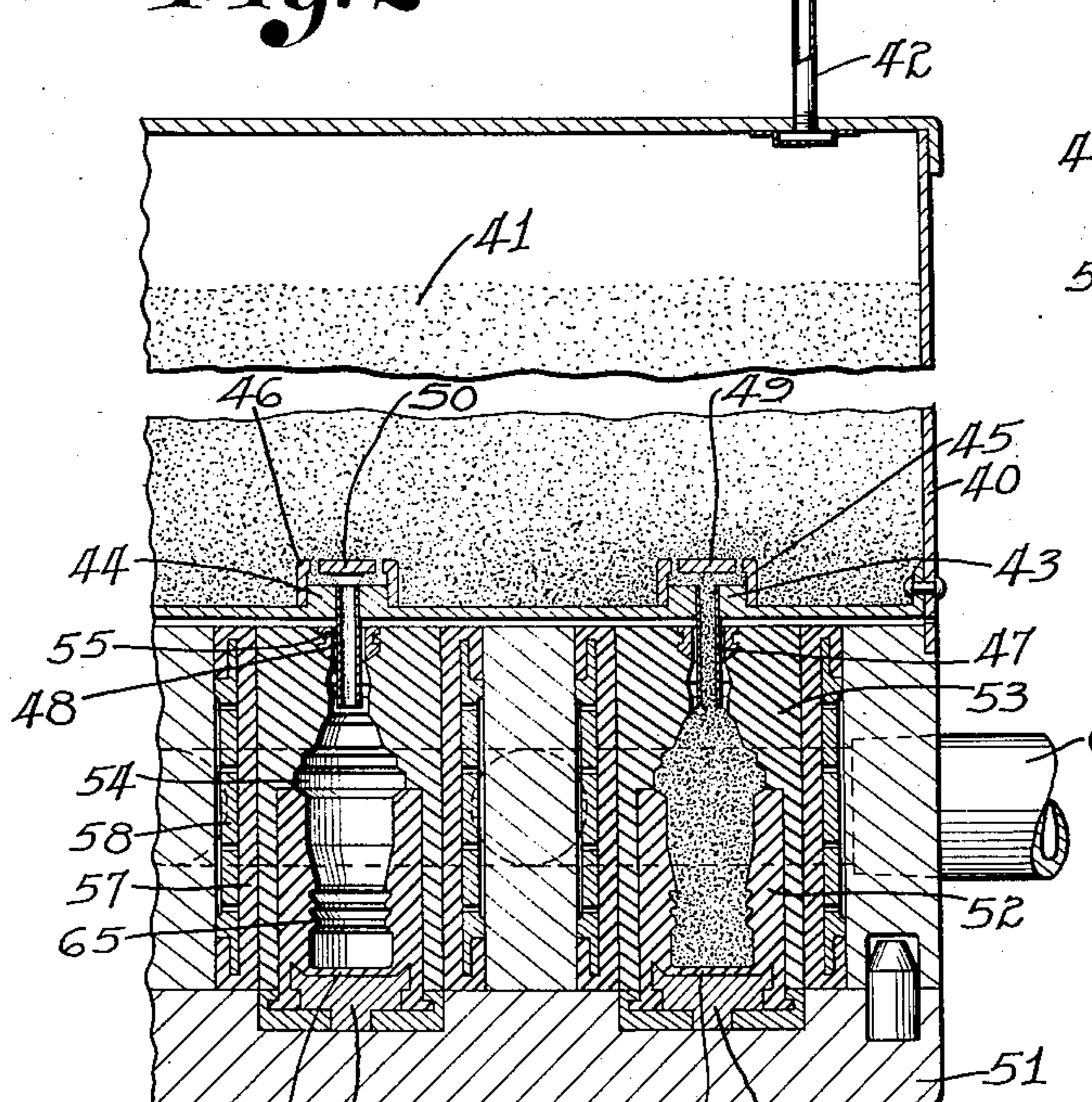


Fig. 3

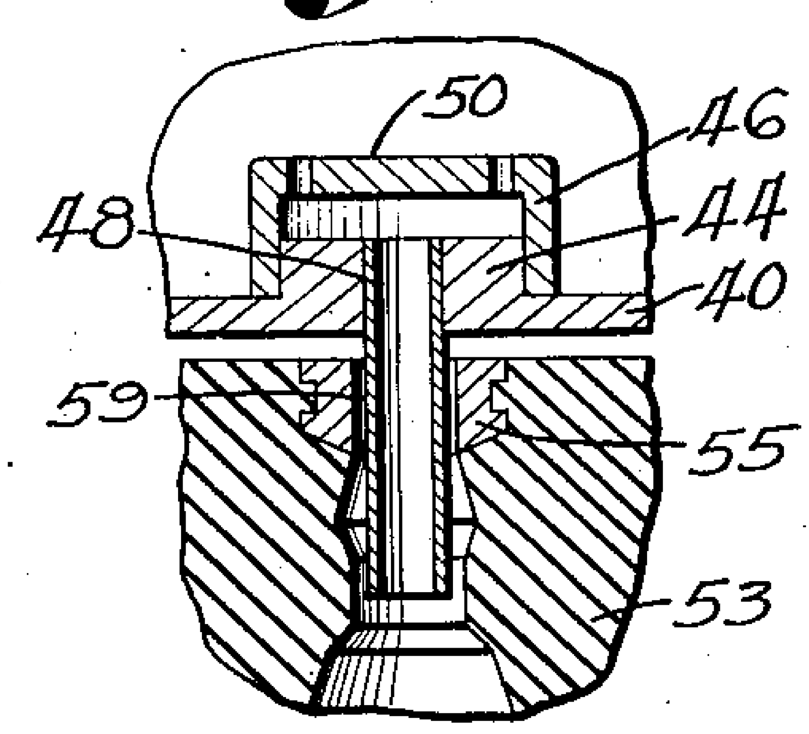


Fig. 4

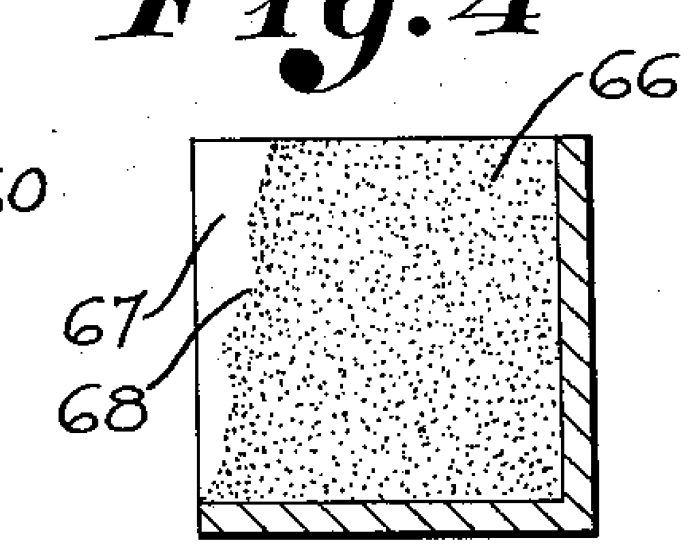


Fig. 5

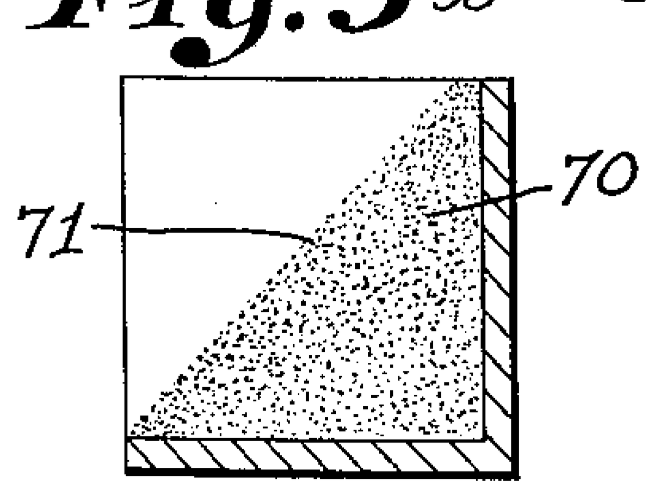
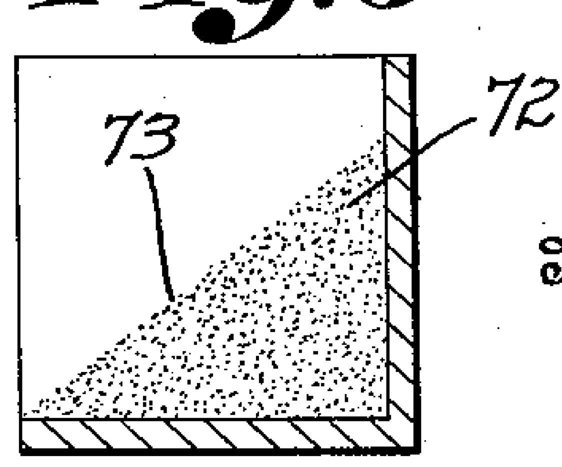


Fig. 6



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Attorneys

Patented Aug. 5, 1941 2,251,454

UNITED STATES PATENT OFFICE

2,251,454

PREPARING AND MOLDING MATERIAL

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Application April 16, 1935, Serial No. 16,601
In Great Britain April 16, 1932

11 Claims. (Cl. 25—156)

This invention relates to preparing and molding material and more particularly to preparing material in rounded pellet form so that it will pour readily and filling material thus prepared into a cavity to give it predetermined shape and then causing the pellets to adhere so as to form a rigid body from the pourable pellets.

The invention has for its object the preparation of the material in such a way that it may be readily filled into a cavity so as to completely fill the cavity and fill it substantially uniformly throughout. The method may be described briefly as consisting in spraying a solidifiable liquid into separated drops of rounded or approximately spherical form, solidifying the separated drops into self-sustaining, non-adherent pellets which form a pourable mass, filling the mass into a cavity of predetermined form and causing the pellets in the cavity to adhere sufficiently to form a rigid mass.

This is a continuation in part of my copending application for "Molding spray dried material," Serial No. 591,531, filed February 8, 1932, and is also a continuation in part of my application for "Method of and apparatus for shaping articles," Serial No. 404,541, filed November 4, 1929, and which eventuated on June 21, 1932, in Patent No. 1,863,854.

It will be readily apparent that this process in its broad aspect may be applied to many different materials, but there will be first described one specific application of the process in the production of porcelain spark plug cores and after the specific disclosure of the method as applied to this purpose, the general scope of possible variations with some specific applications will be more briefly indicated.

The application of the method to the production of spark plug cores will be described in connection with the accompanying drawing, which forms a part of this specification.

Figure 1 is a diagrammatic section of spray drying apparatus;

Figure 2 is a section of one form of apparatus for filling the material into molds;

Figure 3 is an enlarged detail of a portion of Fig. 2; and

Figures 4, 5 and 6 are illustrations of the critical angle of different materials.

In carrying out this particular application of the invention, there is formed a slip comprising ground rock, clay, etc., intimately mixed with water, the ceramic materials being suitable for forming the desired porcelain cores. It is preferable to employ a deflocculating agent, so that the water content of the slip may be low while still retaining the fluidity or pourable condition of the slip. This reduces the amount of water that has to be evaporated, and also results in denser, harder pellets. Sodium silicate is a very effective material for producing this result, but has some tendency, if used in excess, to lower the hot dielectric of the resulting spark plug core, and, therefore, in high duty cores it may be preferable to employ potassium silicate or some other deflocculating agent which does not lower the hot dielectric of the resulting product.

After slip of the character described has been thoroughly mixed, it is preferably screened to remove any particles which might be objectionable, and then introduced into a spraying device. This device, as shown in the drawing, may comprise a hollow stationary tube 10 through which slip may be introduced, and a surrounding rotatable sleeve 11 to the lower end of which there are attached two spaced discs 12 and 13. The lower end of the sleeve is closed at 14 and is adapted to receive the slip from pipe 10. Openings through the sleeve serve to discharge slip from the interior of the sleeve through the space between the discs. Preferably the discs are provided with annular corrugations 16, as shown, by which the slip is thoroughly spread out between the discs and finally discharged in a fine and fairly even spray from the edges of the discs. Wear on the discs may be reduced by facing them with rubber, where contacted by the slip. This spray is thrown outward as indicated at 17, but preferably does not reach the surrounding wall 18. To the sleeve immediately above the discs there is attached a fan 19 which serves to direct air downward and outward adjacent the edges of the discs. The sleeve may be driven by any suitable means. It is preferable to have the delivery tube 10 stationary, so as to avoid segregation of the slip before it reaches the discs.

In the construction shown, there is an annular burner 25 provided around wall 18 and the products of combustion as well as air admitted at the gap 26 flow upwardly from the burner through an annular space 27 between wall 18 and wall 28 and pass through gap 29 above wall 18, and are drawn toward the center of the chamber and forced downward by the fan. Thereafter these gases pass downward to a point adjacent the outer lower wall of the chamber, approximately as indicated by arrows 30, and part of the air then passes through openings 31 into an annular chamber 32 surrounding wall 18 below burner 25. The gases may be drawn off from chamber 32 through an exhaust pipe 33 by a fan 34 or other suitable means. However, the speed of fan 19 is sufficiently high so that the gases are driven downward in the center of the chamber in greater quantity than they are drawn off through the exhaust pipe, and, therefore, considerable quantities of the gas pass upward adjacent walls 18, as indicated by arrows 35, and join the gases coming from the burner and return to the center fan.

The construction and speed of discs 12 and 13 and supply of material thereto is such that spray 17 is in the form of fine drops. All conditions, including force of projection, the diameter of the space within wall 18, and the temperature, moisture content and amount of air or gas introduced for drying purposes are so regulated that the individual drops of the spray are dried into the form of self-sustaining non-adherent pellets before they contact wall 18 or drop to floor 36 of the apparatus.

The powder produced in this way is highly mobile and permeable by gases, since it is composed of tiny self-sustaining balls. Such a powder may be filled into molds readily.

In the construction shown in Fig. 2, there is shown a container 40 for the powder 41. The container is made substantially air tight and is connected by a pipe 42 with a valve, not shown, through which air under pressure may be passed into the top of the chamber or may be allowed to escape into the open air.

The bottom of the container is provided with perforated bosses 43, 44 surrounded by rings 45, 46. Tubes 47, 48 project downward through the bores of the respective bosses. Immediately above tubes 47, 48 there are baffle plates 49, 50, so that the powder must pass through a tortuous course to reach the tube.

A mold carrier 51 may be brought into operative relation with receptacle 40 so that tubes 47 and 48 project downward into molds mounted in the carrier. In the construction shown each mold is composed of a lower part 52 and an upper part 53. The upper part is telescoped over the lower part so that they will divide at the largest diameter 54 of the cavity, which is shaped substantially like the spark plug core which is to be formed. A metal collar 55 is mounted in the mouth or hole portion 53 around the filling tube and a metal plate 56 is embedded in the bottom portion of mold part 52. In the construction shown there is a flexible sleeve 57 surrounding the mold and a perforated wall 58 in turn surrounding the sleeve.

When the mold is not being filled the powder will clog in the outlet, as shown in connection with tube 48. When molds are to be filled, gas under pressure is introduced above the powder through tube 42. Thereupon pressure is exerted upon the material, urging it towards the outlet. This discharging effect is facilitated by the fact that the powder is readily permeable by air so that the air passing through the powder reaches the discharge outlet and carries with it the pellets immediately adjacent the outlet, thus starting a stream of the powder into the mold. While the mold is being filled, air escapes from the upper portion of the mold around the tube, there being an escape passage left as indicated at 59 in Fig. 3.

After the mold has become filled as indicated in connection with filling tube 47, air pressure is released from the receptacle, and the mold carrier and receptacle are separated. During this operation the powder in the tube runs down into the mold and completes the filling of the mouth of the mold, the powder clogging beneath the baffle plates so as to prevent the discharge of material by gravity.

After the mold has been thus filled, a suitable cover plate may be applied and fluid pressure introduced through pipe 60, from which it passes through perforated sleeves 58 to exert pressure upon the molds. These molds are preferably made of rubber which is thick enough to readily sustain itself against gravity and against forces normally exerted upon it by the filling operation, thus maintaining the cavity during the filling in predetermined form; but the construction is of high grade rubber so that, when high pressure is applied to the outside of the mold, the pressure is transmitted through the walls of the mold in substantially the same manner as though they were liquid. In this way the powder within the mold is compressed from all sides with a substantially equal pressure.

With some shapes of articles it may be preferable to construct the mold so that the contraction is along all cross sectional lines of the mold cavity, but in constructing spark plug cores it has been found sufficient to contract the cross section perpendicular to the axis without shortening the axial length of the core materially during the pressing operation. When the pressure is relieved, there is a tendency for the compressed article to expand slightly in all directions, and unless there is a chance for this slight expansion to take place, strain is introduced which may split the article. The thin layer 56[a] of rubber over plug 56 is thickened slightly by the flow of rubber from the side walls of mold part 52 during compression, so as to allow for this slight expansion of the core longitudinally when pressure is relieved. The enlargements of the core are subjected to longitudinal contraction as well as radial contraction and for this reason the normal cavity of the mold into which the powder is filled is not only larger but of somewhat different shape than the finished article, allowance being made for the direction of contraction at each point on the surface of the article.

After the mold and powder therein has been subjected to a sufficiently high pressure to form a compact self-sustaining body, the pressure is relieved and the cover plate separated from the mold, and lower portion 52 carrying the compressed core therein, may be lowered from the upper portion 53. Thereafter the cores may be lifted from the lower portions 52.

In the design shown there are minor grooves 65 in the mold cavity corresponding to similar ridges on the finished core. It will be understood that these are of less depth than the distance through which the walls of the mold are moved during the compressing of the core and, therefore, when pressure is released and the molds return to normal size, the cores may be readily lifted out of portions 52.

It is old to fill ceramic powder into a mold and compress the powder sufficiently to form a self-sustaining article; but hitherto it has been customary to form such powder by drying a batch of proper composition and then grinding and disintegrating the same into power. A powder made in this manner is similar to flour and, when placed in a box and one side of the box removed as indicated in Fig. 4, only a little of the powder 66 drops off of the open side, as indicated at 67, the remaining face 68 being irregular, as indicated. The material has no true critical angle, but avalanches down when loosened.

Ceramic material, prepared by spray drying the slip in the manner described, produces a material such as illustrated at 70 in Fig. 5. When this material is placed in a box and one side of the box opened, the material flows down until it leaves an even face 71 which shows the true critical angle of the material. By introducing a deflocculating agent, such as silicate of sodium, which facilitates the drying and also forms pellets with harder surfaces, the fluent nature of the material can be increased. This is somewhat diagrammatically indicated by the material 72 in Fig. 6 which flows to a critical angle, as indicated at 73, at less than 45°.

The spray dried material is advantageous for filling molds and dry pressing for a number of reasons. It will be readily seen from the foregoing that it can be poured into molds by apparatus which would be totally inoperative with the ground powder. Also, since it is made up substantially of self-sustaining pellets, the voids are determined practically by the relative sizes of those pellets, and where the material is dried under uniform conditions the resulting sizes of the pellets, and consequently the resulting voids, are practically constant for every portion of the dried material, if proper care is taken to avoid segregation of the different sizes during handling. Consequently the same amount of material is readily filled into each mold merely by pouring the mold full, a result which would be entirely impossible with the ground material. Furthermore, since the materials are bound into self-sustaining pellets by the method of formation and each pellet is produced from a drop of a substantially uniform mixture, the resulting material is of uniform composition throughout. The mold being filled with material which is of uniform composition and has uniform voids throughout, a uniform pressure obviously results in a substantially uniform article both as to texture and composition. Furthermore, the definite voids between the pellets allow ready escape of air from the article during compression, and would continue to provide such escape until the voids were substantially destroyed by crushing together the individual pellets. As a result, comparatively little air is trapped in the article, and a compressed core is formed which is more dense than can be formed by equal pressure exerted upon ground powder and, in practice, has been found to be more dense after burning than can be formed in any other way, with the same composition and fired to the same degree. The initial density of the individual pellets, due to the deflocculating agent, is a factor in this final density.

Furthermore, the very uniform composition and texture of the article facilitates the even burning. In practice, the pressure exerted in molding an article to form a self-sustaining body does not need to be sufficiently great to completely crush all of the pellets, and, therefore, certain voids are left in the article, which voids are uniform throughout and facilitate the entrance and escape of gases, especially during the preliminary stages of the firing operation, which also is helpful in securing exactly controlled firing conditions.

While it is recommended that a deflocculating agent be employed, since a mere fraction of 1% of sodium silicate, for example, added to the slip noticeably decreases the critical angle and results in greater ease in filling molds and greater uniformity in results, still it will be understood that in its broader aspects the invention is not confined to the use of such an agent, as any desired composition of slip may be dried in this manner with beneficial results.

The drying method, including the spraying operation, may be conducted so as to control the size or sizes of the pellets within considerable ranges. By proper production of the original spray the pellets may be made larger or smaller and may be confined to fairly uniform size or may be produced in various grades of sizes. For example, larger or smaller drops of spray may be produced in the same apparatus merely by varying the supply of material to the spraying discs or by varying the speed of rotation. Other methods of controlling the range of sizes of the pellets and relative amount of the different sizes relates rather to the question of spray drying than to the broad method, and, therefore, need not be discussed here. It is desired merely to point out that by thus regulating the size of the pellets, or the graded sizes thereof, the texture of the finished article can be varied. For example, by producing graded sizes which fit together to a maximum degree, voids may be greatly reduced and, on the other hand, with pellets of uniform, especially of relatively very large sizes, a comparatively high percentage of voids may be left in the finished article.

The above description has been given particularly with reference to drying slip and forming the resulting powder into spark plug cores, but it will be understood that similar principles may be applied to forming ceramic materials of other kinds with suitable alterations in the composition of the slip and in the shape and operation of the molds. Also, while the preferred method is to employ elastic molds, it will be readily understood that, for some types of articles, it will be sufficient to press or tamp the powder in rigid molds. Also, the pellets might be formed into a rigid body in a cavity in which they remained during use.

The percentage of mechanically combined water does not have as marked effect upon the forming or firing characteristics of spray dried dust as of ground dust, so widely varying percentages can be used. But quite dry pellets can be dry pressed successfully, and are preferred because they form the densest bodies.

It is sometimes desirable to make spark plug cores or other ceramic articles of material that does not contain sufficient clay, or fine particles of colloidal size, to form a good suspension. In such cases, a suitable suspending agent may be employed.

Some materials are difficult to wet with water. In such cases the powdered mass may be wet with water containing a wetting agent. By wetting in this way, each grain is provided with an enveloping film of liquid and air does not cling to the surface, and so may be removed from the mass. After thorough wetting, the excess solution of water and wetting agent may be drained off, and sufficient water added, with a suspending agent if necessary, to make a slip in which the particles will remain suspended sufficiently for forming a uniform mix that can be sprayed and dried into pellets of sufficiently uniform compression.

Ceramic material may vary all the way from that containing sufficient plastic clay to form a good slip without other suspending agents, to powdered alumina, magnesia or the like containing few particles of collodial size and dependent upon a suspending agent to form a slip. In the intermediate stages, sufficient colloidal material of any kind, or any necessary amount of suspending agent, may be added to supplement the natural properties of the material and make a satisfactory slip.

For some purposes, it is desirable to form into pellets a non-ceramic material, for forming into uniform solid bodies. Metal powder may be treated as described above to form a slip and spray dried. Copper powder, or aluminum powder such as is employed in aluminum paint, may be satisfactorily suspended in a 1% aqueous solution of gum tragacanth, after proper wetting. Mixture of powders may be made by mixing the desired proportions into a mixing slip. Any other suitable agent may be employed in place of gum tragacanth.

Instead of adding a suspending agent acting to support the solid particles, in some cases there may be added a reagent that acts upon all or part of the solid particles to facilitate suspension. For example, alumina dust, if sufficiently fine, may be worked into a slip by using a colloid mill or similar methods, or wax, gum, or other suspending agent may be mixed therewith, or it may be treated with hydrochloric acid to facilitate the formation of slip. In one specific instance, alumina fines were treated by mixing with a solution of hydrochloric acid in the proportion of 20 grams of the fines to 9 cc. of water and 3 cc. of hydrochloric acid, allowed to stand heated in a steam bath for about 48 hours, then the surface liquid decanted and the remainder mixed with water to form a slip having a specific gravity of about 2. This slip spray dried well into satisfactory pellets.

In some of the slips described above, part of the material that enters the final pellet is in solution and part in suspension. Obviously, variations may be made both ways, on the one hand to slip that contains in suspension substantially all of the material that remains in the dried pellets, and on the other hand to slip that contains in solution all of the material that forms the final pellets. In either case, drying the separated drops results in solid pellets. For convenience, both in the following portion of the description and in the claims, "slip" will be taken to cover any liquid containing in solution and/or suspension material which, upon spraying into separated drops, and drying of those drops, forms as a residue solid, non-adherent pellets, these being the requisite for forming a readily pourable mass which can be filled into a cavity as a readily pourable mass.

Sometimes it facilitates the formation of the initial bond making a self-sustaining shape to add a bonding agent, such as paraffin, which in small percentages does not interfere with the pourable nature of the mass and yet under pressure binds the particles temporarily sufficiently to form a self-sustaining mass, which may be handled for firing or other treatment.

The invention is primarily directed to the spray drying of slip into a pourable mass and the shaping of such mass, but in its broader aspect it may be applied to liquids that are solidified partly or entirely by means other than the evaporation of the liquid vehicle; for example, liquids may be sprayed into drops and the drops frozen to solid form, or a reaction may be set up or hastened by heat and/or the action of the surrounding gaseous medium to solidify an initially liquid material. In the appended claims the term "solidifiable liquid" is intended to cover any liquid that can be readily solidified as a whole or that leaves a solid residue upon evaporating a portion thereof, or all but the material dissolved or suspended therein.

In the first instance, molten metal or the like can be solidified by spraying into a gaseous medium at ordinary temperatures, while water or the like may be similarly frozen to small hail stones by spraying into a sufficiently chilled space.

While pressing similar to that described is satisfactory for forming at least the initial bond with a wide variety of materials, it will be understood that other means may be employed, singly or in combination, for securing the desired adherence of the particles, and for the broad aspects of the invention the nature of the means for securing adherence is not essential. No attempt will be made to exhaust the list of possible means for securing adherence, because these will be readily apparent from the nature of the material. For example, frozen drops of water could be formed into a rigid mass of a selected degree of porosity by simple pressure, whereas chilled hard metal, such as steel balls, might be made to adhere by electric welding. Heat might be employed, by flowing heated fluid through the porous mass, by heating the walls of the enclosing cavity, by electric current or a combination of these methods to form an initial bond, strengthened by cooling, or to strengthen and render permanent an initial bond, as in ceramic material.

Amongst other uses, carbon or graphite resistors made by compressing powdered carbon mixed with a binder, can be made advantageously by this method and have more uniform electrical resistivity due to the definite control of density that the method offers. Also, by suitably regulating the grain sizes and pressure, various resistances can be produced in pieces of the same size and shape. Obviously, by including more or less metallic powder, the resistance may be cut down to any desired extent. It is one of the clear advantages of this method that powders or materials of different nature can be very evenly distributed through the finished article where it would be difficult or impossible to accomplish this result in other ways.

The production of tungsten carbide articles by this method indicates another class of applications.

Having described specifically an application of the invention and indicated its broader scope and some of its other applications, what I claim is:

1. The method which consists in spraying into separated drops a ceramic slip, drying said separated drops into dense, self-sustaining, normally non-adherent and non-plastic, tiny, rounded pellets, collecting the pellets into a pourable mass of powder, filling the powder into a mold cavity and pressing the powder within the cavity by hydrostatic pressure to form a self-sustaining body.

2. A method which consists in spraying into separated drops a ceramic slip, drying the separated drops into tiny, rounded pellets which form a pourable powder, pouring the powder into a mold cavity, pressing the powder within the cavity to form a self-sustaining body and firing said body.

3. The method which consists in forming ceramic powder into a slip containing a deflocculating agent and a minimum of liquid to make the slip fluent, spraying the slip into separated drops, drying the separated drops into tiny, rounded pellets forming a pourable powder, pouring the powder into a mold, pressing the powder within the mold into a self-sustaining body, and firing the body.

4. The method which consists in mixing powder with a wetting agent and liquid to form a slip, spraying the slip into separated drops, drying the separated drops into tiny, rounded pellets forming a pourable non-plastic powder, pouring the powder into a cavity, pressing the substantially dry powder into a rigid body in the cavity.

5. A method which consists in mixing finely divided ceramic materials of different chemical compositions substantially uniformly throughout a slip, spraying the slip into separate, fine drops, drying the separated drops into rounded, normally non-adherent and non-plastic pellets, pouring the pellets into a mold cavity, compressing the pellets in said cavity by hydrostatic pressure into a self-sustaining body and firing the body.

6. The method which consists in treating different powdered ceramic materials with a wetting agent, mixing the materials uniformly throughout a slip, spraying the slip into separate, fine drops, drying the separated drops into rounded, normally non-adherent and non-plastic pellets, pouring the pellets into a mold cavity, compressing the pellets in said cavity by hydrostatic pressure into a self-sustaining body and firing the body.

7. The method of making spark plug cores and the like which consists in spraying a liquid slip into separated drops, drying the separated drops into self-sustaining, normally non-adherent, rounded pellets, pouring the pellets into a substantially closed mold and uniting them by pressure in the mold to form a rigid body.

8. The method of forming spark plug cores and the like which consists in spraying into separated drops a liquid containing ceramic powder in suspension and material in solution, drying the separated drops to form pellets, the material in solution being adapted to harden the surface of the resulting pellets as it is concentrated at the surface by the drying action, introducing the pellets into a substantially closed mold and uniting them in the mold by pressure to form a rigid body.

9. The method of forming spark plug cores and the like which consists in spraying into separated drops a ceramic slip containing a deflocculating agent, drying the drops, while they are separated, into self-sustaining, normally non-adherent, rounded pellets, introducing the pellets into a mold by pouring, and uniting them within the mold by pressure to form a self-sustaining article.

10. The method of forming spark plug cores and the like which consists in treating powdered ceramic material with a wetting agent, forming the wetted material into a slip, spraying the slip into separated drops, drying the drops while they are separated into self-sustaining, normally non-adherent, rounded pellets, pouring the pellets into a mold, pressing them by hydrostatic pressure in the mold to form a self-sustaining article and firing the article.

11. The method of forming spark plug cores and the like which comprises forming a slip containing ceramic material, a deflocculating agent and a minimum of water to make the slip fluent, spray-drying the material to form self-sustaining, rounded pellets, pouring a charge of the pellets into a cavity of predetermined shape, exerting thereon sufficient hydrostatic pressure to crush at least a portion of the pellets and form a temporarily bonded article, removing the article from the mold and firing the article to form the spark plug core.

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