July 5, 1938. K. SCHWARTZWALDER 2,122,960

REFRACTORY BODY AND METHOD OF MAKING SAME

Filed Jan. 25, 1935 3 Sheets-Sheet 1

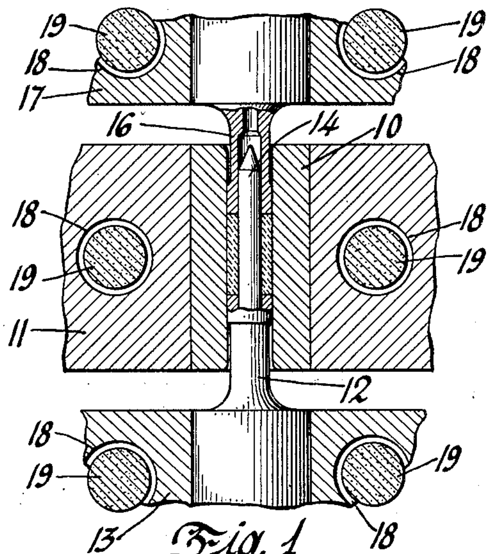

Fig. 1

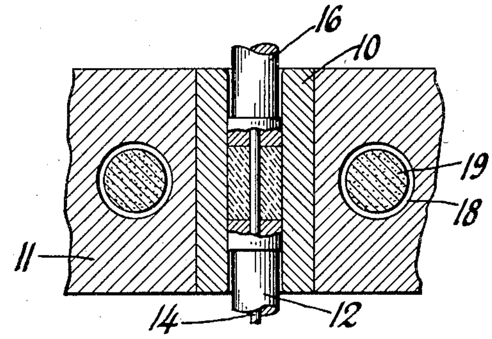

Fig. 2

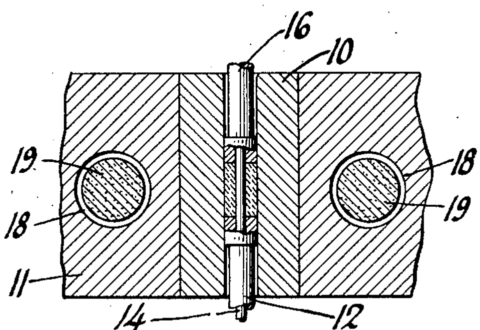

Fig. 3

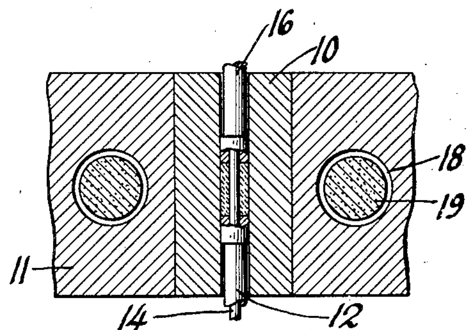

Fig. 4

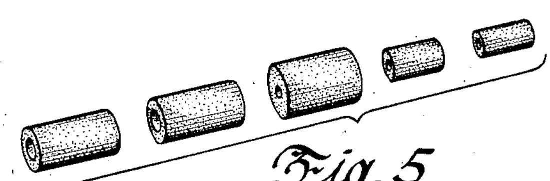
Fig. 5

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REFRACTORY BODY AND METHOD OF MAKING SAME

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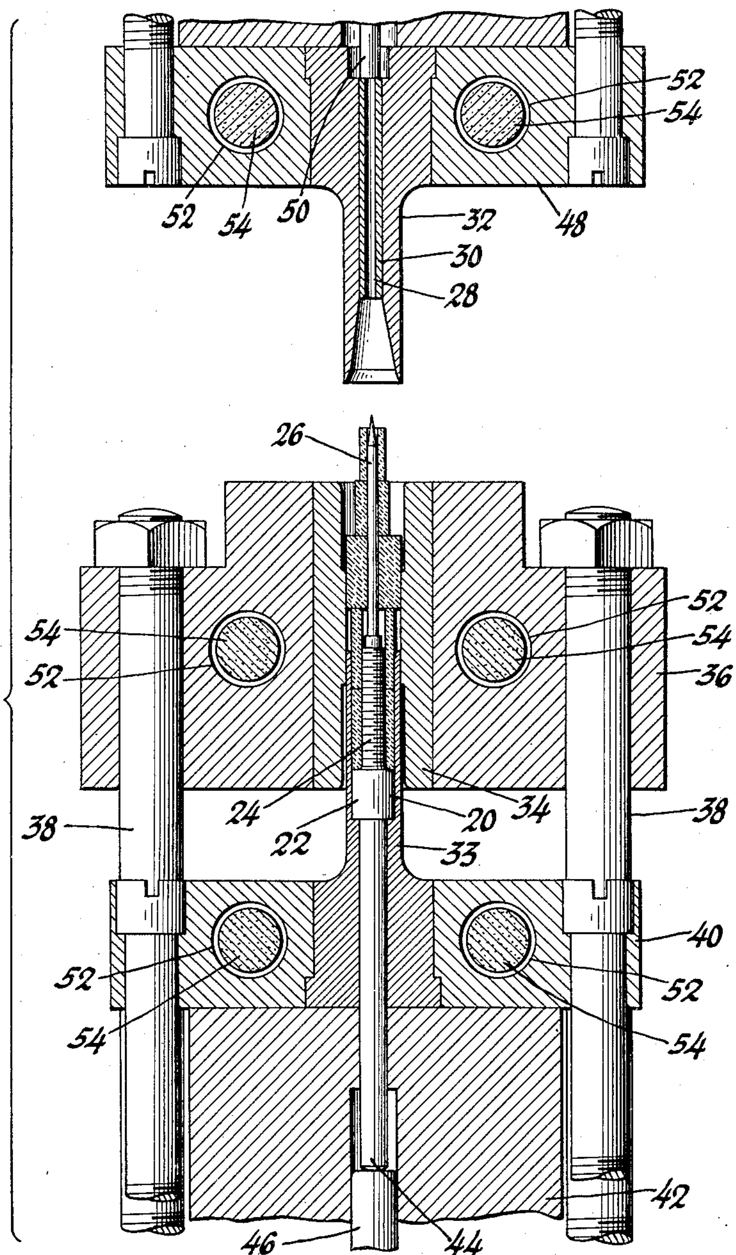

Fig. 6

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REFRACTORY BODY AND METHOD OF MAKING SAME

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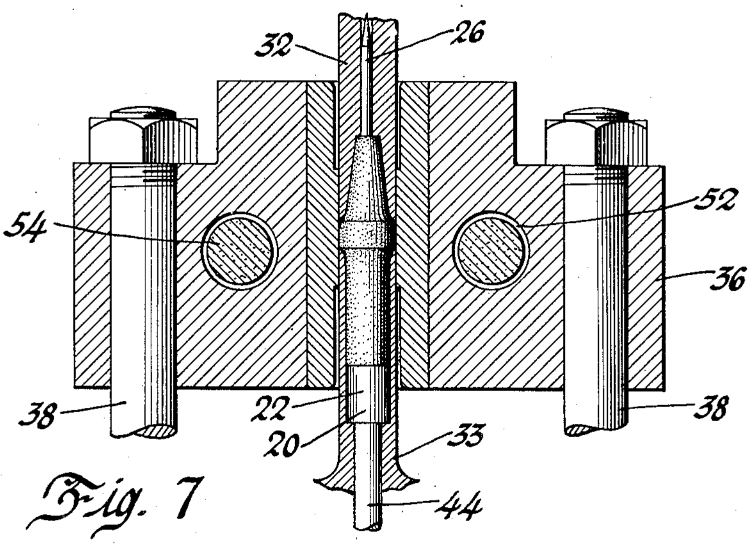

Fig. 7

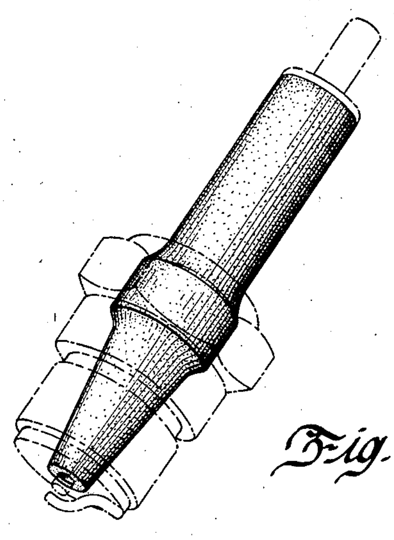

Fig. 8

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UNITED STATES PATENT OFFICE

2,122,960

REFRACTORY BODY AND METHOD OF MAKING SAME

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Application January 25, 1935, Serial No. 3,465

14 Claims. (Cl. 25—156)

This invention has to do with new bodies especially adapted for use as insulators for spark plugs and with methods of making the same. The new insulator material is non-porous, translucent and has a smooth glossy surface which requires no glaze. It is characterized by superior electrical insulating properties even as compared with insulators of the same composition made by other methods. Thus while insulators of alumina made by firing poured bodies,—that is, bodies formed by pouring slip into porous molds,—glow when used in high frequency ignition systems, indicating a partial breakdown of insulating properties, insulators made of the same material by my improved method used in the same system completely insulate the electrodes.

In the case of spark plug insulators the preferred embodiment of the method comprises compressing a material consisting of a mixture of a non-plastic, and a binder, preferably a resin, or mixture of resins, with the addition of a lubricant, into a roughly formed blank or "preform", which is thereafter preferably further compressed between dies into a shape more closely approximating the final article. The insulator shape is then fired in an oxidizing atmosphere to completely burn out the resin or resin-like material and lubricant and the firing is continued to a sufficiently high temperature to recrystallize the non-plastic into a definite-sized crystal structure producing a body that, while retaining its original shape, is somewhat shrunken in size and is non-porous, translucent and possesses a smooth, glossy surface. Microscopic examination shows that the crystals of the final product contain few voids whereas insulators or articles of like composition made by other methods show the presence of a considerable number of voids.

Insulators produced by the new method are extremely dense and translucent. These properties are highly desirable in spark plug insulators since they indicate a definite continuity of structure which should give increased thermal conductivity, more uniform thermal expansion, increased electrical resistance and an increased resistance to thermal shock over insulators of the same chemical composition produced by other methods.

Glazes have customarily been applied to all insulators of ceramic material heretofore used, to prevent adherence of dirt, grease, oil and other foreign substances, as well as to permit of applying trade marks and other ceramic decorations. It has proven somewhat of a problem to provide glazes that will satisfactorily unite with the body composition and give good service. Since the new insulator has a smooth, glossy surface it is not necessary to apply a glaze to it so the various steps required in glazing are eliminated. The surface of the new insulator has all the advantages of a glazed surface with the additional important advantage that it is an integral part of the insulator body. In the manufacture of other ware it will be found possible to dispense with preforms. In this case the prepared material is charged into a suitable mold and formed by application of heat and pressure into the desired shape. The heat employed should be sufficient to soften the resin so as to distribute the forming pressure evenly throughout the body, thereby insuring uniform density throughout. Where thermo-setting resins are employed the application of heat should be continued until the resin sets. Thereafter the article is fired as previously described.

In the drawings:

Figure 1 is a section through a portion of one of the presses showing the method of producing one of the preformed blanks used in making a spark plug insulator.

Figures 2 to 4 are similar sections showing the production of others of the preformed blanks used in making the insulator.

Figure 5 is a perspective view showing all of the preformed blanks used in the production of the final shape.

Figure 6 is a section through the final forming press showing the blanks assembled and in position for forming.

Figure 7 is a section similar to Figure 6 showing the position of parts when forming is completed.

Figure 8 is a perspective view showing the finished insulator as it appears when assembled in the shell.

The first step in carrying out the process consists in the preparation of the raw material.

In general the non-plastic may be any ceramic material which in the fired state meets the requirements for a particular type of ware. The present method, unlike those almost universally employed in the ceramic industry, does not impose any substantial limitation on the non-plastic materials that may be used. Thus the operation of the method is not conditioned upon the existence of such characteristics as plasticity when wet, solubility, grain size, abrasive qualities, etc. Hence with only slight variations the method may be used with a great variety of ceramic materials both natural and synhtetic. While clay has been extensively used in the ceramic industry either as the chief constituent or as a binder to hold bodies together until fired, with the new method it will for the most part be found preferable to employ neither raw nor calcined clay although such materials may be added as a source of their constituents. By "non-plastic material", as used in this specification and in the claims, is meant any material, whether consisting wholly of non-plastics or partly of non-plastics and partly of plastics, possessing insufficient coherence to permit convenient forming by ordinary pressing methods.

In the manufacture of spark plug insulators I have had particular success employing alumina as the non-plastic, as hereinafter described. The use of other refractory oxides such as the oxides of tellurium, thorium, beryllium, magnesium, zirconium, yttrium, and titanium or of refractory compounds such as sillimanite, mullite, and the other minerals of the sillimanite group, is indicated. Other oxides such as vanadium oxide may be found desirable in the manufacture of articles other than spark plugs by the methods herein disclosed. The broad applicability of the method to non-plastics can best be appreciated when it is realized that the temporary binder is substantially completely expelled during the firing of the bodies so that the non-plastic is finally held in shape solely by its own cohesiveness.

The resins or resin-like materials are added to the non-plastic in order to coat the grains or act between the grains so that after the pressing operation the formed body is hard and dense, contributing to the final characteristics of the body as well as rendering it easy to handle.

The resins or mixtures of resins may be either thermoplastic or thermosetting, synthetic or natural, liquid or solid, soluble or insoluble. I have successfully employed synthetic thermo-setting resins such as glyptal (glycerol-phthalic-anhydride) and thermo-plastic resins such as vinylite (vinyly compounds). I have successfully used natural resins such as red and yellow gum accroides, and dragon's blood. The use of various other synthetic or natural thermoplastic resins is indicated. The natural resins may, if desired, be so treated chemically as to enhance their thermoplastic properties. When using thermo-plastic resins it is important that not too much be used so that the softening of the resin on firing the body will not cause the latter to lose its shape before the resin is driven off.

Best results have been obtained by using a thermo-setting phenol-formaldehyde resin. An important advantage arising from the use of thermo-setting resins is the fact that the resin does not soften and tend to flow during firing of the body but volatilizes and oxidizes, leaving behind a firmly pressed mass of non-plastic material in the original molded shape. Plaskon (urea-formaldehyde) as well as other synthetic thermo-setting resins having similar properties may be employed. Natural resins may also be so treated chemically by known methods as to render their action under heat and pressure similar to thermo-setting resins.

Among the resin-like materials with which I have experimented are organic or inorganic-organic compounds such as aluminum stearate, cellulose acetate, and various waxes, but with much less satisfactory results than where resins were used. The term "resinous materials" is used in the specification and claims to cover both the resins and resin-like materials hereinbefore referred to.

Lubricant is added in order to aid in releasing the insulator from the mold after the final pressing has taken place. It forms a coating next to the metal of the mold and so prevents the non-plastic, which may be abrasive, from coming into direct contact with the mold. The lubricant may be an organic compound such as stearic acid, oleic acid, palmic acid, etc., or an inorganic-organic salt of an acid or other materials which have similar action under heat and pressure. The lubricant should have a melting point below the temperature at which softening of the resins begins. The amount to be added is determined by the effect the pore space left by the lubricant on burning has on the final structure of the insulator. Instead of adding the lubricant as a separate ingredient it may be introduced into the mix through incorporation in the synthetic resin during its preparation.

The following is one example of a mixture that has been successfully employed in the manufacture of insulators for spark plugs:

	Per cent
Calcined alumina	88
Bakelite	10
Lubricant	2

The calcined alumina was of low alkali content having been treated with boric acid to remove alkaline impurities in accordance with the process described and claimed in Patent #2,069,060, granted to Albra H. Fessler, on January 26, 1937. The grain size of the major portion of the non-plastic material before agglomerating was not greater than 5 microns. In this application of the invention a thermo-setting resin was found preferable in order to eliminate the heating and cooling cycle characteristic of thermoplastic binders.

The non-plastic, either alone or in combination with the resin or lubricant or both, is preferably agglomerated before molding in order to produce a free flowing, dense material that facilitates filling the preform molds and reduces the compressibility of the loose material to a minimum. This is preferably accomplished by grinding the resin, lubricant and non-plastic material together to the proper grain size, followed by moistening with water, rubbing through a screen of suitable mesh, and drying below 50° C. This maximum drying temperature is maintained so as to avoid setting the resin. According to another method the non-plastic material may be ground with .5 to 1% of furfural followed by addition of the resin and lubricant, after which the material is ground to the proper size. Thereafter the material is moistened and treated as above described in connection with the preferred method. Agglomeration can also be accomplished by first producing a water slip with or without a small percentage of dextrin or similar material and then spray-drying the slip in accordance with known methods; or by drying the slip and then crushing and screening it to the desired agglomerate sizes. If either resin or lubricant, or both, have not been added prior to agglomeration, they may be mixed in dry with the aggregated material. It may be desirable to thereafter tumble the mixture for a short time in order to smear the resin and lubricant on the aggregated material. This aids materially in the final pressing operation.

The next step following the preparation of the raw material is the making of the rough blanks or "preforms". The purpose of the preforming operation is to reduce the reduction in size effected in the final dies. In the case of spark plug insulators the hole for the center wire is made in the preforming dies.

Figures 1 to 4 illustrate diagrammatically the making of preforms. In each figure, 10 indicates a stationary bushing centrally bored and carried by support 11. One end of the bore is closed by the die member 12 mounted in the base 13 and carrying pin 14 providing the aperture for the center wire. In the other end of each bore is fitted the cooperating die member 16 mounted in the movable member 17 of the press and having a central aperture to receive the stationary pin 14.

In operation the lower die member is in the position shown and the upper die member 16 is withdrawn from the bushing 10. The bore is filled with the prepared mixture. Thereafter the upper die member is brought down to the position shown in the figures compressing the material so that its particles adhere. Pressures of the order of 10,000 lbs. per sq. in. will be found satisfactory. Thereafter the upper die member 16 is withdrawn and the lower die member 12 is moved upward, ejecting the blank and at the same time stripping it from the stationary pin 14.

When using thermo-setting resin, such as bakelite, the preforming dies should not be heated as this would set the resin before the bodies are formed to final shape. In the case of thermoplastic resins, it may be desirable to heat the dies. Thus the movable press member 17, support 11 and base 13 may be apertured as at 18 to receive electrical heating elements 19. Or if preferred, steam heating may be employed. However, ordinarily pressure alone will be satisfactory in making the preforms.

The blank shown in Figure 2 is designed for the enlarged central portion of the plug known as the shoulder. The blank shown in Figure 1 is designed for the portion of the plug above the shoulder, called the butt. Two of these preforms will be required for each insulator. The blanks shown in Figures 3 and 4 are designed for the tip of the plug, this being the portion that extends into the combustion chamber.

If desired the preformed pieces may be tapered instead of straight. Tapering the piece, while maintaining the same minimum diameter, increases the volume of material per unit of height and this has some advantages.

The length of the preforms may be varied considerably. Difficulties that may be encountered with longer pieces due to lamination can be overcome by the addition of a small percentage of moisture or furfural to the mix.

The next step consists in assembling on a threaded center wire the number of preforms necessary to form an insulator. Figure 5 shows the preforms in the order in which they are assembled on the center wire while Figure 6 shows them on the wire and in the press. The special center wire 20 is provided with a head 22 connected by a tapered threaded shank 24 to a straight shank 26 extending slightly beyond the last of the preforms encircling it so that it may engage aperture 28 in bushing 30 in the top die member 32. The top die member is of the shape required to form the portion of the insulator that extends into the combustion chamber. 33 indicates the cooperating bottom die member. The bottom die member together with the center wire 20 which fits a recess in it, are formed to give the desired shape to the top of the insulator.

The upper end of the bottom die member 33 fits snugly in a bushing 34 carried in block 36 yieldably mounted on the base of the press by suitable bolts 38 which also pass through the block 40 supporting the bottom die member, and resting in turn upon a suitable support 42. 44 indicates a knock-out pin slidably mounted in an extension of the die cavity in the bottom die member 33. When the pin is actuated by suitable mechanism such as the lever 46, the end of which alone appears on the drawings, it strikes the head of center wire 20, and through the center wire ejects the formed blank from the bottom die.

In like manner the top die 32 is likewise mounted in a block 48 secured in any suitable manner to the moving part of the press, and is also provided with knock-out pin 50 which at the proper time is actuated to force the bushing 30 downwardly to engage the tip of the insulator and release the insulator from the upper die.

Provision is made for heating the dies. Die supporting blocks 48, 36 and 40 are apertured as indicated at 52 to receive electrical heating elements 54. In many cases it will be found preferable to heat the dies by means of steam to reduce the cost of operation.

It will be understood, of course, that with the dies heated, the preforms assembled on the special center wire 20 are inserted in the aperture in bushing 34 in engagement with the bottom die member 33, as shown in Figure 6. Thereafter the upper die member is brought down to the position shown in Figure 7, compressing the preforms into one unitary insulator shape, the heat assisting in even distribution of pressure and in flowing the parts together.

The pressures employed may vary from 25,000 to 100,000 lbs. per square inch, depending upon the character of piece being pressed and the kind of raw material employed. It will be noted that pressure is applied at the butt and shoulder of the insulator as well as the tip in order to obtain uniformity of compression. The body is held under compression i. e., cured, for from one to four minutes depending upon the size and shape of the finished piece.

It will be understood that the special center wire really serves as part of the die, and so assists in distributing the final pressure throughout all parts of the body. It has been found possible to successfully press insulators with center wire holes of less than .050″ at pressures on the order of 100,000 lbs. per square inch.

Following the pressing operation the special center wires are extracted from the bodies while hot, and the insulator is then ready for firing.

Firing may be accomplished by any of the known methods and in any known type of kiln capable of providing the necessary heat treatment. Thus the kiln may be of the continuous or tunnel type or of the periodic type; it may be either of direct-fired or muffle construction.

The firing time, rate of heating and cooling of the ware, etc., must, of course, be selected for best results with the particular body composition being treated in accordance with conventional ceramic practices.

The firing temperature is determined by the characteristics of the non-plastic material. Thus in the case of alumina, temperatures of around 1750° C. are required to recrystallize the materials into one coherent mass. Long before this stage is reached the resin and lubricant have been volatilized and/or oxidized and so driven off by the heat. Careful petrographic study of the fired body reveals no trace of these materials. The firing produces substantial shrinkage. Thus certain bodies shrunk 15.5% in length and 14.5% in diameter. However, the bodies retain their shape within very close limits.

Figure 8 is an enlarged view showing a fired insulator as it appears assembled in a plug. Actually the insulator will be much smaller than the unfired form shown in Figure 7.

The method is well adapted to quantity production. The preforms may be rapidly made by semi-automatic machinery, and will stand rough handling. The final pressing may be rapidly done in multiple cavity molds. Losses due to imperfectly formed insulators have been very small compared with other methods.

The preliminary preparation of the raw material is much simpler than in ordinary ceramic processes. Neither the non-plastic material, nor the resin nor the lubricant require chemical treatment as in casting or grinding (pugging or pressing, etc.) in order to develop plastic properties necessary in other methods of manufacture. The insulator shape is ready for firing immediately after pressing. Since no glaze is required, this entire operation is eliminated.

Spark plug insulators made by the above method employing alumina as the non-plastic material are characterized by dense, homogeneous structure. Individual alumina crystals in nature have small voids or pits in them. Insulators made of alumina by casting methods are characterized by larger voids apparently formed by coalescing of individual crystals. Insulators made by my method have an apparent specific gravity of 3.85, while cast alumina insulators have a specific gravity of 3.65 or 3.75.

The new insulators are characterized by a glossy, homogeneous surface which is apparently due to the finer particles of alumina coming to the surface as a result of pressing. It seems that lubricant squeezed to the surface by the heavy pressure has a tendency to carry with it the finer particles of non-plastic material to form the glossy surface.

Like the cast alumina insulators, the new insulators are practically unbreakable, have excellent resistance to heat shock, high thermal conductivity, and good electrical resistance properties. The new insulators possess all of these properties but in greater degree and with substantial uniformity throughout the same batch, something not always attainable with the cast bodies.

The new method of manufacture offers the possibility of making the insulator of various sections of different non-plastics. In this way each section of the insulator can be made of the material best fitted for it. For instance, an insulator may be formed of an alumina tip and a mullite and glass butt in order to have a tip of high hot dielectric strength and good thermal conductivity while the butt is kept at a lower temperature because it is made of material of poor thermal conductivity. Naturally in such designs the problem of sealing joints between different sections which may be exposed to the compressed gases in the combustion chamber will require attention.

I claim:

1. An unglazed ceramic article of recrystallized non-plastic ceramic material having a smooth, glossy surface and being substantially free of voids rendering it translucent.

2. An unglazed ceramic article made of recrystallized refractory oxide characterized by a dense structure substantially free of voids imparting translucency and having a smooth, glossy surface.

3. An unglazed ceramic article made of recrystallized aluminum oxide characterized by a dense structure substantially free of voids and a smooth, glossy surface, said article having a specific gravity substantially greater than 3.75.

4. The method of making ceramic articles which consists in preparing an intimate mixture of ceramic material and a binder, forming the material into preliminary shape, subjecting the preliminary shape to the combined action of heat and heavy pressure to produce the final shape, and firing the article to eliminate the binder and cohesively unite the ceramic material into a dense structure.

5. The method of making ceramic articles which consists in preparing an intimate mixture of non-plastic ceramic material and a binder, forming the material into preliminary shapes, assembling the number of shapes necessary to form the finished article and subjecting them to heat and to heavy pressure to unite them into the final shape, and firing the article to a degree to eliminate the binder and cohesively unite the non-plastic material into a dense structure.

6. The method of making apertured ceramic articles which consists in preparing an intimate mixture of ceramic material and a bond, forming the material into apertured blanks, assembling the apertured blanks on a spindle, forming the assembled blanks into a unitary structure under heavy pressure, removing the spindle and firing the article to a degree to drive off the bond and cohesively unite the non-plastic material into a dense structure.

7. The method of making spark plug insulators which consists in reducing highly refractory non-plastic ceramic material together with a thermo-setting binder and a lubricant to a fine powder, molding the material into apertured preforms under heavy pressure but with insufficent heat to set the binder, assembling a plurality of preforms on a spindle, molding the assembled preforms under heat and heavy pressure effecting a substantial reduction in the volume of the assembly, setting the binder and forming a durable, compact body, removing the spindle from the body, and firing the body at temperatures sufficient to expel the binder and recrystallize the material into a dense, non-porous structure.

8. The method of making ceramic articles which consists in preparing a mixture of finely ground non-plastic ceramic material, and a resinous binder, forming a body therefrom by application of pressure, reforming said body by application of pressure and heat in sufficient degree to cause the binder to flow thereby distributing the forming pressure throughout the body, permitting the body to harden and firing the hardened body to expel the binder and cause the non-plastic material to sinter together.

9. The method of making composite ceramic articles which consists in pulverizing non-plastic ceramic materials having different properties, mixing each of said pulverized materials with a binder, forming each of the mixtures independently into preliminary shape, assembling a plurality of shapes of different materials to form the finished article, subjecting the assembly to

pressure to unite the preliminary shapes into a unitary assembly, and firing the assembly to drive off the binder and cohesively unite the non-plastic material into a rigid unitary structure characterized by different physical properties in different parts thereof.

10. The process of making articles which consists in preparing a mixture of finely ground non-plastic material and a temporary binder, forming the mixture into the shape of the desired article while applying heat thereto in sufficient degree to cause the binder to flow, thereby distributing the forming pressure evenly throughout the formed body, and firing the body to expel the binder and sinter the non-plastic material into a strong, coherent article of the desired shape.

11. The method of making ceramic articles which consists in preparing a mixture of finely ground ceramic material and a temporary binder, forming a body therefrom by application of pressure and heat in sufficient degree to cause the binder to flow, thereby distributing the forming pressure throughout the body, firing the formed body at a temperature sufficiently high to expel the binder and sinter the ceramic material together into a dense non-porous structure.

12. The method of making articles which consists in preparing an intimate mixture of non-plastic material and a thermo-setting binder, forming the mixture into the shape of the desired article while applying heat thereto in sufficient degree to cause the binder to soften and subsequently set, thereby producing a strong coherent body capable of being handled without likelihood of breakage, and firing the body to eliminate the binder and sinter the material into a strong coherent article.

13. The method of making ceramic articles which consists in preparing an intimate mixture of finely ground non-plastic ceramic material, a thermo-setting phenol-formaldehyde resin and a lubricant, forming the mixture into the shape of the desired article while applying heat thereto in sufficient degree to cause the binder to flow, thereby distributing the forming pressure evenly throughout the formed body, curing the body, and firing the body in an oxidizing atmosphere to eliminate the resin and binder and continuing the firing to sinter the non-plastic material into a strong, non-porous article of the desired shape.

14. The method of making ceramic articles which consists in preparing a mixture of finely ground ceramic material and a thermo-plastic temporary binder, forming a body therefrom by the application of heat and pressure, causing the body to cool and harden to permit convenient handling, and firing the body to expel the binder and cause the ceramic material to cohere into a dense, non-porous structure.

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