

PATENT SPECIFICATION

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(54) IMPROVED CERAMIC ARTICLES

(71) We, WESTINGHOUSE ELECTRIC CORPORATION of Westinghouse Building, Gateway Center, Pittsburgh, Pennsylvania, United States of America, a company organised and existing under the laws of the Commonwealth of Pennsylvania, United States of America, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to ceramic articles and, in particular, to compositions and methods which permit such articles to be made on a mass-production basis at low cost.

Various methods for fabricating different kinds of articles using glass-forming materials, pulverised glass or quartz are known in the prior art.

Generally speaking, the prior art methods and composition were unsatisfactory in that they required high-firing temperatures or long firing times, expensive raw-mix materials and special molds or time-consuming operations which prevented the articles from being efficiently mass produced.

The invention consists in a composition which forms a dense ceramic article when compressed and subsequently fired in air at a temperature of 800°C. to 1100°C for 10 minutes to one hour, said composition consisting essentially of from 25% to 45% by weight of clay, from 25% to 45% by weight of powdered glass having a softening point below 800°C, and from 10% to 25% by weight of at least one of these minerals wollastonite, talc, petalite and potter's flint.

The composition can also contain up to 5% by weight of bentonite and/or up to 5% by weight of an inorganic pigment.

The invention also consists in a ceramic article of dense impervious structure consisting essentially of a composition fired at a temperature of from 800°C to 1100°C for 10 minutes to one hour and, in its raw-mix state, said composition consisting essentially of from 10% to 25% by weight of at least one

of wollastonite, talc, petalite and potter's flint, from 25% to 45% by weight clay and from 25% to 45% by weight of a powdered glass having a softening point below 800°C.

The invention also includes a method of making a ceramic article as defined above, which comprises mixing selected amounts of clay, at least one of the minerals wollastonite, talc, petalite and potter's flint, and powdered glass to provide a finely divided raw-mix composition, placing said raw-mix composition in a mold and subjecting it to a predetermined pressure to form a compact, removing said compact from the mold and heating it to a temperature of from 800°C to 1100°C, and maintaining the compact at said temperature for a period of from 10 minutes to one hour to convert the compact into a hardened ceramic body.

A procedure which is especially suitable for making ceramic insulators of electric lamp bases or similar devices involves mixing from 25% to 45% by weight ball clay, from 10% to 25% by weight wollastonite and from 25% to 45% by weight powdered glass having a softening point below 800°C, preferably powdered soda-lime silicate glass, and forming the compact by subjecting the resulting raw-mix composition to a molding pressure within a range of from 1000 to 7000 pounds per square inch, said compact being subsequently heated to a temperature within the range of from 800°C to 1100°C and maintained at said temperature for a period of from 10 minutes to one hour.

In order that the invention can be more clearly understood, convenient embodiments thereof will now be described, by way of example, with reference to the accompanying drawings in which:

Figure 1 is a side elevational view of an incandescent lamp bulb having a base assembly which includes a preformed insulator component made in accordance with this invention;

Fig. 2 is an enlarged perspective view of the preformed ceramic insulator used in the lamp shown in Fig. 1; and

Fig. 3 is a flow diagram illustrating the various steps in fabricating an article according to a specific embodiment of the invention.

5 While the composition and method of the present invention can be employed to fabricate various types of ceramic articles, the invention is particularly suited for use in connection with the manufacture of base insulator components for electric lamps of the type used for general lighting purposes. As shown in Figure 1, such lamps 10 include the usual glass envelope 12 that protectively encloses a suitable light source such as a tungsten filament (not shown) and has its sealed-neck portion 13 fitted with a suitable base member 14. In the particular embodiment shown, the base member 14 comprises a threaded metal shell 15 and a preformed ceramic insulator 16 that is mechanically locked to the shell 15 by a peripheral indent 17 and has a metal end cap 20 which is force-fitted onto the end of the insulator and serves as one of the lamp terminals.

25 As will be noted in Fig. 2, the ceramic insulator 16 is of button-like configuration and has a flat rim portion 18 and a protruding frusto-conical medial portion 19 which merges with a cylindrical-shaped boss 21 that has a tapered peripheral edge. A centrally-located aperture 22 in the boss 21 permits one of the lead-wires of the lamp 10 to be threaded through the insulator 16 and connected to the end contact 20 during the basing operation.

35 In accordance with the present invention, the raw ceramic insulator (or other article) is formed from a raw-mix composition that contains selected amounts of clay, powdered glass and a refractory mineral (wollastonite, talc, petalite or potter's flint) as essential ingredients. While various types of clay can be used, ball clay is preferred and excellent results have been obtained with a ball clay marketed by the Kentucky-Tennessee Company under the trade designation "Old Mine #4". Ball clays are well known in the ceramic art and are classified according to their properties and origin, as disclosed in the article entitled "Ball Clay Classification" beginning at

page 59 in the July 1970 issue of "Ceramic Age".

Wollastonite is calcium silicate (CaSiO_3), occurs naturally in the form of white-colored fibrous masses and is named after W. H. Wollaston, an English chemist who died in 1828. It melts at 1540°C .

Talc is also a naturally-occurring mineral, specifically, $\text{H}_2\text{Mg}_3(\text{SiO}_3)_4$, which is hydrous magnesium silicate (melting above 1400°C). It is a soft green-to-grey colored material and different varieties are known in the art as potstone, soapstone, steatite, etc.

Petalite is lithium-aluminum silicate, chemical formula $\text{LiAl}(\text{Si}_2\text{O}_5)_2$. It has a melting point of about 1400°C and occurs in nature in the form of colorless or white (and sometimes reddish or greenish) foliated cleavable masses or monoclinic crystals.

Potter's flint is ground sandstone and quartzite and is thus not a true flint. It does, however, contain large amounts of SiO_2 .

While various types of scrap glass can be pulverized and used in the raw-mix composition, a glass which has a low softening point (below about 800°C) is preferred since this will reduce both the firing temperature and time and enable the articles to be quickly produced on a mass-production basis—an essential requirement in the case of the lamp bases. Excellent results have been obtained with glass cullet—that is, the scrap glass produced in electric lamp factories when the filament-mount is sealed to the end of the glass bulb and the excess bottom segment or "cullet" portion of the bulb is removed. Glass cullet is also produced in glass plants which make tubing for fluorescent lamps and large quantities are thus readily available at very low cost.

Technically speaking, glass cullet is soda-lime silicate glass which has a softening point of approximately 693°C and a working range of approximately 1000°C . The chemical composition of this type glass and the ranges of the various constituents, as well as the specific amounts used in a conventional glass of this type employed in the lamp industry for lamp bulbs is given below in Table I, the amounts being expressed in percentages by weight as calculated from the batch.

TABLE I

	Constituent	Amount (Percent by Weight)	Bulb Glass (Percent By Weight)
105	SiO_2	60—75	72.5
	Na_2O	5—18	16.5
	CaO	4—13	5.3
	MgO		3.7
110	Al_2O_3	0.5—3.4	1.7
	K_2O	0.1—2	0.18

Small amounts of As_2O_3 or Sb_2O_3 can also be included as fining agents if desired.

While soda-lime silicate glass is preferred since it is also used in making bottles and other containers and is readily available as scrap glass, other low-melting point glasses such as well known lime or lead glasses can also be used.

A small amount of bentonite can also be included in the raw-mix composition. Bentonite is magnesium-aluminum silicate.



—a naturally-occurring clay-like mineral that

is named after the Ft. Benton formation in the upper Missouri Valley.

A small amount of an inorganic pigment can also be included in the raw-mix to provide a distinctive body color. For example, a very small amount of cobalt oxide will provide a blue ceramic body, manganese dioxide will produce a greyish color, and iron oxide will provide a reddish body color. The natural color of the composition, after firing, is a pleasing milky-white.

The permissible ranges of the various constituents and a preferred composition are given below in Table II.

TABLE II

Raw Mix Composition

Constituent	Range (% by Weight)	Preferred (% By Weight)	Batch Weight (grams)
Powdered Soda-Lime			
Silicate Glass	25—45	40	40
Ball Clay	25—45	40	40
Wollastonite	10—25	16	16
Bentonite	0—5	4	4
Inorganic Pigment	0—5	—	4

While wollastonite is listed in the above table, it will be understood that it can be replaced by the same amount of talc, petalite or potter's flint and mixtures of these minerals (which mixtures could also include wollastonite).

As indicated in the flow diagram depicted in Fig. 3, the glass cullet or other scrap glass is first crushed and ball-milled. As a specific example, the glass cullet is crushed into particles that pass through a 10 mesh screen, a one-gallon ball mill is charged with 1200 grams of the glass particles and 1000 cc. of isopropyl alcohol, and the mixture is milled for 24 hours. The alcohol is then evaporated—thus producing a powdered glass that has an average particle size of about 5 microns, as measured by a Fisher Subsieve Sizer.

Formula weights of the various constituents are then weighed out in 200 gram batches and placed into a one quart ball mill together with 250 cc. of isopropyl alcohol. This mixture is milled for approximately four hours and the alcohol is evaporated off to provide the raw-mix composition which is used for the dry pressing or molding operation. A measured quantity of this composition (for example, between 2 and $3\frac{1}{2}$ grams in the case of a medium-screw type base insulator such as that shown in Figs. 1 and 2) is placed in a hardened steel mold and subjected to a pressure in a range of from about 1,000 to 7,000 pounds per square inch. The resulting green compact is then placed in a furnace and fired

in air at a temperature above the softening point of the glass for a sufficient time to melt or fuse the powdered glass and convert the compact into a hard ceramic article having a very dense impervious structure.

In the case of the raw-mix composition given in Table II which contains powdered soda-lime silicate glass, the green compacts should be fired at a temperature above 800°C and preferably at a temperature in the range of between about 1000°C and 1050°C for from 10 minutes to one hour. Of course, the density and porosity of the finished ceramic article will vary depending upon the particular raw-mix formulation, the molding pressure, and the firing time and temperature which are used.

For the preferred raw-mix formulation given above in Table II, the optimum molding pressure is about 5000 pounds per square inch and the optimum firing schedule is a 15 to 45 minute bake at a temperature of between about 1000°C and 1050°C . Ceramic bodies prepared in this manner have a pressed density of about 1.5 grams per cc., a density of about 1.9 grams per cc. after being fired for 40 minutes at 1050°C , an absorption that ranged from about 0.5% to 6%, and a shrinkage factor of from about 8% to 12% based on their "as-pressed" dimensions.

To facilitate the dry-molding operation and provide stronger pressed compacts which can be handled readily without breaking, a suitable binder-lubricant material is desirably added to the raw-mix composition after the ball

milling operation has been completed. As a specific example, excellent results have been obtained by blending into the raw-mix prior to press molding 2½% by weight of a wax emulsion type binder-lubricant which is marketed by the Mobil Oil Company of petroleum distillation. Of course, this material "burns out" of the compact during the firing operation and is not present in the finished ceramic body.

Removal of the pressed compact from the mold can be facilitated by blending prior to press moulding ¼% by weight of zinc stearate into the raw-mix composition after the aforementioned binder-lubricant has been admixed. The zinc stearate also inhibits excessive wear of the molding dies and thus extends their useful lives.

WHAT WE CLAIM IS:—

1. A composition which forms a dense ceramic article when compressed and subsequently fired in air at a temperature of 800°C to 1100°C for 10 minutes to one hour, said composition consisting essentially of from 25% to 45% by weight of clay, from 25% to 45% by weight of powdered glass having a softening point below 800°C, and from 10% to 25% by weight of at least one of the minerals wollastonite, talc, petalite and potter's flint.

2. A composition according to claim 1 which also contains up to 5% by weight of bentonite.

3. A composition according to claim 1 or 2 which also contains up to 5% by weight of an inorganic pigment.

4. A composition according to claim 3, wherein the inorganic pigment is cobalt oxide, manganese oxide or iron oxide.

5. A ceramic article of dense impervious structure consisting essentially of a composition fired at a temperature of from 800°C to 1100°C for 10 minutes to one hour, in its raw-mix state, said composition consisting essentially of from 10% to 25% by weight of at least one of wollastonite, talc, petalite and potter's flint, from 25% to 45% by weight of clay and from 25% to 45% by weight of a powdered glass having a softening point below 800°C.

6. An article according to claim 5, wherein the raw mix also includes up to 5% by weight of bentonite.

7. An article according to claim 5 or 6, wherein the raw mix also includes cobalt oxide, manganese oxide or iron oxide.

8. An article according to claim 5, 6 or 7, wherein the powdered glass is lime glass, lead glass or soda-lime silicate glass.

9. An article according to any of claims 5 to 8, wherein the composition, in its raw-mix state has a 10% to 25% by weight content of wollastonite, and a 25% to 45% by weight content of ball clay, said article being a ceramic insulator for an electric lamp base or similar device.

10. A method of making a ceramic article as claimed in claim 5, which comprises mixing selected amounts of clay, at least one of the minerals wollastonite, talc, petalite and potter's flint and powdered glass to provide a finely divided raw-mix composition, placing said raw-mix composition in a mold and subjecting it to a predetermined pressure to form a compact, removing said compact from the mold and heating it to a temperature of from 800°C to 1100°C, and maintaining the compact at said temperature for a period of from 10 minutes to one hour to convert the compact into a hardened ceramic body.

11. A method according to claim 10, wherein the raw-mix composition is pressed into a compact while the composition is in a dry state.

12. A method according to claim 10 or 11, wherein the raw-mix composition consists essentially of a mixture of from 25% to 45% by weight ball clay, from 10% to 25% by weight wollastonite, and from 25% to 45% by weight of pulverized scrap glass in the form of lime glass, lead glass or soda-lime silicate glass, the compact being formed by subjecting the raw-mix composition to a moulding pressure within a range of from 1,000 to 7,000 pounds per square inch.

13. A method according to claim 12 wherein a predetermined minor amount of a binder-lubricant material is added to and blended with the dry raw-mix composition, a predetermined minor amount of zinc stearate is also added to and blended with said dry raw-mix composition, both additions being effected before the press-molding operation.

14. A method according to claim 13, wherein the amount of zinc stearate is 0.5% by weight.

15. A method according to claim 13, wherein a wax emulsion type binder lubricant is used and in an amount of 2.5% by weight.

16. A method according to any of claims 10 to 15, wherein up to 5% by weight of bentonite and/or up to 5% by weight of an inorganic pigment is added to and blended with the raw-mix composition before the press-molding operation.

17. A method of making a ceramic article substantially as described herein with particular reference to Figure 3 of the accompanying drawings.

18. Ceramic articles when made by a

milling operation has been completed. As a specific example, excellent results have been obtained by blending into the raw-mix prior to press molding $2\frac{1}{2}\%$ by weight of a wax emulsion type binder-lubricant which is marketed by the Mobil Oil Company of petroleum distillation. Of course, this material "burns out" of the compact during the firing operation and is not present in the finished ceramic body.

Removal of the pressed compact from the mold can be facilitated by blending prior to press moulding $\frac{1}{2}\%$ by weight of zinc stearate into the raw-mix composition after the aforementioned binder-lubricant has been admixed. The zinc stearate also inhibits excessive wear of the molding dies and thus extends their useful lives.

WHAT WE CLAIM IS:—

1. A composition which forms a dense ceramic article when compressed and subsequently fired in air at a temperature of 800°C to 1100°C for 10 minutes to one hour, said composition consisting essentially of from 25% to 45% by weight of clay, from 25% to 45% by weight of powdered glass having a softening point below 800°C , and from 10% to 25% by weight of at least one of the minerals wollastonite, talc, petalite and potter's flint.

2. A composition according to claim 1 which also contains up to 5% by weight of bentonite.

3. A composition according to claim 1 or 2 which also contains up to 5% by weight of an inorganic pigment.

4. A composition according to claim 3, wherein the inorganic pigment is cobalt oxide, manganese oxide or iron oxide.

5. A ceramic article of dense impervious structure consisting essentially of a composition fired at a temperature of from 800°C to 1100°C for 10 minutes to one hour, in its raw-mix state, said composition consisting essentially of from 10% to 25% by weight of at least one of wollastonite, talc, petalite and potter's flint, from 25% to 45% by weight of clay and from 25% to 45% by weight of a powdered glass having a softening point below 800°C .

6. An article according to claim 5, wherein the raw mix also includes up to 5% by weight of bentonite.

7. An article according to claim 5 or 6, wherein the raw mix also includes cobalt oxide, manganese oxide or iron oxide.

8. An article according to claim 5, 6 or 7, wherein the powdered glass is lime glass, lead glass or soda-lime silicate glass.

9. An article according to any of claims 5 to 8, wherein the composition, in its raw-mix state has a 10% to 25% by weight content of wollastonite, and a 25% to 45% by weight content of ball clay, said article being a ceramic insulator for an electric lamp base or similar device.

10. A method of making a ceramic article as claimed in claim 5, which comprises mixing selected amounts of clay, at least one of the minerals wollastonite, talc, petalite and potter's flint and powdered glass to provide a finely divided raw-mix composition, placing said raw-mix composition in a mold and subjecting it to a predetermined pressure to form a compact, removing said compact from the mold and heating it to a temperature of from 800°C to 1100°C , and maintaining the compact at said temperature for a period of from 10 minutes to one hour to convert the compact into a hardened ceramic body.

11. A method according to claim 10, wherein the raw-mix composition is pressed into a compact while the composition is in a dry state.

12. A method according to claim 10 or 11, wherein the raw-mix composition consists essentially of a mixture of from 25% to 45% by weight ball clay, from 10% to 25% by weight wollastonite, and from 25% to 45% by weight of pulverized scrap glass in the form of lime glass, lead glass or soda-lime silicate glass, the compact being formed by subjecting the raw-mix composition to a moulding pressure within a range of from 1,000 to 7,000 pounds per square inch.

13. A method according to claim 12 wherein a predetermined minor amount of a binder-lubricant material is added to and blended with the dry raw-mix composition, a predetermined minor amount of zinc stearate is also added to and blended with said dry raw-mix composition, both additions being effected before the press-molding operation.

14. A method according to claim 13, wherein the amount of zinc stearate is 0.5% by weight.

15. A method according to claim 13, wherein a wax emulsion type binder lubricant is used and in an amount of 2.5% by weight.

16. A method according to any of claims 10 to 15, wherein up to 5% by weight of bentonite and/or up to 5% by weight of an inorganic pigment is added to and blended with the raw-mix composition before the press-molding operation.

17. A method of making a ceramic article substantially as described herein with particular reference to Figure 3 of the accompanying drawings.

18. Ceramic articles when made by a



method according to any of claims 10 to 16. Figures 1 and 2 of the accompanying drawings. 5

19. Ceramic articles substantially as described herein with particular reference to

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