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CERAMIC INSULATOR AND METHOD OF
MAKING THE SAME

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My invention relates to ceramic objects and more particularly to insulators such as electrode spacers of coherent alumina for use in high voltage, high frequency electron discharge devices.

Manufactured ceramic insulators such as spacing members between high voltage parts in electron discharge devices, in which low shrinkage and electrical losses are desirable, are commonly made of oxides of metals such as powdered alumina (Al_2O_3) or magnesia (MgO) mixed with a binder and fired for several hours at high temperatures to vitrify the particles of the mixture. It has been found that the prolonged period of firing necessary to harden the mass to the required degree usually produces an insulator with rather poor electrical characteristics and high linear shrinkage. Changes in firing schedule and proportions of the oxides and binder have not heretofore produced an insulator with the mechanical strength, low shrinkage and good insulating properties necessary in electron discharge devices operating with high voltages at high radio frequencies.

An object of my invention is to provide a ceramic insulator which is mechanically strong, has good insulating properties and low linear shrinkage during manufacture.

Another object of my invention is to provide a ceramic insulator which has good mechanical and electrical characteristics for use in electron discharge devices operating with high voltages at high radio frequencies, and which is easy and inexpensive to make.

The high voltage gradients, of the order of 3000 to 4000 volts per inch, and ultra high frequencies of the order of 10 to 20 megacycles, imposed by the modern radio art upon vacuum tubes cause undue losses in the usual ceramic insulators and spacers made of natural soapstone, known as lava or "lavite", of clay and steatite products, or of metallic oxides. The usual spacers are made of various combinations of these products and are commonly powdered, mixed and pressed or molded into the desired shape and fired at high temperatures to solidify and strengthen the formed bodies. These insulators require such a firing schedule to obtain the necessary strength that the particles of the compounds in the body are substantially vitrified and produce on the surface of the bodies a smooth glassy layer that provides short leakage paths over the surface of the body and appears to cause, due to its imperviousness, difficulty in degasifying the insulator in evacuated envelopes. Such insulators are inefficient at the higher fre-

quencies, causing considerable heating of the insulator between insulated energized electrodes. The efficiency of an insulating material at a given frequency is determined by the relation of the leakage resistance through a unit cube of insulation and the capacitive reactance between A. C. energized plates on opposite faces of said cube and may conveniently be designated as percentage power factor measured by determining the trigonometric tangent of said resistance and reactance.

I make the bulk of my insulator body of finely ground particles of alumina (Al_2O_3). While many binders have been suggested and used in the prior art, none have to the best of my knowledge been suggested which will bind together the particles of the insulator material to make a mechanically strong and low electrical loss body and at the same time leave the body sufficiently porous and non-vitrified to de-gas easily during exhaust. I propose, according to the characteristic features of my invention, to add two binding materials to my insulator which apparently, with proper heat treatment, cooperatively react to bind by surface fusion the interfaces of the particles of the main body. The two binding agents which I add to my insulator have been found to so interlock the particles of the insulator without vitrifying those particles that the finished body has extraordinary mechanical strength, is so porous that the gases in the spaces or voids between the particles may be readily removed, and has exceptionally high efficiency for all frequencies up to about 100 megacycles.

Powdered aluminum oxide, commercially known as alumina, as the main constituent of my insulating material, is combined with small percentages each of powdered acid magnesium meta-silicate, commercially known as talc, and silicon dioxide introduced as binding agents. The alumina, preferably the grade commercially known as "bauxite ore concentrated, special purity" and containing less than .02% alkali, is preferably calcined one hour at from 1500 to 1600° C., ball-milled until 90% of the material is finer than 2 microns in diameter and screened through a standard 100 mesh screen. The talc, commercially known as U. S. P. talc, and neutral to litmus, is powdered and screened through a 325 mesh sieve, and the silica is preferably air-floated to a particle size corresponding to 325 or finer mesh screen, and containing less than about .10% impurities. Good results have been obtained by combining these three powders in the proportion; 90% alumina, 6% talc, and 4%

silica. A slip is conveniently prepared by adding 300 grams of the mixed powder to 250 cubic centimeters of carbon tetrachloride and 18 grams of organic binder material, such as domestic paraffin, in a 1¼ liter porcelain ball mill containing 1000 grams of ¾ inch flint pebbles. To insure complete and uniform coating of the particles of the aggregate with a film of paraffin, the slip is ball-milled for three hours at a speed of 70 to 80 R. P. M., whereupon the slip may be poured from the ball mill and the carbon tetrachloride removed by slowly heating the material in air at about 110° C. for approximately 12 hours. The resulting aggregate, free from carbon tetrachloride may be readily crumbled while slightly warm and screened, preferably through a 40 mesh sieve. The resulting powder may now be pressed in steel molds into the desired insulator shapes by means of a plunger, preferably at a pressure of about 5000 pounds per square inch.

The pressed insulator bodies in their unfired or "green" state are firm and coherent and may be handled without breakage, the particles of the body being held together by their coherent coatings of paraffin. To remove the organic binder, I prefer to fire the insulator bodies in air, as follows: raise temperature of the oven containing the insulators to 220° C. and hold for six hours, then raise temperature to 800° C. and hold at this temperature for 45 minutes, then raise temperature to 1050° to 1100° C. and hold at this temperature for 45 minutes. The insulators may then be removed from the oven after the temperature has dropped to 400° C. or lower. At this stage the material is strong and coherent enough to be drilled or cut if desired.

The insulator body is finally fired and hardened in a hydrogen atmosphere at about 1570° C. for 100 to 140 seconds. An insulator thus produced is exceptionally strong, having a mean modulus of rupture of about 13,000 pounds per square inch as compared to the modulus of rupture of 6,600 pounds per square inch for the usual "lavite" insulator. My improved insulator is not only strong but is porous, much like a porcelain filter, its surface having a uniform satiny white appearance as distinguished from the glassy varicolored surface of the usual "lavite". Further, my insulator is exceptionally efficient at high frequencies, having a power factor of .02% to .10% between .3 and 17 megacycles as compared to .35% to 2.2% for "lavite" in the same range of frequencies, and has a dielectric constant of less than 4 at 25° C., as compared to the dielectric constant of about 6 for the usual "lavite", "Alsimag", and other common metallic oxide insulators.

While 90% alumina has been mentioned with 6% talc and 4% silica, the alumina may be varied within the scope of my invention in its proportion between 88 and 92% of the total and the talc may be varied between 4 and 8%, and the silica varied between 2 and 6%. With these proportions added to the mixture before firing the following proportions of metals after firing will be found by analysis: magnesium .78% to 1.57%, silicon 2.71%

to 4.6% and aluminum 46.7% to 48.8% corresponding to:

	Per cent
MgO-----	1.3 to 2.6
SiO ₂ -----	5.8 to 9.8
Al ₂ O ₃ -----	88.2 to 92.2

Slight variations in these percentages found by analysis will be due probably to varying amounts of impurities.

Electrical insulators prepared with the ingredients in the proportions above specified produce an insulating body that is mechanically strong, has a low linear shrinkage during firing and has extraordinarily high insulating properties. Since the good electric and mechanical characteristics of my insulator may probably be obtained by proportions beyond the limits above specified by the proper adjustment of firing schedule to obtain the partial fusion between the particles in the insulator body, it is desired that my invention be limited only by the prior art and by the terms of the following claims.

I claim:

1. A porous ceramic body of which about 90% by weight is a dense coherent alumina and the remainder is magnesia and silica in the ratio of 1 to 4, said body having a modulus of rupture of about 13,000 pounds per square inch and a power factor of less than .10 per cent at 17 megacycles.
2. An unvitified ceramic body of alumina, silica and magnesia intimately admixed and fired into a dense coherent porous mass consisting of 90% alumina, 8% silica and 2% magnesia by weight, said body having a power factor of .10 per cent at ultrahigh frequencies.
3. A ceramic composed essentially of refractory oxygen compounds of aluminum, silicon and magnesium and containing approximately 48% aluminum, 1.2% magnesium and 3.5% silicon.
4. A ceramic insulating body with a modulus of rupture of about 13,000 pounds per square inch, comprising by weight 88 to 92% alumina, 4 to 8% talc, and 2 to 4% silica.
5. A ceramic insulating body for an electron discharge device, by analysis composed of about 88.2% to 92.2% of aluminum oxide, 5.8 to 9.8% silica and 1.3 to 2.6% magnesium oxide.
6. The method of making a porous ceramic insulator with a modulus of rupture of about 13,000 pounds per square inch for use at high voltages and high radio frequencies which comprises mixing substantially pure alumina particles measuring about 2 microns in diameter, with 4 to 8% powdered silica, and 2 to 6% powdered acid magnesium meta-silicate, pressing the particles under high pressure into intimate contact, and firing in a hydrogen atmosphere at about 1600° C. for a period of about 140 seconds.
7. The method of making a ceramic body which has low electric losses at high radio frequencies which comprises mixing 88 to 92% powdered alumina with 4 to 8% powdered talc and 2 to 6% powdered silica, adding an organic binder, forming by pressing the mixture into the desired shapes, removing said organic binder, and firing in hydrogen at about 1600° C. for a period of 100 to 140 seconds.

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