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METHOD OF MAKING ASBESTOS INSULATING MATERIAL WITH IMPROVED ELECTRICAL PROPERTIES

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Fig. 1.

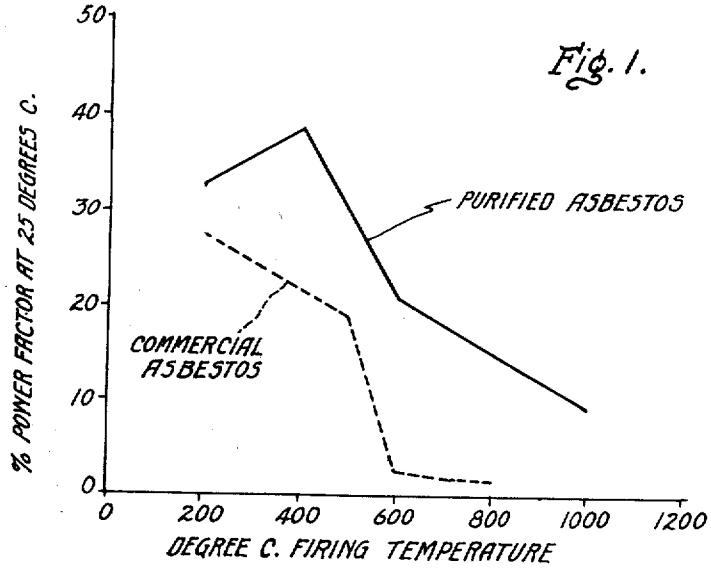
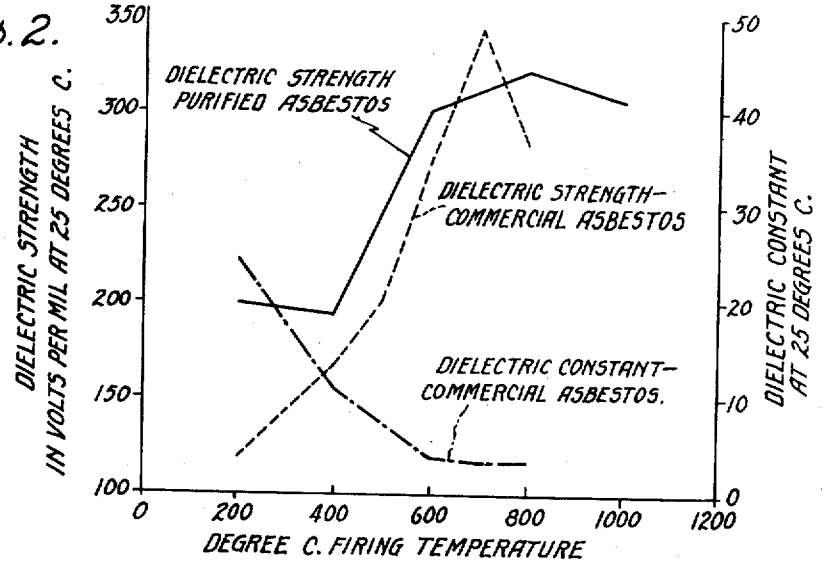



Fig. 2.




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# METHOD OF MAKING ASBESTOS INSULATING MATERIAL WITH IMPROVED ELECTRICAL PROPERTIES

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4 Claims. (Cl. 154—2.6)

The present invention relates to electrical insulating material, and more particularly to insulating material and products composed of asbestos sheet material treated with phosphoric compounds and characterized by improved electrical properties.

It is an object of the present invention to provide phosphoric acid treated asbestos insulating sheet materials and products particularly adapted for high temperature application and having improved electrical properties.

It is a further object of the invention to provide an improved, economical, hard, dense laminated absestos product particularly adapted for high temperature electrical insulation applications and characterized by low power factor, low dielectric constant and high dielectric strength.

A further object of the invention is the provision of an improved method of making insulating asbestos sheet materials and products of the above types.

In the copending application of Spooner, Serial No. 336,885, issued as Patent No. 2,702,068 on February 15, 1955, and assigned to the same assignee as the present application, there is described an aluminum phosphate bonded asbestos insulating material which provides a hard dense laminated product particularly adapted for high temperature electrical insulating applications. In accordance with the present invention the material disclosed by the copending application has been found to be greatly improved by subjecting it to a firing temperature of between 600° to 1000° C., and as a result of such treatment the dielectric strength of the material is markedly increased. The firing apparently drives off not only any organic material present in the paper but also at least a portion of the water of crystallization, leaving a fired product with greatly improved electrical properties as hereinafter shown.

The invention will be better understood from the following description taken in conjunction with the accompanying drawing, in which:

Fig. 1 is a graph showing the power factor of two types of the present fired absestos material in laminated form as a function of the firing temperature; and

Fig. 2 shows the change with firing temperature in dielectric constant and dielectric strength of different types of the present fired asbestos material in laminated form.

The composition and method of making the aluminum phosphate bonded absestos material which is heat treated in accordance with the present invention is described in detail in the above-mentioned copending application, and as disclosed therein the insulating material is obtained by impregnating asbestos sheet material such as asbestos paper with a solution of mono-aluminum phosphate, drying the impregnated material and thereafter forming the impregnated sheet into a laminated structure. The binder used in the laminated structure is a higher aluminum phosphate of the formula $Al_x(H_{3-x}PO_4)_3$, wherein $x$ has a value of from 1.3 to 1.7. The resulting product is a laminated structure in which the individual sheets are



completely impregnated with an aluminum phosphate impregnant and are securely bonded together in the form of an extremely hard, dense product.

Both the mono-aluminum phosphate employed for impregnating the individual sheets of asbestos and the higher aluminum phosphates employed for bonding these individual sheets into a laminated structure are water soluble or water dispersible products which, by removal of all or part of the water, are converted into solid products having excellent bonding and insulating characteristics. In this connection, it is to be understood that the expression "phosphoric acid compound" as used herein is intended to refer to the above aluminum phosphates and the orthophosphoric acid hereinafter described, as well as other phosphoric compounds which decompose under heat in the presence of asbestos to phosphoric acid.

The term "mono-aluminum phosphate" as used in the description of the present invention refers to the aluminum phosphates of the formula $Al(H_2PO_4)_3$ as well as the aluminum phosphates in which the aluminum to phosphate, $PO_4$, molar ratio varies slightly from the 1:3 ratio and may be as high as about 1.2:3. They are water soluble materials which can be employed in the form of relatively low viscosity solutions for the complete impregnation of the asbestos sheet material. They are further characterized by the fact that after removal of the water, in whole or in part, there is obtained an impregnated sheet of good flexibility and high strength which can be readily employed in the manufacture of laminated products.

The aluminum phosphates employed for bonding the impregnated sheet into a laminated structure are those which form a highly viscous aqueous solution having a viscosity which may be 100 times the viscosity of the mono-aluminum phosphate solution. These more viscous products after removal of all or part of the water are converted to relatively hard, resin-like solids and have been found to have excellent bonding action for the mono-aluminum phosphate impregnated sheets.

The absestos sheet material employed in the practice of the present invention may be any of the usually available asbestos sheet materials consisting predominantly of asbestos fibers. The purified asbestos material, as referred to herein, is made of asbestos fibers which have been treated in known manner to remove iron so that the final iron content is less than ½%. The commercial asbestos, as referred to herein, is asbestos material which has not been similarly treated and contains up to 6% of iron. The sheet material may consist entirely of asbestos fiber or it may contain small amounts of cellulose fiber, such as cotton fiber, frequently present in commercial types of asbestos sheets for strengthening purposes. Also, included within the scope of the present invention are asbestos sheets containing minor quantities of bentonite or glass fibers, which sheets are presently employed for electrical insulating purposes. It is not essential in the practice of the present invention that the aluminum phosphate be applied in two steps, i. e., as impregnant and then as a binder, nor in the different forms as described above. If desired, any of the aluminum phosphate compositions described in solutions of suitable viscosity may be applied to one or more asbestos sheets which can then be subjected to sufficient pressure to compact the individual sheets and/or bind the sheets together into a laminated structure, care being taken that the amount of aluminum phosphate impregnant used is sufficient to provide effective binding action between the sheets in forming the laminated structure.

The above types of phosphate compounds and procedures as described are particularly appropriate where it is desired to form laminated structures, for example, those in the shape of wound cylinders or laminated plates. In

making these structures, the asbestos sheets are formed in any suitable or known manner, then impregnated with the aluminum phosphate composition and thereafter shaped into desired form either by winding or stacking.

Instead of the phosphate impregnants described, orthophosphoric acid may be used in combination with asbestos fibers and molded to form a unitary sheet or plate and thereafter fired to a temperature range of 800°–1000° C., in accordance with the invention, to obtain a molded thermoset insulation product with exceptionally improved electrical properties. Due to the rapid reaction of the orthophosphoric acid with the asbestos, this latter modification does not lend itself to the making of laminated structures as well as the aluminum phosphate treated asbestos, where the slower reaction between the phosphate and asbestos permits better control of the binding action of the impregnant in the laminating process. The invention is not limited to any specific orthophosphoric acid-asbestos composition, and heat hardenable products can be obtained for example by using one part by weight of concentrated phosphoric acid to from about 2 to 3.5 parts of asbestos. The optimum ratio will depend to some extent on the texture of the asbestos, i. e., the fiber diameter and length, and the nature of any filler such as zircon which may be included in the composition.

The above-mentioned copending application describes heat treatment of the finished material at temperatures up to 250° C. for decreasing the water content of the laminated material to a desired degree. However, such treatment does not result in effective improvement in the electrical properties of the insulating material. In fact, it has been found that unfavorable results in terms of power factor and dielectric strength may occur on heating in the range of 200°–400° C.

The present invention, on the other hand, which provides for heating the phosphate-treated asbestos material to a temperature range of 600°–1000° C. produces exceptionally marked improvement in terms of lower power factor and increased dielectric strength as well as reduced dielectric constant. It will be seen from Fig. 1 that as the firing temperature is increased the power factor of phosphate treated purified asbestos sheet material in laminated form as measured at 25° C. is substantially increased in the firing temperature range of 200°–400° C. As the temperature is increased further, the power factor of the purified asbestos shows a marked decrease until at about 1000° C. a power factor of about 9.25% is obtained. Even greater is the improvement in power factor achieved in firing phosphate treated commercial asbestos sheet material in laminated form, and as shown in Fig. 1 the power factor of this material treated with aluminum phosphate in accordance with the invention shows a sharp drop in the temperature range between 500°–600°, and at a temperature of between 600°–800° C. a very low power factor of about 1%–4% is achieved.

Likewise, improvements in dielectric strength and dielectric constant of the aluminum phosphate impregnated material were produced by firing at the elevated temperatures. As shown in Fig. 2 with respect to the phosphate impregnated and laminated purified asbestos sheet material, the dielectric strength decreased slightly between 200°–400° C. but it increased sharply thereafter until maximum values of dielectric strength were achieved in the temperature range of 600°–1000° C. In the case of phosphate treated asbestos of the commercial type, the dielectric strength rose with increasing temperature from 200° C., with maximum results achieved from 600°–800° C. Fig. 2 also illustrates the decrease of dielectric constant with increase of firing temperature, this factor also reaching a minimum value between 600°–800° C. As will be understood by those skilled in the art, low dielectric constant is a desirable characteristic for insulating material, in view of the high electrical resistance of insulating material and the tendency of electrical stress to concentrate in low dielectric constant materials, since in this way the greater proportion of electrical stress is exerted on the material better able to withstand it.



The length of firing time at the elevated temperatures to produce the improved electrical properties described and shown will vary depending on the thickness and shape of the material fired. The results illustrated in Figs. 1 and 2 were obtained by firing for 15 hours laminated material having a thickness of about .20–.25". This period represents a maximum firing time beyond which the improvement in electrical properties would be negligible. Material of the same approximate thickness could be fired for a minimum time of about 2 hours to obtain somewhat less improvement in the electrical properties. It is necessary only to provide a sufficiently long firing time to heat the entire mass of material to the elevated temperatures and to drive off the hydrates nad water of crystallization. In general, within the above range of firing periods, the required time of firing will be longer the lower the temperature, and vice versa, to obtain substantially the same improvement in electrical properties of like material of the same approximate thickness. Material of thickness less than .20" could be heated for a period between 1–2 hours at the elevated temperatures to obtain substantial improvement in electrical properties. It is important to note, however, that heating of the material of any thickness at temperatures lower than about 600° C. no matter how prolonged will not produce the improved electrical characteristics obtained in accordance with the present invention.

Tests made on weight loss of the phosphate treated asbestos material at various temperatures between 100°–1000° C. showed three general temperature zones in which different types of losses appeared to have taken place as follows:

*a.* 100–150° C.—free water given off.

*b.* 200–500° C.—organic matter diminished by oxidation or thermal breakdown.

*c.* 600–1000° C.—chemically bound water and hydrates driven off.

Other experimental data have shown that when the phosphate treated asbestos material was heated to 300–600° C., poorer dielectric properties were produced due probably to the carbonization which takes place of the small content of organic material. However, heating at temperatures above 600° C. for a reasonable period causes further oxidation of the carbon and driving off of any residual chemically bound water, thereby producing the improved electrical properties described and shown. The present method of firing asbestos treated with phosphoric acid compounds produces, especially in the laminated types of structure, a strong hard compact fired insulating material and provides in this respect a considerable advantage over the known types of asbestos insulation materials. The present process provides, in addition, for a more complete chemical reaction between the aluminum phosphate and asbestos than obtained by the process of the copending application and insures a more effective bond therebetween. Furthermore, the elevated firing temperature has the effect of insolubilizing the aluminum phosphate and thereby renders the binder more stable under high moisture conditions. Moreover, by virtue of the present invention the phospho-asbestos material is ready for use after firing and requires no further treatment such as impregnation, curing or other processing other than what might be desirable to prevent absorption of free moisture due to porosity of the fired material.

While the products produced by the present invention are useful for various electrical applications, they are particularly suited for use as insulation in dry-type transformers. Without further treatment, the fired laminated materials can be employed in sealed dry-type transformers either as cylinders on which the coils are wrapped, or as flat plates, spacers, end-rings and various forms of volt-

age barriers. Parts made from asbestos sheet material impregnated with orthophosphoric acid as described above are particularly useful as arc chutes for circuit interrupting devices.

It is possible to produce not only molded compositions and products but also laminated products in various shapes and forms, as indicated in the description above. Since the raw materials are relatively cheap the product is comparatively inexpensive and may be easily produced. It will be understood that various modifications may be made by those skilled in the art, particularly in the specific phospheric acid compositions used, without actually departing from the scope of the invention. Therefore, the appended claims are intended to cover all such equivalent variations as come within the true spirit and scope of the invention.

What we claim as new and desire to secure by Letters Patent of the United States is:

1. The method of making a hard, dense electrical insulating product having low power factor, low dielectric constant and high dielectric strength, which comprises impregnating asbestos sheet material within aluminum phosphate superimposing a plurality of layers of the thus treated asbestos sheet material to form laminated structure, and heating the thus formed laminated structure for at least one hour at a temperature of at least 600° C. until hyrates and water of crystallization are driven off therefrom.

2. The method of making a hard, dense, moisture-resistant electrical insulating laminated asbestos product having low power factor, low dielectric constant and high dielectric strength, which comprises impregnating asbestos paper with an aqueous solution of mono-aluminum phosphate, coating the thus impregnated sheet of a solution of aluminum phosphate of the formula $Al_x(H_3-_xPO_4)_3$ wherein $x$ has a value of from 1.3 to 1.7, forming a laminated product from the thus treated asbestos sheet material, and heating the thus formed laminated product to a temperature of between 600°–1000° C. until hydrates and water of crystallization are driven off therefrom.

3. The method of making an insulating product which comprises heating a molded mixture of asbestos fibers and orthophosphoric acid for at least one hour at a temperature of from 800°–1000° C. until hydrates and water of crystallization are driven off therefrom.

4. The method of making electrical insulating material having low power factor, low dielectric constant and high dielectric strength, which comprises heating for at least one hour at a temperature of at least 600° C. asbestos material treated with a compound selected from the group consisting of aluminum phosphate and orthophosphoric acid until hydrates and water of crystallization are driven off therefrom.

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