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MICA PAPER

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This invention relates to the preparation of mica paper. More particularly, this invention relates to sheets of mica paper which are characterized by improved tensile strength and to the process by which these improved products are obtained.

Heretofore, the preparation of mica paper has been known in the art. For example, mica paper preparation is described in Patents 2,549,880—Bardet, 2,614,055—de Senarclens, and 2,709,158—Bouchet. Mica paper prepared by the methods of these patents as well as by other known methods is gaining commercial acceptance because of the excellent electrical properties of the mica paper and because of its inertness at elevated temperatures. However, one serious drawback to the universal acceptance of mica paper for many applications is the fact that the mica paper does not have as high a tensile strength as desired.

It is an object of the present invention to provide an improved mica paper.

A further object of my invention is to provide a mica paper of extremely high tensile strength.

These and other objects of my invention are accomplished by subjecting comminuted mica particles to the action of barium ions in an aqueous barium salt solution and subsequently forming a paper-like sheet from the resulting particles.

The term "mica paper" as used in this application is used in its usual sense to refer to a sheet-like aggregate of mica particles which are usually heated at an elevated temperature, ground into fine particles in an aqueous solution, and then formed into paper by conventional paper-making techniques.

The mica paper of the present invention differs from that of the prior art in that the mica particles have been subjected to the action of barium ions before they are formed into the final sheet.

The mica paper of the present invention can be prepared by firing or heating mica, which may comprise phlogophite, lepidolite, or preferably muscovite, at a temperature and time sufficient to partially "dehydrate" the mica. In general, the heating of the mica is carried out at a temperature of about 800° C., e. g., from about 750–850° C. for a time of about 10 minutes, e. g., from about 5 to 20 minutes. This heating step causes a loss in weight of the mica equal to about 2 percent by weight of the original mica. This heating has the effect of softening the mica while at the same time delaminating and increasing the bulk volume of the mica. This heat treated mica is then added to an aqueous medium and agitated by any suitable device, such as a high speed comminuter or mixer to convert the mica into small particles or platelets. The communition of the mica can be carried out in an aqueous suspension containing from about 0.1 to 10 percent, and preferably about 1 percent by weight, of mica. This results in a pulp-like suspension of mica in which the particle size of the mica flakes has a wide distribution. The extra-fine and extra-coarse particles in this comminuted aqueous suspension may then be

removed and the resulting slurry is formed into sheets of paper on conventional paper-making apparatus wherein the mica particles are allowed to settle on a screen and the aqueous medium which suspended the particles is removed from the particles by suction or evaporation.

The purpose of the firing or heating step described above is to facilitate the comminution of the mica. Although I prefer to include this firing step in my process, it should be understood that the comminution can be accomplished without firing.

According to the method of the present invention, the mica particles while in comminuted form are brought into contact with barium ions in an aqueous barium salt solution. The method of bringing the barium ions into contact with the mica particles is not critical, and this step may be accomplished by any desirable means. Although I do not wish to be bound by theoretical consideration, it is believed that the barium ions which are brought into contact with the mica particles replace some of the potassium ions which are normally found on the surface of mica and that the presence of the barium ions on the surface of the mica tends to bind the particles of mica together with a greater force than is found with untreated mica containing only potassium ions on its surface.

The barium ions can be brought into contact with the mica particles by suspending the particles in an aqueous solution containing these ions. The formation of the suspension of mica particles in the aqueous barium ion solution may be accomplished at any time between the time the mica is fired up to the time the mica is formed into paper sheets. Thus, after firing of the mica, the fired mica may be dispersed in an aqueous solution containing barium ions and comminuted while in this solution. Alternatively, the fired mica may be comminuted in plain water and a suitable barium salt may be added to the water to bring the barium ions into contact with the particles. As a further alternative, the fired mica particles may be comminuted in plain water, filtered from the grinding medium, and then added to another aqueous solution containing barium ions. A still additional method of adding barium ions to the mica particles is to comminute the fired mica in water, separate the extra-coarse and extra-fine particles from the resulting solution and then either add a barium salt to the resulting slurry, or the mica particles may be filtered from the slurry and added to another aqueous solution containing barium ions.

In any event, the contact of the braium ions with the surfaces of the mica particles leads to the formation of mica paper having a higher tensile strength than mica paper formed without the barium ion treatment.

Unexpectedly, I have discovered that all solutions containing barium ions are not satisfactory in the process of the present invention. Treatment of mica particles with a barium hydroxide solution results in no improvement in the physical properties of a subsequently formed sheet. However, when a solution of barium salt is employed to treat mica, marked improvement of the physical properties of the resultant mica paper is observed. The particular barium salt employed is not critical, the only requirement being that the salt be soluble in water at room temperature (i. e. about 20° C.) to the extent of at least about 0.5 to 1.0 parts by weight of salt per 100 parts of water. The amount of barium salt employed may vary over a wide range, e. g. from about 0.1 to 10, and preferably about 1.0 part by weight per part of mica. Although I prefer to use barium chloride in the practice of my invention, other satisfactory barium salts include, for example, barium acetate, barium nitrate, barium fluoride, etc. The reaction between the mica particles and the barium ion solution is substantially instantaneous so that the time of contact between the mica particles and the barium ion is not critical. Thus,

a time of a few minutes up to several hours or more is satisfactory for causing the replacement of some of the potassium ions in the mica with barium ions.

After contacting the mica particles with the barium ions, the mica particles may be filtered from the treating solution and resuspended in pure water. This pure water solution may then be used to form sheets of paper from the mica on conventional paper-making machines. Alternatively, the suspension of mica in the barium treating solution may be employed directly in forming the sheets of mica paper. Regardless of whether a pure water suspension or a barium ion containing aqueous suspension of mica particles is used, the sheets are formed by dispersing the suspension on top of a screen and drawing the suspension medium through the mica particles by means of suction. The remaining aqueous medium may then be removed from the mica particles by evaporation at room temperature or at elevated temperatures. Where elevated temperatures are employed, it is desirable to use temperatures of from about 110–150° C. since this temperature has been found satisfactory to remove all of the suspension medium from the sheet. After removal of the water, the resulting mica particles are in the form of a fairly rigid sheet which is extremely resistant to elevated temperatures. In addition to forming the final mica paper by evaporation of the suspension medium from the particles, satisfactory mica paper is obtained by calendering the damp mica sheet resulting from sucking the suspension medium through the mica particles on the paper-making machine screen. This calendering operation may be carried out at a temperature of from 110–175° C. to facilitate removal of remaining moisture from the sheet.

The mica paper of the present invention may be formed of any desired thickness. However, for most applications it is preferred to prepare sheets having a thickness of from 1 to 50 or more mils and preferably from about 2 to 25 mils. This mica paper is very useful in applications which require the combination of a high dielectric strength and a good resistance to elevated temperatures. Thus, the mica paper of the present invention may be employed as slot insulation for dynamoelectric machines, as insulation in capacitors, transformers and the like, as well as insulation and separators in high temperature vacuum tubes.

One important advantage of the method of the present invention is that the barium ion solution employed to treat the mica particles may be separated from these particles and reused to treat additional mica particles. The same treating solution may be used over and over again as long as additional barium-containing material is added to the solution to keep the barium ion concentration to whatever value is selected.

The following examples are illustrative of the practice of my invention and are not intended for purposes of limitation. In each of the examples a 1 percent by weight slurry of mica in water was employed. The slurry was formed by firing Bengal Ruby muscovite at a temperature of about 800° C. for about 10 minutes, at which time there had been a weight loss of about 2.7 percent of the original weight of the mica. After cooling, this mica was then added to distilled water and the resulting slurry was agitated violently to comminute the mica particles. The extra-fine and extra-course particles of mica in the slurry were removed, leaving a 1 percent slurry of mica in water. All "parts" mentioned in the examples are by weight.

Example 1

A control was prepared by adding a 1 percent by weight slurry of mica in water to a Buchner funnel and sucking the water through the funnel, leaving a 3 mil wet sheet of mica paper on the surface of the funnel. This wet sheet was then pressed at a pressure of 1000 p. s. i. for one minute and dried at 110° C. and its tensile strength was measured. To another portion of the 1 percent mica solution, 1 part of barium chloride per part of mica was added. This barium chloride treated slurry was allowed to stand for 64 hours, at which time the mica was filtered and added to 99 parts of distilled water. A 3 mil sheet was formed following the same procedure employed with the control. The tensile strength of this sheet was about 4500 p. s. i. as compared with a tensile strength of about 2150 p. s. i. for the control.

Example 2

One part of anhydrous barium chloride was added to a 1 percent slurry of mica in water containing 1 part of mica. This barium chloride-mica solution was allowed to stand for about 18 hours, at which time the mica particles were filtered from the barium chloride solution, washed several times with water, and made up into a slurry containing 1 percent by weight of barium chloride treated mica. A sheet of mica paper about 1.8 mils thick was then prepared by the method of Example 1. A control was also prepared from the 1 percent mica slurry to which no barium chloride had been added. The thickness of the control was about 2.3 mils. The barium chloride treated mica sheet had a tensile strength of about 7900 p.s. i. as compared with the tensile strength of about 4900 p. s. i. for the control.

Example 3

This example illustrates the treatment of mica with barium ions from the chloride and the acetate and also illustrates the treatment of mica with barium hydroxide and shows that barium hydroxide is not a suitable treating agent for mica. All of the sheets of mica paper prepared in this example had a thickness of about 2.5 mils. One part of barium chloride or barium acetate was added to a 1 percent slurry of mica in water containing 1 part of mica. In each case, the barium-containing solution was allowed to stand for 3 hours, at which time a sheet was formed from the slurry without washing the slurry. The table below lists the material added to the mica slurry and the tensile strength observed.

Addition:	Tensile strength (p. s. i.)
None (control)	2520
$BaCl_2$	5660
$Ba(OCOCH_3)_2$	4010
$Ba(OH)_2$	2520

As the table shows, the treatment of mica particles with either barium chloride or barium acetate markedly improves the tensile strength of the resulting mica paper. Treatment with barium hydroxide leads to no improvement whatsoever in the tensile strength of the mica sheet.

Although the foregoing examples illustrate the treatment of mica with only several of the possible barium salts, it should be understood that barium salts not specifically illustrated may also be employed. Thus, in addition to using barium chloride as the treating agent, mica particles may also be treated with barium fluoride, barium bromide and barium iodide to obtain the improved mica paper of the present invention. Similarly, the mica particles may be treated with other barium salts such as barium nitrate, barium formate, barium propionate, etc. The only limitation upon the barium salt which may be employed is that the barium salt must exhibit at least a reasonable solubility in the mica slurry. By reasonable solubility is meant that the barium salt must be soluble in a one percent mica slurry to the extent of about 0.5 to 1.0 percent by weight of the salt based on the weight of the water present in the slurry.

Although the examples have not illustrated the full range of all of the variables of the present invention, it should be understood that in the practice of the present invention, slurries of mica containing from 0.1 to 10 percent by weight of mica may be employed. Sim-

ilarly, the amount of the barium salt employed to treat the mica particles may vary from 0.1 to 10 parts per part of mica in the slurry.

The mica paper prepared by the method of the present invention has the same utility as other mica paper. Thus, this mica paper can be employed as slot insulation in dynamoelectric machines, can be cut into tape and employed as insulation for electrical conductors, can be employed as electrical and thermal insulation in electron tubes, etc.

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

1. The method of preparing mica paper of improved tensile strength which comprises contacting comminuted mica particles with an aqueous barium salt solution, forming a wet paper-like sheet from said particles, and drying said sheet, said barium salt being characterized by a room temperature solubility of at least about 0.5 part by weight per 100 parts by weight of water.

2. The product prepared by the method of claim 1.

3. The method of claim 1 in which the barium salt is barium chloride.

4. The method of claim 1 in which the barium salt is barium acetate.

References Cited in the file of this patent

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