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[54] LARGE FLAKE RECONSTITUTED MICA INSULATION

9 Claims, No Drawings

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[56] References Cited

UNITED STATES PATENTS

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ABSTRACT: A reconstituted mica insulation sheet comprising large mica flakes has improved physical properties, particularly resistance to cut-through and sever mechanical abuse without danger of electrical failure. The sheet can be impregnated with resin, laminated to webs, adhesive-coated, slit, and coiled to form insulating tapes.

LARGE FLAKE RECONSTITUTED MICA INSULATION

BACKGROUND OF THE INVENTION

This invention relates to reconstituted mica insulation. More particularly, it relates to reconstituted mica insulation sheets and tapes having improved physical properties, particularly resistance to cut-through and severe mechanical abuse.

Cut-through of electrical insulation occurs when, during application or use, a sharp edge or corner of an insulated part forces its way through and physically separates the insulation, causing electrical failure. A particularly acute cut-through problem exists in which voltage turbine generators having rectangular copper conductors which must be insulated from each other and from ground. The insulation utilized must have exceptional electrical and mechanical properties, particularly resistance to cut-through and severe mechanical abuse, and must withstand being wrapped around small angle bends and being forced into tight crevices without cracking, splitting, or developing voids which would cause electrical failure. It has been found that resistance to this type of failure can be predicted from the results of a laboratory test which measures the force, in pounds, required to force a sharp edge through an electrical insulation.

Mica has excellent electrical, mechanical, and thermal properties, but is thick and inflexible in its naturally occurring state. Laminar mica crystals have been manually delaminated into noncohesive splittings, laid in overlapping pattern by hand or machine, and bonded with resinous material to form an insulation sheet having resistance to mechanical abuse and cut-through adequate for most purposes. However, machine laid sheets are thick, often discontinuous, have a tendency to flake, are not uniform and must be used in thick layers to obtain adequate electrical properties. Hand-laid insulation sheets, are expensive, difficult to make, have a tendency to flake, are in short supply domestically and consequently must be obtained from import sources.

U.S. Pat. Nos. 2,405,576, 2,549,880, and 2,614,055 disclose reconstituted mica insulation sheets made by reducing naturally occurring mica into thin cohesive flakes, in the absence of a deactivating atmosphere, pressing the flakes together, and drying under heat and pressure. U.S. Pat. No. 3,131,114 discloses the broad flake size range, about 3.5 to 400 mesh, utilized in reconstituted mica flake insulation sheets having glass flakes included therein. Such prior art reconstituted mica insulation sheets have good electrical properties and are considerably more flexible and uniform than sheets of mica splittings, but are opaque and are dependent upon an impregnating resin for resistance to cut-through and to physical abuse. When impregnated with soft resin the sheets lack resistance to physical abuse and deformation while sheets impregnated with hard resin crack and split during use with a resultant decrease in electrical insulation properties.

Despite the long-recognized desirability of mica sheet or tape insulation having both the resistance to cut-through and physical abuse of mica splittings, and the electrical superiority, uniformity, low caliper and flexibility of reconstituted mica, such a product has never heretofore existed.

SUMMARY

This invention provides reconstituted mica insulation sheets and tapes that combine excellent resistance to cut-through and physical abuse with the electrical superiority, uniformity, low caliper, and flexibility of reconstituted mica.

Insulation sheets and tapes prepared in accordance with this invention have excellent electrical and physical properties. They are flexible, dense, uniform, continuous, translucent, nonflaky, and retain their electrical properties such as arc and corona resistance, dielectric strength and low power factor, in severe use. Further, these sheets are ideally suited for insulating sheets, wrappers, and tapes having substantially improved resistance to cut-through and mechanical abuse during application and use. The insulation sheets of the invention are admirably suited for commercial use in insulating direct current

traction motors, alternating current motors, and transformers, and are particularly well suited for insulating the rectangular conductors of high-voltage turbine generators. These mica insulation sheets can be bent around sharp angle bends, including the right angle bends at the edges of rectangular stator conductors, and can be forced into small crevices without danger of electrical failure.

Surprisingly, it has been discovered that a reconstituted mica insulation sheet can be made from large mica flakes so as to have a resistance to cut-through, when unimpregnated, more than 200 percent greater than broad flake size prior art reconstituted mica sheets, while retaining the advantageous uniformity, flexibility, and electrical properties. The mica flakes used to make these sheets are considerably larger than the average size of mica flake used to make prior art sheets. At least 40 percent by weight of the flakes are larger than 14 mesh, at least 70 percent are larger than 35 mesh and at least 90 percent larger than 60 mesh. Preferably, at least 50 percent of the flakes are larger than 14 mesh, at least 80 percent larger than 35 mesh and at least 95 percent larger than 60 mesh. Prior art reconstituted mica insulation sheets utilize a broad flake size distribution from about 3.5 to 400 mesh, primarily from about 35 to 400 mesh. It is believed that the large surface area of individual flakes permits a large overlap area which increases cohesion between flakes, eliminates internal voids in the sheet to increase the specific gravity and provides a continuous sheet. It is also thought that the large surface area of the large flakes contributes the excellent resistance to cut-through and physical abuse.

The strength and resistance to abuse of mica insulation sheets are illustrated by tensile strength (ASTM Test D-828). Prior art insulation sheet made with a broad flake size range ("Acim" Brand) has an average tensile strength of about 400–500 p.s.i. Prior art insulation sheet made with very small flakes ("Samica" Brand) has an average tensile strength of about 1,200–2,400 p.s.i., while sheets made according to this invention have an average tensile strength of about 3,500–5,000 p.s.i.

The freedom from internal voids which characterizes the sheet of this invention is illustrated by its specific gravity of about 1.6–2.5, which approaches the 2.7–2.8 specific gravity of solid muscovite. Prior art reconstituted mica insulation sheet has a specific gravity of about 0.9–1.5, which clearly indicates the large number of voids contained therein. The specific gravity of mica insulation sheets is readily determined by the Mercury Intrusion Method described in Bulletin 2405-A of the American Instrument Company.

Muscovite and phlogopite mica larger than about 3 mesh can be utilized to produce these large mica flakes. After a preliminary water washing to remove dirt and debris, the mica blocks are split by means of water jets striking the mica blocks at an angle substantially parallel to the plane of cleavage as disclosed in U.S. Pat. No. 2,405,576. The flakes are classified with the proper mesh in U.S. standard sieve and reconstituted by standard papermaking techniques into a mica insulation sheet having an overlapping arrangement of large mica with their surface in contiguous relation. Flakes split in this manner are very thin and have a large surface area as compared to their thickness. When reconstituted, the individual mica flakes adhere to each other by natural cohesive forces, as contrasted with mica splittings which must be bonded together with resin.

After the reconstituted mica insulation sheet is made, it may be impregnated with inorganic resin such as boron phosphates and potassium borates and organic resin such as shellac, epoxy, alkyd, polyester, silicone, etc., the choice of resin depending on a balance of cost, temperature resistance, flexibility, and electrical resistance required. Inorganic or organic binders, fibers, and filaments may be incorporated into the sheet, if desired. For some industrial applications, it is desirable to laminate the large flake impregnated mica insulation sheet to a web such as polymeric film and woven or nonwoven fabrics, either by using the impregnating resin as an adhesive or by applying a separate adhesive between the web and the insulation

sheet. The improved cut-through resistance of large flake reconstituted mica insulation sheet is not dependent upon the saturating resin or backing web, whereby permitting the utilization of numerous soft resins which retain the flexibility of the insulation sheet.

Resistance to cut-through is determined by installing a ½" x ½" x 3" mild steel bar, having a 32 micro inch finish on all sides, in each jaw of an "Instron" tensile tester containing a compression cell. Both bars are installed on edge at right angles to each other so that only point contact will occur when the bars touch. The sample specimen is placed between the bars and the machine jaws closed at the rate of 0.5 inch/minute. The right-angle edges of the steel bars are forced through the mica sheet until they contact each other closing an electrical circuit to light a bulb which indicates when cut-through is complete. The "Instron" recorder provides an accurate reading of the force, in pounds, required to force the sharp edges through the mica sheet.

The following examples, in which all parts are by weight unless otherwise noted, illustrate preparation of the insulation sheets and tapes of this invention without limiting the scope thereof.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

EXAMPLE 1

This example describes splitting mica-blocks into flakes and making a reconstituted large flake mica insulation sheet. Indian muscovite punch scrap is split into thin flakes by subjecting it to jets of water as described in U.S. Pat. No. 2,405,576. The slurry of wet mica flakes was classified by washing the flakes through a series of U.S. standard screen sieves, all flakes below 140 mesh being discarded. The classified flakes are recombined in a 1 percent mica slurry in water such that 52 percent by weight of the flakes in the final slurry are larger than 14 mesh, 85 percent are larger than 35 mesh and 93 percent are larger than 60 mesh.

The reconstituted sheet was then made by using commercial paper mill equipment comprising, in connected series, an agitator, a storage chest, and a cylinder-type paper machine having an endless wet press belt which transfers the wet mica flake layer from the cylinder screen to a steel wet press roll. The speed of the cylinder screen was 16 ft./minute. An endless woven cotton belt transferred the wet paper web into a drying section of the machine where the paper was dried by passing through a series of steel cylinders heated to between about 140° F. and 180° F. The delivery rate of mica flake slurry to the paper machine determines the thickness of the insulation sheet obtained. Sheet thickness can be varied from about 0.5 mil to about 30 mils. If thicker sheets are desired, sheets can be laminated or sandwiched together. Mica insulation sheets produced in this manner have an unusually high degree of uniformity of flake structure as is evident from the uniform translucency observed when a sheet is held up to a light source.

An insulation sheet made in the aforedescribed manner had an average thickness of 2.45 mils when tested according to ASTM Test D-374, Method C, a tensile strength of about 4,000 p.s.i. when tested according to ASTM Test D-838, a dielectric strength of about 643 volts per mil when tested according to ASTM Test D-149, a cut-through resistance of about 7 lbs., a specific gravity of about 1.7, and provided a useful insulation sheet without impregnation. This mica sheet was impregnated, laminated, coiled and slit as described in examples 2-5.

An insulation sheet utilizing the broad 3.5 to 400 mesh flake size distribution of the prior art, disclosed by U.S. Pat. No. 3,131,114, was made and tested in the same manner. It has an average thickness of 2.85 mils, a tensile strength of about 200 p.s.i., dielectric strength of about 350 volts per mil, a cut-through resistance of about 2 lbs., and a specific gravity of about 1.2.

EXAMPLE 2

This example describes impregnation of a portion of the large flake reconstituted mica insulation sheet of example 1 with a polyester/epoxy resin. A mixture of 100 parts of polyester/epoxy resin and 1 part of tertiary amine [tris-(2,4,6-dimethyl amino methyl)-phenol] was prepared according to example 2 of U.S. Pat. No. 3,027,279. The impregnating resin was diluted to 25 percent solids with methyl ethyl ketone and applied to the mica sheet by means of a conventional dip and flow method. The impregnated paper was then dried at 150° F. for about 15 minutes and subsequently cured for 10 minutes at 400° F. The resultant insulation sheet was tough, flexible, and in a fully cured state. The resin content of the impregnated sheet was determined by weighing a sample both before and after coating and was found to be about 20 percent by weight.

The impregnated sheet, when tested in the manner of example 1, was found to have an average thickness of 4.4 mils, a tensile strength of about 10,400 p.s.i., a dielectric strength of about 690 volts per mil, and a cut-through resistance of about 7.7 lbs.

Impregnation of a reconstituted mica insulation sheet utilizing the broad 3.5 to 400 mesh flake size distribution of the prior art in a similar manner with the same resin produced a sheet that was 4.7 mils thick, contained 20 percent resin and had a cut-through resistance of 3.6 lbs.

EXAMPLE 3

A portion of the reconstituted large flake mica insulation sheet of example 1, was impregnated in the manner of example 2, with an isooctyl acrylate/acrylic acid/epoxy terpolymer resin and 0.45 percent uranyl nitrate hexahydrate catalyst.

The copolymer resin was made by first mixing 1,997.5 pounds of isooctyl acrylate, 29.0 pounds of acrylic acid, and 17.9 pounds of tertiary dodecyl mercaptan in a stainless steel tank. A charge of 2,787.5 pounds of toluene was then placed in a 1,500 gallon glass-lined kettle, after which 147 pounds of the mixture in the tank was added. The kettle was purged with nitrogen and heated to 175° F. with constant agitation, all the while maintaining a slight nitrogen flow through the kettle. Next, three separate 22-pound charges of azo-bisisobutyronitrile dissolved in toluene were added at equal intervals over a period of approximately 50 minutes; each charge being 22 percent solids. During this period the mixture from the tank was continuously added at a rate of about 37 pounds per minute. After waiting another 50 minute period, 20 pounds of 20 percent azo-bis-isobutyronitrile in toluene was added and the temperature maintained at 175° F. for 2½ additional hours. Then, 124.1 pounds of 3,4-epoxycyclohexylmethyl-3,4-epoxy-cyclohexanecarboxylate having an average molecular weight of about 260 and a viscosity of about 500 centipoises at 24° C. (Union Carbide ERL-4221) was added and thoroughly mixed. The mica sheet was impregnated as in example 2, and after drying for 10 minutes at 220° F., and curing 10 minutes at 400° F., the resin content was determined to be 20 percent. The tacky impregnated insulation sheet was then joined to a 1.5 mil nonwoven web of heat bonded polyethylene terephthalate fibers by means of laminating rolls.

The cut-through resistance of this laminate is substantially greater than that of a prior art mica insulation sheet, as described in example 1, similarly impregnated and laminated.

EXAMPLE 4

This example illustrates the impregnation of a portion of the mica insulation sheet of example 1 with soft flexible silicone resin by lamination to a resin impregnated woven glass cloth and subsequent lamination to polyethylene terephthalate film.

Polysiloxane resin (General Electric SR-32) was diluted to 35 percent solids in toluene. A 2 mil woven glass cloth was impregnated with this resin by the common dip and flow method and laminated to a portion of the mica sheet of example 1 by means of laminating rolls, and the laminate dried for 4 minutes

at 400° F. The laminate contained 10 percent resin, was 7.5 mils thick, and had a cut-through resistance superior to that of a prior art mica insulating sheet, as described in example 1, similarly saturated and laminated.

The unlaminated exposed mica surface was then coated with the silicone resin saturant disclosed above, by reverse roll coating technique, and dried for 4 minutes at 250° F. The tacky mica surface was then laminated to 0.25 mil biaxially oriented polyethylene terephthalate film by means of laminating rolls, coiled into a jumbo roll, and subsequently slit into ¾ wide rolls of tape. The resin content of the laminate was 15 percent and the cut-through resistance was superior to that of a prior art mica insulating sheet, as described in example 1, similarly impregnated and laminated.

EXAMPLE 5

This example illustrates the lamination of a polyester film web to the impregnated large flake reconstituted mica sheet of example 2. After impregnation and following drying at 150° F. for 15 minutes, the impregnated mica sheet was cooled and laminated to 0.5 mil biaxially oriented polyethylene terephthalate film by means of rotating pressure rolls. The cut-through resistance of the laminate was superior to that of a prior art mica insulating sheet, as described in example 1, similarly impregnated and laminated.

I claim:

1. In a self-supporting reconstituted mica insulation sheet comprising an overlapping arrangement of unimpregnated mica flakes, the improvement comprising:

the mica flakes having at least 40 percent by weight of the individual flakes larger than 14 mesh,

at least 70 percent by weight of the individual flakes larger than 35 mesh, and

at least 90 percent by weight of the individual flakes larger than 60 mesh,

whereby the insulation sheet has increased density, excellent tensile strength, outstanding electrical properties and resistance to cut-through and physical abuse.

2. The insulation sheet of claim 1 impregnated with resin.

3. The insulation sheet of claim 2 wherein the resin selected from the class consisting of epoxy resin, polyester resin, silicone resin, alkyd resin, and acrylic resin.

4. The insulating sheet of claim 3 laminated to a fibrous web.

5. The laminate of claim 4 wherein the web is polyethylene terephthalate.

6. The insulation sheet of claim 3 laminated to a polymeric film.

7. The laminate of claim 6 wherein the polymeric film is biaxially oriented polyethylene terephthalate.

8. As a new article of commerce, an insulating tape wound convolutely upon itself in roll form and capable of being unwound therefrom without delaminating or offsetting, said tape comprising the insulation sheet of claim 1 or 2 laminated to one side of a thin flexible web, whereby said insulating tape has increased specific gravity, excellent tensile strength, outstanding electrical properties, and resistance to cut-through and physical abuse.

9. The tape of claim 8 wherein the insulation sheet has a second thin flexible web laminated on its other side.

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