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METHOD OF TREATING MICA

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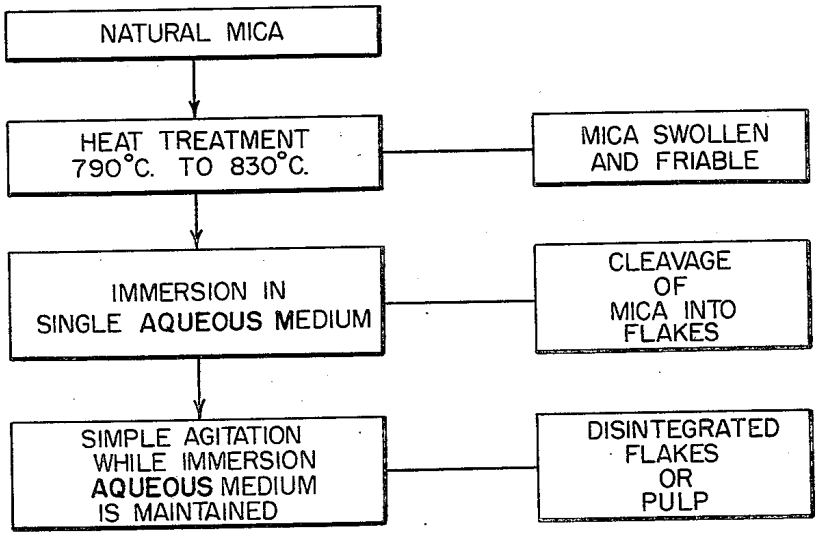

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METHOD OF TREATING MICA

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The present invention has for its object to provide a method for the manufacture of useful insulating elements of any shape and size from mica, and more particularly from sheet fragments or splittings and scraps of mica.

According to the method of the invention the mica is first heated to a temperature of about 790 to 850° C., whereby it is caused to swell and becomes more friable, after which it is placed in the presence of an aqueous liquid in which the swollen mica assumes a divided state in which the mica is in the form of flakes of a thickness in the order of about one micron, and said pulp is then processed in a manner similar to paper-making pulp, so as finally to obtain insulating elements having the desired shapes and sizes.

The mica can be immersed into the liquid immediately upon being heated and while it still is at a temperature near the maximum temperature attained in the heat treatment.

Alternatively the heated mica can be allowed to cool and subjected to the action of the liquid after an indefinite period, provided it has been maintained for at least 10 to 15 minutes at said maximum temperature.

The wet process to which the heated mica is subjected may comprise immersing it into a body of cool water, within which the mica is subjected to agitation causing it to disperse throughout the body of water and to form a pulp having the above-indicated characteristics. The water may have a mineral or an organic acid added thereto during the above-described treatment.

An alternative way of obtaining the pulp is to subject the heated mica after quenching it in water to the subsequent action of a relatively concentrated acid solution. The water used to quench the hot mica could even if desired be replaced by an acid solution.

Another alternative procedure for carrying the invention into practice comprises quenching the heated mica in a body of alkali solution rather than water, then dividing the mica through agitation within said solution; the mica is then drained dry, washed, and then treated with acidified water.

The mica after having been heated to about 800° C. to 850° C. then allowed to cool may be treated with an acid solution within which it is subjected to a mechanical treatment adapted to complete the dividing thereof.

Such treatment can consist of reducing the mica to a pulp while in the treating solution, or subjecting it while in said solution either to the action of a vacuum or to a succession of

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alternating applications of pressure and vacuum.

The cooled mica can also be reduced to a pulp by mixing it in a body of water to which an acid is gradually added. It has been found however that in general, in order to obtain a pulp capable of eventually providing insulating materials having satisfactory cohesion, it is desirable to avoid too violent a mechanical action, and preferably even avoid too thorough a reducing process.

The preferred procedure is to reduce the mica in the presence of a large body of liquid and to carry off the fine flakes with a current of said liquid as fast as they are produced.

The accompanying drawing constitutes a flow sheet in which the figure illustrates the preferred process for the production of the mica pulp, the left hand blocks illustrating the steps of the process and the right hand blocks showing the condition of the mica as it undergoes the steps of the process.

Prior to conversion into insulating elements organic or inorganic binders and other ingredients adapted to confer any desired specific properties to the final insulating materials or reenforce or enhance any such properties as they may already possess, can if desired be added to the pulp.

Sheets of mica-base "paper" or "board" produced from a pulp in accordance with the methods of the invention can be laminated together in any desired number with the interposal of a binder between the adjacent successive layers, the laminated assembly being subjected to the action of a press, at high temperature if necessary. Also such a sheet may be laminated through the use of any suitable binding substance to a backing or support comprising cellulosic paper or asbestos paper, or a film of plastic material, or a fabric formed of natural or synthetic fibres of vegetable, animal or mineral origin.

Some examples will now be given merely with the purpose of illustrating the above-described operating procedures used in carrying out the present invention.

Example 1

Muscovite mica in the form of splittings or scrap is oven heated at a temperature of 800° C. for about 10 minutes oven. It is then rapidly thrown into a concentrated sodium carbonate solution. The mixture is allowed to cool and is then agitated until the mica is reduced to a pulp. This pulp is dried as through centrifugal

means so as to eliminate the near totality of the alkali solution therefrom. The cake thus obtained can be washed and even dried. The divided mica is then suspended in a 2% sulfuric acid solution. The mixture is thoroughly mulled so as to obtain a thorough distribution of the acidified water throughout, then the resulting pulp is centrifuged or otherwise dry drained an quickly washed to separate the very small amount of sodium sulfate formed as well as any excess acid.

If and when desired a levigation may be effected in order to classify the particles of mica according to size.

The aqueous mica suspension thus treated may be processed like paper-making pulp either in papermaking forms or in a continuous machine, or further by means of appropriate moulds without the addition of any binder or agglomerating agent, the sheets, boards or molded articles thus obtained having a very satisfactory degree of cohesion.

Example 2

The mica is heated as described above in Example 1 and is then immersed as quickly as possible in a body of water. The mixture is allowed to cool and is then mixed until a pulp of finely divided mica is formed. To this pulp there is added an aqueous emulsion of a synthetic resin and the resulting mixture is treated like ordinary paper pulp. The resulting products are subsequently heated to a sufficient temperature to cause softening or melting of the resin, which imparts the necessary cohesion to the final product.

Example 3

The procedure of Example 2 is followed except that lead borate is substituted for the resin. Desirably said lead borate is directly formed in situ within the mica suspension with the addition thereto of salt solutions adapted to react together to yield lead borate, as for instance sodium borate and lead acetate in solution. The resulting products are dried and then heated to a temperature of 400° C. to melt the lead borate.

Example 4

The mica is heated and then allowed to cool, all as described in Example 1 and is then treated with a 2% solution of sulphuric or hydrochloric acid. The mixture is introduced into a tank and subjected to mixing. A current of water is caused to carry away the flakes formed, into a settling tank in which they settle. The flakes are then collected and used to produce sheets or molded articles without it being necessary to add an agglomerating substance thereto.

Example 5

Mica splittings are subjected to a heat treatment of 30 minutes at 830° C., then are suddenly immersed in water. They are then removed from the water and immersed in 20% sulphuric acid. The divided mica pulp is removed from out of the acid and thoroughly washed. The procedure of Example 6 is then followed. The resulting "mica paper" has quite a good mechanical or tensile resistance and its dielectric properties are excellent.

Example 6

The procedure of Example 5 is followed. However the mica pulp obtained is suspended in a large amount of water, after which an acidified solution of a melamine-formaldehyde condensation resin is added to it. The resin precipitates upon contacting the water and mixes with the mica pulp. Paper is made from this pulp according to usual procedure and a "mica paper" is obtained having excellent tensile strength and increased dielectric properties.

Example 7

Exactly the same procedure as in Example 6 is followed. However the mica pulp containing the resin, instead of being transferred to a papermaking machine is thoroughly dried as by centrifugal means, then compressed in molds until a complete polymerisation of the resin has occurred. There is thus obtained an extremely strong insulating material having excellent dielectric properties.

Example 8

Mica splittings are subjected to a heat treatment of about 20 minutes at 800° C., then suddenly immersed in water. A small quantity of sulphuric acid is then added to the water and the mixture is then vigorously agitated for about 30 minutes. The mica is then in a thoroughly divided state. By filtering this mica there may be produced a mica base paper having very high tensile characteristics.

Example 9

Mica splittings are subjected to a heat treatment for about 20 minutes at 800° C., then suddenly immersed in a normal hydrochloric acid solution. This is then agitated until the mica assumes a thoroughly divided condition; the pulp is washed and filtered.

Example 10

Splittings are subjected to a heat treatment for 30 minutes at 790° C., then are suddenly immersed in a normal sulphuric acid solution containing 2 per mill of a wetting agent such as Nekal BX which is the sodium salt of sulfonic diisobutylnaphthalene acid. The mixture is subjected to agitation until the mica splittings are thoroughly divided, this being considerably facilitated by the presence of the wetting agent.

Example 11

Mica splittings are subjected to a heat treatment for 30 minutes at 820° C. and are then air-cooled. They are then immersed in a hydrochloric acid solution at 2 N concentration containing 3 per mill of a wetting agent such as Sandozol SB which is the sodium salt of the sulfuric ester oxystearic acid. The mixture is subjected to agitation until total division of the mica. A resinous melamine-formaldehyde condensation product is then added to it and the resulting mixture is filtered as described in Example 8.

Example 12

Mica splittings are subjected to a heat treatment for 30 minutes at 820° C., then air-cooled. They are then immersed in a hydrochloric acid solution at 2 N concentration. The mixture is agitated until in a totally divided state and a melamine-formaldehyde resinous condensation product is added to it and the resulting mixture is filtered as indicated above.

Example 13

A web of mica paper about 1 meter wide and 0.07 mm. thick is impregnated by dipping it into an alcoholic solution of formophenol resin, then

dried in a drying oven until complete solvent evaporation. Sheets one meter square are then cut out of the web. Some 20 such sheets are piled on top of each other and pressed between sheet-metal plates in a suitable platen-press. A temperature of about 150° C. with a pressure of 100 kg. per square cm. is maintained for an hour. There is obtained after cooling an extremely rigid laminated board having excellent thermal and dielectric properties.

Example 14

A web of mica paper is surface-coated with a shellac base varnish in such a way that the resin content does not exceed 20% after the coat of varnish has set.

The coated sheets are then placed in a platen-press as in Example 13 and pressed therein at a temperature of 130° C.

The pressed sheet is allowed to cool and there is obtained a hard board adapted to be hot-shaped in molds so as to yield mica insulators in a very wide range of shapes.

Example 15

A shellac solution is applied to one side of a web of kraft paper by means of an inking-roll, a brush or a sprayer, then the solvent is removed by passing the coated paper through an oven heated at 80° C. The shellac coated paper is then led into and through the nip between two heated rollers in which it is passed in contact with a web of mica paper. The latter is caused to adhere to the paper because of the liquefaction of the shellac whereby there is obtained, in a continuous process, an excellent insulating material comprising a kraft paper backing and a sheet of mica paper laminated thereto, the thickness of the latter being selected with regard to individual requirements for the insulation. A coating of shellac varnish can then be sprayed into the mica whereby tubes, sheets and the like may be subsequently produced.

Example 16

A web of mica paper 0.03 mm. in thickness is led into the nip of two heated cylinders where it is brought into contact engagement with a web of silk fabric which previously has been caused to pass through an insulating varnish coating material having a high adhesive power. The two webs are caused to adhere to each other and are led through a tunnel oven in which the insulating varnish is thoroughly dried or cured. An insulating sheet-material is thus obtained which comprises a varnished silk web to which is strongly laminated a very thin sheet of mica. The break-voltage of such an insulating sheet material is in the order of about 5000 volts.

Example 17

A glass fabric sheet about one meter square is carefully coated with a glycerophthalic base varnish and the solvent is allowed to evaporate in the atmosphere. Over this varnished fabric there is then applied a sheet of mica paper of similar dimensions. The mica paper is then covered with a glass fabric sheet preliminarily immersed in a glycerophthalic base varnish and

the assembly has applied to it a pressure of a few kg. p. sq. cm. The product is oven-dried at 120° C. and an insulating laminated material is thus produced which comprises a sheet of mica interposed between two sheets of glass fabric and which forms an excellent insulating material for notches in magnetic armatures.

What I claim is:

1. A method for the conversion of mica, initially in the form of sheets, splittings and the like, into a pulp suitable for conversion into sheet form by conventional paper making methods and also for conversion into molded form, which consists of heating the mica to a temperature of about 790–830° C., immersing the heat-treated mica in a single aqueous medium which is a non-solvent therefor, maintaining such immersion in said single aqueous medium until cleavage of the mica into flake form is accomplished, and then disintegrating the resultant flakes by simple agitation thereof in said single aqueous medium until a mica pulp is formed.

2. A method for the conversion of mica, initially in the form of sheets, splittings and the like, into a pulp suitable for conversion into sheet form by conventional paper making methods and also for conversion into molded form, which consists of heating the mica to a temperature of about 790–830° C., cooling the heat-treated mica, immersing the cooled mica in an aqueous medium which is non-solvent therefor and which contains a wetting agent, maintaining such immersion in said single aqueous medium until cleavage of the mica into flake form is accomplished, and then disintegrating the resultant flakes by simple agitation thereof in single aqueous medium until a mica pulp is formed.

3. A process according to claim 1, wherein the aqueous medium is water.

4. A process according to claim 1, wherein the aqueous medium is an alkaline solution.

5. A process according to claim 1, wherein the aqueous medium is an acid solution.

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REFERENCES CITED

The following references are of record in the file of this patent:

UNITED STATES PATENTS

Number	Name	Date
109,416	Johnston	Nov. 22, 1870
845,450	Dobler	Feb. 26, 1907
1,194,155	Edgecomb	Aug. 8, 1916
1,578,812	Dawes	Mar. 30, 1926
1,578,813	Dawes	Mar. 30, 1926
1,702,896	Frederick	Feb. 19, 1929
1,870,835	Coffey	Aug. 9, 1932
1,922,448	Miner	Aug. 15, 1933
2,108,577	Brough	Feb. 15, 1938
2,265,358	Denning	Dec. 9, 1941
2,306,292	Atwood	Dec. 22, 1942
2,377,868	D'Alelio	June 12, 1945
2,405,576	Heyman	Aug. 13, 1946
2,549,880	Bardet	Apr. 24, 1951

FOREIGN PATENTS

Number	Country	Date
72,209	Norway	June 23, 1947