

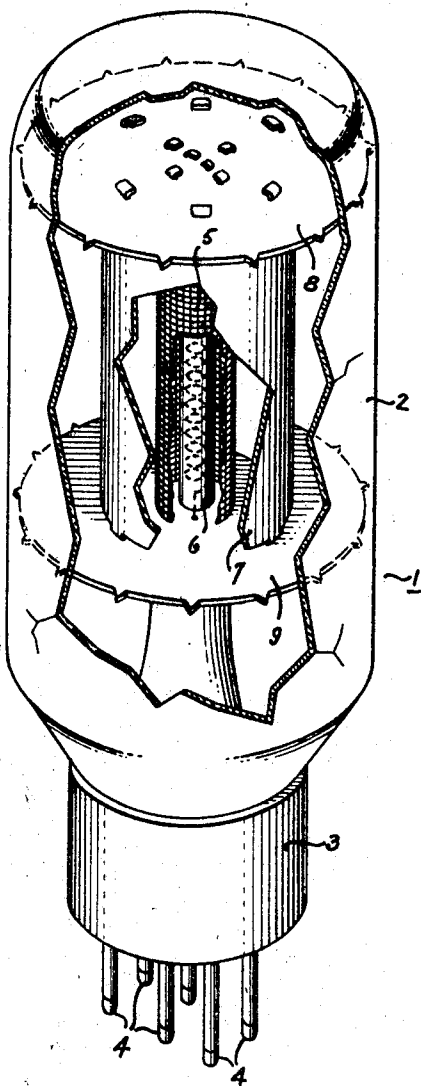
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PROCESS FOR PREPARING BONDED RECONSTITUTED MICA

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1

3,183,115

PROCESS FOR PREPARING BONDED RECONSTITUTED MICA

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This invention relates to silica-bonded materials. More particularly, it relates to reconstituted micaceous materials bonded with silica which are characterized by increased handleability, chemical inertness and absence of volatiles.

Reconstituted micaceous materials, mica paper or mica mat, as it is variously known, is described in various patents such as Bardet 2,549,880, April 24, 1951 and elsewhere. Generally speaking, such reconstituted mica mat is made by heating mica to a temperature sufficient to produce cleavage and then comminuting by various methods to a finely divided state which is then reconstituted in the form of a mat or sheet by well known paper making methods. The mica mat so prepared consists essentially of mica in its pure state. Other methods of making such mica are well known to those skilled in the art. However, it is so fragile and lacks tensile strength to such a degree that from a practical point of view it is difficult to fabricate structures from the material as is. Consequently, it is usual practice to impregnate the mica mat with resinous or other binder material and to lay up thin laminae of such impregnated material to form a mica board which has desired physical strength. In this form the mica board is useful in many electrical insulating applications but there are some uses, notably as spacers in vacuum tubes and the like, where absence of metallic ions and of organic or other gas producing materials should as would be in ordinary impregnants is indicated.

It is an object, therefore, of this invention to provide reconstituted mica and similar materials which are bonded together with an inorganic bonding agent which is characterized by good insulating qualities and at the same time is free from metallic ions or gaseous material even at elevated temperatures.

Briefly, the invention relates to reconstituted micaceous material which is impregnated with a solution of ammonium silicate, the material then being heated to drive off the ammonia, leaving a mica mat which is bonded together with pure silica.

Those features of the invention which are believed to be novel are set forth with particularly in the claims appended hereto. The invention will, however, be better understood and further advantages and objects thereof appreciated from a consideration of the following description and the drawing in which the single figure shows a typical application of the present materials.

Broadly speaking, reconstituted mica paper, according to the present invention, is treated by roll-coating with a water solution of ammonium silicate containing from about five percent to thirty percent by weight of ammonium silicate. It has been found that the mica paper when saturated will have picked up about 1.7 times its own weight of a solution containing about 15 percent ammonium silicate. It will be realized, of course, that in lieu of roll-coating the mica paper can be immersed in the solution, the solution may be sprayed, brushed or otherwise applied to the mica paper in manners well known to those skilled in the art. In general, the mica paper, depending upon the mode of application, picks up from 0.25 to 3.5 times its own weight of impregnant.

When the paper has become saturated with the ammonium silicate solution, it is dried at a low temperature varying from about 20° C. and 100° C. to a leathery condition having approximately 1.1 to 2.0 times the origi-

2

nal weight of the mica mat. Depending upon the thickness of final mica mat or board desired, the appropriate number of sheets are placed together and pressed at from about 25 to 2000 p.s.i., for from one-half hour upward at 75° C. to 200° C. and cooled under pressure. The assembly can, if desired, be postpressed for one-half hour upward at 100° C. to 200° C. and cooled while under pressure. This procedure produces a mica board of desirable integrity which has a flexural strength of over 30,000 p.s.i., and even post baking for several hours at 500° C. lowers the flexural strength only to the order of about 25,000 p.s.i. It has also been found convenient to impregnate the board with the ammonium silicate solution after it has been laid up from the layers of mica mat.

The wet strength of the mica paper which is treated with the above ammonium silicate solution is markedly increased over that which is not so treated. Since ordinary mica mat is very fragile and hard to handle, this increase in wet strength greatly simplifies the handling of the material.

The following examples illustrate the preparation of a mica board according to the present invention. Reconstituted mica paper made in the usual manner and having a thickness of 0.004" mil was laid up in four thicknesses to form a mica board and brush-impregnated with a 15 percent ammonium silicate solution to the point where it had absorbed 1.7 times its own weight of the solution. The board so treated was partially dried at a temperature of 55° C. for fifteen minutes to a leathery condition at which point it weighted 1.35 times the original weight of the mica paper. The laid-up board was pressed at 500 p.s.i. for two hours at 93° C. and for two hours at 200° C. and then cooled under pressure. The very uniform board having a thickness of .011" mil which was so produced was found to have a flexural strength of about 31,000 p.s.i. This compares favorably with a flexural strength of like material having organic binders. Furthermore, the flexural strength of the material was essentially retained even after treatment at elevated temperatures. For example, when the material was heated to a temperature of 500° C. over a period of two hours and held at that temperature for one hour, the flexural strength had decreased to only 25,000 p.s.i.

Another board 30 mils thick was laid up from mica mat as above and painted with a 15 percent by weight solution of ammonium silicate. The board which absorbed its own weight of the solution was dried to a leathery state at 55° C. for ten minutes. It was then pressed at 500 p.s.i. for one hour at 200° C. and cooled while under pressure. The resultant board had very desirable characteristics.

As pointed out above, while the present materials are useful in any electrical insulating or heat insulating application requiring such a material, they were particularly useful for applications which demand inertness to elevated temperature and at the same time require that no metallic ions or gas be given off at such temperatures. They are thus especially adapted to use as insulating spacers for electrical tubes.

Shown in the single figure of the drawing is a simple triode electron tube which illustrates the use of the present invention as a tube spacer. In the figure is electron tube 1 having envelope 2 of the usual type and base 3 with contacts 4 leading into the tube envelope in well known manner to the grid 5, cathode 6 and plate 7. Tube spacers 8 and 9, respectively, at the top and bottom of the interior elements are punched and provided with projections or points to hold the elements in relative position one to the other and also firmly in place with respect to the tube elements.

Among the other specific uses for the present invention are electrical applications where exposure to arcs is spec-

3

ified such as in arc chutes. The bonded micaceous material described herein is also useful in electrodynamic machine applications, in insulating applications such as slot liners, slot wedges, commutator retaining rings, phase spacers, segment plates and the like. In general, the materials are useful in any application where high electrical insulating characteristics are desired, particularly where such characteristics are mandatory at elevated, as well as, ordinary temperatures.

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

1. The process of bonding reconstituted mica mat which comprises impregnating said mat with a solution of 5 to 30 percent by weight ammonium silicate in water, heating said mat to drive off ammonia and pressing at a pressure of from 25 to 2,000 p.s.i. at a temperature of from about 75° C. to 200° C. to form a unitary structure.

2. The process of bonding reconstituted mica mat which comprises impregnating said mica with a solution of 5 to 30 percent by weight ammonium silicate in water, heating said mica to drive off ammonia and pressing under a pressure of from 25 to 2,000 p.s.i. at a temperature of from 75° C. to 200° C., post-pressing at 100° C. to 200° C. and cooling under pressure.

4

3. The process of bonding reconstituted mica mat which comprises impregnating said mica with a solution of ammonium silicate, heating to drive off ammonia and pressing with heat to form a unitary structure.

4. The process of bonding reconstituted mica mat which comprises impregnating said mat with a solution of 15 percent ammonium silicate in water, heating said mat at a temperature of about 55° C. to a leathery condition to drive off ammonia, pressing at a pressure of about 500 p.s.i. for two hours at 93° C. and for two hours at 200° C. and cooling under pressure.

5. The process of bonding reconstituted mica mat which comprises impregnating said mica with a solution of 5 to 30 percent by weight ammonium silicate in water, heating said mica to drive off ammonia and pressing under a pressure of from 25 to 2,000 p.s.i. at a temperature of from 75° C. to 200° C. and cooling under pressure.

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25 EARL M. BERGERT, *Primary Examiner*.