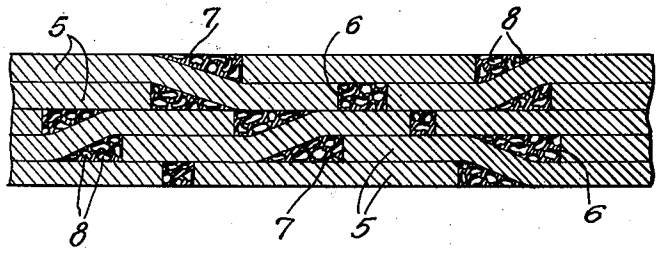
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MICA BASE INSULATING SHEET AND METHOD FOR PRODUCING THE SAME

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MICA BASE INSULATING SHEET AND METHOD FOR PRODUCING THE SAME

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This invention relates to a sheet comprising an integrated mica base and a hard inorganic skeleton occupying the spaces or pores of said base. The invention also relates to a method for producing such a sheet. This application is a continuation-in-part of my pending application, Serial No. 286,700, filed May 8, 1952, now abandoned.

In the electronic field there are many places of application for disc-like elements that are strong yet light, hard yet non-abrasive, highly heat resistant, highly dielectric, and free of occluded gases or, at least, capable of being evacuated of any gases or air therein. An example of use for such disc-like elements is in electronic tubes to support and space the various metal parts and fine wires therein. Inasmuch as such tubes are under high vacuum and generate considerable heat, it will be evident that the mentioned properties are important for such discs, particularly the property of freely giving up any occluded gases when a vacuum is drawn in the tube. The hard and non-abrasive property is also important because it makes for good machineability and permits production by die cutting without abnormal wear of the dies and also provides an element that does not have undue wear or attrition on the fine metal parts in contact therewith. Those skilled in the art will readily understand the desirability of providing a sheet of the character indicated from which such disc elements may be produced for the indicated and similar purposes. It is an object of the present invention to provide a mica base insulating sheet having the properties above mentioned.

The present sheet, in addition to the listed properties, is form-retaining when immersed in liquid. The sheet also is cellular in its internal construction and, therefore, is hygroscopic. Consequently, the sheet may have use as a wick material in which the cells serve as capillaries. Another indicated use is as separator plates in storage batteries.

Another object of the invention is to provide a novel method for producing a sheet comprised of integrated mica in the spaces or pores of which a hard cellular skeleton of inorganic material is provided.

Another object of the invention is to provide such a method in which the inorganic material is conveyed to the pores of the integrated mica by an organic material and heat employed to evaporate or burn out all except the inorganic skeleton.

Reference is made to applicant's Patent No. 2,405,576, dated August 13, 1946, in which integrated mica is disclosed.

The foregoing objects and other objects, features and advantages of the invention will become more clearly evident from the following description of a preferred form of sheet and method for producing the same. It is to be understood, however, that the description, which is based on the accompanying drawing, is given by way of example or illustration only.

In the drawing:

The figure is a greatly enlarged fragmentary sectional view of a mica base insulating sheet according to the present invention.

Integrated mica comprises extremely thin mica flakes having virgin or activated surfaces, said flakes being arranged in random fashion with their surfaces in contiguous relation enabling the natural cohesive forces resident in the flakes to become effective to integrate the flakes into a sheet having spaces or pores among the flakes. The drawing shows such a sheet having flakes 5, the same also showing that some of the flakes lie in more than one plane, and that spaces or pores 6 are formed among the flakes. The term "integrated mica" in this disclosure and in the appended claims is intended to define any sheet or mass of mica flakes or particles that hold together in a handleable manner and in which said flakes or particles are integrated without a binder or other extraneous material.

The flakes comprising integrated mica, being substantially incompressible, the sheet itself cannot be compressed to such degree that the pores among the flakes are eliminated and the same are, therefore, retained even under application of pressure applied to the opposite faces. This incompressible property of integrated mica differs from the inherent compressibility of fibrous materials and is herein employed to provide a labyrinthine maze of spaces and passages capable of housing a hard cellular skeleton of inorganic material in which the cells are retained.

According to the invention, the spaces or pores 6 are occupied by a skeleton 7 of inorganic material which embodies a multiplicity of cells, holes or pores 8. The problem has been to provide the inorganic skeleton to the substantial exclusion of all matter that would break down or gasify under heat and/or vacuum. During my work with this material, I have attempted to provide an inorganic skeleton by direct introduction into the pores 6 of silica in the form of glass particles of colloidal size but found that the resultant sheet was gritty and highly abrasive, would unduly wear dies punching parts therefrom, and having wearing attrition on metal parts in contact therewith.

During my work with this material, I found that organo-inorganic material in a liquid state so as to be capable of impregnating integrated mica, whether or not vacuum is used, provides a satisfactory means for conveying inorganic material to pores 6 and that a suitable skeleton of inorganic material remains in said pores when the organic matter is removed. The liquid organo-inorganic material, which I have found will satisfactorily impregnate integrated mica is commonly known as silicone resin which broadly comprises the metal silicon—the inorganic component—and a hydrocarbon—the organic component. These silicone resins have diverse formulae and, according to their formulae, different names or groups of names.

I have used as an impregnant siloxanes which are compounds of silicon, oxygen, carbon and hydrogen and containing in their molecules the structural unit R_2SiO in which R is usually CH_3 and may be C_2H_5, C_6H_5, or even more complex substituents. In commercial silicones R is usually CH_3, i. e., they are methyl siloxanes. These siloxanes are here given as merely an example of organo-silicon compounds that are produced in the form of a resin capable of being held in suspension by various solvents, such as those of the group of aromatic solvents, e. g., benzene, toluene, xylene, naphtha, etc. A common silicone resin is methyl phenol polysiloxane.

Hereinafter I shall call an organo-inorganic material, such as above mentioned, and dissolved or in suspension in a solvent, a silicone resin, the same being intended as a

generic term for the organo-silicon impregnant employed in the following manner to provide a skeleton of silica in a porous sheet comprised of mica flakes.

A fabricated sheet of mica is impregnated with silicone resin as by dipping the sheet in the resin until saturation is attained. For quicker saturation, vacuum impregnation may be employed. Time is then allowed for the solvent in the resin to evaporate. The drying time will depend on the proportion of solvent in the solution. In this manner, the silicon of the impregnant is brought into the pores of the sheet since silicone resin comprises both silicon and an organic material.

The impregnated mica is then subjected to a temperature of approximately 1000° F. Since mica may dehydrate at temperatures higher than 1000° F., the same should not be exceeded unless the possibility of dehydration is not present. It may be stated that the heating temperature may be raised to but before the temperature of dehydration is reached. However, 1000° F. has been found satisfactory.

In actual practice, the above temperature is not provided immediately, but the impregnated sheet, after drying, is first baked at a temperature between 400° F. to 500° F. for approximately one hour. At this stage, the impregnant still contains elements of trapped moisture, air and gas, but a reaction has taken place that has cured the silicone resin. Said action thermo-sets the resin in the pores of the sheet.

After this baking period, the temperature may be raised to 1000° F. and maintained for one hour to three hours or, as an alternative, the temperature may be first raised to 600° F., kept there for about one-half to one hour and then raised to 1000° F. for the one to three hour period. It is during this period of higher temperatures that the organic matter or carbon in the resin and the gases released thereby is burnt out to leave the skeleton 7 with the multiplicity of cells or pores 8. Improved results may be obtained by increasing the baking time, as desired, and as long as forty eight hours. The heat above-mentioned may be applied in the presence of oxygen to improve elimination of the organic component or radical.

As a result of the foregoing method, the organic component or carbon radical in the silicone resin impregnant is burnt out and driven off through the cells 8; a shell or skeleton of silica remains and lines said cells; there is provided a sheet that is hard, quite stiff and of such strength as to have a tensile strength of 8500 p. s. i.; and there is provided a sheet that has the properties set forth in the preamble to this specification.

I have also found that certain silicates, although not as efficient as silicone resins, may be used as the impregnant. These silicates are essentially inorganics as distinguished from the silicones which are organo-inorganics. Nevertheless, these silicates, of which ethyl silicate is an example, if suspended in a suitable liquid carrier, such as alcohol, may be used to impregnate integrated mica to carry silica to the pores of said mica and will leave a cellular silica skeleton after the carrier has been evaporated out and the silica cured or thermo-set.

From the foregoing, it will be seen that the inorganic material is brought into the pores of the mica base sheet by means of either a liquid or solvent and organic matter, or by a liquid alone, and that suitable heat is used to drive off substantially all matter and gases except the organic material. Therefore, the present method employs a carrier for the inorganic material that then comprises the skeleton which occupies the pores or spaces of the mica base sheet.

The herein-described sheet is hygroscopic because of the cellular nature of the silica skeleton 7. Therefore, said sheet is capable of being again impregnated and burnt out, as described, and, if necessary, re-impregnated and burnt out a third, fourth and, even, a fifth time. The resultant sheet is particularly valuable for providing mica supports or bridges in electronic tubes that operate at extremely high ambient temperatures—temperatures so high

that the same causes dehydration of the mica. Since such dehydration of natural mica releases moisture and generates steam, a reduction of the vacuum in the tubes results and the tubes rapidly deteriorate in their electronic function.

Dehydrated mica alone is much too soft for the above-described use. By fortifying the sheet of mica flakes with successive impregnations of silica or comparable inorganic material, the mass of the sheet may be made to contain as much as twenty percent of silica. With such a large proportion of silica to fortify the mica, the sheet can be heated above the temperature at which the mica dehydrates to insure substantially complete expulsion of all moisture. In practice, the heating range may be between 1400° and 1800° F. Also, the high proportion of silica in the sheet renders the same stiff and strong. It has also been found that, by applying pressure to such a sheet while the same is being heated, the strength of the material is further increased, and said sheet has been found to have a tensile strength up to twenty thousand pounds per square inch.

While this disclosure describes the impregnation of a sheet from which disc-like parts are to be punched or otherwise produced, the method may be carried out on mica parts already punched or formed and not requiring further punching or machining operations. Therefore, the term "sheet" as used herein is intended to include such disc-like parts.

While I have described preferred products and methods that exemplify the invention, the same are subject to modification within the concepts of my invention. Therefore, I desire to reserve to myself all modifications of product and method that may fall within the spirit and scope of the appended claims.

Having thus described my invention, what I claim and desire to obtain by Letters Patent, is:

1. A sheet comprising a base of integrated mica flakes arranged in random fashion and having therein a multiplicity of spaces, and a cellular skeleton of silica occupying said spaces.

2. A sheet comprising a base of integrated mica flakes arranged in random fashion and having therein a multiplicity of spaces, and a cellular skeleton comprising the silica component of silicone resin occupying said spaces.

3. A method for producing a mica base insulating sheet that consists in impregnating a porous integrated sheet comprised of mica flakes with a solution consisting of a silicone resin and a carrier material of volatile liquid, and, while retaining the porosity of the sheet, by application of heat below the temperature at which the mica flakes dehydrate and above the temperature at which the silicone resin cures, driving off the carrier liquid to leave a cellular skeleton of silica in the pores of the sheet.

4. A method for producing a mica base insulating sheet that consists in impregnating a porous integrated sheet of mica flakes in a solution containing silicone resin and a solvent, evaporating the solvent to leave said silicone resin in the pores of the sheet, applying heat to the sheet sufficient to thermo-set the resin, and, thereafter, applying higher heat than the thermo-setting heat to said sheet at a temperature below the temperature at which the mica flakes dehydrate to drive off from said thermo-set resin substantially all organic matter contained therein to, thereby, leave a cellular skeleton of silica in said pores.

5. A method according to claim 4: the low heat approximating 400° F. to 500° F.

6. A method according to claim 4: the higher heat approximating 1000° F.

7. A method according to claim 4: the low heat approximating 400° F. to 500° F., and the higher heat approximating 1000° F.

8. A method according to claim 4: the step of impregnating cells of the skeleton with a silicone resin after the carrier liquid has been removed from said skeleton.

9. A method according to claim 3: the steps of impregnating the skeleton with successive and additional

amounts of silicone resin, and, finally, applying heat above the temperature at which mica dehydrates to expel a substantial proportion of the waters of crystallization from the sheet.

10. A method according to claim 9: the last mentioned temperature ranging between 1400° F. and 1800° F.

11. A method according to claim 9: the step of simultaneously applying pressure to the sheet while the last-mentioned heat is being applied.

12. In a method for producing a sheet comprised of integrated mica flakes and a cellular skeleton of silica among the flakes, the steps of introducing silicone resin into the sheet in the company of a non-silica evaporative carrier, and, thereafter, applying heat sufficient to remove the carrier by evaporating the same and utilizing said heat to simultaneously eliminate the carbon radical of said resin to leave the mentioned skeleton of silica among the flakes.

13. In a method for producing a sheet comprised of integrated mica flakes and a cellular skeleton of silica among the flakes, the steps of introducing silicone resin into the sheet in the company of an evaporative organic material, and, thereafter, applying heat sufficient to remove the organic material by evaporating the same and

utilizing said heat to simultaneously eliminate the carbon radical of said resin to leave the mentioned skeleton of silica among the flakes.

14. A method for producing a mica base insulating sheet that consists in impregnating a porous integrated sheet of mica flakes with ethyl silicate in solution in a volatile liquid, baking the impregnated sheet to thermoset the ethyl silicate while retaining the porosity of the sheet, and then subjecting the sheet to a temperature below the temperature at which the mica flakes dehydrate and above the temperature at which the organic component of said ethyl silicate is driven off to, thereby leave a cellular skeleton of silica in the pores of the sheet.

References Cited in the file of this patent

UNITED STATES PATENTS

1,578,812	Dawes	Mar. 30, 1926
2,204,581	Denning	June 18, 1940
2,352,974	Rochow	July 4, 1944
2,405,576	Heyman	Aug. 13, 1946
2,524,932	Schulman	Oct. 10, 1950
2,546,474	Peyrot	Mar. 27, 1951