United States Patent Office

3,032,444

Patented May 1, 1962

3,032,444

PROCESS FOR IMPROVING THE ELECTRICALLY INSULATING PROPERTIES OF COMPACTED, PULVERULENT, INSULATING MATERIALS

Marvin D. Scadron, Skokie, and Otto Joseph Cousins, Chicago, Ill., assignors to Aero Research Instrument Company, Inc., Chicago, Ill.

No Drawing. Filed Aug. 13, 1959, Ser. No. 833,381

4 Claims. (Cl. 117—213)

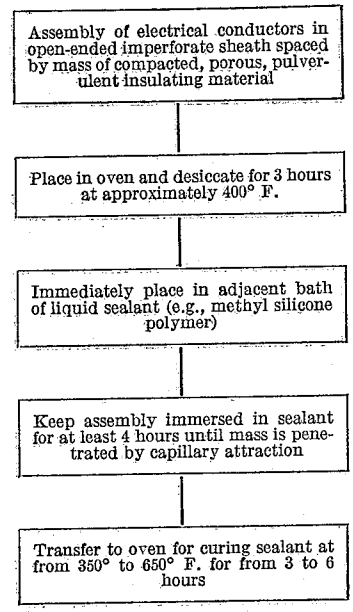
This invention relates to a process for enhancing the electrically insulating properties of tightly-compacted, pulverulent material serving as an electrical insulator and support.

In the manufacture of sheathed and otherwise housed electrical conductors, e.g. single or multiple wires carried within a tubular sheath, dual-conductor thermocouple elements, electrical heating elements and similar sheathed components it has been the practice to position the inner elements in their relation to the tubular sheath, to charge the space therebetween with a comminuted or pulverulent, electrically-insulating material and to compact the material by vibration or otherwise to provide a comparatively dense matrix maintaining the inner elements in predetermined relation to the sheath or housing and to each other. The materials thus used as matrices may be magnesia, alumina, zirconia, beryllia, thoria, etc., or combinations thereof. In most cases the application in which the thus-assembled component is to be used the ohmic resistance between conductors or between conductors and sheath must be held within close tolerances for what must be regarded, for all practical purposes, as an indefinite period of time. Since many of these applications are subject to the normal humidity of the ambient air or even to immersion in a liquid, hygroscopicity of the porous, compacted matrix can deleteriously affect the insulating value thereof.

Heretofore various substances and procedures have been availed of for sealing the porosities to reduce the hygroscopicity whereby the desired ohmic resistance originally established may be maintained notwithstanding a humid environment. However, these prior procedures have left much to be desired; principally because adequate initial penetration of the compacted material has not been possible or the sealing material has not been applied in a manner to yield permanence. Our invention relates to an improved process for effecting the foregoing desideratum.

Regarded in one sense the invention comprises the use of an acceptable fluid incapable per se of adversely affecting the ohmic value of the porous insulating material, and having a viscosity such that the porosities of the material will, by capillary attraction, be infiltrated by the fluid and will, upon solidification by curing, polymerization or the like, permanently seal the pores against entry of water or of other fluids to which the sealant is chemically inert. The gist of the invention resides in extending the times of the steps of the treatment and the temperature at which the same are effected to an extent heretofore not practiced or contemplated in this branch of the art. Moreover, the invention contemplates not only the heating of the fluid but of the work piece as well.

A simplified flow diagram of the process is as follows:



For conciseness of exposition the invention will be described with reference to a thermocouple element, specifically one which comprises a rigid, outer, metallic sheath enclosing a pair of parallel conductors and between which several elements a compacted mass of pulverulent material is interposed, e.g. magnesium oxide. However it will be understood from what follows that the invention is of general application where the electrical insulating qualities of a porous mass of pulverulent, nonconductive material is to be improved by sealing the voids therein and that, moreover, the shape of the housing and the shape and disposition of the conductive elements are not inventively material. That is to say, the inner elements may be rectilinear, spiralled, helical or otherwise formed and may be variously oriented with respect to each other and the outer element.

The work piece to be treated is first desiccated in an air oven at approximately 400° F. for three hours or longer to assure that all occluded moisture is driven off, the time varying in accordance with the moisture content of the insulating material per se, and the extent to which the porosities of the material may have theretofore absorbed moisture.

Immediately upon removal from the oven the work piece is dropped into a trough containing a sufficient quantity of the sealing medium to completely immerse the part. It is important to minimize the delay between removal from the oven and immersion in the fluid. To this end the oven and trough are installed closely adjacent. By so doing the work piece may be transferred immediately from the oven to the bath of sealant with no perceptible time delay which could allow the reabsorption of moisture to defeat the basic purpose of the process. As a sealing liquid we prefer to employ a methyl silicone polymer, which is polymerizable by heat and/or catalysts and which has a viscosity at 77° F. of from 4 to 40 centistokes and at 100° F. of from 36 to 163 centistokes. This fluid is non-corrosive on metals or other materials of construction and is comparatively

unreactive. The piece remains immersed in the sealant for at least 4 hours to insure that penetration of the fluid into the accessible portions of the piece is sufficient. This time may be greater depending upon the degree of porosity and the viscosity of the fluid.

Following this step the piece is transferred to an oven for polymerization. We have found that optimum benefit from the curing stage is obtained at a temperature of 500° F. and for a time of at least 5 hours, and that a circulating air type of oven is preferred. Under some circumstances a time as little as 3 hours may suffice, and the temperature may range from 350° F. to 650° F.

After completion of polymerization a test is made by immersing the sealed areas in water for a few minutes, removing the piece, blowing off the water with compressed air and performing a test for insulating resistance.

We have found that a component treated in accordance with the invention is greatly improved over those processed by prior methods in the atainment of prescribed ohmic resistance and permanence of such condition.

While we have described a particular embodiment of our invention, it will be understood, of course, that we do not wish to be limited thereto since many modifications may be made and we, therefore, contemplate by the appended claims to cover any such modifications as fall within the true spirit and scope of our invention.

We claim:

1. A process for improving the electrical insulating properties of a mass of compacted, porous, pulverulent, finely-divided electrical insulating material in an electrical assembly comprising one or more electrical conductors housed in a rigid, imperforate sheath coextensive longitudinally with the conductors, said mass of compacted, porous, pulverulent electrical insulating material spacing said conductors from each other and from said sheath, the ends of the sheath being open to expose the insulating material, said process consisting essentially of the steps of: placing said electrical assembly in an oven and desiccating the mass of insulating material in said oven under applied heat at a temperature of from

350° to 450° F. for at least three hours to drive off all occluded moisture from the porosities of said material; providing, in close proximity to the oven, a bath of a heat-curable liquid sealant open to the atmosphere and at room temperature, said liquid sealant having a viscosity at that pressure and temperature sufficiently low that the porosities of the insulating material will be infiltrated by the liquid solely by capillary attraction; removing said assembly from said oven and immediately placing it in said bath; allowing the assembly to remain immersed in said liquid sealant for at least substantially four hours until said insulating mass is completely penetrated by said sealant by capillary attraction; transferring the assembly to an oven for curing the sealant at a temperature ranging from 350° to 650° F. for a period of from three to six hours; and removing the assembly from said last mentioned oven.

2. The method in accordance with claim 1 wherein the sealant has a viscosity at 77° F. ranging from 4 to 40 centistokes and at 100° F. ranging from 36 to 163 centistokes.

3. The method in accordance with claim 1 wherein the sealant is a polymerizable methyl silicone.

4. The method in accordance with claim 1 wherein the finely-divided porous insulating material is from the class of refractories comprising magnesia, alumina, zirconia, beryllia and thoria.

References Cited in the file of this patent

UNITED STATES PATENTS

2,664,364	Thom	Dec. 29, 1953
2,709,692	Gainer	May 31, 1955
2,804,404	Thiessen	Aug. 27, 1957
2,821,518	Edelman et al.	Jan. 28, 1958
2,849,527	Rogers at al.	Aug. 26, 1958
2,864,728	Predota et al.	Dec. 16, 1958
2,872,344	Mees	Feb. 3, 1959
2,930,106	Wrotnowski	Mar. 29, 1960

FOREIGN PATENTS

807,746	Great Britain	Jan. 21, 1959