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Nanodielectric Surface Performance When Submitted to Partial Discharges in Compressed Air

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Abstract: Bulk samples of a nanodielectric material were synthesized. This insulating material consisted of a mix of epoxy resin, a SiO_2 load and a percent fraction of inorganic nanoparticles. The aim of the present experiment was to determine the performance of the nanodielectric surface when exposed to partial discharges as compared to that of an epoxy containing only the micrometric SiO_2 load. A discharge situation featuring a triple-junction condition was used. Low-intensity discharges were produced along a gap formed by the interface between compressed air and the bulk sample. The material containing a small amount of nanoparticles was found to resist much more the present discharge conditions, showing an improved performance as compared to that observed in the case of the epoxy without nanoclay.

Introduction

The broad field of nanoscience has shown signs of convergence at the turn of the century. This was considered to have a great potential for insulating systems and dielectrics. It was put forward then [1] that new and improved dielectrics would be found among the nanostructured ceramics and tailored nanocomposites. Nanodielectrics were perceived as emerging as a new class of materials.

Since then, the trend showing an increase in activity has been maintained (see for instance [2-4]), the various efforts producing a diversified wide spectrum of results, and the hope that this would elicit a momentum transfer in the field of electrical insulation and dielectric phenomena has quickly materialized. In the past few years, the number of published scientific contributions in this particular domain has increased considerably. For instance, at the CEIDP2005 alone and for the second year in a row, there will be about 20 papers dealing more or less with nanodielectrics [5]. One of the key points of the recent years, in the field of nanoscience or specifically for the nanodielectrics, has been the recognition of the need for a multidisciplinary approach and the success of implementing it. Past experience permits to speculate that the time counter is running fast for the field to produce some breakthroughs in order to keep the money flowing. A success involving nanodielectrics could be to find and achieve a profitable electrotechnical application.

The present contribution looks at an extrapolated situation dealing with an existing insulating material. It is rather revolution from the inside that is sought. For high-voltage applications, epoxy is often used with a high-percent load of silica. The silica load is sometimes of crystalline nature or could be, but the size of the particles ranges mostly in the micrometric scale although it is observed to be distributed in values sometimes reaching down to nanometers [6]. A down-sizing of the crystals into the nanometric scale and its adjunct to the epoxy could in principle improve the performance of the material. In addition, the percent-load could be reduced considerably as it was observed in many instances that a small quantity of nano-additives brings a substantial change. In this effect, it is contended that surface area associated with the nanoparticles is a leading parameter. But here a different approach is followed. The question is rather how a current system, a two-phase bulk consisting of dispersed particles in a polymeric matrix, can be affected by adding to it nanostructures. Would the performance of it be affected, improved and to what extent? How does the nanometric additive interact with such a two-body system?

In this work, a comparative study was conducted seeking to establish the relative surface resistance to partial discharges of two insulating material systems. The referential system was prepared consisting of epoxy with a high-content silica load. A similar system containing in addition a small amount of nanoclays constituted the nanodielectric. The experimental conditions are essentially those corresponding to low-intensity discharge interactions with dielectric surfaces for which exposure can be maintained over time.

Experimental context

Material preparation

The reference dielectric material consisted of micrometric silica dispersed in an epoxy matrix at a loading of 60% by weight. The epoxy resin consisted of a common resin based on DGEBA (diglycidyl ether of bisphenol A) cured with an anhydride-type harden-

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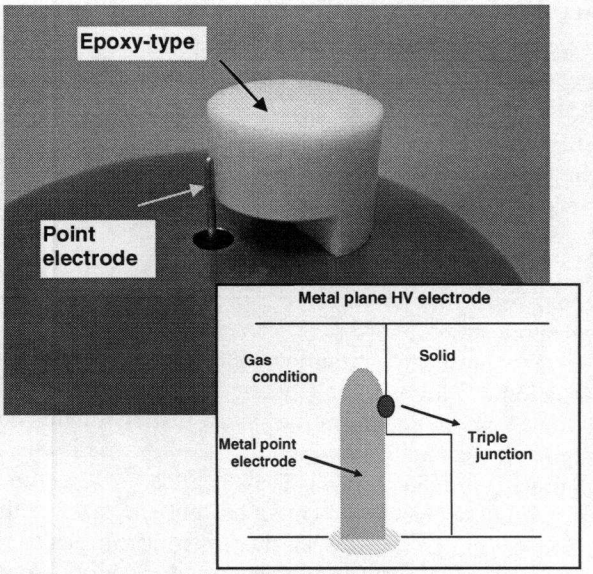

Figure 1: View of the experimental electrode arrangement and schematical definition of the triple junction.

er. The nanodielectric material was identical except that a commercially available nanoclay (a layered silicate treated with organic intercalant) was well dispersed in the epoxy resin before it was mixed with hardener and silica and cured. The amount of nanoclay was chosen so that the content of nanometric silicate in the final product corresponded to 0.45% of the total weight, or 1.1% of the epoxy matrix weight. When such nanoclays are fully well dispersed, they give rise to silicate platelets with a thickness of about 1 nm and a diameter in the range of 100-500 nm. Cylindrical bulk rods were molded in plastic containers. The thus obtained surfaces were used as such in the experiment. However, the cylinders were tailored to the proper dimensions and cleaned for grease and moisture.

Experiment

A detailed description of this type of experiment was provided over the years [7-10], as it forms a basic context for discharge interactions with an insulating surface. A discharge situation featuring a triple-junction condition was used. Low-intensity discharges were produced along a gap formed by the interface between compressed air and the bulk sample. A plane/point/plane electrode configuration was used. Alongside the point electrode that extended from one of the planes, was the solid bulk surface. The gap length was constituted by the interface between the gas medium and the solid material surface. The distance between the point electrode and opposed plane amounted to 5 mm. Figure 1 illustrates the experimental arrangement. The gas pressure was fixed at 5 bars. AC voltage was applied and a voltage-value giving reasonable discharge activity was set. At times, this applied value was re-adjusted to maintain the discharge activity. Discharge characteristics were monitored using an ICCD camera and an ICM. Voltage was applied over extended periods of time, in a continuous or uncontinuous manner, giving a total exposure time.

Discharge conditions

One of the elements of importance from the standpoint of the experiment was to succeed in controlling the applied stresses to the surface under study. The basis of the experiment is to know what type of stresses is applied to the surface. Then, to be able to reproduce and control these stresses over time is an essential in such a comparative study.

Much effort has been devoted to establish a controlled-discharge environment. Knowledge of the discharge situation was clarified. A fibre-optic intensified charge-coupled display detector (FOICCD) with a spectral response between 200 and 600 nm was focused on the area of interest. The camera aims at the point electrode through a sapphire window. The camera is an ICCD type with a possible gain up to 10^6, with a fast response (a max of 1 Msample/s) and is UV enhanced. In addition, the incoming light went through a telephoto (5X) equipped with quartz lenses. Typically, the camera was operated in a continuous mode and opened gate with a gain set at 150 (max being 255). The 16-bit preamplified signal is transmitted via a high-speed serial cable to a dedicated computer via a FO interface. Over a time frame during 100 ms, light was integrated and the resulting image recorded. Figure 2 shows an ICCD rec-

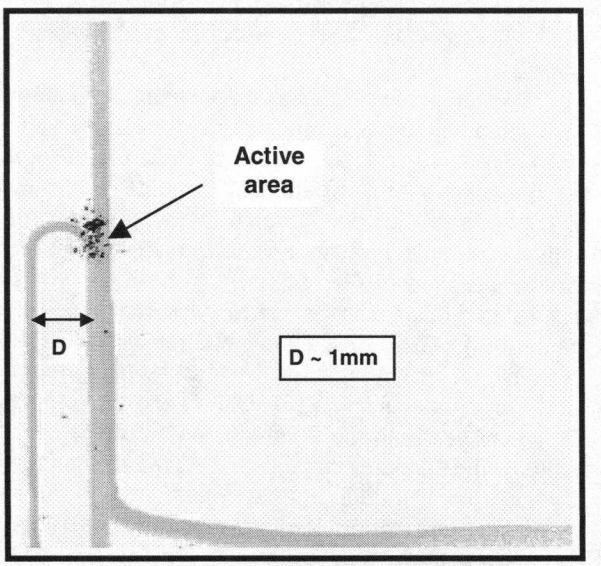

Figure 2: Localization of the light activity observed during the discharge exposure.

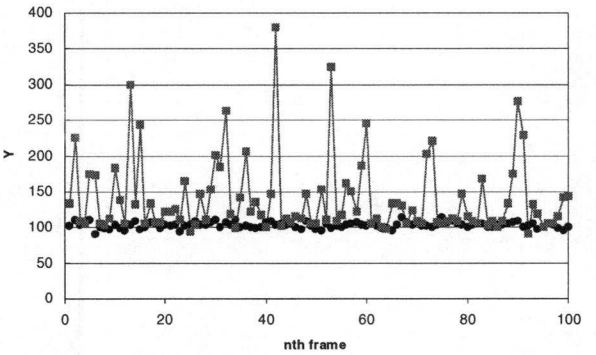

Figure 3: Measured light intensity (Y in a.u.) as a function of consecutive ICCD time frames (nth).

ording of the light that corresponds to a time frame of 100 ms. In the background, in light shade, are featured the epoxy-type bulk and the electrode profile. The use of AC voltage, as opposed to DC voltage, produces a discharge located at the triple junction, which is clearly shown in Fig. 2. Evidence presented in Fig. 3 establishes that the discharge emits almost continuously. In this graph, light intensity in arbitrary unit was displayed as a function of the consecutively recorded ICCD time frame. The darker symbols are ascribed to the background noise.

In such an electrode configuration, the voltage span between corona threshold and breakdown is usually narrow. The applied voltage value was set as to get the partial-discharge pattern as exemplified in Fig. 4. Figure 4 features an ICM recording in which the peak charge is displayed as a function of time. Typically, rates exceeding 25 negative-pulse counts per second with peaks larger than 5 pC were encountered.

Material characterization

The case of epoxy without nanoclay was processed first.

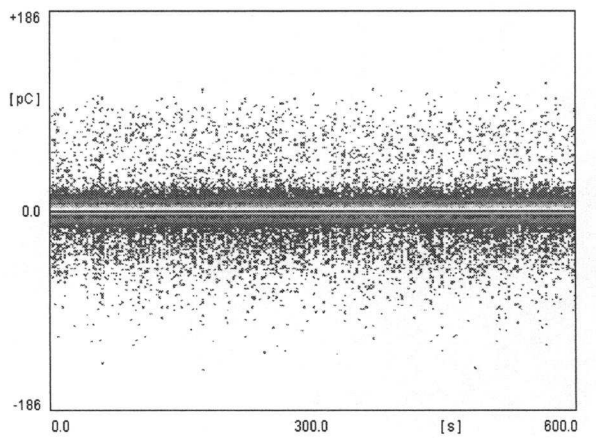

Figure 4: Partial-discharge activity expressed as apparent charge in pC displayed as a function of time.

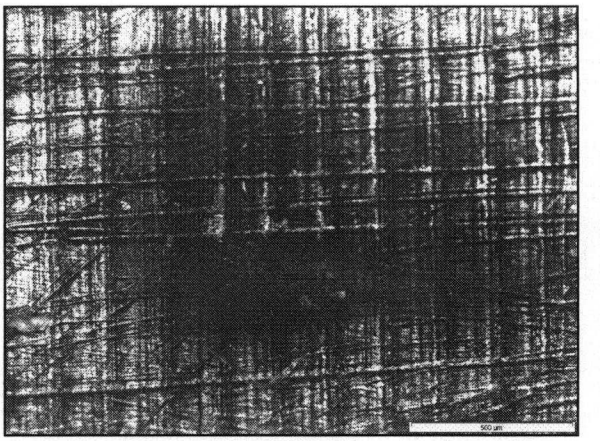

Figure 5: View of the nanodielectric surface after discharge exposure taken with an optical microscope.

The duration of discharge exposure amounted to 13.5 hours. Surface conditions were inspected periodically. At this time, erosion of the surface was apparent. This fixed the reference time line for the exposure duration to be applied to the epoxy containing nanoclay. After the same discharge duration, no apparent surface degradation was observed. Exposure continued to reach a total duration of 25 hours. Then, the bulk was removed and its surface imaged using a depth-of-field compensated optical microscope.

As can be seen in Fig. 5, minimal damage occurred and the original marks associated with the mould are still visible. On the contrary, the surface of the two-body epoxy, thus without clay, showed a substantial damaged area: An oval shape measuring about 500 µm by 1400 µm. Various microscopy techniques were used to characterize the material microstructure and to further investigate this great resistance exhibited by the epoxy containing nanoclay.

The bulk without clay having the eroded surface was cut perpendicularly to its surface with a diamond

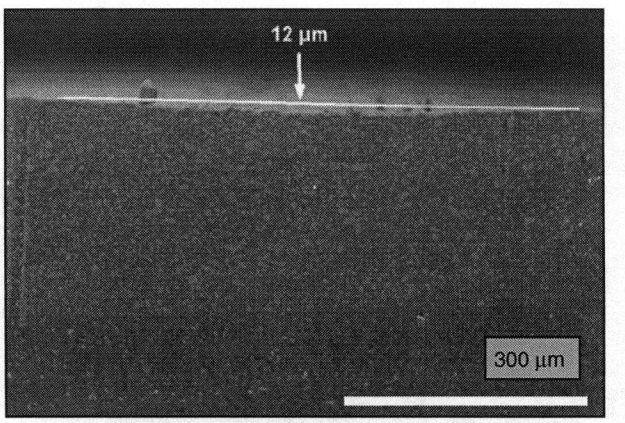

Figure 6a: SEM side view of a perpendicular plane to the eroded surface of the epoxy without nanoclay.

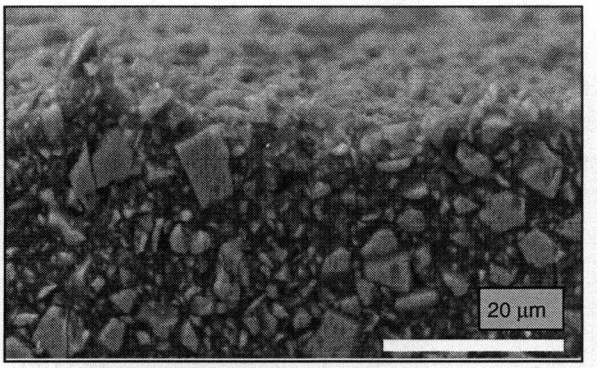

Figure 6b: SEM micrograph of a perpendicular cut of the eroded surface; Epoxy without nanoclay and side view with an angle.

blade. This gave access to the side view of the valley associated with the surface degradation. Observation made with a scanning electron microscope (variable-pressure SEM, 50 Pa) is presented in Fig. 6a where a 12-µm lowering is found at the apex of the degradation valley. Further enlargement as shown in Fig. 6b, again a side view but with an angle this time, permits to image and distinguish the microstructure. Whitish structures below the surface are silica particles. Their nature has been confirmed by running X-ray scattering. They are distributed in size. It has been shown [6] that nanometric silica particles (a few) can exist. But the majority is in the micrometric range. The darker areas seen in Fig. 6a are the organic epoxy matrix. They reflect less and appear darker. Again it was confirmed that in such a situation the larger micrometric particles at the surface or close to it offer the greater resistance to discharges [6].

What is happening with the 2-body epoxy containing nanoclay? What brings the improved performance? The above analysis was applied to the nanostructured epoxy. The SEM micrograph presented in Fig. 7a shows a side view of the sliced bulk. The cut was perpendicular to the discharge-exposed surface. As in the former case, observations are very similar except for the presence now of dark rounded structures having approximate circular diameter of tens of microns. X-rays scattering performed on these structures indicates the presence of alumino-silicates (with traces of iron). Therefore, these are not blobs of polymers. These could be associated with a modification of the fabrication process due to the agglomerated presence of nanostructures or be an artifact of synthesis since the material (and its process) is not optimized. Analysis was pushed further by using a transmission electron microscope. Thin slices having a surface of 30 µm X 30 µm and thickness of 80 nm were obtained from the side plane shown in Fig. 7a using ultramicrotomy. Many of these slices were analyzed by TEM. Two examples are provided in Figs. 7b and 7c. Figure 7b is a typical image of the situation: Nanometric structures have appeared in-between micrometric silica. Typically, the nanostructures are not found close to the micrometric particles or aggregates.

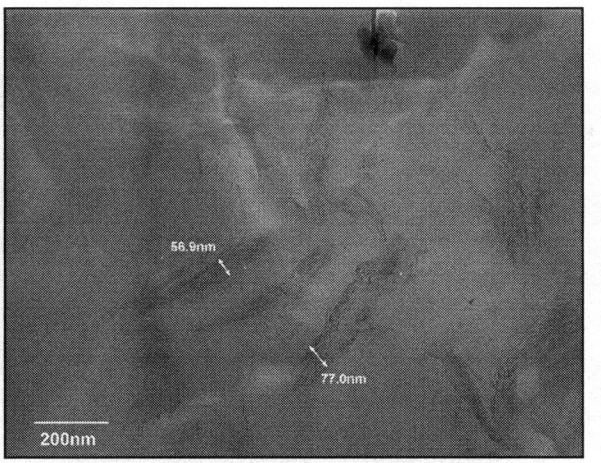

Figure 7b: TEM micrograph of the epoxy containing nanoclay. Grouping of the well dispersed platelets into the epoxy matrix.

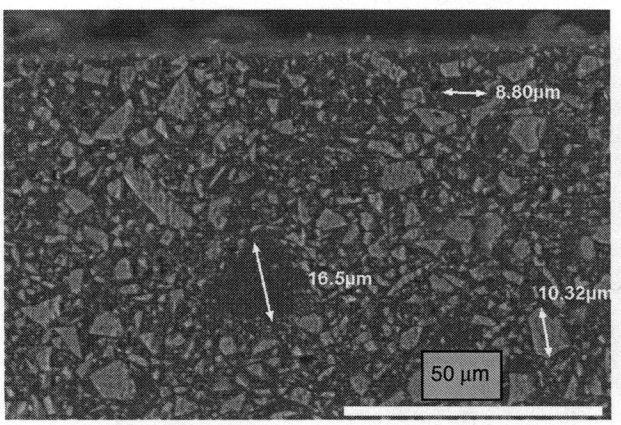

Figure 7a: SEM micrograph showing a side view of the epoxy bulk with nanoclay.

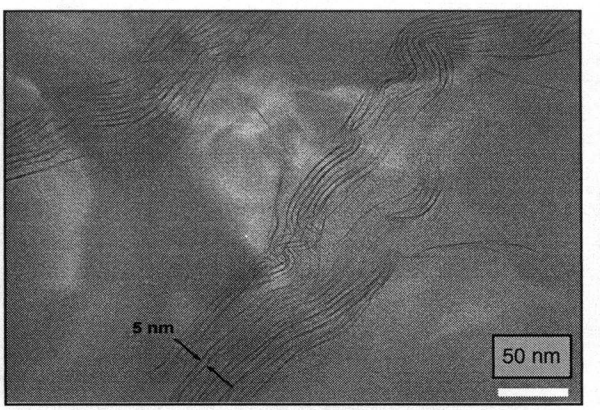

Figure 7c: TEM micrograph of the epoxy containing nanoclay showing the well dispersed platelets of alumino-silicates.

They stand alone in the matrix. Having longer extension (as long as 400 nm) than width (in the range of 50 to 80 nm), they show some structure. These results from the well-achieved dispersion of the nanoclay. Figure 7c presents an enlarged view of the dispersed platelets. The value of 5 mm would tend to indicate some exfoliation, as it exceeds natural inter-cavern distances. However, it is not a measure of the exfoliation status of the bulk. It would appear that the adjunct of nanometric inclusions multiplies the number of bonds and changes the bonding nature with the surroundings.

Closing remarks

Electrotechnical applications make use already of 2-phase epoxy. The present contribution has sought to investigate if adding a small amount of nanometric additives to an existing recipe could revolutionize its performance. In the context, it was found that the adjunct resulted in a substantial improvement of the surface performance resisting to degrading effects associated with partial discharges. The well dispersed platelets were found to group into the epoxy matrix and not observed to interact with the micrometric silica. It gives the image of ligamentary re-enforcement of the weak medium, i.e. the epoxy matrix. The results look promising with the reserve that the material is not optimized and that the properties may depend much on the synthesis process [11-12].

Acknowledgment

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[5] CEIDP stands for Conference on Electrical Insulation and Dielectric Phenomena. The 2005 annual meeting was held in October in Nashville, Tennessee. The 2004 version was in Boulder, Colorado. See the web site: http://ewh.ieee.org/soc/dei/ceidp/

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