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Epoxy-Based SiC Composites with Non-Linear Electrical Properties for Field Grading Applications

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Abstract- Different methods of dispersion for fabrication of SiC/epoxy resin composites were studied in order to obtain an homogeneous filler dispersion. Composites with different filler concentration were also fabricated in order to develop the nonlinear electrical conductivity. The filler concentration was between 0-30 vol%. The thickness of composites was 300 μm. The composites were characterized using a current-voltage technique with an applied voltage between 35-12500 V. The effects of the filler concentration in the nonlinear behavior of materials is presented.

I. INTRODUCTION
In high voltage (HV) applications the insulating material is one of the weakest elements of the electrical system, leading to reliability loss [1]. It is known that the rate of aging depends on the nature of the applied stress. The most important stress types to which insulation is subjected are electrical, thermal, and mechanical stresses [2-4]. When high electric stress is applied to most polymers, charges are injected and accumulated within them, distorting the internal electric field distribution. Consequently, insulation degrades because of space charge accumulation, partial discharge, and electrical treeing across the whole material, which may lead to breakdown [5,6].

One of the key subjects in HV engineering is the control of the electric field strength. The aim is to achieve the lowest possible electrical field strength at a fixed system voltage [7]. One effective technique to control the distribution of the electric field is the nonlinear stress control. It uses materials whose resistivity varies with the applied voltage [8]. These materials can be developed using a base material, which is usually an insulating polymer with an additive that provides nonlinear functionalities. Common additives used are SiC, ZnO, carbon black, or blends of different oxides such as BaTiO₃, TiO₂, SiO₂, Fe₃O₄, and mica [7,9,10]. Finite element method simulations showed that the application of a nonlinear resistive layer at the metallization edge of power electronic substrates significantly reduces the electric field at the critical point between the copper metallization, ceramic substrate, and silicone gel or polyimide encapsulation [11].

In general, nonlinear resistive field-grading materials have a field-dependent conductivity, σ(E), which increases strongly from a low conductivity value, σ(0), to a high value in a narrow field region ΔE near the switching field (Eₛ) [9,12,13]. The value of Eₛ indicates where field grading becomes active [9]. Efficient nonlinear resistive field grading needs a sufficiently large nonlinearity coefficient, α [9]:

\[
\alpha = \frac{d \ln(f)}{d \ln(E)} = 1 + \frac{d \ln(\sigma)}{d \ln(E)} \tag{1}
\]

The final properties of the composite (switching field and nonlinearity coefficient) depend on the characteristics of the filler, such as the filler concentration, size, morphology, and composition.

Due this background, we present the fabrication and characterization of composite materials formed by an epoxy matrix and SiC particles, studying the effect of the filler concentration in the nonlinear properties of the composites.

II. MATERIALS AND METHODS

A. Materials
For composite manufacturing, silicon carbide micropowder (SiC) with a diameter < 1 μm, purity 99+%, crystal type 85% β was utilized as filler. The two component epoxy resin (ER) Polytec EP 630 from was used as the matrix material.

B. Fabrication of homogeneous materials with different filler dispersion methods
In order to study the impact of the fabrication method on the particle dispersion, SiC/ER composites with a filler concentration of 10 vol% were prepared using different dispersion methods. For all the process the particles were dried at 300 °C for 1 h and after kept at 100 °C for one day.

Process 1: The dried fillers were mixing with the epoxy resin part A in a concentration of 10 vol% using a planetary vacuum mixer with a vacuum of 1.0 kPa and 800 rpm for 3 min, then 1.0 kPa and 1800 rpm for 1 min. After, 10 wt% (with respect to part A) of epoxy resin part B (hardener) was added and mixed using the same process in the planetary vacuum mixer.

Process 2: The dried fillers were dispersed on epoxy part A with a filler concentration of 10 vol% using the shear mixer at 500 rpm for 15 min. Then, 10 wt% of epoxy resin part B was added and mixed using the planetary vacuum mixer with vacuum of 1.0 kPa and 800 rpm for 3 min, then 1.0 kPa and 1800 rpm for 1 min.

Process 3: The dried fillers were dispersed on epoxy part A with a filler concentration of 10 vol% using the ultrasonic probe with a power of 950 watts, amplitude of 40%. Then, 10 wt% of epoxy resin part B was added and mixed using the planetary vacuum mixer with vacuum of 1.0 kPa and 800 rpm for 3 min, then 1.0 kPa and 1800 rpm for 1 min. The effective time of ultrasound was 15 min, with a cycle of 5s on/15s off.

For all the process, samples with a thickness of 300 μm and diameter of 44 mm were manufacturing using a pressing process. The composites were fabricated using a temperature of...
100 °C, a pressure of 150 N/cm² for a time of 55 min. Finally, the samples were post-curing in an oven at 120 °C for 1 h.

C. Fabrication of homogeneous materials with different filler concentration

In order to study the impact of the filler concentration, SiC/ER composites with different filler concentrations were prepared. The particles were dried at 300 °C for 1 h and after kept at 100 °C for one day. Then, the dried fillers were dispersed on epoxy part A with filler concentrations of 0, 5, 10, 15, 20 and 30 vol% using the ultrasonic probe with a power of 950 watts, amplitude of 40 %, 15 min of effective time with 5s on/15s off cycle. After, 10 wt% of epoxy resin part B was added and mixed using the planetary vacuum mixer with vacuum of 1.0 kPa and 800 rpm for 3 min, then 1.0 kPa and 1800 rpm for 1 min. After, samples with a thickness of 300 μm and diameter of 44 mm were manufacturing using a pressing process. The composites were fabricated using a temperature of 100 °C, a pressure of 150 N/cm² for a time of 55 min and post-curing in an oven at 120 °C for 1 h.

D. Morphological and electrical characterization

In order to analyze the dispersion and distribution of fillers in the epoxy resin, scanning electron microscopy (SEM) was used.

In order to describe the electrical behavior of composites and to find the nonlinearity, the current-voltage (I-V) dc characterization was carried out. Samples of 300 μm were metalized with gold using a configuration of three electrodes (see Fig. 1). The diameter of top electrode was 33 mm and the guarded electrode was 25 mm.

For I-V characterization, a dc voltage source “FUG HCN 35-3500” was used for electrical characterization between 10-3500 V and a dc voltage source FUG HCN 350-12500 for electrical characterization between 1.0-12.5 kV. The current was measured using a picoammeter Keithley 6485. The connection was made in a Faraday cage. The voltage was applied by steps with logarithmic increment, using 20 points per decade. For each step, the voltage was applied for 300 s (polarization). Then, the corresponding current for the applied voltage was measured. After, the voltage was set to 0 V for 300 s (depolarization). Then, the voltage was set to the next value and the process was repeated. After I-V measurements, the electric field $E$ and conductivity $\sigma$ for each sample were calculated, and the graph of $\sigma$ as a function of $E$ was plotted. Finally, the switching field $E_b$ and the nonlinear coefficient $\alpha$ were calculated.

III. RESULTS AND DISCUSSIONS

A. Effect of the fabrication method on filler distribution on composite materials

In order to compare the filler dispersion difference between the dispersion made with the planetary mixer, the shear mixer and ultrasonic probe, the SEM images for 10 vol% SiC/ER composites for process are presented in Fig. 2. It can be observed in Fig. 2a y b that composites fabricated with planetary and shear mixer presented SiC agglomeration with a considerable size. On the other hand, for composites prepared with the ultrasonic probe (Fig. 2c) no agglomerate was observed; for that reason, composites with different filler concentrations were prepared with this method.

B. Influence of filler on the nonlinear properties of composite materials

In order to describe the influence of SiC concentration on the electrical behavior of composites, the $\sigma$ as a function of $E$ is presented in Fig. 3 for different filler concentrations. The nonlinear behavior of composites was analyzed, the switching field and the nonlinear coefficient were calculated and presented in Table 1.

As we can observed in Fig. 3 and Table 1, for epoxy resin material the conductivity is constant for low electric field. When the electric field arrive around 12.7 kV/mm the conductivity behavior changes and the nonlinearity begins. As it can be observed, after the switching, field the electrical conductivity increases as a function of electric field applied with a nonlinear coefficient of 2.39. When 5 vol % of SiC is added to epoxy resin, it can be observed that the switching field reduce to 9.4 kV/mm and the nonlinear coefficient is bigger than for epoxy material. As it can be observed in Table 1, the switching field reduces when the filler concentration increases.
The influence of SiC concentration on the nonlinear electrical behavior of composites were studied for composites fabricated with filler concentration between 0-30 vol%. The switching field for the composites reduces when the SiC concentration increases and it was between 12.74 and 4.2 kV/mm. The nonlinear coefficient increases slightly when the concentration of SiC increases and it was between 2.39 and 6.40.

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