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An original in-situ way to build field grading materials (FGM) with permittivity gradient using electrophoresis

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*Abstract***—This paper reports on an original method developed at LAPLACE to structure field grading materials (FGM) by local handling of high permittivity ceramic (SrTiO3) particles in an epoxy resin using electrophoresis (i.e. DC voltage application). This new way of structuring composites allows building FGM with a high permittivity region (region with high particle concentration) while the rest of the composite remains with lower permittivity (region with low particle concentration). The resin curing enables to 'freeze' the particles with this spatial arrangement. Process details and dielectric characterization of each region of the FGM composites are reported. Finally, a clear demonstration of the FGM performances, used as a field grading encapsulation material for high voltage power electronics modules, is shown supported both by field repartition simulation and experimental breakdown voltage improvements of encapsulated direct bonded copper (DBC) substrates.**

Keywords—Functionally graded materials; FGM; Epoxy; SrTiO3; Composites; Permittivity; Electrophoresis; Field grading; Encapsulation; Power electronics; Power modules.

I. INTRODUCTION

In power electronics, the recent development of high power semiconductor devices such as Insulated Gate Bipolar Transistor (IGBT) in silicon or Metal Oxide Semiconductor Field Effect Transistor (MOSFET) in silicon carbide needs higher performances of the encapsulation electrical insulation materials (higher dielectric breakdown and partial discharge resistance) in order to increase the voltage switching and/or to enhance the reliability of the power module assemblies. Usually, such encapsulation is achieved using silicone gel, even if integrated modules could allow using epoxy resin. However, during the high voltage (HV) operation of a module, partial discharges and electrical treeing can be initiate at the *triple point*, namely at the junction between the ceramic substrate, the metallization and the encapsulation (see red circle in Fig. 1). Treeing can then propagate at the interface between the substrate and the encapsulation towards the grounded counter electrode [1-4]. This triple point acts as an electrical field-strengthening region where the propagation of these discharges causes electrical degradation of the encapsulation. This may lead to the failure of the insulation in the power modules.

The need of electric field control materials, named *field grading materials* at sharp edges is a common issue in HV electrical engineering to use the material properties to reduce the field reinforcement. In power electronics, various FGM have been tested so far to reduce local enhancements of the electrical field at the triple point [5-7] with different efficiencies. However, none of them has tried so far to directly functionalize a FGM, based on a polymer/ceramic composite, by a local auto-tailoring of the electrical properties of the encapsulation only in the region where it is necessary: at the triple point. From the simple observation that the constraints in the electronic systems are mainly localized, it should be more relevant to design the composite properties in an anisotropic way or in the form of a gradient by structuring them locally. In that purpose, *functionally graded materials*, used for HV field grading, are a class of materials that possess a continuous spatial evolution of at least one property (permittivity or conductivity) in one direction thanks to a progressive evolution in their structuration (the filler content in the case of composites) [8, 9], as shown in Fig. 2.

Fig. 2. Effect of spatial particle arrangement on the composite permittivity: (a) homogenous composite, (b) functionally graded composite material.

Electrophoresis is a physical process, which consists in handling particles within a fluid medium under the application of an electrostatic force (i.e. the electrophoretic force) on the compound. Even if electrophoresis has been extensively used so far for

co-ceramic composites, it has never been used to design polymer/ceramic FGM composites, neither applied to field grading in power modules.

In this paper, the authors propose an original way to tailor by electrophoresis advanced FGM polymerbased composites with permittivity gradient, recently patented [10]. A particular example of a FGM composite with a permittivity gradient based on epoxy resin with strontium titanate $(SrTiO₃)$ is reported. Process details and dielectric characterization of each region of the FGM composites are given. Finally, the FGM is applied, as a field grading encapsulation, on DBC substrates to evaluate the enhancement of the breakdown voltage for power electronics application.

II. EXPERIMENTAL

A. Materials

The polymer used as the host matrix is a diglycidyl ether of bisphenol F (DGEBF) epoxy resin. It has been crosslinked using an amine-type hardener with a ratio of 10:1 (resin:hardener). The viscosity of the epoxy resin is 3000 mPa.s at room temperature and its density after curing is 1.23 $g/cm³$.

The high permittivity fillers used are strontium titanate ($SrTiO₃$) particles with an average diameter of 850 nm and a density of 5.11 $g/cm³$.

B. Electrophoresis process

A homogeneous epoxy/SrTiO₃ composite mixture was processed by direct blending of 10 vol% of $SrTiO₃$ particles into the liquid epoxy resin containing the hardener. Firstly, the mixture was homogeneously dispersed using a planetary mixer at 3000 rpm during 8 minutes. Secondly, it was then degassed in a primary vacuum. Finally, it was poured between two stainless steel plates $(7\times7 \text{ cm}^2)$ separated each others by a 1 mm-thick insulating spacer (see Fig. 3a). The FGM are obtained by applying a DC electric field on the liquid composite compound, before curing it in order to 'freeze' the particles exactly where they have been accumulated (see Fig. 3b). DC voltages have been applied on the electrodes using a Keithley SM 2410 voltage source ranging 125 V up to 500 V in order to build the particle concentration gradient at the HV electrode. After switching the voltage off, the sample cell was stored into an oven in order to cure the epoxy/SrTiO₃ FGM compound and to 'freeze' the particles. The samples were cured at 150 °C for 20 min in air for crosslinking the epoxy resin.

C. Measurements

The permittivity (*ε'*) of the FGM regions has been measured by broadband dielectric spectroscopy using a Novocontrol Alpha-A spectrometer with a ZGS testing cell. Measurements have been performed at room temperature in a frequency range from 100 mHz to 1 MHz. Each region of the FGM samples was characterized after a mechanical polishing of the other one and gold sputtered electrodes deposition.

AC breakdown voltage measurements were achieved between two lateral Cu metallization tracks (separated by 500 μm) of encapsulated alumina DBC substrates, similar to the configuration presented in Fig. 1 The tests were performed at 50 Hz and 25 °C using a 100 kV $_{\rm rms}$ transformer at a rising voltage of 1.5 kV/s. For the measurement, each sample was immersed in Galden fluorinert to avoid surface flashover.

Fig. 3. Electrophoresis process principle under DC voltage for structuring FGM polymer/ceramic composites.

III. RESULTS AND DISCUSSION *A. Particle gradient formation*

Fig. 4 presents cross-section SEM images of the FGM epoxy/SrTiO₃ composites structured by electrophoresis under different DC electric fields for 15 min. In DC field, one can observe the formation of a SrTiO₃ particle layer collected at the HV electrode. This layer progressively increases in thickness with increasing the magnitude of the field to reach between 50 μm and 110 μm. In a homogeneous field (parallel electrodes), this layer has a conformal geometrical aspect and covers the entire electrode set at HV. The rest of the FGM composite (i.e. depleted bulk region) remains with a $SrTiO₃$ filler content very close to the initial one with homogeneously dispersed particles.

Fig. 4. Cross-section SEM images of the epoxy/SrTiO₃ composites (initial filler content: 10 vol %) for different DC electric field during the electrophoretic process for 15 min.

Fig. 5 shows the expansion in the FGM accumulated particle layer thickness for different electric field and time conditions. The thickness of the particle layer increases logarithmically with time for a given field. Moreover, the thickness inhomogeneity increases as well (see error bars around the average thickness) for the higher fields. These plots advice us that it is better to design the FGM during a short time at higher fields or to use low fields.

Fig. 5. Thickness changes of the epoxy/SrTiO₃ accumulated particle layer for different electric field and time conditions.

B. Permittivity of the FGM in each region

Fig. 6 presents a comparison between the dielectric permittivity of the 2 regions of the FGM epoxy/SrTiO₃ composite (process at 250 V/mm for 15 min) with the homogeneous epoxy/SrTiO₃ composite (10 vol) and the neat epoxy.

Fig. 6. Permittivity values in each region of the FGM (process at 250 V/mm for 15 min) and comparison with the homogeneous composite and the neat epoxy.

While the neat epoxy and the homogeneous $epoxy/SrTiO₃$ composite have permittivity values of about 4 and 6.5, respectively, the FGM exhibits two very different behaviors. Firstly, one can observe that the permittivity of the depleted bulk region is close to the one of the homogeneous composite with a slight decrease. Such a value is related to the fact that the particle filler content of the depleted bulk region is roughly unchanged during the electrophoretic process. This has been confirmed by volume density measurements in this bulk region with a value of 9 vol% close to the initial filler content before the

process (10 vol%). On the contrary, when this 1 vol% of depleted particles is accumulated in the 80 μmthick particle layer at the HV electrode, the particle concentration is increased up to 40 vol% in these process conditions. Consequently, the permittivity of the accumulated layer is strongly increased with values ranging between 22 and 24 in the frequency range under test.

C. FGM application for field grading in power electronics modules

The FGM composites has been applied between the two lateral Cu electrodes of DBC alumina substrates with sharp profiles representative of those observed in power modules with a classic chemical etching, as shown in Fig 7a. In this lateral configuration, one can observe that with the electrophoresis process the particle layer with the highest permittivity covers the HV electrode mainly around the two sharp tips where the field reinforcements are definitively the highest.

Fig. 7. (a) Application of the FGM to metallized alumina DBC substrates used in power electronics modules. (b) Electrostatic simulations of the positive effect of the FGM high permittivity layer on the field grading at the triple point.

Such a self-adaptation of the particle layer with high permittivity in the vicinity of the 'needle effect'

regions of the module appears as a great novelty in the design of tailored insulating materials because it paves the way to advanced materials able to selectively 'heal' the defects that can occur during the system packaging assembly.

The field grading efficiency of the FGM with permittivity gradient has been firstly shown using finite element simulations (see Fig. 7b), where the field reinforcement at the triple point is largely attenuated by a factor 3 compared to that of the homogeneous composite.

To confirm experimentally the field grading efficiency at the triple point with the FGM, AC breakdown measurements between the two lateral Cu electrodes of various encapsulated DBC substrates with neat epoxy, homogeneous composite and the FGM with permittivity gradient were performed (see Fig. 8). The results highlight clearly the large enhancement of the breakdown values in the case of the FGM with permittivity gradient with 70% and 30% of improvement compared to neat epoxy and the homogeneous composite, respectively.

Fig. 8. Weibull plot of the AC breakdown voltage of encapsulated DBC substrates between Cu tracks separated by 500 um. Comparison of the type of encapsulation: neat epoxy, homogeneous composite and FGM with ε-gradient.

However, the improvements should be even more important than that because an analysis of the breakdown location afterwards has shown that the FGM has not broken down between the two Cu tracks of the substrate but the failure occurred above the FGM at its interface with the Galden fluorinert liquid. On the contrary, both the neat epoxy and the homogeneous composite always failed between the two tracks. This highlights the efficiency of this new field grading approach.

IV. CONCLUSION

For the first time, this paper reports on an original method to tailor innovative functionally graded materials (FGM) by local handling of high permittivity ceramic particles in a polymer using electrophoresis. This new way of tailoring composites allows building FGM with permittivity gradient between the high voltage electrode (high *ε*) and the ground (low *ε*) directly in-situ of a power systems.

The accumulated and concentrated particle layer exhibits permittivity several times higher than the depleted bulk wherein the concentration is quasi unchanged. A clear demonstration of the FGM performances has been reported through both electrostatic simulations and breakdown voltage measurements on encapsulated DBC substrates used in power electronics modules. The results have shown an impressive attenuation of the field reinforcement at the triple point while the breakdown voltage has been largely enhanced. Such an auto-tuned FGM, in-situ tailored directly using the electrodes of an electronic systems, paves the way to adress the field reinforcement issues (exactly where they are located) at the origin of the electrical aging of the insulating materials.

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