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Field Grading Composites Tailored by Electrophoresis – Part 1: Principle and Permittivity Gradient in Uniform Electric Field

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ABSTRACT

A series of three articles present an innovative way to build advanced functionally graded materials (FGM) based on polymer/ceramic composites tailored by electrophoresis from the process principle to their field grading application in power electronics. In this Part 1, the process is presented and relies on applying a DC voltage on liquid composite compound before curing in order to ‘freeze’ the particles where they have been accumulated. To exemplify this principle, FGM composites involving an epoxy resin with SrTiO₃ high-*k* particles with a permittivity gradient are presented. The methodology to build and characterize them is carried out. An accumulated particle region is observed at the high voltage electrode while the depleted bulk region remains unmodified. This accumulated particle layer both increases in thickness and in filler densification with increasing the field and/or time allowing tuning its permittivity. This work paves the way to the development of more robust electronic systems where the electrical fringe field in critical regions can be mitigated.

Index Terms — functionally graded materials (FGM), composite, electrophoresis, insulation, encapsulation, high voltage, field grading, power electronics modules

1 INTRODUCTION

WITH the rapid and recent progresses in the development of advanced electronics systems and active devices, the polymer materials often used for the packaging of such systems or as active materials are facing physical limits thermally, electrically and mechanically during aging under combined multi-stress conditions [1],[2]. This is the case for polymeric dielectrics and insulators, organic joints or adhesives and printed circuit board substrates and encapsulation. Polymers such as epoxy resin, polyvinylidene fluoride, polypropylene, polyimide, silicone gel, silicone rubber are usually under consideration in these applications.

Composite materials, based on the mixing of both a polymer matrix filled with inorganic particles (ceramic or metallic), appear in recent years more and more useful in electronic applications to mitigate the physical constraints and to push up the polymer limits (e.g. enhanced intrinsic performances, higher stability, increase in the lifetime) [3, 4]. So far, composites enable a fair tradeoff in the material properties trying to improve the weakest one without being, as much as possible, detrimental for the other ones. Polymer-based composites should become ubiquitous to enhance electrical, thermal and mechanical performances of energy

conversion systems in many electronic application fields such as high voltage engineering [5-7], power electronics [8, 9], microelectronics [10], capacitor technologies [11, 12], batteries [13].

However, in most of these applications, one can observe that the physical constraints to which these materials (composites or not) are subjected are mostly non-homogeneous with strong anisotropic characteristics: e.g. local electric field reinforcements at metal track edges, unidirectional heat extraction, in-plane CTE mismatching between materials, etc. To overcome these issues, the community working on composite materials has always had, so far, a standard functionalization approach. Particularly, researchers have developed a lot of composites with homogeneous distribution of the inorganic particles within polymers, thus modifying one (or more) property *isotropically* in the entire bulk. From the simple observation that the constraints in 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.

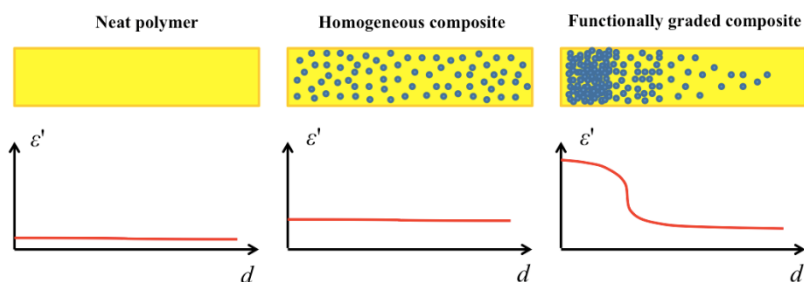


Figure 1. Impact of high permittivity particles arrangement in a polymer composite on the permittivity gradient along a distance: (a) neat polymer, (b) particle homogenous distribution, (c) particle gradient distribution (FGM with gradient).

Functionally graded materials (FGM) are a class of materials that are tailored to have a spatial evolution of at least one property in one direction thanks to a gradual evolution in their structuration. They can be processed either by multilayer deposition (i.e. each layer owns specific properties) or in the case of composites by creating a particle spatial distribution thus offering a property gradient within an initial isotropic matrix [14]. In this latter case, a certain gradient in the electrical conductivity, wear resistance or other mechanical properties can be created in order to smooth or adapt the constraints between materials. Figure 1 exemplifies the principle of the effect of high permittivity particle spatial distribution in a matrix on the overall composite permittivity in the direction of the gradient. Several studies have already reported that such FGM polymer-based composites with particle distribution gradient, processed using centrifugation, exhibit properties gradient in the direction of the applied centrifugal force. Those materials found applications in high voltage engineering for electric field grading [15, 16], as well as in space-craft applications by tuning the elastic modulus [17 18]. However, for more integrated electronic systems (as power modules), such centrifugation process is no longer suitable to tailor locally a property gradient exactly where the electric field constraints are the most severe. A well-known and potential suitable process to accumulate particles, but never used for such applications, could be the electrophoresis.

Electrophoresis is a physical process, which consists in displacing particles within a liquid medium under the application of an electrostatic force (i.e. the Coulomb force) on the compound [19]. The trapping of electric charges at the interface between the particles and the liquid enables the particles to move towards the electrode of opposite potential. Even if electrophoresis has been extensively used to develop co-ceramic FGM composites [20], it has recently been firstly used and reported for polymer-based composites and in that purpose by our LAPLACE research group [21-23]. Since then, another study has reported also the interest of similar epoxy/BaTiO₃ FGM for HV application [7].

In this paper, the authors propose an original way to tailor by electrophoresis advanced FGM polymer-based composites with properties gradient [21]. Such FGM are obtained by applying a DC voltage on the liquid composite compound before curing it in order to ‘freeze’ the particles exactly where they have been accumulated. Such process is very versatile and adaptable from small size to large-dimensions electronic and electrical systems by directly

using the electrodes of the systems themselves. A particular example of a FGM composite with a permittivity gradient (epoxy/SrTiO₃) is developed all along this series of papers for explaining the process, the characterization methodologies and its application for the encapsulation of power modules.

2 EXPERIMENTAL

2.1 MATERIALS

In this study, the polymer used as the host matrix is a diglycidyl ether of bisphenol F (DGEBF) thermosetting epoxy resin (cf. Figure 2). It is crosslinked using an amine-type hardener with a ratio of 10:1 (resin:hardener). The viscosity of the epoxy resin and the harder mixture is 3000 mPa.s at room temperature and its density after curing is 1.23 g/cm³ and a large and high continuous operating temperature range (-55 to 230 °C) compatible with standard power electronics applications (125 °C).

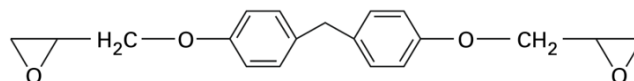


Figure 2. Chemical structure of DGEBF epoxy.

The high permittivity fillers used here are strontium titanate (SrTiO₃) particles with a density of 5.11 g/cm³ and a BET specific surface of 1.83 m²/g. SrTiO₃ has the twofold advantages of a negative Curie temperature not interfering with the application range, and of a higher dielectric strength (35 kV/mm) compared to BaTiO₃ (approx. 5 kV/mm).

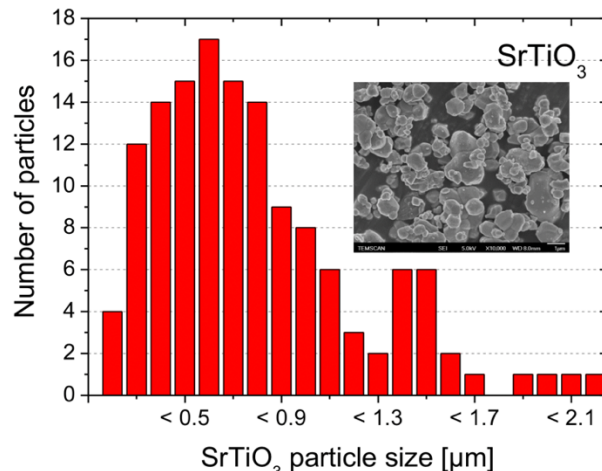


Figure 3. SrTiO₃ particle size distribution. The inset shows a SEM image of the particles (the scale bar is 1 μm).

SrTiO₃ particles used are polyhedral-shaped and present an average diameter of 600 nm, as shown in Figure 3.

2.2 ELECTROPHORESIS PROCESS

A first step in the composite mixture elaboration process was achieved by a direct blending process of the SrTiO₃ particles into the liquid epoxy resin containing the hardener. The liquid composite mixture, with an initial filler content of 10 vol%, was homogeneously dispersed using a planetary mixer at 3000 rpm for 8 minutes. The mixture was then degassed in primary vacuum (<1.5 kPa).

For real-time optical microscopy observations of the SrTiO₃ particle movements in the resin, the mixture was poured between two Cu parallel plates (1mm×5mm×20mm) separated by 1 mm-thick glass spacers and glued on a glass slide (see Figures 4a and 4b). DC electric fields were applied on the Cu electrodes using a voltage source Keithley SM 2410 ranging 125 V up to 500 V and for durations from 1 to 60 minutes (see Figure 4c). After switching the voltage off, the sample cell was put into an oven in order to cure the FGM epoxy/SrTiO₃ compound and to ‘freeze’ the particles where they have been accumulated. The samples were cured at 150 °C for 20 min in air for crosslinking the epoxy resin (see Figure 4d). For making larger FGM samples suitable for dielectric characterizations, the same process was achieved in stainless steel molds of larger area (7×7 cm²). In this case, the DC voltage was applied between the two sides of the mold separated by an insulating joint spacer of 1 mm-thick. The same curing protocol was used.

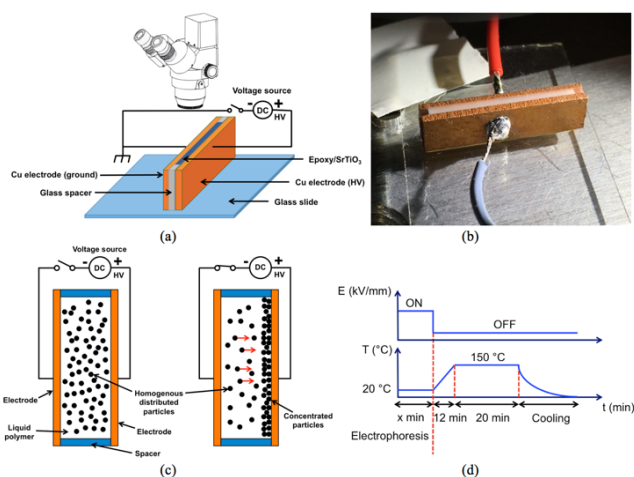


Figure 4. Principle of the electrophoresis process under DC voltage applied to a liquid polymer/ceramic compound and curing process.

2.3 MEASUREMENTS

The FGM accumulated layer thickness and the particle distribution were characterized using a field-emission gun scanning electron microscope (FEG-SEM JEOL JSM-7800F) coupled to energy dispersive spectroscopy (EDS), which enables to qualitatively and quantitatively identify the elemental distribution of Sr and Ti atoms from the SrTiO₃ particles. The weight fractions of SrTiO₃ particles present in

each region (accumulated layer and depleted zone) of the FGM epoxy/SrTiO₃ composites were determined by thermogravimetric analysis (TGA) using a Perkin-Elmer Instruments Diamond analyzer. The measurements were achieved on around 10 mg of composite matter picked from each region of the FGM after polishing mechanically the other one. The measurements were performed with a temperature rising of 10 °C/min up to 800 °C in air atmosphere in order to totally degrade the epoxy matrix and to only conserve the particle fraction. The SrTiO₃ volume fraction was then calculated using the particle and epoxy densities. The dielectric permittivity (ϵ') 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 was characterized after polishing the other one and deposited gold sputtered electrodes.

3 RESULTS AND DISCUSSION

3.1 PARTICLE GRADIENT FORMATION

During the experiments, it was possible to highlight that the dynamics of the particle concentration towards the higher potential electrode was strongly influenced by many parameters such as the field magnitude, time, the field polarity, the initial filler content, the particle size, the particle permittivity/ conductivity, the matrix permittivity/ conductivity, the zeta potential around the particles and the viscosity of the resin.

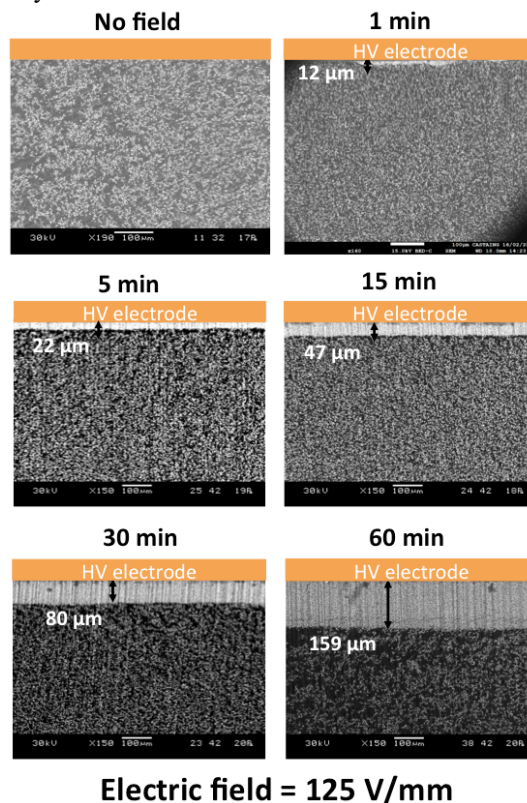


Figure 5. Cross-section SEM images of the epoxy/SrTiO₃ composites (initial content: 10 vol%) structured by electrophoresis in a DC electric field of 125 V/mm for different times.

Even though the complete study that deals with the influence of all those parameters was carried out [23], only the influence of the electric field magnitude and time is detailed in the present report.

Figure 5 presents cross-section SEM images of the FGM epoxy/SrTiO₃ composites structured by electrophoresis under a DC electric field of 125 V/mm and for different times. Under DC field, the formation of a SrTiO₃ particle layer collected at the HV electrode is observed. This layer progressively increases in thickness with time starting from few microns up to more than 100 μm after 1 h showing a first possibility to tune this layer in the desired dimension. This layer exhibits conformal geometrical aspects and covers the entire electrode set at HV. The rest of the FGM composite (i.e. the depleted region) still contains SrTiO₃ particles homogeneously dispersed in the epoxy bulk.

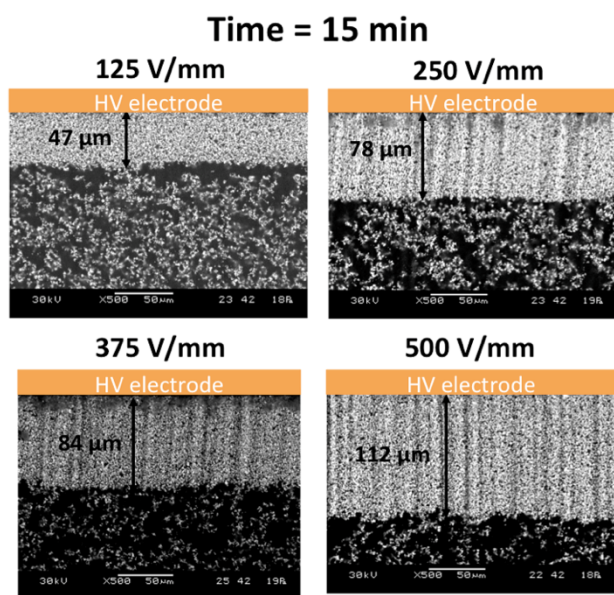


Figure 6. Cross-section SEM images of the epoxy/SrTiO₃ composites (initial content: 10 vol%) structured by electrophoresis at different DC electric fields for 15 minutes.

Figure 6 shows cross-section SEM images of the FGM epoxy/SrTiO₃ composites after the structuration during 15 minutes under different DC electric fields. In this case, one can observe the effect of increasing the electric field on the dynamics of the particle layer deposition. For such a short duration of the field application, the conformity of the layer remains homogeneous. Moreover, as the layer thickness increases with the field, the depleted region seems to present a clear decrease in its filler density. Such higher depletions of the SrTiO₃ particles from the bulk of the composites could be related either to a more important densification in the particle layer with the field increasing or just to its thickness increase at a constant content inside. This will be emphasized in the following parts.

3.2 INHOMOGENEITY ISSUES AT LONG TIMES

Figure 7 shows cross-section SEM images of the FGM epoxy/SrTiO₃ composites after the structuration during 60 minutes under different DC electric fields. It is worth to note

that combining both high electric fields and long application times is detrimental in terms of the particle layer homogeneous conformation.

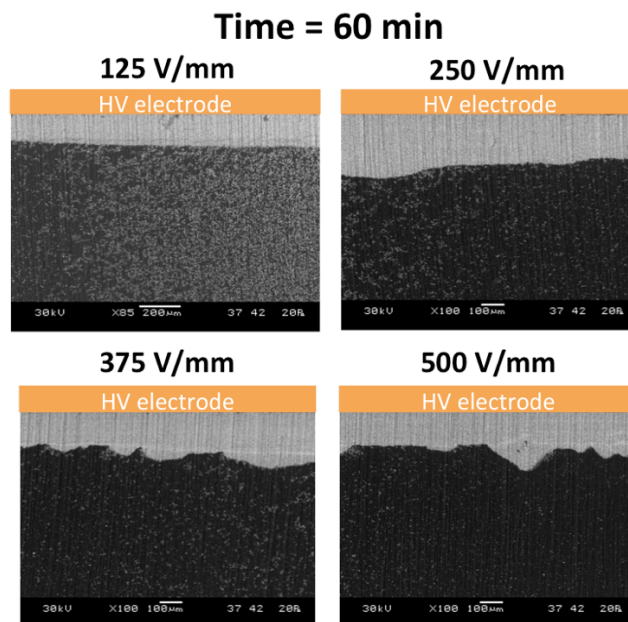


Figure 7. Cross-section SEM images of the epoxy/SrTiO₃ composites (initial content: 10 vol%) structured by electrophoresis at different DC electric fields for 60 minutes.

Actually, after 1h of electrophoresis, one can clearly observe from 250 V/mm and above that the FGM epoxy/SrTiO₃ composites exhibit non-uniformities in the growth of the particle layers. These last ones present large variations of their thickness along the HV electrodes. Such thickness inhomogeneities could be explained by the progressive occurrence during the electrophoresis process of localized regions of higher particle concentrations leading to high permittivity/conductivity regions. Thus, these particle aggregations in the layer could act as higher electric field regions that locally enhance the field viewed by the other particles in movement. Consequently, they could be preferentially attracted by these higher electric field areas, thus building peaks of particles within the accumulated layer. Moreover, such a combination between the high field and its long application time leads at the end to an almost complete depletion of particles from the rest of the composite (e.g. at 500 V/mm). Such conditions structure the FGM composite in the way of a high permittivity layer and a bulk of lower one probably similar to that of the epoxy matrix.

For building FGM composites with a less abrupt permittivity difference between the two regions (i.e. accumulated and depleted), a strategy would be to work under lower applied electric fields and to use the application time as a thickness tuning parameter.

3.3 PARTICLE LAYER THICKNESS

Figure 8 presents a summary of the thickness changes in the accumulated particle layer of the FGM epoxy/SrTiO₃ composites versus the application time and for different applied electric fields.

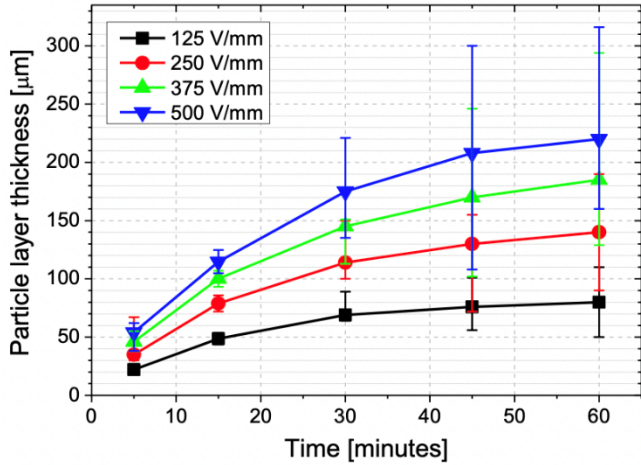


Figure 8. Thickness changes of the accumulated particle layer versus time for different applied electric fields.

The thickness of the accumulated particle layer increases asymptotically with time at a given field. Moreover, the initial increase (initial slope) appears more pronounced when the applied field is higher. As explained previously, the thickness inhomogeneity leads to larger error bars (dispersion) around the average thickness of the layer for the longest times and the highest electric fields. The present results confirm the kinetics tendency of what is typically observed in electrophoretic deposition following the deposited weight of the accumulated layer [24]. The Sarkar-Nicholson kinetics Equation [25] for the deposited weight, can be easily modified by a similar equation describing the increase in the accumulated particle layer thickness (e) and expressed as an exponential function of time:

$$e(t) = e_{\infty}(1 - \exp(-kt)) \quad (1)$$

where e_{∞} is the maximum particle layer thickness reachable at infinite time and k a parameter describing the accumulation kinetics. Table 1 summarizes for each electrical field the maximum thickness reachable and the kinetics parameter of the layer deposition.

Table 1. Parameters describing the kinetics of the particle layer accumulation extracted from eq. (1) for the different applied fields.

Applied field (V/mm)	e_{∞} (μm)	k (s^{-1})
125	82.4	5.80×10^{-2}
250	145.0	5.08×10^{-2}
375	188.5	4.76×10^{-2}
500	249.9	3.72×10^{-2}

3.4 PARTICLE DISTRIBUTION

In order to confirm the profile of the particle distribution in the FGM, EDS measurements were achieved on the SEM images of the samples. Figure 9 presents an example of the EDS profiles of Sr and Ti elements along the particle gradient for an epoxy/SrTiO₃ FGM composite processed at 500 V/mm for 15 minutes.

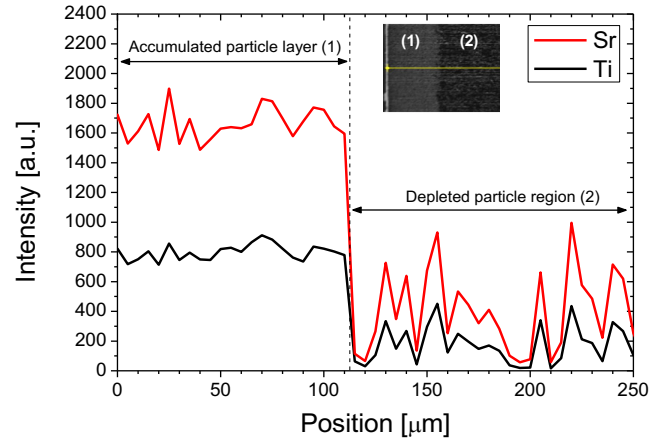


Figure 9. EDS profiles of Sr and Ti elements along the particle gradient for an epoxy/SrTiO₃ FGM composite processed at 500 V/mm for 15 minutes. Position '0' corresponds to the HV electrode location. The measurement is only given along the first 250 μm of the sample. The inset corresponds to the SEM image where (1) is the accumulated particle layer and (2) is the depleted region.

As expected, the intensity detected for both Sr and Ti atoms are larger in the accumulated particle layer than those in the depleted region of the FGM. This is in good agreement with the concentration of SrTiO₃ particles at the HV electrode. Moreover, it is worth to note that the elementary profiles in the accumulated layer is constant all along the thickness emphasizing a homogeneous particle concentration of the particle in this layer. The abrupt decrease in the Sr and Ti atom intensities detected when entering into the depleted region confirms a well-marked transition between the two regions of the FGM composite, which should have different dielectric properties. In the depleted particle region, apparently the noise level of the EDS signal appears as higher, this is due to the presence of SrTiO₃ particles or aggregates within the resin that increase very locally the X-ray emission. However, in the present case, the results highlight the FGM composites structuration exhibiting two regions with different particle concentrations (i.e. high and low).

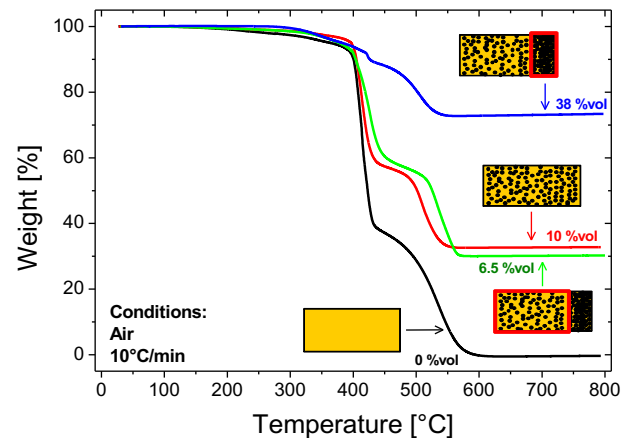


Figure 10. TGA thermographs in the two regions (accumulated and depleted) of an epoxy/SrTiO₃ FGM composite processed at 250 V/mm for 60 minutes. Comparison with the homogeneous epoxy/SrTiO₃ composite (10 vol%) and the neat epoxy.

To characterize precisely the SrTiO₃ filler content in each region, weight loss measurements were performed on samples after polishing the other one. Figure 10 shows TGA thermographs up to 800 °C in air for the two regions (accumulated and depleted) of an epoxy/SrTiO₃ FGM composite (initially 10 vol%) processed at 250 V/mm for 60 minutes. From the residual weight of SrTiO₃ particles obtained above 600 °C, after a complete degradation by oxidation of the epoxy matrix, it is possible to calculate the equivalent volume filler content. This technique seems to be efficient, since we confirmed that for an homogeneous epoxy/SrTiO₃ composite prepared with 10 vol% of particles the TGA analysis yielded also 10 vol%. Thus, for the FGM depleted region, it was obtained that the filler content decreased slightly of only few vol%. The concentration of this small content coming from a large volume and moving into a smaller one (i.e. the accumulated layer) largely increases the SrTiO₃ volume content up to 38 vol%.

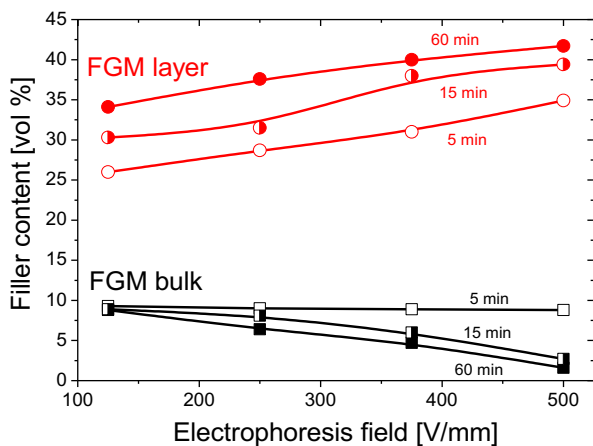


Figure 11. Filler content in the two regions (accumulated and depleted) of various epoxy/SrTiO₃ FGM composites versus the electrophoresis field and for different times.

Figure 11 presents the filler content changes in each region (layer and bulk) of the FGM as a function of the electrophoretic field and for different times. One can observe that the filler content in the particle layer is increasing with the electrophoresis field and process time while it is decreasing simultaneously in the bulk region. This confirms the observations of SEM images. Moreover, this confirms that not only the thickness increases but also the FGM layer density increases through an enhancement of the particle packing.

3.5 PERMITTIVITY

Figure 12 shows the dielectric permittivity and losses for the two regions (accumulated and depleted) of an epoxy/SrTiO₃ FGM composite (initially 10 vol%) processed at 250 V/mm for 60 minutes. While the neat epoxy and the homogeneous epoxy/SrTiO₃ composite have isotropic permittivity values of about 4 and 6.5, respectively, the FGM exhibits two very different behaviors.

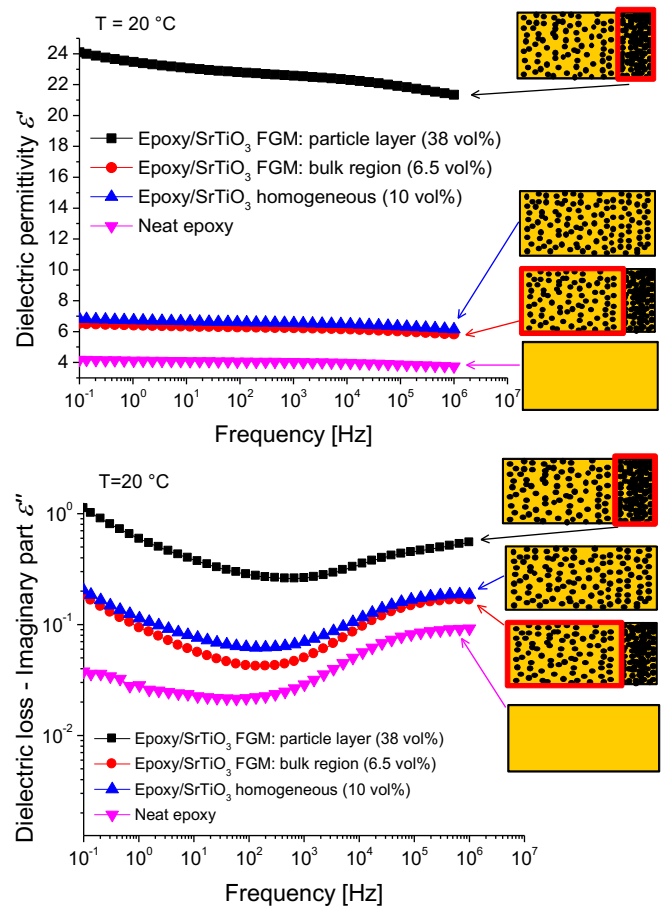


Figure 12. Complex dielectric permittivity versus frequency in the two regions (accumulated and depleted) in the epoxy/SrTiO₃ FGM composite processed at 250 V/mm for 60 minutes. Comparison with the homogeneous epoxy/SrTiO₃ composite (10 vol%) and the neat epoxy.

Firstly, one can observe that the permittivity and the dielectric loss of the depleted bulk region are almost similar with the ones of the homogeneous composite. Such properties are related to the fact that the particle content of the depleted bulk region is roughly unchanged during the electrophoretic process, as confirmed before. On the contrary, when this few vol% of depleted particles is accumulated in the particle layer at the HV electrode, the particle concentration is largely increased. Consequently, the permittivity and loss of the accumulated layer is significantly increased at the HV electrode with values of the permittivity above 20 in these tested conditions and in the frequency range under consideration. The dielectric losses, even if slightly higher, are not controlled by a conduction process but mainly by a dipolar polarization process, as the low frequency slope is largely lower than -1.

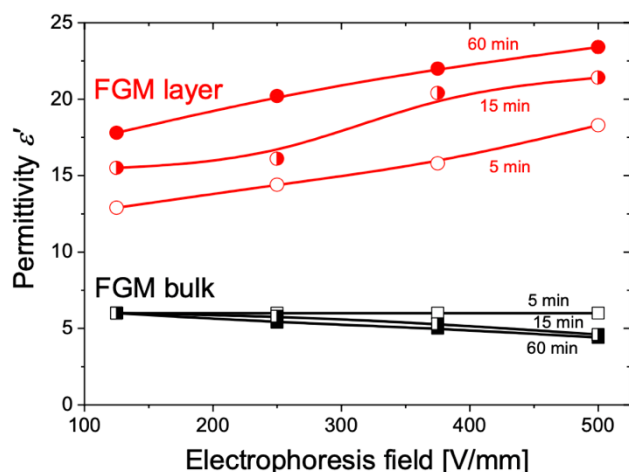


Figure 13. Dielectric permittivity in the two regions (accumulated and depleted) of various epoxy/SrTiO₃ FGM composites versus the electrophoresis field and for different times.

Finally, Figure 13 shows a summary of the permittivity changes in the two regions (accumulated and depleted) of initially 10 vol% epoxy/SrTiO₃ FGM composites as a function of the electrophoresis process parameters (i.e. field and time). One can observe the possibility to tune the permittivity particularly in the particle layer region in a wide range of values in order to adapt it for a given application. Thus, the permittivity gradient of the entire FGM can be tailored to optimally minimize an electric field reinforcement region in the vicinity of the high voltage electrode. This principle can be easily extended to other physical properties applying the same methodology than the one developed here. In that purpose, other particle types could be used for the construction of property gradients by this electrophoretic method such as BaTiO₃, TiO₂, ZnO, SiC, etc. These particles have either high permittivity (ferroelectric) $\gg 100$ or non-linear conduction effects. This new way of tailoring FGM composites paves the way of the possibility to adapt and relax the physical constraints in any system with multiple materials in contact each other's. This could participate to improve their reliability in extreme working conditions.

4 CONCLUSION

In this first paper, it was reported a novel way to build advanced functionally graded materials (FGM) based on polymer/ceramic composites using an electrophoretic deposition process. It relies on applying a DC voltage on the composite compound at the liquid state before curing it in order to 'freeze' the ceramic particles exactly where they have been accumulated. To exemplify this principle, a FGM epoxy/SrTiO₃ composite with a permittivity gradient is presented and the analysis methodology to build and characterize it is reported. An accumulated particle region is observed at the high voltage electrode with a field-dependent thickness, while the depleted bulk region remains almost unmodified. Moreover, the accumulated particle region exhibits a filler densification with the electrophoresis field and/or time, which enables the tuning of its permittivity. Such process can be very versatile to various electronic

systems by directly using their own electrodes. This process is easily transferable and adaptable to a wide range of polymer/particle combinations to tailor their electrical conductivity, mechanical and thermal properties in a gradient form. This work paves the way to the development of more robust electronic systems using such FGM with properties gradient in order to relax the physical constraints in critical regions and to finally improve their overall reliability. The next Part 2 of this article series will present permittivity gradient features under a non-uniform electric field.

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