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Structure modification of 0-3 piezoelectric ceramic/polymer composites through dielectrophoresis

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Short title

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Abstract

Anisotropic material properties can be induced in ceramic-polymer composites by applying an alternating electric field of moderate strength during processing. Under suitable conditions, particles of a ceramic filler material that are randomly dispersed in a liquid polymer or pre-polymer can be polarized and they then exhibit a collective response to localised gradients in the electric field. Typically the particles experience a mutually attractive force which causes them to form 'pearl-chains' or columnar structures spanning the gap between electrodes. If the fluid is solidified, for example by curing the polymer resin, then the newly formed structures can be fixed in place to produce a composite with directional electrical and mechanical properties. Direct visual observations were made for low volume fraction dispersions of pure lead titanate in an epoxy pre-polymer under the influence of an electric field. The observed interaction was correlated with low-field dielectric measurements and existing theory to identify optimum assembly conditions. The dielectric properties of the fluid are predominant and the formation of chain-like structures is found to be both field strength and field frequency dependent. The dielectric permittivities of a range of structurally modified composites were measured and compared with existing theoretical models of di-phasic materials.

Classification Numbers:

72.80Le, 72.80Ph, 72.80Tm, 72.80Sk, 77.22.-d, 77.65.-j, 77.84Py, 81.05Lg, 81.05Qk, 81.20Ev, 81.40Rs, 83.80Gv, 83.80Hj, 83.80Rs

Keywords:

Dielectrophoretic assembly, dielectrophoresis, electric-field structuring, 1-3 composites, 0-3 composites, piezoelectric, lead titanate, PZT, electrohydrodynamic flow, EHD, delectric cure monitoring, logarithmic, polarizability, nanowires

1. Introduction

The technique outlined in this paper is based on a process variously referred to as 'electric-field structuring' [1] or 'dielectrophoretic assembly' [2]. In this process, a dispersion of filler particles in a liquid polymer or pre-polymer is exposed to a moderate a.c. electric field. Under suitable conditions, the filler particles become polarised and exhibit a mutually attractive force, which causes them to form chain-like structures between the electrodes. The liquid can then be solidified by means of a chemical reaction or a change in temperature and the newly-formed structures fixed in place to form a composite material with anisotropic properties. This method has the potential advantage that materials having the directionality of 1-3 type composites (an array continuous rods in a 3D matrix) could be made, whilst retaining the simplicity of 0-3 (randomly dispersed) manufacturing [3]. Recently it has been suggested that this could be exploited to produce nano-scale conductors (nanowires) [4].

In operation, the 'electric-field structuring' technique exploits the dielectrophoretic force, which is responsible for the electrorheological effect [5-9]. It can be modelled in terms of: a) the dielectrophoretic or polarisation force, which is directed to produce the desired particle structure; b) viscous drag in the fluid, which resists particle motion; and c) settling or sedimentation of the polarised material, which must be controlled. Alternating electric fields are used, by preference, to suppress electrophoresis. Applied electric-field strength is deliberately moderated to preclude such effects as electrically-induced turbulence in the fluid and accelerated curing of the fluid pre-polymer. The frequency of the applied electric field is dictated by the dielectric properties of the fluid and the filler. It can be determined through consideration of the complex polarisability parameter by the method outlined in [10].

One of the major pitfalls associated with the electric-field structuring technique is sedimentation. When particles of the filler component have a higher density than that of the surrounding fluid they will tend to fall out of suspension under the influence of gravity. The rate of sedimentation depends on particle size and shape and also on the viscosity of the surrounding fluid. In practice, the dielectrophoretic force can be arranged to overshadow viscous drag and also the effect of gravity. However, the forces acting on different sized particles are not of the same magnitude. This makes precise control over the shape of the electric-field-induced structures difficult and irregularities commonly occur. A further difficulty concerns the viscosity of the surrounding fluid, which is not constant over the course of the processing cycle. For example, thermosetting polymers such as epoxy resins exhibit a progressive increase in viscosity with time as polymerisation proceeds. At the same time, the polymerisation reaction itself is exothermic and heat is generated. The fluid experiences a rise in temperature and consequently its viscosity decreases. Furthermore, the rate of reaction is increased at the higher temperature. These competing effects make precise control over fluid viscosity difficult to achieve.

2. Experimental

Direct visual observations of low volume-fraction suspensions subject to an applied electric field can yield valuable information in the current context. In particular, direct observation is used here to study the influence that applied field strength and

frequency has on structure formation. The potential difference applied across a curing suspension is one of the more readily controllable parameters in the field structuring technique, and tailoring the applied waveform opens up a wide range of possibilities.

Observations were made of low volume fraction (2%) suspensions of lead titanate powder in an epoxy resin, as they cured under the influence of an electric field. The effect of applied field strength and applied field frequency on the ceramic particle distribution was recorded. In a separate experiment the dielectric spectra of an epoxy resin and of several suspensions of lead titanate ($\leq$ 40% vol.) in the resin were measured as they cured. The relative importance of cure temperature and ceramic volume fraction was explored. The results of the two experiments were correlated to explain the observed electro-kinetic behaviour. Optimum process parameters were identified and were used to produce a range of electric-field structured composite materials.

2.1 Powder Preparation

High purity lead titanate (PT) was prepared by solid phase reaction of lead oxide (Aldrich: PbO <10μm, 99.9%+) and titanium dioxide (Aldrich, TiO_2 325 mesh, 99%min.) in molar quantities of 11:10. The addition of 10% excess lead oxide in this way was found by experiment to produce a powder with a narrow particle size distribution. The oxides were first ball-milled in acetone for 4hrs using zirconia elements, then dried and formed into 5g pellets by holding at a pressure of 20MPa for 5mins. The pellets were fired at 800°C for 2hrs and immediately pulverized by quenching in distilled water at room temperature. The powder so formed was etched in a solution of nine parts distilled water to one part concentrated nitric acid at 80°C for 1hr using an etchant to powder ratio of 25ml/g. The effect of this was two-fold. Firstly, excess lead oxide was removed from the surface and this was confirmed by x-ray diffraction. Sharp facets and surface damage were also removed and nano-sized particles were completely dissolved (Figure 1). The yield for the process was 82% by weight. The particle-size distribution was inspected using an X-ray Sedigraph and was found to be very narrow, with ~96% of particles in the range 2-8μm equivalent spherical diameter. Density was measured by pycnometry as 7.771kg/m^3.

2.2 Epoxy Resin

An optically clear, low conductivity epoxy resin (Epotek 302-3M - Epoxy Technology Inc., Billerica Ma.) was selected for the experiments. This is a two component fluid system. The epoxy pre-polymer is based on diglycidyl ether of bisphenol-A (DGEBA) and the curing agent is based on the multi-functional aliphatic amine, poly(oxypropyl)-diamine (POPD). Both parts of Epotek 302-3M are clear liquids at room temperature. The viscosity of the resin was measured using a rotational viscometer (Contraves Rheomat 115) as 2.48Pas immediately after mixing at 20°C. The initial viscosity is relatively high for a commercial epoxy resin and this is an advantage in the current context as it serves to inhibit sedimentation of the dense ceramic powder. The glass-rubber transition temperature (T_g) of the resin is 52°C when cured at 65°C for 90mins. Post-curing the resin at 150°C for 1hr. raises T_g to 96°C. Refractive index is given as 1.5340 and transmission of light at optical frequencies is >96%. Figure 2 shows isoviscosity curves for Epotek 302-3M measured over a range of cure temperatures.

2.3 Direct visual observations

Direct visual observations of electrically induced structure formation in low volume-fraction (2%) suspensions of lead titanate powder in a curing epoxy resin pre-polymer were made using an optical microscope. The suspensions were held in a shallow glass cell which was purpose made for the experiment. The resin was first degassed under vacuum at 50°C for 1 hour, the powder was added and the suspension degassed for a further 1 hour. The curing agent was added and thoroughly stirred before being introduced to the cell. An alternating electric field was applied between parallel copper plates set 2mm apart, using a Trek 610C High Voltage Power Amplifier in conjunction with a signal generator (Thurlby Thandor TG501). The influence of applied voltage was examined in the frequency range 0-5kHz as the resin cured at room temperature. The suspensions were randomised by stirring between observations to ensure that they did not retain a 'history' of previous processing conditions. The experiment was subsequently repeated using lead titanate powder which had been vacuum-dried for 24hrs at 100°C.

2.4 Dielectric cure monitoring

By measuring the frequency dependence of complex permittivity (ε^*) it is possible to separate out and monitor parameters governing both ionic and dipolar mobility in the curing resin and suspensions. A 4g sample of each fluid, in a glass test-tube, was placed in a temperature-controlled cell. An interdigitated, fringing-field type capacitive sensor (Dek-Dyne) was positioned in the fluid and an a.c. signal voltage applied using an impedance analyser (Solartron SI1260 Gain-Phase). The resulting current was compared to the applied voltage in respect of amplitude ratio and phase difference. The impedance analyser scanned twenty-six individual frequencies, in the range 10Hz - 1MHz, each sweep taking 30s. The data was recorded automatically and was used to deduce relative permittivity and dielectric loss. The fringing field of the capacitive sensor penetrates 25μm into the fluid making it suitable for both the resin and suspensions. Measurements were made on the curing resin and a range of suspensions (to 40% vol.) of dry lead titanate in the resin as they slow cured at 40˚C. In all cases measurements began 5 minutes after initial mixing.

e) Fabrication of model composites

Structured lead titanate-epoxy resin composites were prepared according to the following method. A dispersion of known ceramic volume fraction was made up for each sample. This was divided into two identical parts. One part was processed by field-structuring between two charged copper electrodes. The other was simultaneously put through an identical thermal cycle but with no field applied. The method by which the processing conditions were derived is outlined below (Section 3). After curing, a layer of material adjacent to each electrode was skimmed off to a depth of 250μm as a precaution against a build-up of impurities in that area. The density of each sample was measured by the Archimedes method and the volume fraction of the ceramic was re-calculated. This step was felt necessary to ensure that sedimentation had not caused the actual composition to drift away from its nominal value during processing. The composites were sectioned, using an annular saw both perpendicular and parallel to the applied field. Similar sections were made from the

unstructured composites. The specimens were furnished with electrodes using air-drying silver paint. A 1mm perimeter, around the edge of each specimen, was left exposed in order to minimise the influence of the fringing field. Dielectric measurements were made using a GenRad 1689M RLC Digibridge. Transmission light microscopy was used to reveal the chained microstructure in low volume fraction composites as illustrated in Figure 3.

3. Results and Discussion

3.1 Visual observations

The electrically-induced patterns that were observed to form in the suspensions can be conveniently described in terms of the structure-types illustrated in Figure 4**.** Type I is characterized by a large number of randomly positioned short chains consisting of only a few particles. These are aligned with the applied field. Type II consists of single strand 'pearl-chains' which completely span the inter-electrode gap indicating a stronger dielectrophoretic attractive force. Some of these may be branched. In Type III the individual chains have been drawn together and have coalesced to form more substantial 'columnar' structures.

Figure 5 shows the observed behaviour at different applied field strengths and frequencies when untreated particles are used. The experiment was repeated, as shown in Figure 6, using particles which have been vacuum-dried to remove adsorbed moisture.

Refering to Figure 5**.** At 250 V/mm the field strength is sufficient for structuring to take place but initially, at all frequencies, significant electrohydrodynamic flow of the fluid prevents full chains from forming. The dielectric properties of the fluid change as curing progresses and after 30 minutes full chaining becomes possible at higher frequencies (≥3kHz). At 500 V/mm fluid flow is initially violent which precludes any structure formation. At the higher frequencies (≥2kHz) the field strength is such that full chaining is observed despite significant fluid flow. After 30 minutes, fluid flow subsides at ~4kHz and chaining readily occurs. NB: After 40 minutes violent streaming returned at the higher frequencies. This was ascribed to exothermic heating, leading to a temporary reduction in viscosity and hence increased mobility of charged species. Full chains formed rapidly and were immediately disrupted. After one hour under these conditions bubbles started to form in the resin and foaming ensued. The lead titanate particles clearly exhibited electrophoresis at lower frequencies. At 1000 V/mm electrical breakdown occurred initially at all frequencies. Some evidence of chaining, at 4kHz, was observed after 30 minutes.

Figure 6 demonstrates the changes that occur when using lead titanate powder which has been scrupulously dried. Chains are seen to form much more readily and, again, this is initially favoured at the higher frequencies. Overall, it can be seen that fluid flow is substantially diminished and the chains that form as a consequence of polarization are disrupted to a lesser extent. The presence of adsorbed moisture would, therefore, seem to be an important factor to consider when defining process parameters. It could be that the effect of drying is twofold. Clearly, by removing

absorbed moisture the total number of charged ions is reduced. In addition, a number of reactive sites on the particle surfaces become available and ionic species may be adsorbed, thereby further reducing the number of free ions in the fluid. In the light of the above findings, the highest field strength (1000V/mm) was omitted for this experiment. After 30 minutes a slight increase in fluid flow was observed at 1kHz and this is again explained by an observed increase in temperature due to the reaction exotherm. Curing progressed in this case without the occurrence of excessive bubbling.

Figure 7 illustrates the measured dielectric loss of Epotek 302-3M at 20ºC alongside the observed frequency dependence of streaming due to electrohydrodynamic (EHD) flow. The measurements were taken 5 minutes after curing began. Analysis shows that in the frequency range 10^2-10^5 Hz a plot of log(ε") v - log(f) has a slope of very close 1. The dominant role of ionic conduction at these frequencies is thereby confirmed.

With reference to Figure 6. At 250V/mm there is a clear reduction in EHD flow which occurs between 0.5kHz and 1kHz. At 500V/mm, the same change is seen between 2kHz and 3kHz. The significance of this is apparent on the dielectric loss curve (Figure 7), as it corresponds to a range where, due to relaxation, the loss approaches its high frequency value. Such behaviour provides clear evidence that ionic conductivity in the fluid is predominant in the early stages of cure and in general it can be said that: 1) the abatement of streaming is at a higher frequency at the higher field strength and 2) that as time passes streaming occurs at progressively lower frequencies.

3.2 Dielectric cure monitoring

A more complete data set is available for the resin and suspensions curing at 40°C and the results of cure monitoring are reproduced below as Figure 8. These plots show the initial low frequency spike that is indicative of electrode polarization, which appears stronger for the 40% vol. lead titanate in comparison with the resin itself. An inflection in the curves at high frequency (log f = 6) is seen after curing for 1 hour and, as time goes on, this moves to progressively lower frequencies. This marks the onset of gelation in the resin. Figure 9 shows the initial values and the changes that have occurred after 1 hour. Note that measurements relating to a suspension of 10% vol. lead titanate are little different from those of the resin alone. The effect of raising the temperature is twofold. Firstly, at a given field strength, a higher field frequency is required to suppress EHD flow because of increased charge mobility. However, the reduced viscosity of the fluid will ensure that chaining is possible at lower field strengths. This is significant because the viscous drag force scales linearly with viscosity, whereas the polarization force scales quadratically with applied field strength. Further work would be required to find exactly where the applied field strength exceeds the threshold for chain formation and the corresponding field frequency at which EHD flow is suppressed.

The above findings can be related to established theory and an important factor to consider is the frequency dependence of the polarizability parameter. Figure 10 shows collected data and calculated values of the polarizability parameter in its complex

form; it having a real part referred to as Re[f(ε*)] and an imaginary part Im[f(ε*)] [10]. The ideal, perfect dielectric, form of the polarizability parameter (β) is also calculated for comparison. The plots show that the Re [f (ε*)] mirrors the form of the initial relative permittivity curve for the resin. The Im [f (ε*)] on the other hand mirrors the corresponding dielectric loss curve. Interestingly, because (ε_p - ε_f) and (σ_p - σ_f) have opposite signs, the function Re [f (ε*)] passes through zero. This occurs at ≈1kHz and consequently the dielectrophoretic force is zero at this frequency. Below it the particles experience negative dielectrophoresis and above it positive dielectrophoresis. Given that the interparticle forces scale according to $(\beta)^2$, the point at which the traces of Re [f (ε*)] and Im [f (ε*)] cross, ~2.5kHz, may prove to mark a lower limit for composite assembly. Clearly, the ideal value of beta (β), quoted in some research papers, does not appear to be a true indicator of dielectrophoretic behaviour below c.10kHz.

Benguigui and Lin [11] showed through calculation that the frequency dependence of the dielectrophoretic force is divided into a dielectric regime and a conductivity regime. The dielectric regime exists where $t << \tau$ for the d.c. case or where $\omega.\tau >> 1$ for the a.c. case (where τ is the Maxwell-Wagner time constant $(\varepsilon_p + 2\varepsilon_f)/(\sigma_p + 2\sigma_f)$). The dielectrophoretic force then depends only on the mismatch in the dielectric constants of the two components. The conductivity regime is defined by $t >> \tau$ for d.c. and $\omega.\tau << 1$ for a.c. The dielectrophoretic force is then defined by the difference in their respective conductivities. Figure 11 shows the frequency dependence of $\omega.\tau$ This indicates that the point where $\omega.\tau = 1$, marking the transition between the two regimes, occurs at 1290Hz. For the case in point, this is identical to the frequency at which Re [f (ε*)] = 0 and the calculation is somewhat simpler. Structure formation in seen to occur when $1 < \omega.\tau < 2$ and this may represent an alternative assessment of particle polarizability when processing parameters are defined.

3.3 Processing parameters for composite assembly

The chief processing variables that must be defined for structuring to take place are: a) time scale; b) temperature; c) electric field strength and d) electric field frequency. In particular, it is essential to identify the field strength - frequency relationship, which highlights the onset of significant EHD flow. Given that the dielectric properties of the fluid are predominant in the early stages of cure, this could be determined directly from measured conductivity or alternatively from log (viscosity), which has been shown to mirror the extent of conversion of the reactants over the range in question [12]. Measurements would encompass a suitable range of temperatures. On the side of caution, a question remains as to whether the parameters identified in this way would reliably translate to very high volume fraction suspensions, where interfacial effects may assume greater prominence. Furthermore, there is a concern that measurements made under low field conditions may not accurately reflect high-field behaviour. For the purposes of this study it can be said that suitable frequencies for composite assembly lie beyond the elbow in the dielectric loss curve and, in the absence of EHD flow, visual observations show that full chaining can occur at 250V/mm. At 500V/mm columns form readily indicating a second threshold has been passed. When establishing the processing time and

temperature, it should be recognized that sedimentation of the relatively heavy particles must be avoided. A simple way to overcome this is to partially cure the resin and then re-mix to re-establish a uniform dispersion. It has been observed that the resin is still sufficiently fluid for structure formation to occur at 10Pas. Accordingly the resin can be allowed to cure for 1 hour at room temperature. It is then re-stirred under vacuum. The temperature is then raised to 35ºC for 45 minutes with a field of 250V/mm applied at 4kHz. The temperature is then raised to 55ºC for 45 minutes to complete the cure. A post-cure at 120ºC for 1 hour can be performed if required to raise T_g. The relative permittivities of a range of composites prepared in this way are shown in Figure 12 and these are compared to calculated values for the same system using some standard models for di-phasic composites.

In common with many composites, the interface between the filler and matrix components has a controlling influence on the physical properties of materials produced by electric-field structuring. The surface electrical properties of the filler particles, in particular, are of prime importance. Normally particles are completely surrounded by a layer of adsorbed polymer, giving true 0-3 type connectivity [3]. Where these layers form insulating barriers between the particles, the useful electrical properties of the composite material are adversely affected. Furthermore, where variations in the sizes of individual particles exist, chain branching is found to occur. Anisotropy in the electrical properties will then be a function not only of ceramic loading, but also of the degree of chain branching [2]. In practice, some disparity in the spatial distribution of the filler particles is found to occur between otherwise identical composite samples. Hence, some variability in the physical properties of powder composites prepared by electric-field structuring can normally be expected.

4. Conclusion

The electric-field structuring technique has been explored for a model composite system and it has been demonstrated that it can be used to induce directional materials properties. The main processing parameters have been explored and it has been demonstrated that the dielectric properties of the fluid are of prime consideration in the early stages of cure. Clearly electric-field structuring is a highly controllable, low energy technique which could be used effectively to induce locally reinforced or functional-gradient materials. The technique is not restricted to the model composite system investigated here, but is applicable to a wide range of fluid/filler combinations.

5. Acknowledgements

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Figure legends

Figure 1a - $PbTiO_3$ + 10% excess PbO, quenched (x1000)

Figure 1b - $PbTiO_3$ + 10% excess PbO etched in nitric acid (x1000)

Figure 2 – Isoviscosity curves for Epotek 302-3M at different cure temperatures

Figure 3 - 'Pearl chains' in a field-structured 10% vol. lead titanate / epoxy composite.

Figure 4 - Types of electrically-induced structure observed to form in low-volume fraction suspensions of lead titanate in a curing epoxy resin.

Figure 5 – Observed behaviour of a 2% vol. suspension of lead titanate (undried) in curing Epotek 302-3M. Where 0 indicates that no structure formation is observed. I II III indicate Types I, II and III structure formation respectively

Figure 6 – Observed behaviour of a 2% vol. suspension of lead titanate (dried) in curing Epotek 302-3M

Figure 7 - Initial dielectric loss of Epotek 302-3M epoxy resin at 20°C

Figure 8 - Dielectric cure monitoring of Epotek 302-3M epoxy resin (a) and a 40% vol. suspension (b)

Figure 9 – Relative permittivity (a, c) and dielectric loss (b, d) of Epotek 302-3M epoxy resin and suspensions at 40°C - initially (a, b) and after 1 hour (c, d)

Figure 10 - Initial polarizability parameter of a dispersion of lead titanate
in Epotek 302-3M curing at 40°C
Figure 11 – An alternative assessment of polarizability for a dispersion of lead titanate in Epotek 302-3M curing at 40°C
Figure 12 – Relative permittivities of structured and unstructured composites in comparison with some standard models for randomly dispersed di-phasic materials

Figures

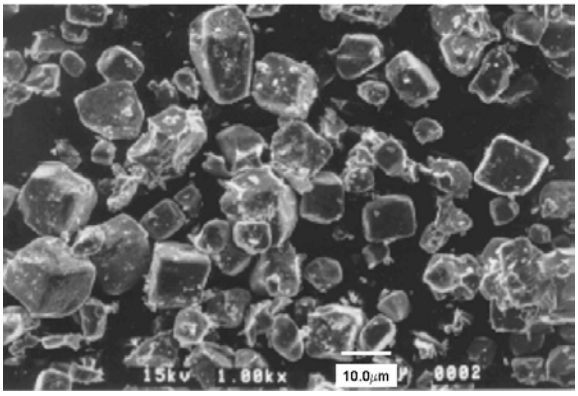


Figure 1a - $PbTiO_3$ + 10% excess PbO, quenched (x1000)

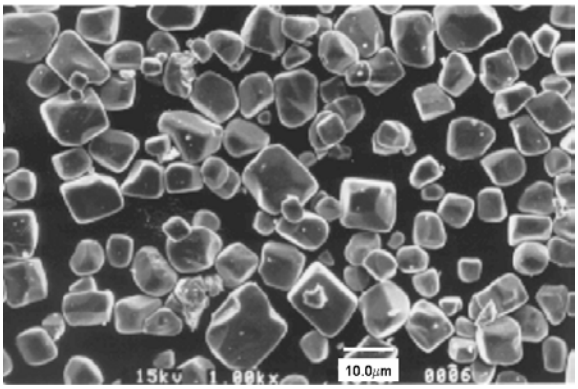
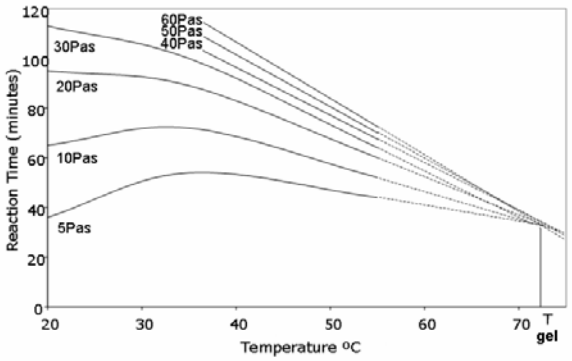


Figure 1b - $PbTiO_3$ + 10% excess PbO etched in nitric acid (x1000)



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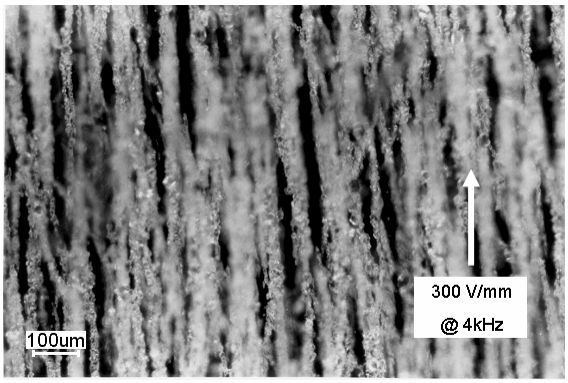

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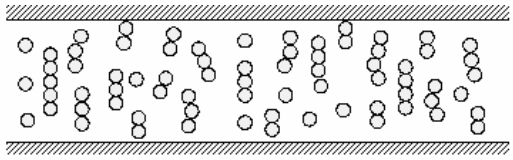
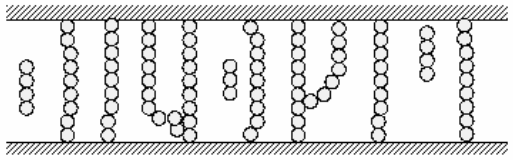
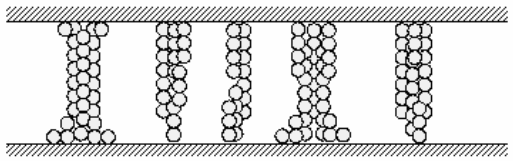


Type I) Weak interaction of polarised particles

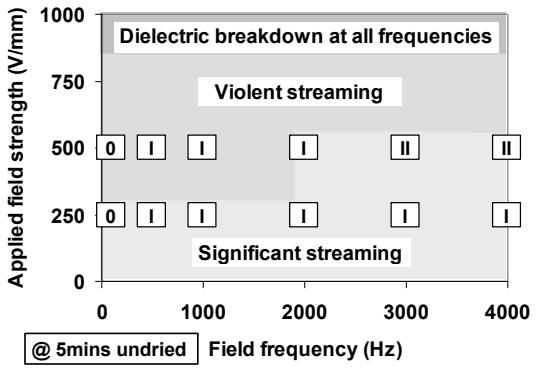


Type II) Randomly located 'pearl chains'



Type III) Chains aggregate into 'columns'

Figure 4 - Types of electrically-induced structure observed to form in low-volume fraction suspensions of lead titanate in a curing epoxy resin.



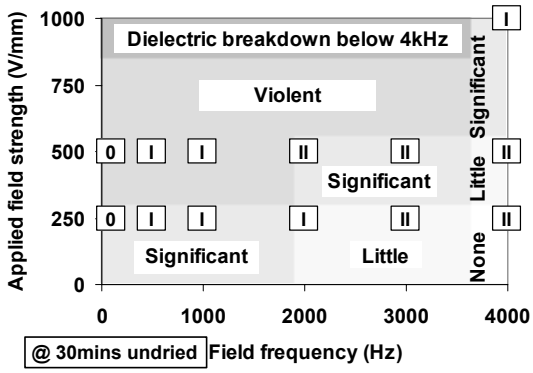
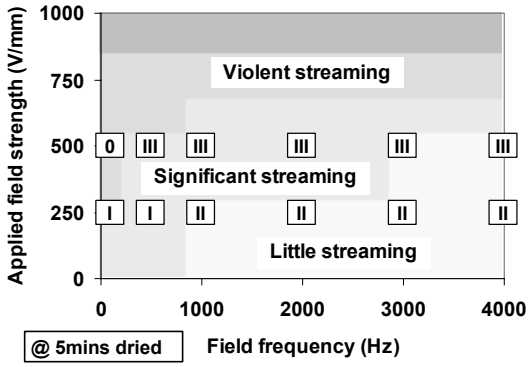
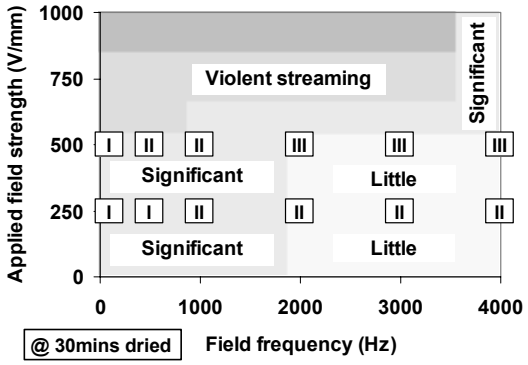


Figure 5 – Observed behaviour of a 2% vol. suspension of lead titanate (undried) in curing Epotek 302-3M Where 0 indicates that no structure formation is observed. I II III indicate Types I, II and III structure formation respectively





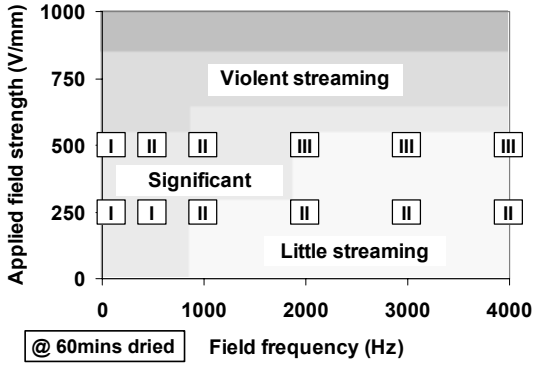


Figure 6 – Observed behaviour of a 2% vol. suspension of lead titanate (dried) in curing Epotek 302-3M

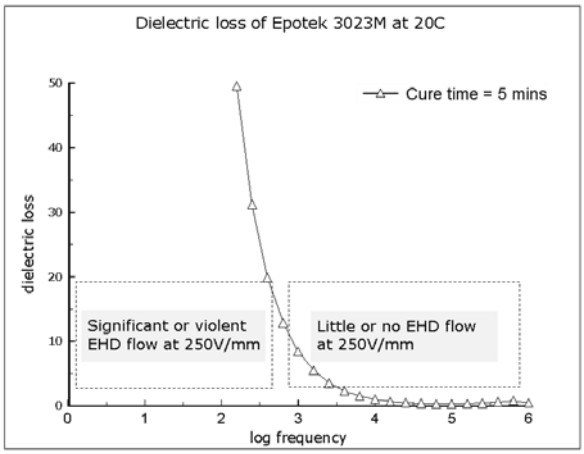
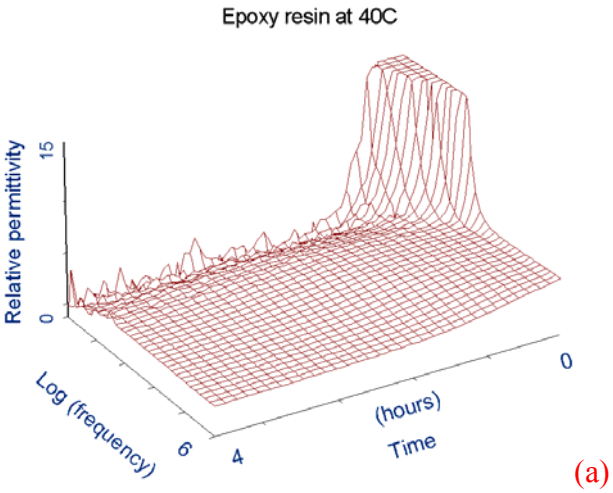
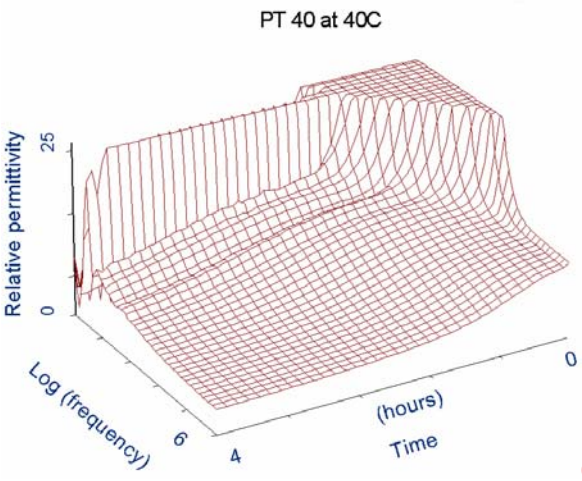


Figure 7 - Initial dielectric loss of Epotek 302-3M epoxy resin at 20°C

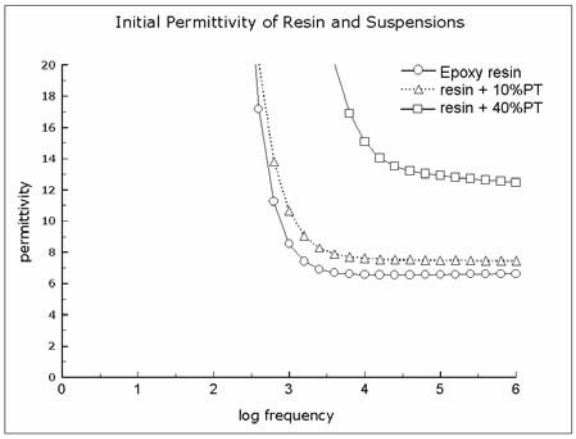


(a)

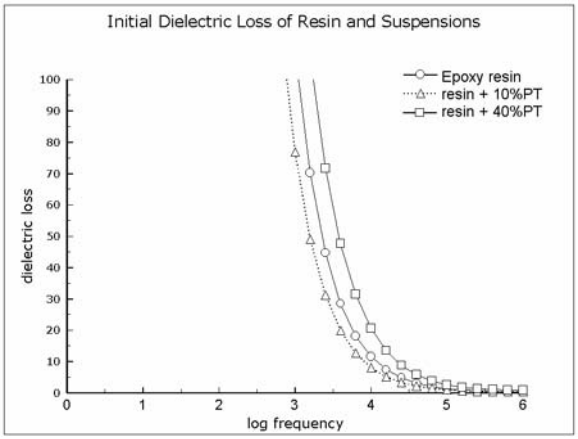


(b)

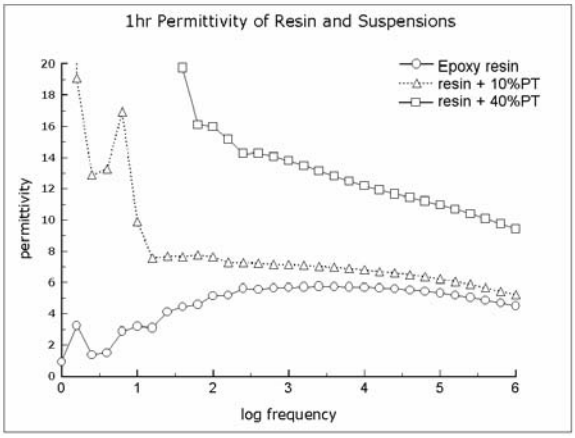
Figure 8 – Dielectric cure monitoring of Epotek 302-3M epoxy resin (a) and a 40% vol. Suspension (b).



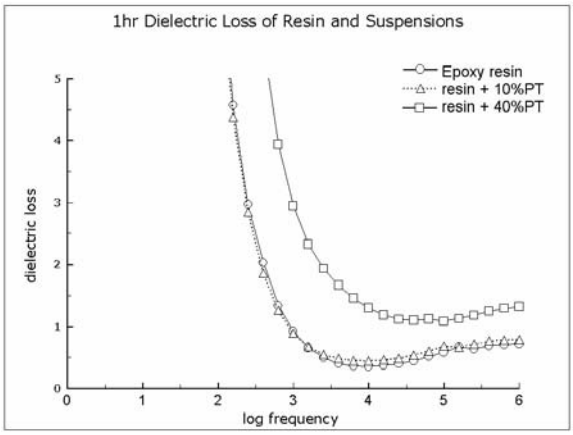
(a)



(b)



(c)



(d)

Figure 9 – Relative permittivity (a, c) and dielectric loss (b, d) of Epotek 302-3M epoxy resin and suspensions at 40°C - initially (a, b) and after 1 hour (c, d)

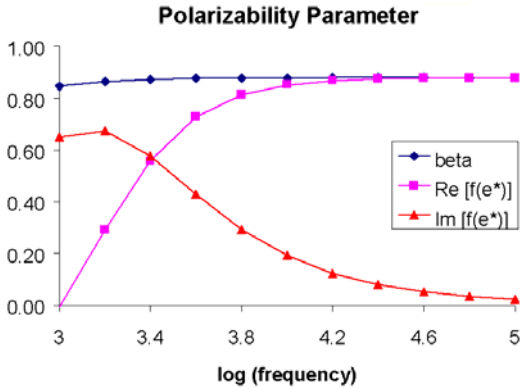


Figure 10 - Initial polarizability parameter of a dispersion of lead titanate in Epotek 302-3M curing at 40°C

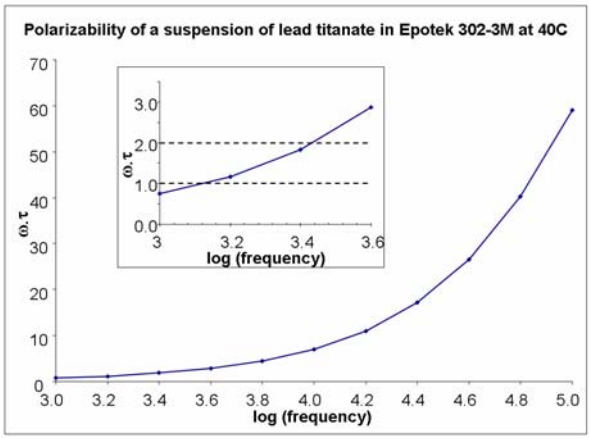


Figure 11 – An alternative assessment of polarizability for a dispersion of lead titanate in Epotek 302-3M curing at 40°C

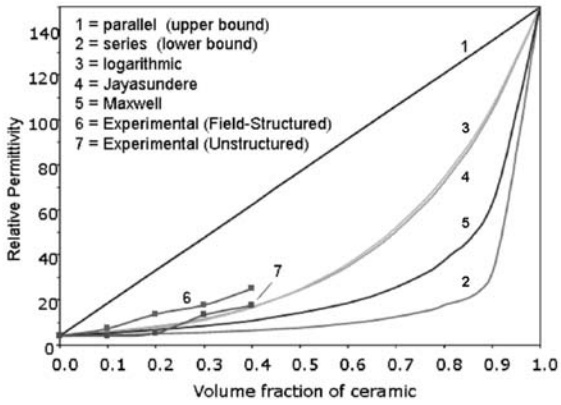


Figure 12 – Relative permittivities of structured and unstructured composites in comparison with some standard models for randomly dispersed di-phasic materials

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Structure modification of 0–3 piezoelectric ceramic /polymer composites through dielectrophoresis

Wilson, Stephen A.; Maistros, George M.; Whatmore, Roger W.

2005-01-21
Institute of Physics (IOP)
https://doi.org/10.1088/0022-3727/38/2/001
Wilson SA, Maistros GM, Whatmore RW. (2005) Structure modification of 0–3 piezoelectric ceramic/polymer composites through dielectrophoresis. Journal of Physics D: Applied Physics. Volume 38, Issue 2, pp. 175-182

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