A pilot study on the Relationship between Mechanical and Electrical Loss Tangents of Glass Powder Reinforced Epoxy Composites Post-cured in Microwaves

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Abstract. The mechanical and thermal properties of hollow glass powder reinforced epoxy resin composites have been measured and evaluated in earlier studies. This basic but critical and important data have caused interests in the relevant industry in Australia. This study is therefore carried out to measure and evaluate the dielectric properties of the composites with a view to benefit the relevant industry. The relationship between the dielectric and thermal properties will also be studied and correlated. The original contributions of this paper are that samples post-cured in conventional ovens have higher electrical as well as mechanical loss tangent values than their counterparts cured in microwaves only. The storage modulus of all samples post-cured conventionally is higher than its counterpart. This is in line with the fact that they are softer material with lower glass transition temperatures. For all percentages by weight of glass powder, the glass transition temperature for the microwave cured sample was higher and the composite was stiffer; the opposite was true for the conventionally cured samples.

Introduction

The Centre for Excellence in Engineered Fibre Composites (CEEFC) at the USQ seeks to facilitate the development and introduction of lightweight composite materials into engineering commercial applications. The most widely used and least expensive polymer resins are the polyesters and vinyl esters; these matrix materials are used primarily for glass fiber-reinforced composites. The epoxies are more expensive and, in addition to commercial applications, are also utilized extensively in polymer matrix composites for aerospace applications; they have better mechanical properties and resistance to moisture than the polyesters and vinyl resins [1]. In this study, the dielectric properties of the prepared composites were investigated and were correlated with the thermal properties. The percentage by weight of glass powder studied was varied from 5 to 15 %. Half of the samples were post-cured conventionally and the other half of them was post-cured in microwaves.

The materials

The epoxy resin used in this study is Kinetix R246TX Thixotropic Laminating Resin, an opaque liquid, and the hardener used is kinetic H160 medium hardener which has a pot life of 120 minutes. Other hardeners like H126, H128, H161 and H162 can also be used [2]. The glass powder was first mixed with epoxy resin, after this the hardener, kinetic H160 medium was added. The by weight ratio of resin to hardener used was 4:1 [2]. The composite was then cast to moulds of loss tangent test pieces and left to cure under ambient conditions for 24 hours. The loss tangent test specimens were taken out of the moulds and then post-cured in oven at 40 ^{o}C for 16 hours, and then at 50 ^{o}C for 16 hours and finally at 60 ^{o}C for 8 hours. This is to ensure the heat distortion temperature (HDT) is above

63 ^{o}C. To bring the ultimate HDT to 68 ^{o}C, another 15 hours of post-curing will be required [2]. The dielectric losses of specimens were then measured.

The glass powder used is SPHERICEL® 60P18 (spherical) hollow glass spheres. They are used to enhance performance and reduce viscosity in paints and coatings and as lightweight additives in plastic parts. They are chemically inert, non-porous, and have very low oil absorption. SPHERICEL® 60P18 hollow spheres products offer formulators flexibility in polymer composites. The addition of hollow spheres to fiberglass reinforced plastics (FRP), epoxy, compounds, and urethane castings can provide weight reduction cost, savings and improved impact resistance. Insulating features of hollow spheres also work to the chemists' advantage in thermal shock and heat transfer areas. Two densities available are 0.6 to 1.1 g/cc; it provides choices to best fit mixing and target weight requirements [4]. The density of the hollow glass powder used in this research is 0.6 g/cc because the other filler, ceramic hollow spheres or SLG used in similar study is 0.7 g/cc; this will give a better basis for comparison of results obtained in the future. When used in polymer concrete, hollow spheres provide a cost effective alternative without degrading physical properties.

The Composite Samples

The reinforcer was glass powder (glass hollow spheres) particulates and they were made 5 % to 15% by weight in the cured epoxy composite, EP/GP (X %), where X is the percentage by weight of the filler. The glass powder percentages by weight vary in step of 5 %. As the raw materials of the composites are liquid and glass hollow spheres, the specimens of 120 mm x 120 mm x 6 mm were cast to shape. The resin is an opaque liquid and is first mixed with the catalyst. After that the glass powder is added to the mixture, they are then mixed to give the uncured composite. Table 2 shows the mass in grams of resin, catalyst and glass powder required respectively to make 1000 grams of uncured composite of 15 % by weight of glass powder. The mixture of glass powder, resin and accelerator was blended with mechanical blender to ensure a more homogenous mixture. The uncured composite was then cast into the moulds curing in ambient conditions. After initial 24-hour curing, half of the test pieces were removed from the mould and post-cured conventionally in an oven for 40 hours. Their dielectric loss tangent values then were measured. The other half of the samples were post-cured in microwaves for 4 minutes using a power level of 320 W and the temperature reached was 40 ^{o}C. The temperature was measured using an Oakton TempTestr Infra Red handheld thermometer. Allow the samples to cool in the oven cavity to room temperature. The samples were again heated to 50 ^{o}C by exposing them to a power level of 320 W for 6 minutes. Allow the samples to cool in the oven cavity to room temperature. The samples were again heated to 60 ^{o}C by exposing them to a power level of 320 W for 8 minutes. Allow the samples to cool in the oven cavity to room temperature. The processes were equivalent to heating the samples in a conventional oven with the above parameters. In both cases, the intermittent and final temperatures were made the same in post-curing the composite samples. The dielectric loss tangent of specimens was then measured as well.

Dielectric tangent loss

Dielectric properties can be used to classify materials as conductors, quasi conductors or insulators. The alternating polarisation of the molecules can consume energy, this is polarisation loss. A complex permittivity is required to characterise these materials. Materials can be good conductors at some frequencies while becoming a dielectric at other frequencies. A dielectric material is a substance that is a poor conductor of electricity, but an efficient supporter of electro static fields. An important property of a dielectric is its ability to support an electrostatic field while dissipating minimal energy in the form of heat. The lower the dielectric loss, the proportion of energy lost as heat, the more effective is a dielectric material.

The dielectric tangent loss of the samples of glass powder filled epoxy composites cured under ambient conditions and ambient conditions plus post-curing in an oven were measured.

The complex relative permittivity of a dielectric is $\varepsilon = \varepsilon' - j\varepsilon''$; the real part is the dielectric constant; the imaginary part is referred to as the loss factor. The ratio of these two values is the loss tangent, $\tan\delta = \frac{\varepsilon'}{\varepsilon''}$ [5]. The method used to obtain the dielectric tangent loss will be by measuring the capacitance and conductance with a meter.

Figure 1 shows the schematic parallel connection of C and G. The distributed shunt capacitor, C and the conductance, G are both dependent on the properties of the dielectric material which separates the line conductors. The currents flowing through the parallel combination of C and G are shown in Figure 1(a) and their phasor relationship in Figure 1(b).

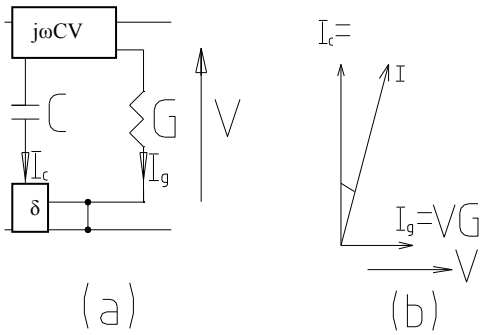

Fig.1 (a) parallel connection of C and G (b) Phasor diagram

From the measurements, the real and imaginary parts of the dielectric loss can be calculated from the following relationships [6]:

$$C = \frac{\varepsilon_0 \varepsilon_r A}{s} \quad (1)$$

where C is the capacitance in Fm^{-1};

ε_0 is the dielectric permittivity of free space = $\frac{1}{36\pi}$ x 10^{-9};

ε_r is the dielectric constant of the composite;

A is the surface area of the samples in mm^2;

s is the thickness of the composite sample in mm^2.

and the loss tangent [6],

$$\tan\delta = \frac{\varepsilon''}{\varepsilon'} \quad (2)$$

Loss tangent is frequency dependant. This experiment will be made at one hundred, 100, 120, 1k, 10k, 20k 100k. This is within the range of the measuring device.

If the properties of the dielectric are constant over the frequency range of interest, then C will be constant and G will be proportional to frequency, and the loss tangent can be easily calculated by the formula [7]:

$$G = \omega C \tan\delta \quad (5)$$

Results and discussions

Figures 2 and 3 are the loss tangents of glass powder filled epoxy composites with 5 and 15% percent of by weight glass powder post-cured conventionally and in microwaves respectively. For the composites, it is necessary to divide the results into two groups: low frequency group (up to 1 kHz) and high frequency group (10 kHz and up). In low frequency group, for both powder by weight, the oven cured specimens have lower electrical loss tangent values than their counterparts. The opposite is true for the higher frequency group. It can be argued that the dielectric behaviour of the low frequency group is due to the fact that the change in polarity used in the study is too slow to initiate the change of polarity for water molecules; this results in low loss tangent values for materials supposed to have high water content. On the other hand, the dielectric behaviour of the other group

(higher frequency) was normal because samples post-cured in microwaves should have less water content and hence lower electrical loss tangent values. This may be due to higher degree of curing and less water content. The higher frequency group is more important because for most microwave processing of materials, the frequency used will be high, e.g. 2.45 GHz. Since the electrical loss tangents of the oven cured samples are relatively high, they can be further processed by curing them in microwaves to 100 % cured to make them stiffer in a much shorter time [8].

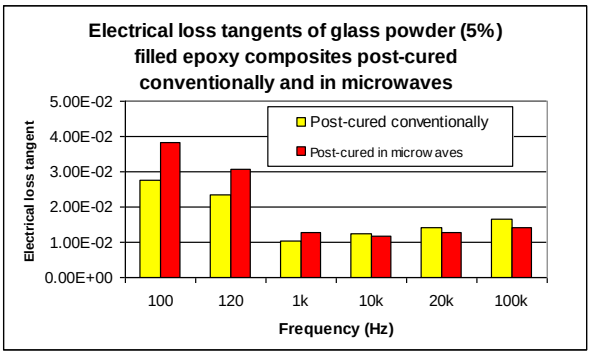

Fig. 2 Loss tangent of epoxy-glass powder (5%) post-cured in oven and microwaves

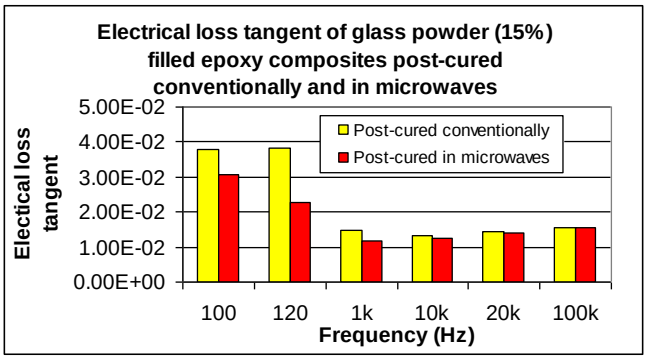

Fig. 3: Loss tangent of epoxy-glass powder (15%) post-cured in oven and microwaves

Referring to Figures 4 through 7, for all samples, irrespective the percentage by weight of glass powder, samples post-cured in microwaves have higher glass transition temperature than their counterparts, which imply that samples post-cured conventionally are softer material than their peers and this is at par with the tan delta values found. The higher electrical loss tangent values are due to the presence of water and lower degree of curing. Higher mechanical loss tangent values mean softer material, which in itself may have higher water content and less degree of cure.

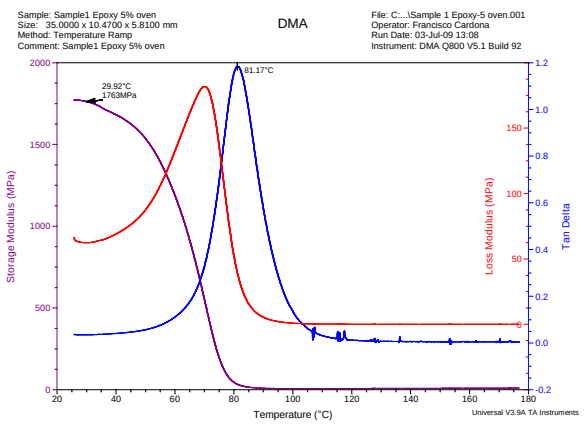

Fig. 4: DMA results of 5% glass powder filled epoxy resin post-cured in an oven for 40 hours

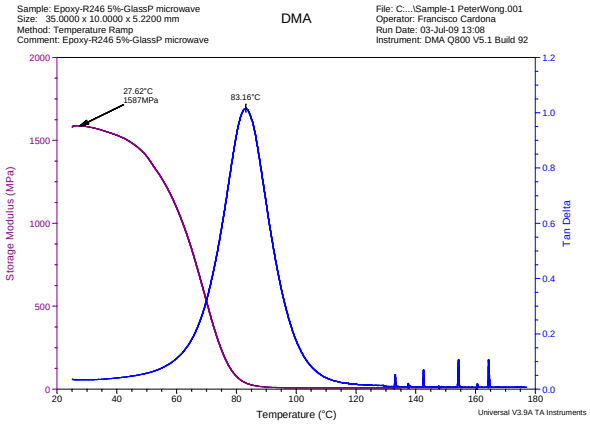

Fig. 5: DMA results of 5% glass powder filled epoxy resin post-cured in microwaves

The values of electrical loss tangent to that of the mechanical ones were now compared. Samples post-cured in microwaves, irrespective of glass powder content by weight, have lower electrical loss tangent (Figures 3 through 4) and mechanical loss tangent (Figures 5 through 8) and it can be argued that by measuring electrical loss tangent of a material, it is possible to deduce its mechanical loss tangent [9].

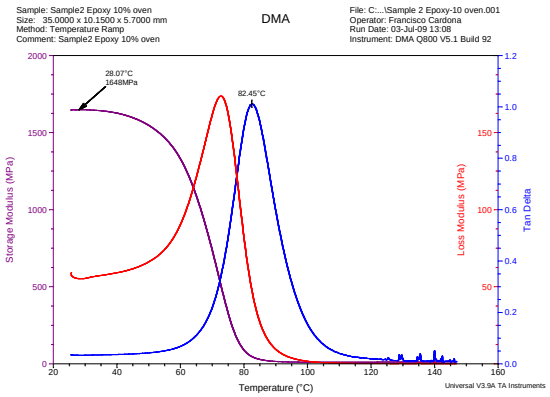

Fig. 6: DMA results of 15% glass powder filled epoxy resin post- cured in an oven for 40 hours

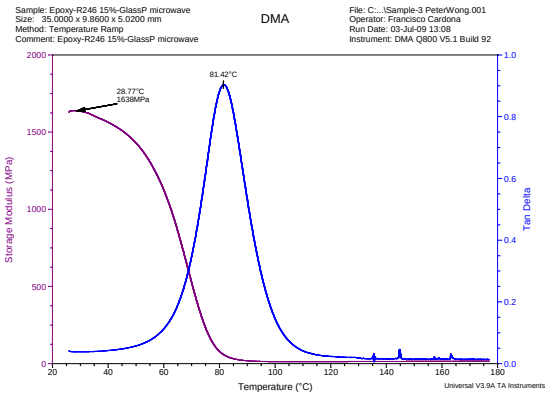

Fig. 7: DMA results of 15% glass powder filled epoxy resin post- cured in microwaves

Conclusions

From electrical loss tangent measurements, it was found that the samples post-cured in microwaves have lower electrical loss tangents than their counterparts in higher frequency range and the samples would be less able to convert the microwave absorbed into heat in microwave processing. The main contributor to this phenomenon was the absence of or very little water molecules in the specimens because of higher degree of curing. It can be argued that the absence or very little molecules in the samples make the samples stiffer.

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