



A STUDY OF THERMO MECHANICAL PROPERTIES ON EPOXY/ALUMINA NANOCOMPOSITES

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Abstract. *The modification of polymer properties through the addition of nanoparticles has lead to the development of the so-called nanocomposites. The purpose of this study was to observe the improvement of a thermo mechanical property in epoxy/alumina nanocomposites, the Glass Transition Temperature. Differential Scanning Calorimetry was carried-out for different volume fractions, ranging from 2.5% to 10% and three different nanoparticles sizes – 35nm, 150nm and 200nm and showed that this property is not only depended of the chemical bonding between the organic matrix and inorganic particles.*

Keywords: *Epoxy, Alumina, nanocomposites, DSC.*

1. INTRODUCTION

With the recent increase in nanoscience researches, it was observed a promoting interest in nano-sized reinforced polymers for high technological applications, as coatings, electronic devices, adhesives, automotive and aerospace industries (Jiang et al., 2012; McGrath et al., 2008; Omrani and Rostami, 2009).

Polymer nanocomposites are usually defined as a combination of a polymer matrix and nano-sized particles (Moreira et al., 2011). The possibilities to improve mechanical or thermal properties, developing a new material, by nanoparticles, have found applications in both academia and industry. Were performed several experimental and theoretical studies about micro and nanocomposites mechanical properties but there have been few studies on the thermo-mechanical properties of these materials. Many papers report an improvement in the properties of polymers more effectively through the use of nanoparticles than with microparticles (Shukla et al., 2008).

A property that has been widely used to predict the change in mechanical properties of these materials as a function of temperature is the glass transition temperature (T_g), a second-order pseudo transition which constitutes a high interesting parameter of amorphous and semi-crystalline materials (Gracia-Fernández et al., 2010). The glass transition temperature basis is the onset of coordinated molecular motion is the polymer chain. In the region of glass transition temperature (T_g), the polymer softens, the modulus drops three orders of magnitude and the polymer becomes rubbery (Sperling, 2006). The T_g is usually the first nanocomposite property to be determined because gives a first idea about the maximum temperature this new material can support.

2. EXPERIMENTAL

2.1 Nanocomposites fabrication

The nanocomposite materials used in this study are composed of a polymeric matrix and metal oxides (Al_2O_3) nanoparticles used separately as fillers. The properties and sources of the composite material components are described as follows.

The employed polymer was RR515 (provided by SILAEX), an epoxy resin (ER) based on diglycidylether of bisphenol A (DGEBA). This resin was polymerized by the addition of an aliphatic amine hardener in a portion of 25 phr by weight.

The ER properties are presented in Table 1.

Table 1. Properties of epoxy resin.

Property	Epoxy
Viscosity at 25°C (cP)	12,000-13,000
Density (kg/m ³)	1160
Heat distortion temperature, HDT (°C)	50
Modulus of elasticity, E (GPa)	2.4-5.0
Flexural strength (MPa)	60
Tensile strength (MPa)	73
Maximum elongation (%)	4

The nano-particles, spherical alpha aluminum oxide nanoparticles with mean diameter of 35nm, provided by NanoAmor (www.nanoamor.com), containing 5-10% theta, were employed as filler. The nanoparticle properties are summarized in Table 2.

Table 2. Properties of nanoparticles.

Property	Al ₂ O ₃		
Particle size (nm)	30-40	150	200
Modulus of elasticity, E (GPa)	300	300	300
True Density (kg/m ³)	3700	3700	3700
Morphology	Spherical	Spherical	Spherical
Specific surface area, SSA (m ² /kg)	35,000	5,000-15,000	3,900
Purity	≥99.9%	99.9%	99.9%

The samples were manufactured by adding different amounts of nanoparticles from 0% to 10% in volume of the total mixture to the liquid resin. The nanoparticles volume fractions were calculating based on true densities data provide by the manufactures and the rule of mixtures. The nanoparticles drying process was performed at 120°C for 24 hours before added to the liquid resin. Homogenization by a planetary ball milling was performed during 1h at 200rpm. After mixing, the hardener has added and the resulting blends were manually homogenized and poured into the mold.

The mold was composed of a metal frame with tree channels between two glass plates. The specimens were cured at room temperature for 24 hours. The demolding of the samples occurred after the first 24 hours, and the samples remaining in a post cure process at room temperature for at least 7 days. After this time, the samples were grounded for DSC analyses.

2.2 Experimental setup

The glass transition temperature of the ER/nano-Al₂O₃ composites was measured by DSC (NETZSCH, DSC 200 MAIA) at temperature ranging from 30 to 150°C at a heating rate of 10°C/min under a nitrogen atmosphere in a 50ml/min flow rate. Three analyses with approximately 10mg of the material in an aluminum pan were performed for each sample (pure epoxy resin, nano-Al₂O₃/ER 2.5% v/v; nano-Al₂O₃/ER 5% v/v, nano-Al₂O₃/ER 7.5% v/v, nano-Al₂O₃/ER 10% v/v) in order to obtain an average value.

According to ASTM D 3418 (2012), the test was composed by three sections: first heat curve, to erase previous thermal history, or post-cure process; cooling, that can be used to know the crystallization temperature, for semi-crystalline polymers; and the second heat step, same as the first, which was used in this paper to provide the glass transition temperature for each nanocomposite.

3. RESULTS AND DISCUSSION

3.1 DSC Results

As observed by Tanaka et al. (2005), organic-inorganic bonding force seems to be relatively week in epoxy/alumina nanocomposites. Some authors, as Zhang and Singh (2004), are studying the addition of silane as a treatment to improve the particle (inorganic)/matrix (organic) adhesion.

Nanocomposite properties are directly related to the microstructure and particle dispersion obtained during manufacturing and and the physical and chemical interactions among particles and matrix are related to the superficial interaction of these materials (Luo and Daniel, 2003).

It is expected an smaller increase in the T_g the higher the particle diameter used since the adhesions forces between the nanoparticles and the matrix is directly proporcional to the specific surface area.

Table 3 displays the average T_g measured for each nanocomposite.

Table 3. DSC results.

Sample	T_g (°C)		
	Al ₂ O ₃ 30-40nm (A)	Al ₂ O ₃ 150nm (B)	Al ₂ O ₃ 200nm (C)
Pure ER	70.3	70.3	70.3
nano-Al ₂ O ₃ /ER - 2.5%	78.1	59.8	70.6
nano-Al ₂ O ₃ /ER - 5.0%	70.9	60.2	79.3
nano-Al ₂ O ₃ /ER - 7.5%	75.9	56.7	76.2
nano-Al ₂ O ₃ /ER - 10%	76.8	52.2	74.1

Only the sample nano-Al₂O₃ 30-40nm/ER 5% v/v not presented a result consistent with the literature and the responses of the other samples. During demolding and milling for the manufacture of DSC samples was observed the presence of bubbles inside the nanocomposite. Such bubbles can be regarded as points of concentration of alumina and adversely interfere in the analysis by DSC, were tried to avoid as much that they be added to the DSC samples. That is why the T_g results were omitted in the next figure.

Figure 1 displays a samples comparison and the T_g behavior for each mixture.

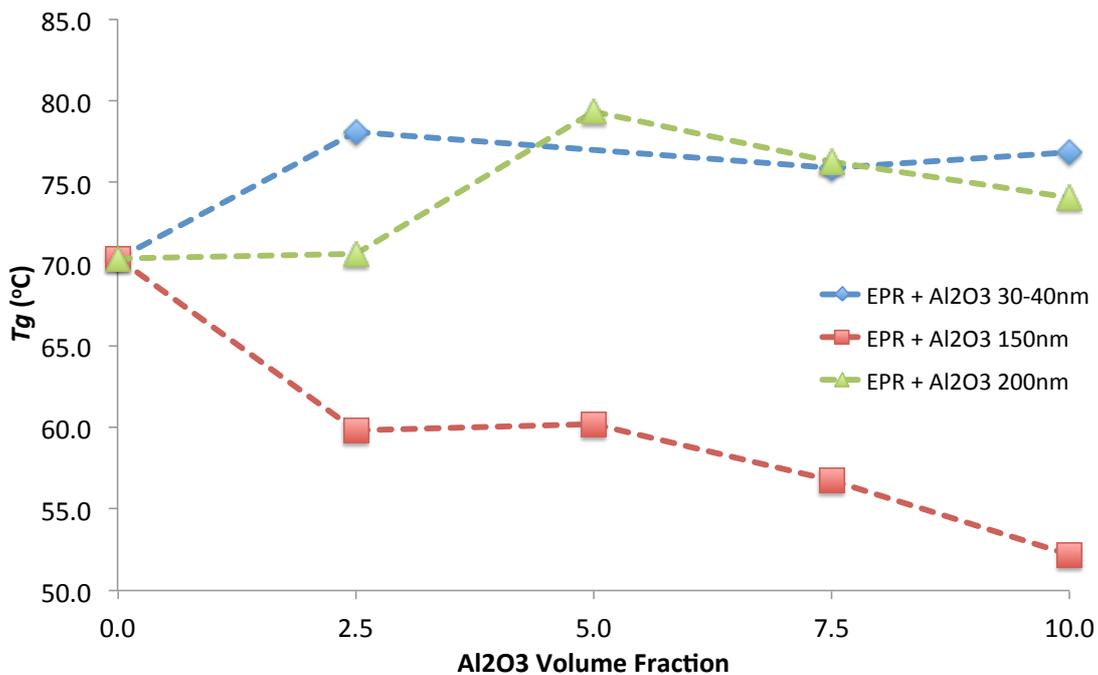


Figure 1. DSC results.

Glass transition data showed in Table 3 and figure 1 are apparently according to what have been obtained recently for nanocomposites without silane treatment. However, the results do not follow the theories of interaction on the surface.

For a same particle diameter, it was expected an increase in T_g with increasing volume fraction of particles. The behavior for the T_g observed in this study was also observed by Shukla et al. (2008) for tensile strength, caused by the low chemical bonding between the inorganic particles without treatment and the organic matrix.

Since DSC analyses shows the microscopic behavior of the materials, the T_g decrease in the samples reinforced with Al₂O₃ 150nm can be explained assuming a lack in the adhesion forces between nanoparticle and matrix, in that the nanoparticle will behave as a defect, reducing the T_g and not as a particle that would increase adherence between polymer molecules, which would increase the glass transition.

4. CONCLUSIONS

The purpose of this study was to observe the glass transition temperature by Differential Scanning Calorimetry (DSC) in epoxy/alumina nanocomposites. Was observed that the glass transition is unchanged between different volume fractions. For different particle diameter, only the variation of the specific surface area of the particles is not enough to understand the behavior of the variation of T_g with particle diameter and the adhesion theory is important in the way to predict this property in composite materials. Thus, Dynamical Mechanical Analysis (DMA) and Fourier transform infrared spectroscopy (FTIR) studies will be realized hereafter, enabling an understanding of fundamental molecular interactions to study nanocomposites and the macromolecular mechanical behavior of these materials.

5. ACKNOWLEDGEMENTS

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