Influence of Carbon Fiber Addition in Physical-Thermal Properties of Composites Obtained by RTM

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Abstract: This work aims to study the influence of carbon fibers addition in physical-chemical properties of bicomponent epoxy resin, produced by POLIPOX, in a composite obtained by RTM. Thermal analyses (DSC (Differential Scannig Calorimetry), TGA (Thermal Gravimetric Analysis) and DMA (Dynamic-mechanical Analysis) were performed in order to compare the results and observe the reinforcement influence on the resin properties. Increased glass transition values and degradation temperatures show that fibers cause a reduction in the molecular motion of polymer chains.

Keywords: Composites materials, RTM, thermal analysis, carbon fibers, epoxy resin.

1. INTRODUCTION

The use of composites materials has increased worldwide since the 50s, being mostly considered an alternative to traditional metals [1]. Composite materials have been commonly used by several industries because they show properties as lightness, flexibility, durability, strength, and adaptability. They are constituted by two or more materials, reinforcements and matrix, which together result in a material with higher mechanical and thermal properties, rather than the same materials being separately used. The matrix involves the reinforcement, protecting them against external ambient influences, distributing the strain applied to fibers and resulting in the component's final form. The reinforcement relates to the physical properties of the obtained composite [2-5]. Among the manufacturing process used in polymer matrix composites, the one which is mostly applied is the manual blading with autoclave cure. Knowing that the composite quality depends on the processing, the optimization involves optimizing and replacing the manufacturing process [6]. Many processes have been developed for the manufacturing of composite materials. Among these techniques, RTM (Resin Transfer Molding) is regarded as fast, flexible and capable of producing parts with good surface finishing on both sides. The RTM stands out, which is a technique capable of reaching the imposed quality for the aviation and automotive industry by a structural application of polymer matrix composites [6-12].

In the RTM process, a dry fibrous preform is placed in the mold cavity, being subsequently impregnated with a liquid resin. The resin enters the mold through a number of injection ports (holes) and slowly flows within the mold cavity [1].

The liquid resin of low viscosity is injected in the mold until a total fiber impregnation is reached, using low pressure. After that, the metallic mold can or cannot be heated for the cure process start, depending on the characteristics of each resin. At the cure ending, the mold is opened and the processed composite is removed [8,9].

The thermal analysis is an important analytical technique that measures the physical behavior of materials in relation to temperature, when these materials are submitted to a thermal cycle. This technique has been used for better understanding the structure-properties relationship, as well as evaluating the composites thermal stability. The techniques set supplies data, as the temperature limit in which the composite can be applied, the elastic properties, as well as to possibility carry out a kinetic study for the determination of other parameters [13-15].

In this work, the influence of carbon fibers addition (using the HexForce™ AGP193-P plain weave) is to evaluate the thermal behavior of the bicomponent epoxi resin, produced by POLIPOX. The composite was obtained by RTM process by FEG/UNESP Fatigue and Aeronautics Materials Group.

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2. MATERIALS AND METHODS

2.1. Resin Thermal Analyses

The POLIPOX bicomponent resin was thermally analyzed with DSC, TGA and DMA techniques. The resin was cured before than analyses.

Analysis by differential scanning calorimetry (DSC) under dynamic conditions were performed in an equipment Seiko Model 6220 SII Nanotechnology, calibrated with indium and zinc, based on ASTM D3418 [16] to evaluate glass transitions (T_g). The heating rate used for the dynamic analysis of epoxy polymer was 20°C/min, as recommended by ASTM D3418 - *Standard Test Method for Transition Temperatures and Enthalpies of Fusion and Crystallization of Polymers by Differential Scanning Calorimetry* [16]. As sample containers, have been used sealed aluminum pans with mass about 10 mg. The software on the central module of the equipment is Muse Measurement - Version 6.2U (1989 - 2006). This program enables the management of the analysis, as well as the evaluation of the obtained curves.

The TGA test was realized in a nitrogen atmosphere (gas flow rate: 100 mL/min), with a heating rate of 10°C/minute, between 30 and 800°C, using the TG/DTA 6200 SII Nano Technology INC equipment, using approximately 15 mg. The definition of the initial temperature degradation was based on ASTM E2550 - *Standard Test Method for Thermal Stability by Thermogravimetry* [17] whereby the temperature is determined when the first deflection on the baseline of the curve is observed TGA.

Three resin samples were analyzed by DMA techniques, between 25 and 140°C, with a heating rate of 5°C/min, using a nitrogen atmosphere and a frequency of 1.00 Hz and deformation mode Dual Cantilever in accordance with ASTM D7028 - *Standard Test Method for Glass Transition Temperature (DMA T_g) of Polymer Matrix Composites by Dynamic Mechanical Analysis (DMA)* [18], of T_g determination in a nitrogen atmosphere, using a gas flow rate of 100 mL/min. The samples size for analysis of DMA were defined according to the standard ASTM D4065 - *Standard Practice for Plastics: Dynamic Mechanical Properties: Determination and Report of Procedures* [19] and the fixture used to test the machine's DMA 6100 SII Nanotechnology Inc.

2.2. Composite Thermal Analysis

As the resin, the composite was analyzed also through the TGA, DSC and DMA techniques, on the same bicomponent resin POLIPOX test conditions with the purpose of to verify the influence of the addition reinforcement in the resin thermal properties.

3. RESULTS AND DISCUSSIONS

3.1. DSC Analysis-Resin

The resin analysis in a dynamic DSC contributed to determining the glass transition temperature (T_g) of the

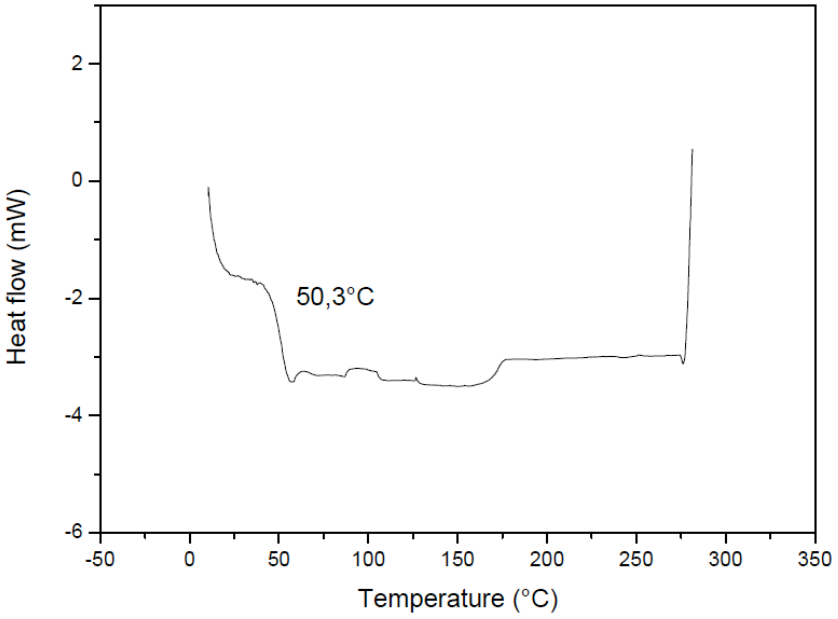

Figure 1: Glass transition of bicomponent resin.

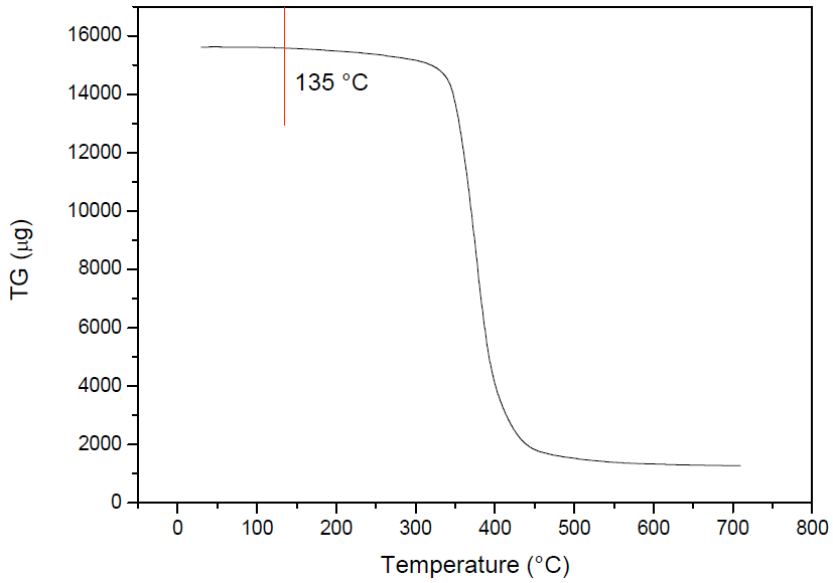

Figure 2: TGA curve of bicomponent resin.

resin. The Figure **1**, shows the POLIPOX bicomponent resin T_g. It occurred at 50,3°C. It can be stood out that for structural calculations purposes the used value is between 20 to 30% of the T_g.

3.2. TGA Analysis-Resin

The Figure **2** shows the POLIPOX bicomponente resin TGA analysis indicating start degradation around 135°C (ASTM 2550-07). From this temperature, the polymer's molecular chains primary links begin to be broken, causing a loss in its structural integrity, leading to possible failures.

3.3. DMA Analysis-Resin

The Figure **3**, for the POLIPOX bicomponente resin, shows the sample number one DMA curves.

The T_g values of the other samples, obtained in DMA analyze, are shown in Table **1**. For a comparation, the T_g value obtained in DSC analyze can also be found in Table **1**.

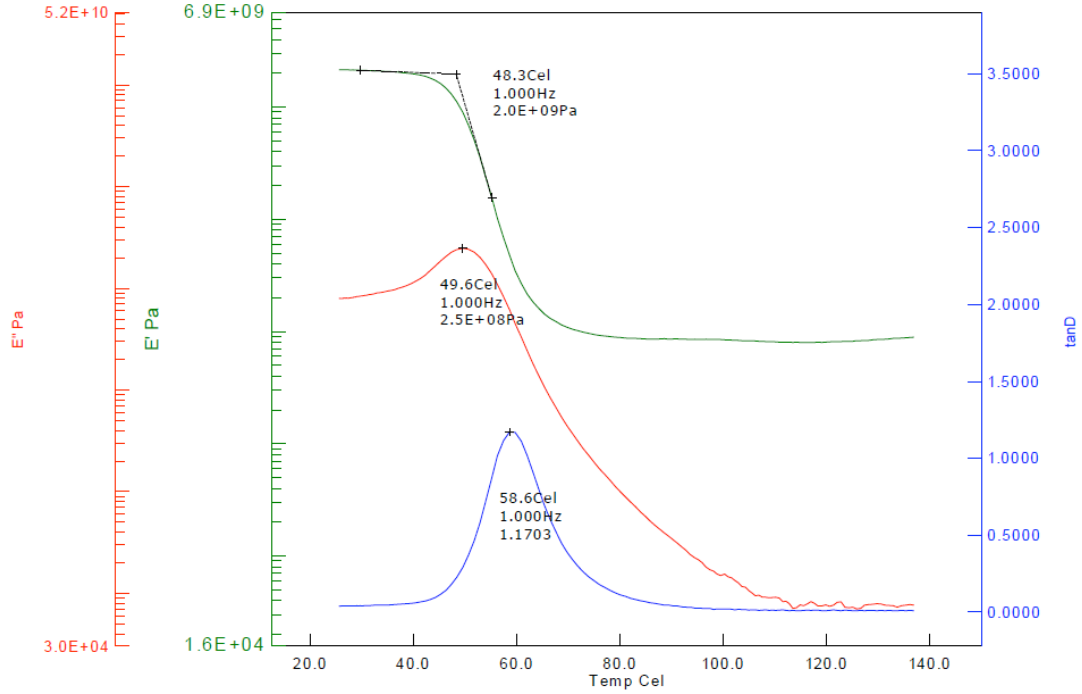

Figure 3: DMA curves of the bicomponent resin.

Table 1: T_g Values for the Resin

Sample	E' (°C)	E'' (°C)	tan D (°C)	DSC (°C)
1	48,3	49,6	58,6	50,3
2	49,9	51,3	59,2	
3	52,9	54,4	62,1	
Average	50,4	51,8	60,0	

3.4. DSC Analysis-Composite

The DSC test provides information about the glass transition temperature (Figure **4**). In the composite analysis, the determined T_g was 48,2°C. The value of the found T_g for the composite was lower than that found for the unreinforced resin. This fact can be explained by the presence of fibers which, in turn, caused an impediment in the movement of the resin polymer chains, making it harder for the relaxation of these.

3.5. TGA Analysis-Composite

The Figure **5** shows the TGA curve of the composite. It can be observed that the degradation

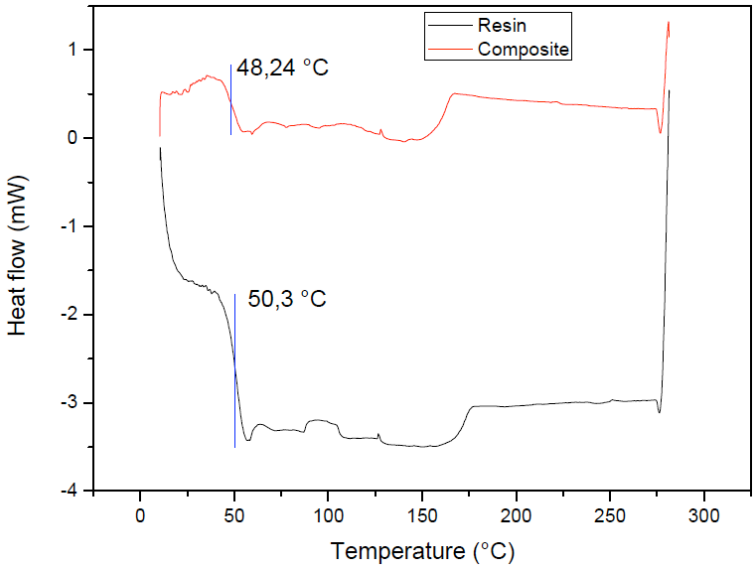

Figure 4: Glass transition of the composite and bicomponent resin.

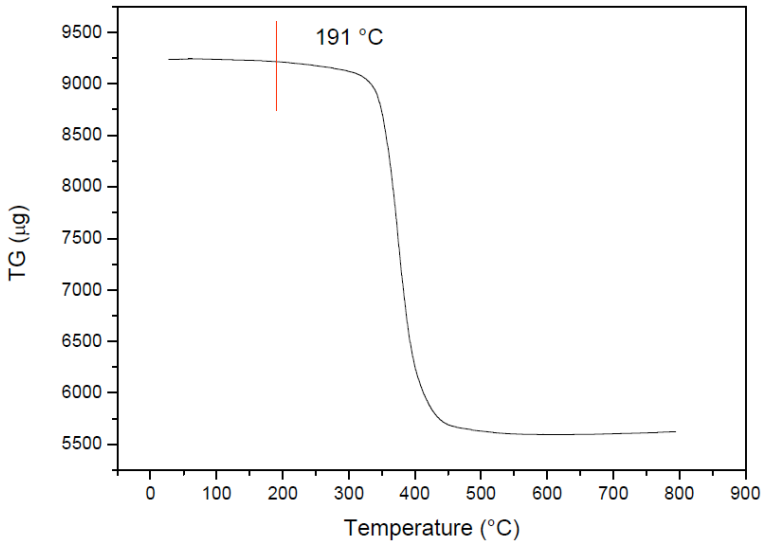

Figure 5: TGA curve – degradation of the composite.

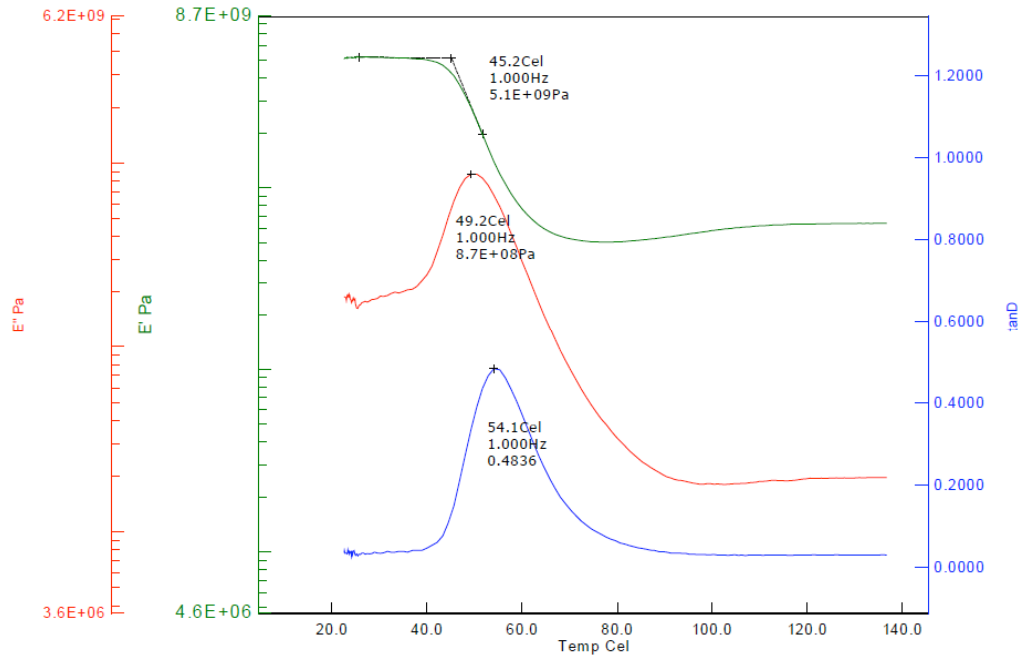

Figure 6: DMA curves of the composite.

Table 2: T_g Values for the Composite

Sample	E' (°C)	E'' (°C)	tan D (°C)	DSC (°C)
1	45,2	49,2	54,1	48,2
2	43,7	48,9	53,2	
3	42,3	46,6	50,9	
Average	43,7	48,2	52,7	

process began at 191°C. This temperature is higher than the degradation of the resin without the reinforcement. The interaction between the fibers and the matrix favored the thermal stability of the sample. It can be stood out that the fibers can function as a obstacle, rendering difficult the diffusion of volatiles products generated by degradation process, such as was realize by *Carvalho et al.* with grapheme nanosheets [20].

3.6. DMA Analysis-Composite

The Figure **6** shows the DMA curves of the composite sample number one.

The T_g values of the other samples, obtained in the same analyze, are shown in Table **2**. For a comparison, the T_g value obtained in DSC analyze can also be found in the table above.

For a better comprehension of the experimental data, the Table **3** was constructed of T_g values averages, obtained by DSC and DMA tecniques.

Table 3: Comparison of Resin and Composite

	Composite	Resin
E'	43,7 °C	50,4 °C
E''	48,2 °C	51,8 °C
tan D	52,7 °C	60,0 °C
DSC	48,2 °C	50,3 °C

4. CONCLUSIONS

The thermal analysis showed that the addition of carbon fiber reinforcement to the POLIPOX bicomponent epoxy resin influenced the thermal properties. The composite glass transition temperature was lower when compared to the resin without reinforcement, in the both analyzes (DSC and DMA). While the degradation temperature was higher.

Therefore, the addition of the carbon fiber was advantageous, because it provided an improvement in the thermal properties of the composite.

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