EFFECTS OF INTERFACIAL ADHESION ON THERMAL AND MECHANICAL PROPERTIES

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Abstract

Carbon fiber/epoxy matrix composites produced by resin transfer molding (RTM) were investigated in order to define the influence of interfacial adhesion on mechanical and thermal properties. The adhesion was studied through DMA, DSC and TGA thermal analyses and tensile test. SEM analyses of fibers surface and tensile fractures support the conclusion that the composite with higher degradation temperature and Tg value has better interface boundary. Besides, the DMA results corroborate with the same conclusion.

1 Introduction

The fiber-matrix interface has been the great challenge of composites manufacture since the mechanical and thermal properties are closely linked to it [1-2]. Good interfacial adhesion can improves mechanical properties and thermal stability of polymer composites through effective stress transfer and decrease of chain polymer mobility [2-3].

The interfacial adhesion can be divided in physical and chemical interaction. The first one is based on mechanical interlocking and the other is based on polar and acid-base linking [4-5]. Carbon fibers (CF) are usually coated by sizing, a polymeric solution applied to improve the adhesion with resin matrix, since the CF are normally inertness and promote a very weak interface. Some fibers show roughness and porosity, since a good interfacial adhesion can be obtained through the physical interaction. [4;6]. Dai [6] (2011) concludes in his study that interfacial properties depends not only on chemical but physical interaction. Some authors have shown that the processing technique has too influence on interface and, consequently, composites mechanical properties, mainly due to parameter processes as injection pressure and vaccum [7].

Using the DMA technique for interfacial fiber/matrix evaluation was proposed by Akbar and Yiu [8]. These showed that tan δ peak can be related to interfacial adhesion strength and is a true indication of molecular motion. The higher the molecular motion, greater the area under the tan δ peak and weaker interfacial adhesion [8-9]. Ko et al [10] also reported a decrease of damping peak with the highest intensity of interface adhesion.

The TGA and DSC are complementary analysis to evaluate the interface. Higher temperatures of Tg and degradation are signals of better interfacial interaction, considering composites with the same matrix or reinforcement of types [11-12].

Traditionally, mechanical properties are linked with quality of interface and its strength magnitude [1;13]. Some authors have shown that a slightly variation of interface properties can lead to significant altered performance of composites [1;9].

Many phenomena associated with composites interface and your influence are understood yet. In this work the interface of two carbon/epoxy composites processed under same conditions by RTM using Cycom 890 RTM epoxy system and two types of intermediate modulus carbon fiber were characterized through TGA, DSC, DMA, and tensile test. The SEM was used to confirm the interfacial adhesion difference observed on tests and show the fiber surface, tensile fracture standard and interface of produced composites.

2 Materials and methods

2.1 Materials

Resin transfer moulding (RTM) process was used to prepare two different composites sets with biaxial fabrics. The Plain Weave (PW) and Twill Weave (TW) fabric were manufactured by Sigmatex Material Innovation with 200gms. The commercially available carbon fiber used to prepared the PW (T800HB 6K 40B) and TW (IM7 GP 6K) fabrics were, respectively, T800HB 6K 40B (Toray Innovation by Chemistry) and IM7 GP 6K (Hecxel). Cycom 890 RTM monocomponent epoxy resin, produced by Cytech Technology, was applied as matrix. All composites were post-cured as manufacture´s recommendation. The fiber volume fraction on composites are 51,6% for PW-T800HB and 52,4% for TW-IM7, defined through acid digestion in a previous work.

2.2 Thermogravimetry

Three samples with nearly 10 mg were analyzed using a Seiko equipment model TGA-50, under nitrogen flow, between 30ºC and 800ºC with 10ºC/min heating rate.

2.3 Differencial Scanning Calorimetry

Three samples with nearly 6 mg were analyzed under nitrogen by using a DSC-Q10, TA Instrument. The test samples were heated from 40°C to 250°C at the rate of 10°C/min. Two analyzes were conduct, the first to eliminate the previous thermal history and the second to verify glass transition temperature.

2.4 Dynamic Mechanical Analysis

Following ASTM D7028, samples with (50x7,2x3,0) mm were measured using DMS 6100 model EXSTAR 6000 equipment (SII Nanotechnology Inc.). The analysis were done in three-point bending mode with besides 10 μm of amplitude, at frequency of 1 Hz and 3°C/min heating rate between 30°C and 300°C.

2.5 Tensile test

The tensile test specimen was prepared according to ASTM D3039 and carried out in a universal testing machine Instron 8801, with a 100kN load cell. The cross head speed of the specimen was 2 mm/min. Five samples were tested for each specimen.

2.6 Scanning Electron Microscopy

Carbon fibers surfaces of both composites and specimens subjected to the tensile test were analysed using a JEOL JSM5310 scanning electron microscope (SEM). All specimens were sputtered with a layer of gold prior to SEM observations. The accelerating voltage of 15 kV was employed. The SEM analyses of both composites were compared.

3 Results and discutions

3.1 Dynamic Mechanical Analysis

A comparison of dynamic mechanical properties of PW-T800HB and TW-IM7 composites is given in Fig. 1, which presents well defined curves. The E´ show clearly the limit between glassy and rubbery region, characterized by decreasing of E´ value. This same region is coincident with Tg in a conservative form, because indicate a increasing on chain molecular mobility. An observation of two composites curves, is evident the PW-T800HB higher modulus, however, the modulus drop takes place at close temperature, indicating a similar Tg. Higher modulus can means better interfacial adhesion.

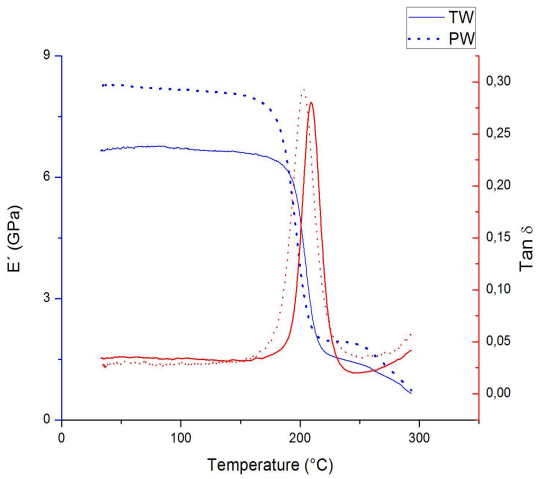


Figure 1. Dynamic mechanical properties of PW-T800HB and TW-IM7 composites.

The Tg curve (Fig. 1) indicates the beginning of relaxation process, coincident point with drop of E´ curve. This process is caused by combination of microbrownian chain motion and stress relaxation. The Tg peak shows a proportion of chain that starting their movement at each temperature, and the peak a temperature of most of chain segments are in movement. It can be explained for different store energy potential of chain.

According to Hameed et al. (2007) “the damping properties of the material give the balance between the elastic phase and viscous phase in a polymeric structure. In composites, damping

is influenced by the incorporation of fibers".
Besides, the energy dissipation occurs on interface, so a strong one is characterized through low energy dissipation, i.e., lower Tg height peaks what means more restricted movements and effective stress transfer.

Both Tg curves on Fig. 1 show similar height and temperature. According Table 1, the height is slightly lower and Tg temperature is higher for TW-IM7 (Table 1), what can means more effective interfacial bonding. The fact of the tan δ peak width at halt height be broader for PW-T800HB is an indicative of better adhesion, nevertheless, the other two aspects discussed leads to consider that TW-IM7 interfacial adhesion more effective.

	PW-T800HB	TW-IM7
E' (GPa)	8,0	7,8
Tg (tan δ)	202	209
Tan δ peak height	0,265	0,242
Tan δ peak width at half height	25	21
E" (GPa)	1,1	0,8

Tabel 1 – DMA data for both composites

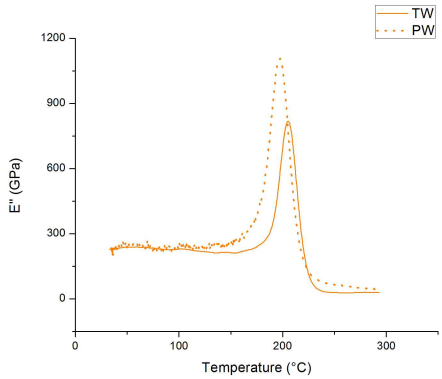


Figure 2. Loss modulus *vs* temperature for PW-T800HB and TW-IM7 composites

The Fig. 2 shows a viscous response of materials. Considering that E" curve is a measure of dissipated energy and its high is related to relaxation process, higher E" values, i. e., higher peaks, indicating more energy dissipation. In composites this is attributed to internal friction increasing.

Dislocated peaks to shift temperature are an indicative of more chain segments mobilization on fiber surface. Here, the PW-T800HB composite dissipated more energy, corroborating with Tg and E' peaks analysis, i. e., weak interface. The TW-IM7 showed more chain mobilization, pertaining to higher interfacial adhesion seen with SEM analyzes.

3.2 TGA and DSC Thermal Analyzes
Fig. 3 shows the TGA curves analyze of both composites. The point at with the TW-IM7 begin to decompose is greater, as well the Tg temperature observed on DSC analyses and described on Table 2. This results lead to confirm the DMA results which indicated the better interfacial adhesion to TW-IM7.

The Fig. 3 detail is a broadening of the curve beginning. The region I shows a small mass loss until about 150°C attributed to water loss. Regions II and III corresponds to stable mass region for PW-T800HB and TW-IM7, respectively. From this point, mass is lost and the weight sample becomes less than 99% of initial one. Although PW-T800HB reaches shift temperatures before to fall quickly, the TW-IM7 was considering more stable for support higher temperature without losing weight.

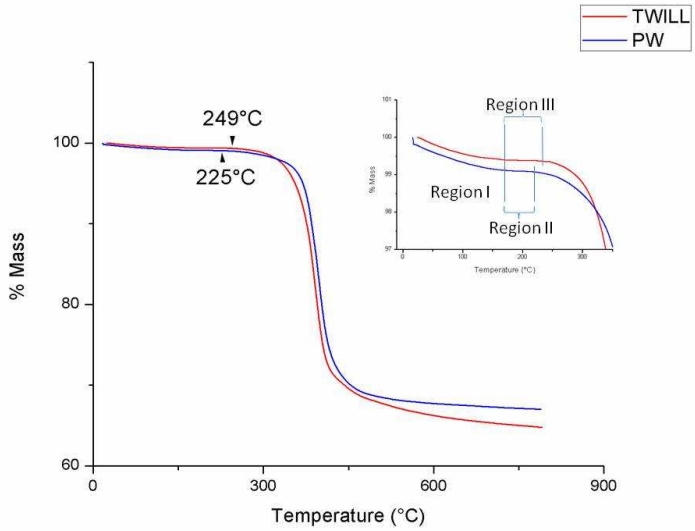


Figure 3. TGA analysis of both composites

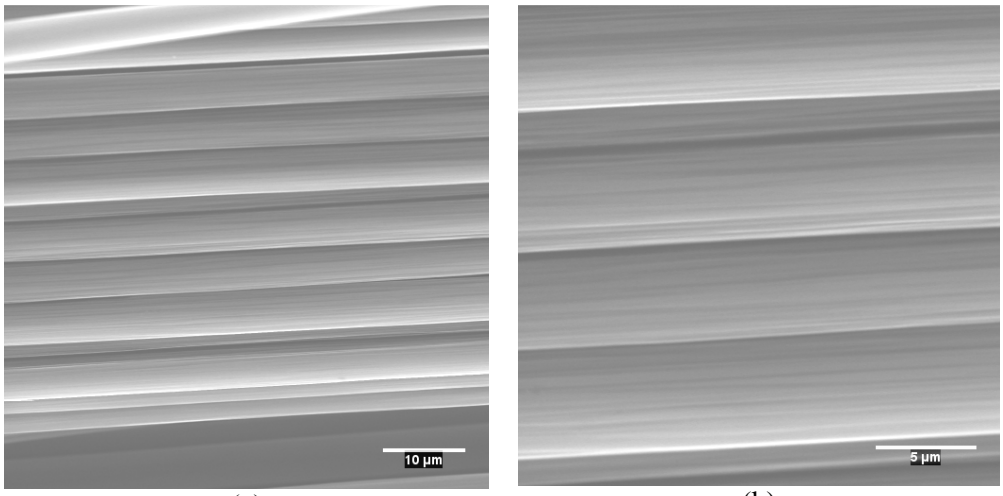
Fiber morphology affects the thermal properties since interfere on contact area and, reinforces with higher contact area promotes better thermal stability [12]. Interfacial defects can reduce these properties. So, the interface can delay the polymer degradation through decreasing of molecular motion, which increases with temperature.

	PW-T800HB	TW-IM7
Decomposition onset temperature by TGA	250°C	265°C
Tg temperature by DSC	208°C	213°C

Table 2. TGA and DSC composites data.

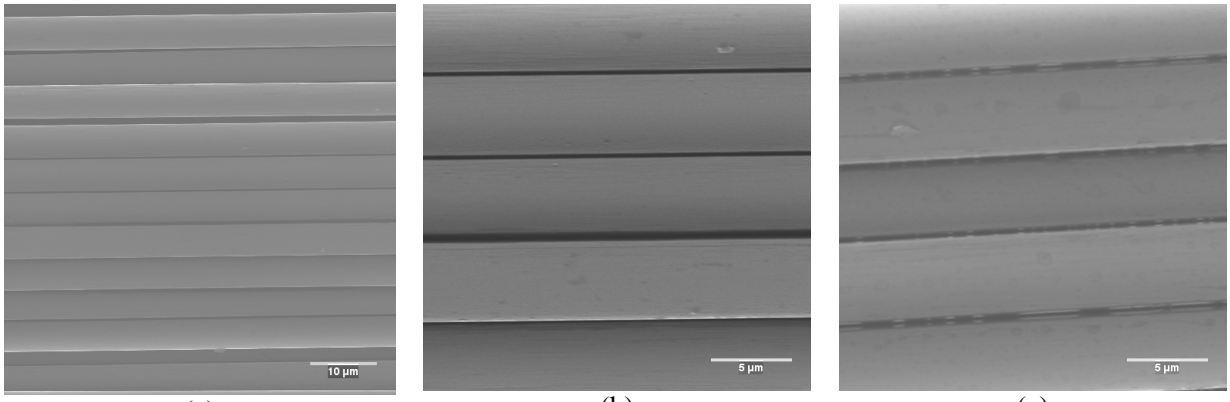
Observing the Fig. 4 and Fig. 5, PW-T800HB seems to be rougher than TW-IM7. This fact induces to believe that PW-T800HB will be offer better conditions to interfacial adhesion. However, according to Dai [6] (2011), cited on introduction, the interfacial properties depend on the chemical and physical interactions.

The TW-IM7 surface fibers are clearly recovered by resin with different chemical nature regarding the matrix (Fig. 5c). However this phenomenon could mask their original roughness (produced during Pan stretching stage) and provided an improvement of adhesion by chemical interlocking. Fig. 5b shows a TW-IM7 fiber region without coating resin excess.



(a) (b)

Figure 4. Roughness of PW-T800HB fiber surface with (a) 2000X and (b) 5000X.



(a) (b) (c)

Figure 5. Roughness TW-IM7 fiber surface with (a) 2000X and (b), (c) 5000X.

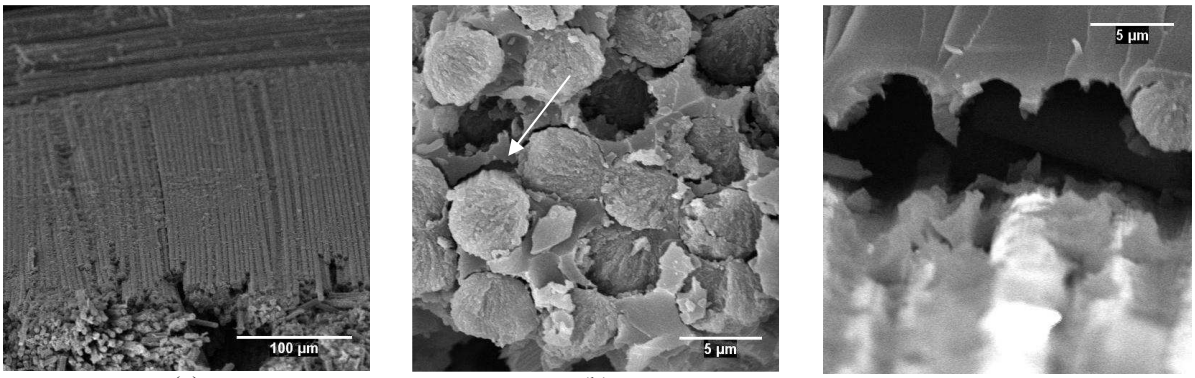
3.3 Tensile test

The values for composites tensile strength are showed on table 3, been higher for TW-IM7, although considering the Standard Deviation (SD) values are quite similar. SEM was applied on fractures to allow the accurate evaluation of interface, shows on Fig. 6 and Fig.7.

	PW-T800HB	TW-IM7
Tensile Strength (MPa)	773	827
SD (%)	9	14

Table 3. Tensile strength of composites.

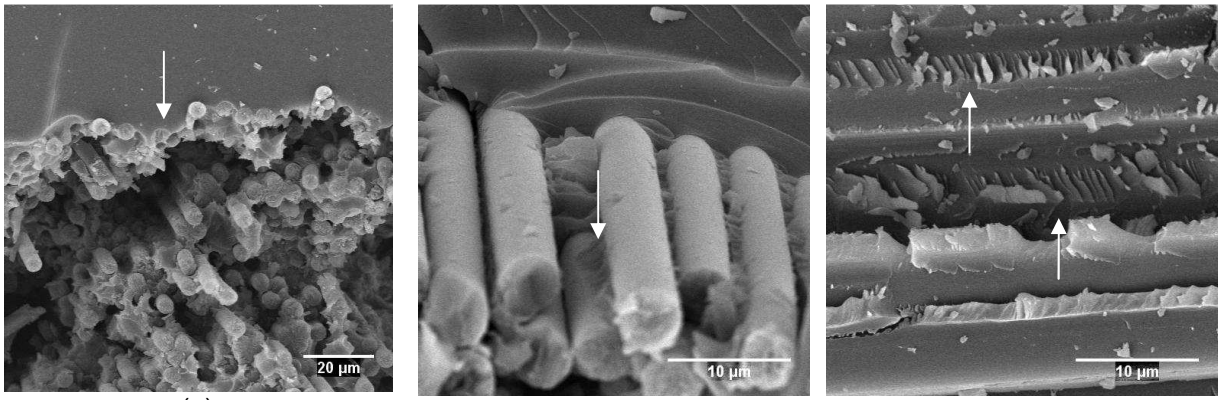
The Fig.6a shows a macroscopic view of PW-T800HB, where is possible seeing a good matrix and fiber distribution. On Fig. 6b can be seen a top of fiber tow and the matrix debonding (white arrow). The radial topography is a signal of individual crack propagation in each fiber. A delaminated region is presented on Fig. 6c.



(a) (b) (c)

Figure 6. Tensile fracture SEM of PW-T800HB composites with (a) 350X, (b) and (c) 5000X.

As for TW-IM7 composite, the SEM analysis shows a strong and uniform interfacial adhesion throughout the sample, as seen in Fig. 7a, which presents no failure by delamination. On Fig. 7b can be observed the presence of resin between fibers and small pieces of resin adhered on the exposed fibers surface. This fact leads to the conclusion about the fracture was initiated into the resin. So, is this case, the strength of interfacial adhesion is higher than matrix cohesive strength. Hackles caused by fiber separation have been observed on Fig. 7c.



(a) (b) (c)

Figura 7. Tensile fracture SEM of TW-IM7 composite with (a) 1000X, (b) and (c) 3500X.

Considering this analyze, the TW-IM7 composite shows higher interfacial adhesion, probably due to great chemical linking promoted by this type of fibers.

4 Conclusion

The PW-T800HB and TW-IM7 composites were shown to have different interfacial adhesion, what affected the mechanical and thermal properties of the polymer composites. It was reveled that composites made with IM7 Hecxel fibers promote higher interfacial interaction with Cycom 890 RTM epoxy resin compared with T800HB, a carbon fiber produced by Toray. Results of TGA and DSC show great thermal stability and Tg temperature for TW-IM7, results attributed to higher fiber/matrix interaction. The DMA shows lower energy dissipation through chain motion for TW-IM7, besides higher Tg temperature and peak height can means more effective interfacial bonding. Finally, the tensile fracture SEM analyses of composites confirm the higher interface formed between IM7 (Hecxel) fibers and Cycom 890 RTM epoxy resin, which is attributed to surface coating of them, although the PW-T800HB seems to be rougher.

Acknowledgments
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