# STUDY OF THE HYGROTHERMAL EFFECTS ON THE COMPRESSION STRENGTH OF CARBON TAPE/EPOXY COMPOSITES

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**Abstract**. *The wide range of composite material applications results in an almost inevitable contact with liquids and vapors, either organic or aqueous, which can affect both the immediate and the long-term performance of the material. In this study, the influence of the hygrothermal effects on the compression strength of carbon unidirectional tape/epoxy 8552 composites ([0/0]s) has been investigated. The moisture absorption was monitored placing the samples for a long time (until saturation) in a humidity-controlled chamber (80°C and 95% relative humidity (RH)) and other samples in a salt spray chamber (36°C and 95% RH). The compression tests were carried out in two temperatures: the room temperature (22°C) and the high temperature (82°C). It was observed that the samples tested at room temperature and submitted to the salt spray chamber presented a decrease of 8% on the compression strength (1667.8 ± 107.3MPa) when compared with the non-conditioned specimens (1812.2 ± 30.6MPa). On the other hand, the samples tested at high temperature and submitted to the humidity-controlled chamber (1129.0 ± 32.5MPa) presented a decrease of 25.7% on the compression strength, when compared with non-conditioned samples (1519.1 ± 52.6MPa). However, the specimens submitted to the salt spray chamber (1237.8 ± 36.9MPa) presented a decrease of 18.5% on the compression strength when compared with non-conditioned specimens at high temperature. These results showed that the combined effect of the humidity with the high temperature test, decrease the compression strength of the composite materials.*

**Keywords***: polymeric composites, hygrothermal effects, compression strength, salt spray.*

## 1. Introduction

The polymeric composites, when placed in service, are exposed to a variety of ambient conditions and different types of mechanical loading. The main atmospheric agent causing s ambient attacks are the temperature, the relative humidity of air, the effect of ultraviolet radiation, the chemical exposition, the saline water and the contact with hydraulic fuels, gases and fluids. Thermosetting polymeric composites must perform under both environmental conditional combined with deformations and mechanical stresses (Demonte, 1987).

The environmental effect caused by temperature and relative humidity of air can be reversible, when the period of exposition is of short duration. However, when the exposition occurs in long cycles, where the combination of humidity with temperature change is present, the produced effect can be irreversible due to the water affinity with specific functional groups of polymeric matrices of polar nature. In this case , generally destructive alterations in the reinforcement/polymeric matrix interface occur due to the degradation of the physical and chemistry interactions between the resin and the fiber. In consequence, the displacement of fibers can occur, causing the delamination of the composite with the reduction of the mechanical and thermophysical properties of the composite material (Demonte, 1987; Ashbee, 1993; Wolff, 1993; Cândido, 2001).

The present work aims to contribute with the study of the mechanical behavior of thermosetting polymeric composites submitted to the humidity-controlled chamber and to the salt spray chamber.

## 2. Materials and methods

### 2.1. Materials

The material used in this study is prepreg of carbon/epoxy produced by Hexcel Composites and supplied by EMBRAER (Empresa Brasileira de Aeronáutica S.A.) in the form of continuous unidirectional tape. This material is commonly used in the manufacturing of aircraft primary structure. The used prepreg material has the 8552 modified epoxy resin system. The reinforcement of the prepregs used in this work was the IM7 carbon fiber of high resistance,

with density of 1,75 g/cm³, maximum deformation of 1.4 % and previously treated for a better chemical compatibility with the epoxy resin system.

The cure cycle used for the composite consolidation was suggested by the prepreg supplier. This procedure involved 4 steps. Firstly, the heating from room temperature up to 116ºC at 2.5ºC/min, after an isothermal period in this temperature for 60 min; heating to 181ºC at 2.5ºC/min and finally an isothermal period of at least 120 min.

The samples had been divided in three families with six specimens for each: the first one was kept in the room condition of the laboratory, called as dry or non-conditioning, the second family was conditioned in a humidity-controlled chamber and the third was conditioned in a salt spray chamber until the humidity saturation. Later compression strength tests have been performed in two conditions: at room temperature (22 ± 3ºC) and high temperature (82 ± 1ºC). The tests carried out at high temperature aim to comply with the requirements recommended by the standard MIL-HDBK-17 – 02 (2002), considered the most critical condition for the polymeric composite test.

### 2.2. Hygrothermal conditioning

The samples were submitted to two different hygrothermal conditionings: in a humidity-controlled chamber and in a salt pray chamber. The moisture absorption was controlled by weight changes of traveler specimens (representative specimens used to measure the mass gain) according to procedure B of ASTM D 5229/D5229 M-04 (2004). In this study the moisture absorption was monitored placing the samples for nine weeks (until saturation) in a humidity-controlled chamber at 80°C and 95% of relative humidity (RH). The mechanical test specimens were kept in the environmental chamber until saturation. The specimens were then removed from the environmental chamber and tested at room temperature.

The conditioning in salt spray chamber was carried out according to ASTM 117 – 03 (2003) to verify the strength of the sample in saline condition for a period of nine weeks. The traveler samples and the test samples were suspended between 15º and 30º in a parallel vertical line, guaranteeing no contact between them inside the chamber. The main direction of the mist flow in the chamber is horizontal. The saline solution was prepared by the dissolution of 5 sodium chloride parts (w/w %) in 95 distilled water parts, with pH in the range of 6.5 to 7.2. The samples were submitted to the temperature of 35°C.

### 2.3. Compressive strength

Compression tests were carried out according to the IITRI experiment (developed for the Illinois Institute of Technology Research Institute), which follows the ASTM D 3410/D 3410M-03 (2003), in a universal machine of test MTS with load cell of 100kN. This device uses a relatively small sample with bearing and steel columns to guarantee the alignment of the loading. The grips in the form of wedges are lodged in machined sockets (Fig. 1).

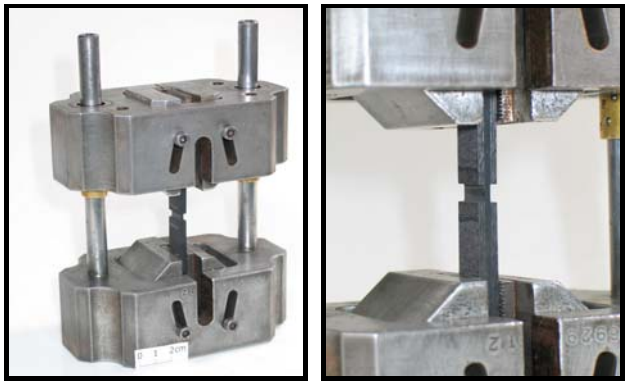

Figure 1 - Device of compression test used.

## 3. Results

### 3.1. Moisture absorption

The humidity increment can be gravimetrically measured through the change in the weight of the polymeric composite. The content of humidity absorbed for the families of unidirectional composites of carbon fiber IM7 impregnated with epoxy resin 8552 is presented in Tab. 1, as resulted of the weight average gain weekly in the traveler samples.

Table 1 - Results of humidity absorption in the composites submitted to the humidity-controlled and the salt spray chambers.

| | Average mass gain (%) | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| **Conditioning** | **1st week** | **2nd week** | **3rd week** | **4th week** | **5th week** | **6th week** | **7th week** | **8th week** | **9th week** |
| Salt Spray Chamber | 0.16 | 0.28 | 0.40 | 0.37 | 0.38 | 0.41 | 0.39 | 0.40 | 0.40 |
| Environmental Chamber | 0.23 | 0.31 | 0.33 | 0.33 | 0.36 | 0.37 | 0.39 | 0.39 | 0.39 |

It is observed, in Fig. 2, that in the initial period of exposition, it practically exists a linear relation between the absorption of humidity and the square root of the time. For short periods of time, the humidity content increases linearly until reaches a known state as pseudo-equilibrium, approximately between three and four weeks of exposition. This period of pseudo-equilibrium is practically the same for the majority of the thermosetting polymeric composites. Therefore this is characteristic of a behavior of humidity absorption according to the Fick´s diffusion law. The water remains free in the composite and it tends, with the time, to penetrate in the resin through the concentration gradient. Above of this linear portion, the humidity absorption starts to confer a concave format in the curve indicating a positive shunting line of the state of Fick´s pseudo-equilibrium. With the continuous exposition, the process of humidity absorption becomes slower, and many authors attribute to this period, the beginning of the process of relaxation of the polymeric chain (Costa, 2002; Choi, 2001; Cândido, Rezende and Almeida, 2000, Paplham et al., 1995; Thomason, 199a, 1995b; Chateauminois and Vicent, 1994; Harper, Staab and chen, 1987; Ishai, 1975).

For the composites submitted to the humidity-controlled chamber and salt spray chamber it was observed a weight average gain between 0.3-0.4%.

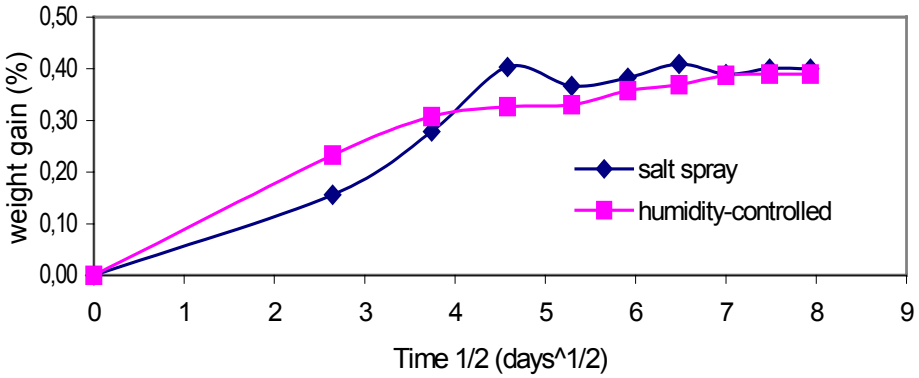


Figure 2 - Weight average gain of the composite submitted to the humidity-controlled and the salt spray chambers.

### 3.2. Tensile strength

Table 2 presents the results of the compressive strength tests, at room and elevated temperatures, of the samples submitted to the hygrothermal conditioning and the salt spray chamber. The result has been obtained using six samples for each family that were depicted in Figures 3 and 4.

Table 2 - Compressive strength results.

| **Room temperature tests** | **Maximum (MPa)** | **Minimum (MPa)** | **Average (MPa)** |
|---|---|---|---|
| Non-conditioned | 1841.7 | 1770.0 | **1812.2 ± 30.6** |
| Salt spray chamber | 1850.4 | 1538.7 | **1667.8 ± 107.3** |
| **Elevated temperature tests** | **Maximum (MPa)** | **Minimum (MPa)** | **Average (MPa)** |
| Non-conditioned | 1587.5 | 1461.7 | **1519.1 ± 52.6** |
| Hygrothermal chamber | 1159.9 | 1072.6 | **1129.0 ± 32.5** |
| Salt spray chamber | 1304.1 | 1179.9 | **1237.8 ± 36.9** |

Despite of the data dispersion, it was observed in Tab.2 and Fig. 3, that the composites tested at the room temperature (22ºC), previously submitted to the salt spray chamber (1667.8 ± 107.3MPa), presented the reduction of 8.0% on the compressive strength, when compared with the non-conditioned specimens (1812.2 ± 30.6MPa).

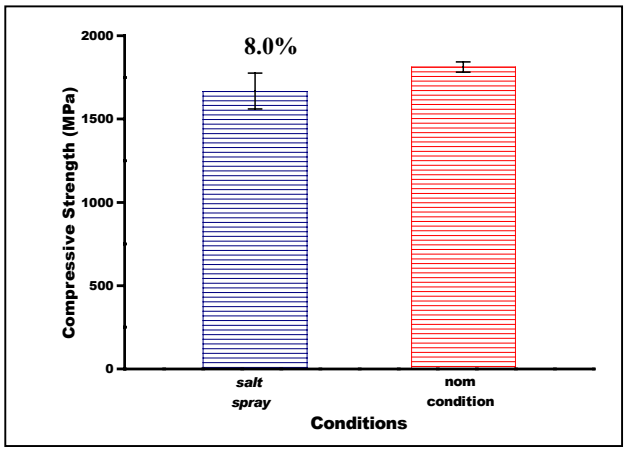


Figure 3 - Compressive strength values at room temperature.

The sample compression strength results (Tab.2 and Fig. 4), tested in elevated temperature (82ºC), show that the composites submitted to the humidity-controlled chamber (1129.0 ± 32.5MPa) presented a decrease of 25.7% in the strength, when compared with the specimens non-conditioned (1519.1 ± 52.6MPa). The literature confirms this behavior (Tang and Springer, 1989). On the other hand, the composites submitted to the salt spray chamber (1237.8 ± 36.9MPa) presented a reduction of 18.5% on the compressive strength in comparison with the non-conditioned composites.

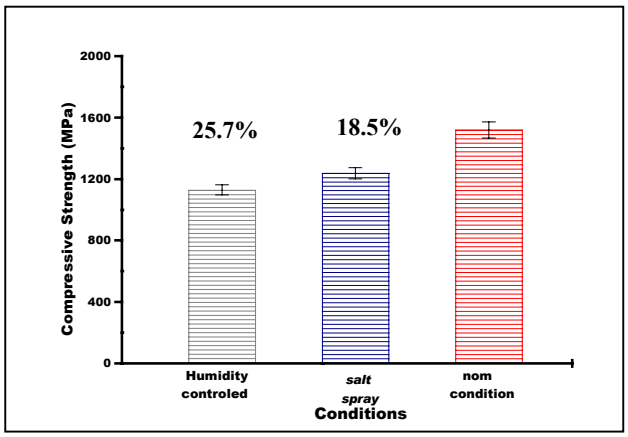


Figure 4 - Compressive strength values at elevated temperature.

The compression strength results of the composites tested at room and elevated temperatures (Tab.2 and Fig.5) show that non-conditioned composites tested at elevated temperature (1519.1 ± 52.6MPa) presented a decrease of 16.2% when compared to the specimens tested at room temperature (1812.2 ± 30.6MPa). Once again, this fact indicates that the temperature has influence on the composite mechanical properties, as shown by Tang and Springer, 1989. Comparing the results of the samples submitted to the salt spray chamber and tested at room temperature (1667.8 ± 107.3MPa) with the values of the specimens tested in elevated temperature (1237.8 ± 36.9MPa), it was observed that the composites tested at high temperature show a reduction of 25.8% on the strength.

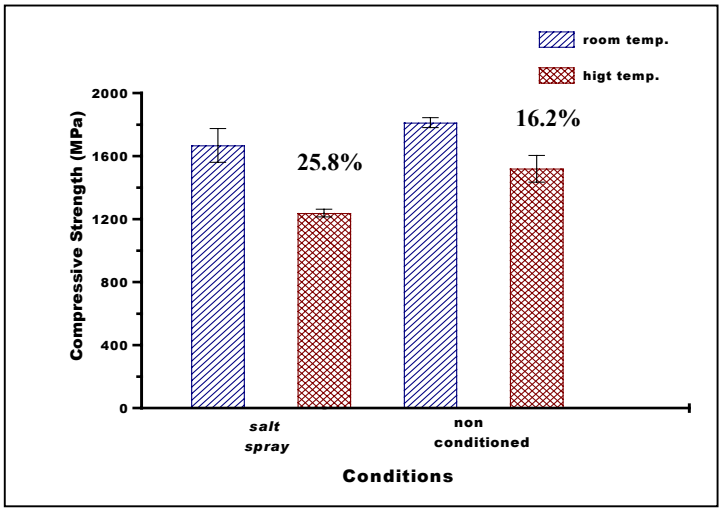


Figure 5 - Compressive strength values at room and elevated temperatures.

### 3.3. Failure mechanisms

Due to the anisotropy of the construction of the laminates, the process of failure in the thermosetting composites submitted to the loading in compression is very complex. Different failure mechanisms can occur and they are influenced by four main factors: the fiber characteristics, the polymeric matrix behavior, the angle lamination and the properties of the fiber/matrix interface.

Figure 6 presents representative photos of the samples tested in compression. All specimens present kink band failure. It is observed that the failure modes observed for the laminates submitted to the room conditioning are very similar to those ones observed for the specimens submitted to the humidity conditionings. It is also observed that the failure is perpendicular to the applied load, revealing interlaminar and translaminar failures.

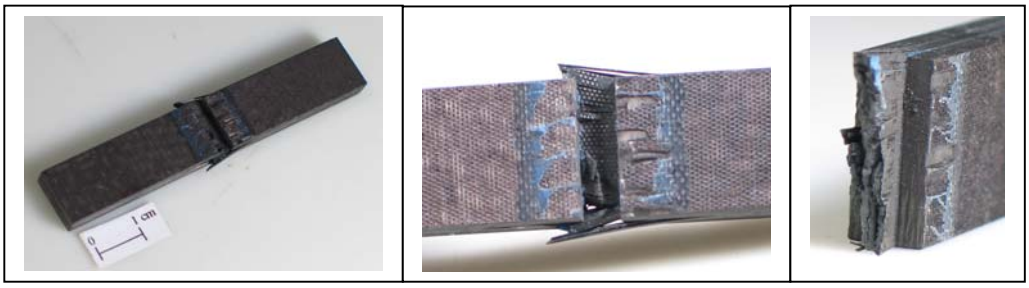

Figure 6 - View of the samples after the compression test.

The different environmental conditionings cause changes on the resin physical and chemical characteristics and on the fiber surface. The decrease on the compressive property indicates composite degradation. The fractography analyses were carried out to elucidate what are the probable causes that contribute to the decrease of the compressive strength.

The transversal section analysis shows the occurrence of crushing (Fig. 7) and the significant presence of matrix residues on the failure surface, due to the carbon fiber rigidity and the fragile characteristic of the epoxy resin. Figure 8 presents the failure surface of the unidirectional carbon fiber composite with a clear fragile fracture, due to the overload for compression that leads to the fiber breaking in a fragile way (without plastic deformation). Franco, 2003, shows that this type of fracture involves possible aspects of flexure in the top of fibers. Aspects of cusps in the matrix are also observed (Fig. 9), revealing that the resin remains adhered to the fibers in large extension, disclosing a good interface between fibers and resin. The majority of the failure surfaces of the specimen is presented damaged, due to the compression effect, which promotes the surface crushing.

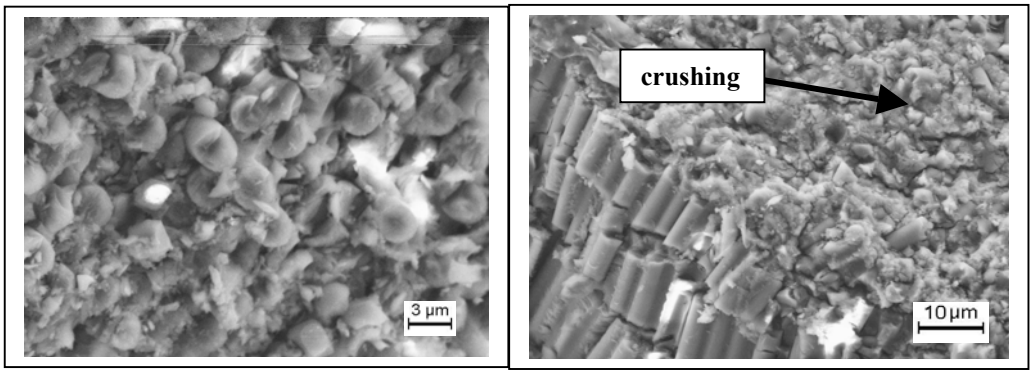


Figure 7 - Micrograph of non-conditioned sample after compression test, at room temperature.

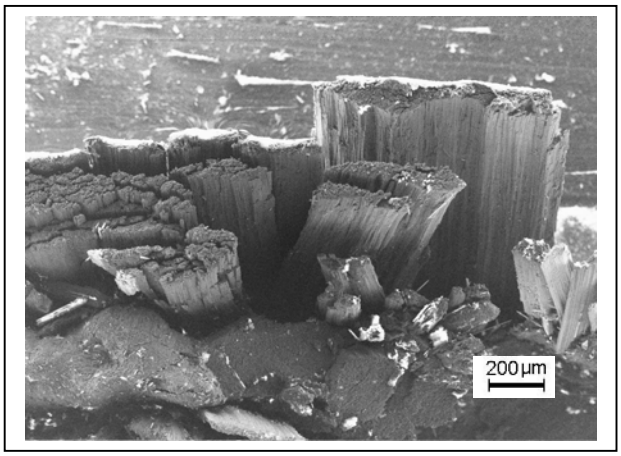


Figure 8 - Surface fracture of non-conditioned sample after compression test, at room temperature.

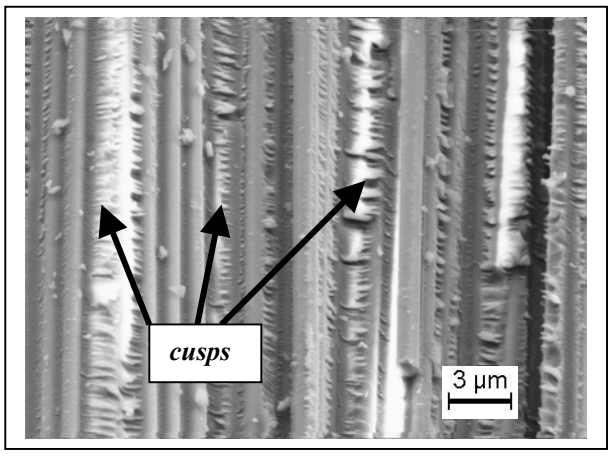


Figure 9 - Longitudinal section of non-conditioned sample after compression test, at room temperature.

Figure 10 shows a typical aspect of the composite submitted to the hygrothermal chamber and tested at elevated temperature, presenting a fragile fracture promoted by the overload in compression. Figure 11 presents the longitudinal section of the same sample (failure area), disclosing a larger amount of fibers without resin, indicating that the hygrothermal conditioning affected the fiber/resin interface and/or promoted the matrix degradation. Figure 12 is representative of the composite submitted to the salt spray chamber and tested at elevated temperature, which shows a significant amount of matrix residues that difficult the analysis of the failure region.

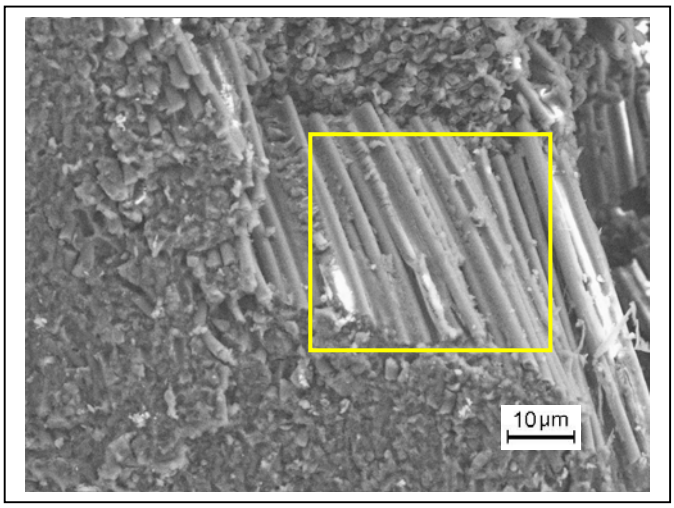


Figure 10 - Transversal section of the fracture surface of the sample submitted to the hygrothermal chamber and tested in elevated temperature.

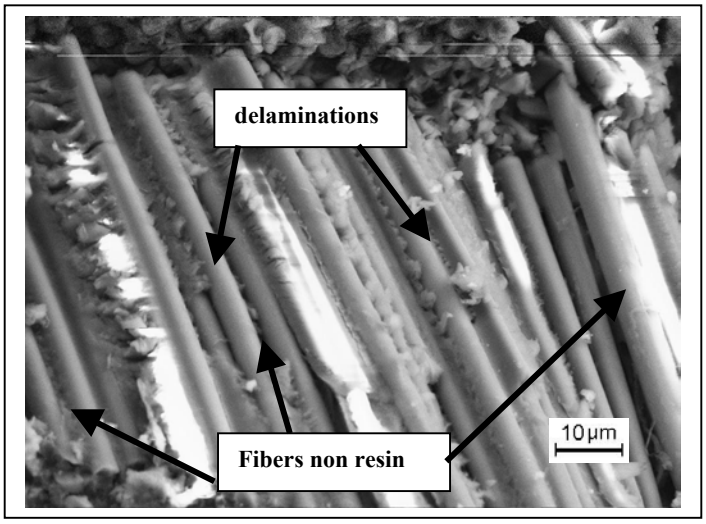


Figure 11 - Longitudinal section of the fracture surface of the sample submitted to the hygrothermal chamber and tested in elevated temperature.

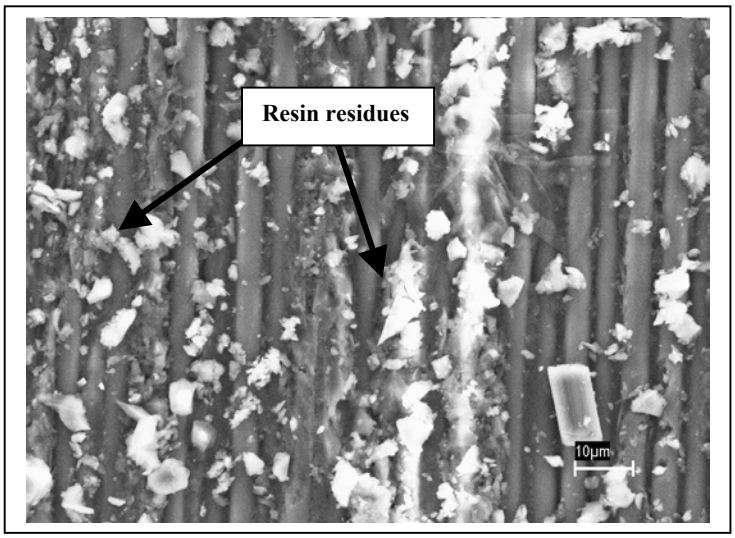


Figure 12 - Longitudinal section of the fracture surface of the sample submitted to the spray chamber and tested in elevated temperature.

## 4. Conclusion

The study of the hygrothermal effect on the compressive strength of unidirectional carbon fiber impregnated with resin epoxy 8552 composites showed that the polymeric matrix is severely affected in the tests at high temperature.

The composites non-conditioned and tested at the room temperature present a decrease of 16.2% in relation to the tested at high temperature. On the other hand, the samples conditioned in chamber of salt spray and tested at the high temperature show a reduction of 25.8% in the resistance in relation to the samples tested at room temperature. This fact confirms that the effect of the high temperature of test reduces the compressive strength, due to degradation of the polymeric matrix and/or the fiber/matrix interface.

## 5. Acknowledgements

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