24th ABCM International Congress of Mechanical Engineering
December 3-8, 2017, Curitiba, PR, Brazil

COBEM-2017-0573
CHARACTERIZATION OF THERMAL EFFECTS IN THE MODULUS OF ELASTICITY OF CARBON-EPOXY LAMINATE COMPOSITE, USING THE IMPULSE EXCITATION TECHNIQUE

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Abstract. *The composite term refers to a compound of two or more components, in macroscopic level, which does not have solubility with each other and that combined form a union of the best features of each one. The objective that generated the development of these materials was structural. Hence, the mechanical properties are the parameters that get further of study interest, such as the modulus of elasticity. The study of the thermal influence in this property is very important, which objective is verifying the viability of the material in different projects. This paper evaluated the thermal effects on longitudinal and transverse modulus of elasticity of a carbon-epoxy laminate using the Standard ASTM E1876.Two methods of characterization of laminated were analyzed, macromechanical and micromechanical composites, as well as the glass transition temperature of the material. As found in the literature, it was demonstrated by experimental data that the thermal effects reduce more the transverse stiffness value (41.67%) in relation to the longitudinal stiffness value (14.12%) up to a certain temperature.*

Keywords: *composite materials, modulus of elasticity, impulse excitation*

1. INTRODUCTION

The composite material depicts the creation of a compound that brings together the best features of different materials, in macroscopic scale, in just one. It can display any combination of properties that are required for material selection, which includes the highest specific resistances that can be achieved (Norton, 2013). According to Gibson (1997), it is being observed that certain composites have mixed mechanical properties, resulting in a material that allows an easier molding, low density and high resistance to corrosion.

The constituents of composites have been distinguished in two phases in macroscopic scale: the matrix and fiber that represent continuous and disperses phase of the material, respectively. The matrix and the fiber are responsible for the longitudinal and transverse modulus of elasticity of the material, respectively. If the distribution of the fiber in the array isn't equal at all points, there is a higher probability of failure in points with less distribution (Daniel and Ishai, 1994).

The characterization of these materials, as stated by Jones (1999), can be made through micromechanical or macromechanical analysis. In the first characterization, the study of the interaction of the constituent materials is examined in detail as part of a behavior of the heterogeneous composite material and it is applied by the mixing rule. In the second characterization, the material is assumed as homogeneous, it is presented an average of properties of the composite and it is applied by the Classical Laminate Theory (CLT).

As these materials have applications in many areas of industry, since the production of simple parts to high-performance race cars, it is possible that the environment with adverse conditions would degrade mechanical rigidity of the material. This deterioration could have been motivated by the action of temperature and humidity (Rodrigues, 2007).

In this context, the study of the hygrothermal influence on the composite is very important to verify the viability of the material. Therefore, this paper was developed with the objective of being a preliminary behavior study of the stiffness of carbon-epoxy laminate composite, with thermal variation using the impulse excitation technique.

2. HYGROTHERMAL EFFECTS

As the polymer matrix composite materials serve many areas of engineering, the possible degradation of the material is a very important study. In this way, the possible degradation of the material is a very important study, since the surrounding environment and the time-dependent phenomenon might have their mechanical properties dramatically affected. Among the many environmental conditions that can influence on the mechanical behavior of composite material, in conformity to Gibson (1994), the change in temperature and moisture content are parameters, which can result in significant effects on the mechanical properties of polymer matrix materials. The hygrothermal term represents both effects on the material.

On the research of Cunha, et al, 2006, it was confirmed that the heating effect is one of the most aggressive agents in the mechanical behavior of polymer matrix materials. When the temperature is increased to a high value, the fragmentation of the molecular chains causes chemical reactions with gaseous product and reduces the weight of the material. According to Canevarollo (2006), the deterioration derived from temperature causes random fission in the polymer chain. In addition, the tensile strength and the longitudinal modulus, which is the responsibility of fiber, are laminated parameters that suffer less aggression by the temperature than by the loading. Therefore, the matrix is the component that suffers degradation in the first order and has the properties damaged by the temperature (Vasiliev and Morozov, 2001).

As mentioned before, the fiber is responsible for the longitudinal modulus of elasticity E_1 (GPa); consequently, the matrix is responsible for the transverse modulus of elasticity E_2 (GPa) of the system. In Figure 1. Normalized experimental data of modulus of elasticity it is reported experimental data of modulus of elasticity of a unidirectional graphite-epoxy laminate at different temperatures that had been normalized. It is possible to check the most significant reduction in transverse modulus E_2 compared to longitudinal modulus $E_1$with the increase of temperature. The normalization was used for presenting distinct values between themselves and in each different temperature. This method represents the ratio of the actual value for the theoretical value of the modulus of elasticity in each point.

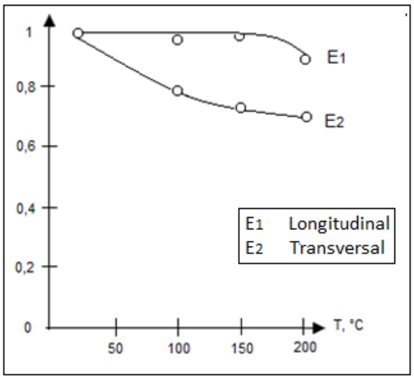

Figure 1. Normalized experimental data of modulus of elasticity.
Source: Adapted from Vasielev e Morozov, 2001.

Load orientation is a factor to consider in thermal degradation. The composites that undergo a request in the direction of the fibers, present their tension-strain curve little affected by the temperatures changes, once the stiffness and resistance of the fibers are predominating in the system. For cross-loaded composites, their tension-strain relationship is significantly affected (Figure 2), which the modulus of elasticity of the material decreases with increasing temperature in a dry environment (Gibson, 1994).

In conformity with Callister (2002), the polymeric material presents some resistance to this kind of deterioration called thermal stability, which has been related to the energy level of bonding between the atomic constituents of the polymer, i.e., higher the bonding energy, better is the thermal stability. The thermal conductivity of a laminate is greater along the fibers than through the thickness. Therefore, it turns out that the outer layers are the most damaged. On the other hand, in the material interior there is also the development of internal stresses between the blades that, when the mechanical resistance has been exceeded, can cause cracks in the matrix (Campus, 2012).

For the polymer matrix composites to attend the requirements for which they had been developed, in addition to their high properties, it should be verified their maximum service temperature, based on the glass transition temperature Tg. According to Canevarollo (2006), this temperature isn't an absolute value, but an average value of a region of temperatures. When the polymeric material is heated, it allows the polymer chains of the amorphous phase to acquire movement freedom. The polymer passes from vitreous state to rubber state.

For temperatures below the Tg, there are intermolecular forces that prevent the chains movement because they don't have enough internal energy to allow the displacement. When the polymer is heated, the temperature weakens these molecular forces and it allows the molecules to movement (Canevarollo, 2006). According to Epoxy Technology (2015), to obtain the reference value of the glass transition temperature, several factors are observed: chemical structure of the epoxy resin, type of hardener and degree of cure. Another important factor to determine experimentally the glass transition temperature is to evaluate the variation of the specific volume, that is, of the total volume that the polymer chains occupy (Canevarollo, 2006).

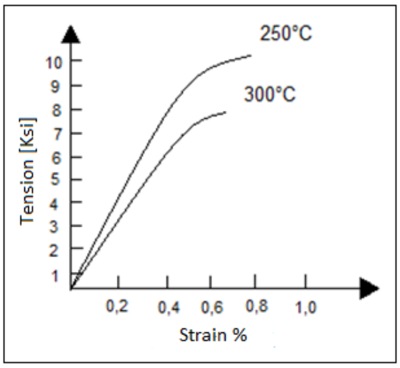

Figure 2. Tension-strain curve of a graphite-epoxy composite with transverse stresses under different temperatures.
Source: Adapted from Gibson, 1994.

To do the specific volume analysis, the polymers have been divided into amorphous and semi-crystalline. Due to the thermal expansion of the material, Figure 3, the increase of the temperature at a constant rate causes a progressive increase in the mobility of the polymer chains, generating a linear thermal expansion. After Tg, the mobility of the chains increases differently for each material. The inflection in the linear behavior of the different materials is defined as the glass transition temperature (Canevarollo, 2006).

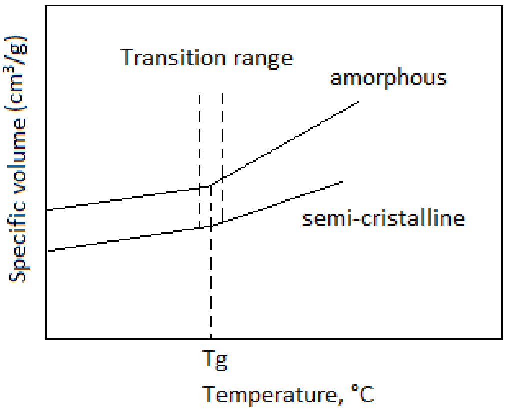

Figure 3. Specific volume variation with increasing temperature
Source: Adapted from Canevarollo, 2006.

Thus, a restriction of the use of the composites is not to be exposed to temperatures close to Tg, which the physical and mechanical properties of the polymers could be impaired (Matthews and Rawling, 1994). According to Baker; Dutton and Kelly (2004), if the service temperature exceeds the glass transition temperature, the modulus of elasticity of the material will decrease considerably.

3. MATERIALS AND METHODS

3.1 Materials

Epoxy resin had been used as matrix material with epoxy hardener and unidirectional carbon fabric with weight of 300 grams per square meter as fiber material. The method chosen for the manufacture of the composite was the hand lay-up and Vacuum Bag procedure. According to Carvalho (1992), the hand lay-up consists of depositing the fiber on the mold and impregnating it with the previously prepared resin, the application of the liquid resin being with paint rollers or brushes. This process has presented great efficiency and low cost because it does not require robust equipment and it is possible to obtain complex geometries of pieces.

The scaling of the specimen had been chosen carefully in accordance with ASTM E1876 and Sonelastic equipment manual. It has been selected a rectangular cross-section geometry, with a thickness of 2 mm, width featured

15 mm and 100 mm of length. These measures had been chosen to use the fewest possible layers that would prevent the waste of material and harmful effects of exothermic reaction.

In the cutting procedure, excess temperature hadn't been allowed because it could degrade the matrix before the test and the presence of burr could influence in the response to the excitation. Hence, two cutting equipment were tested: guillotine and cutting disc. The shear test was performed and the guillotine equipment obtained the best cut.

The laminate had been cut differently for each modulus of elasticity. To find the longitudinal elastic modulus E_1, the laminate has been cut with cutting oriented parallel to the direction of the fiber material and to find the transverse modulus E_2, the laminate has been cut, with the orientation of fiber material perpendicular to the specimen, as shown in Figure 4.

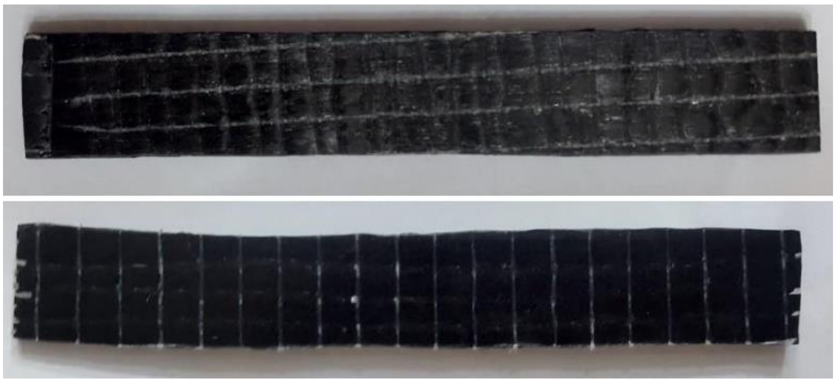

Figure 4. Specimen of longitudinal and transverse elastic moduli, top-to-bottom.

4. EXPERIMENTAL PROCEDURE

The excitation by impulse technique has been ruled by ASTM E1876, and aims to determine the modulus of elasticity experimentally of regular geometry materials in function of their natural frequencies of vibration. These frequencies are related to their mass, size and elastic properties.

The excitation of the material occurs by means of a punctual mechanical impulse of short duration and their acoustic response has been captured by a sound sensor, which with the aid of the mathematical method, such as the Fast Fourier Transform, identifies the natural frequency spectrum. This test can also be applied for high temperatures (ASTM E1876).

The equipment for tests of excitation by impulse is called Sonelastic, which consists of an automatic button, an acoustic pickup and a support for different geometric shapes. However, as the overall objective of this paper has directed to the impulse excitation technique at temperatures higher than the room temperature, the test was conducted in a furnace, whose button and acoustic pickup are integrated.

Moreover, there is Sonelastic software, which is responsible for characterizing the modulus of elasticity. The software allows the estimation of resonance frequency and harmonics of the material calculates standard deviation automatically for three measures of each dimension and calculates the uncertainty of measurement automatically.

5. RESULTS

5.1 Experimental data validation by non-destructive impulse excitation test

The non-destructive impulse excitation test has been ruled by ASTM E1876, in which the specimen isn't damaged due to the action of temperature, to obtain the longitudinal modulus of elasticity $E1$. The motivation to perform this test was to compare the experimental test with the analytical result calculated by the literature. The procedure of this test was performed using only specimen, whose fibers are oriented longitudinally, Figure 4 (a). First, in the software Sonelastic are added specimen information that are presented in the Table 1. Specimen data with their respective uncertainties.

After the setup procedure is completed, the excitation was performed. In the main screen of the program is presented the graph of the specimen response, which from the second peak it recognizes the natural frequency and calculates the modulus of elasticity. For the specimen used in this test, the value of the longitudinal modulus of elasticity E_1 of 87.25 ± 3.33 GPa is obtained.

Table 1. Specimen data.

Mass (g)	5.910 ± 0.001
Length (mm)	100.77 ± 0.05
Width (mm)	15.87 ± 0.16
Tickness (mm)	2.90 ± 0.03

After finding the experimental value of the modulus of elasticity E_1 demonstrated earlier, it had been compared with the analytical value obtained by CLT and mixing rule to make sure that the equipment has presented the expected value.

Thus, the information to find the analytical value, Table 2. Parameters for analytical calculation, had been taken from the fiber and resin datasheet provided by the manufacturer, except for the Poisson coefficient ν_{12} that had been selected as the typical value for carbon-epoxy composites of the literature by Vasielev and Morozov (2001). To find the Poisson coefficient ν_{21}, it had been used the Poisson relation.

Table 2. Parameters for analytical calculation.

Resin elastic modulus (GPa)	115.020
Matrix elastic modulus (GPa)	3.158
Longitudinal Poisson's ratio ν_{12}	0.09
Transverse Poisson's ratio ν_{21}	0.002

In the micromechanics analysis the ideal volumetric fraction was applied with 20% for matrix and 80% for fiber due the laminate fiber hadn't been pre-impregnated matrix (ABMACO, 2009). Then, the analytical modulus of elasticity E_1 of the laminated composite has been obtained by CLT. This value had been compared, Table 3, with the experimental modulus of elasticity previously obtained.

Table 3. Comparison of the analytical and experimental modulus of elasticity.

Parameter	Value
Analytical (GPa)	92.64
Experimental (GPa)	87.25 ± 3.33

Considering the simplifications established in CLT, the mixing rule and the errors associated with the experimental test, the values obtained in both methods were close. Therefore, impulse excitation technique had been validated.

5.2 Destructive impluse excitatio test

The test is termed as destructive because the temperature increases damages the specimen and it couldn't be reused to perform another modulus of elasticity measurement. The impulse excitation technique follows the previous procedure but now it had been inside the furnace.

The polymer composite has a maximum service temperature based on the glass transition temperature. According to the datasheet of the matrix, the Tg is located approximately 98 ° C, so the temperature range of the test had been limited from room temperature (25 °C) to 150 ° C. This reference value of Tg was taken from the technical data of the material, but it may have undergone changes in the experimental method by several factors, such as the environmental conditions, which the specimen was laminated and tested.

First, the thermal analysis had been performed on the longitudinal modulus of elasticity E_1 with the same specimen used in the non-destructive impulse excitation, which is demonstrated in Figure 4 (top) and their information has been presented in Table 1.

The impulse excitation was performed each time the oven reached a certain temperature that generates the graph demonstrated in Figure 5. Following what had been previously mentioned, the graph resulting from this test had been also normalized to be comparable with the graph presented in the literature.

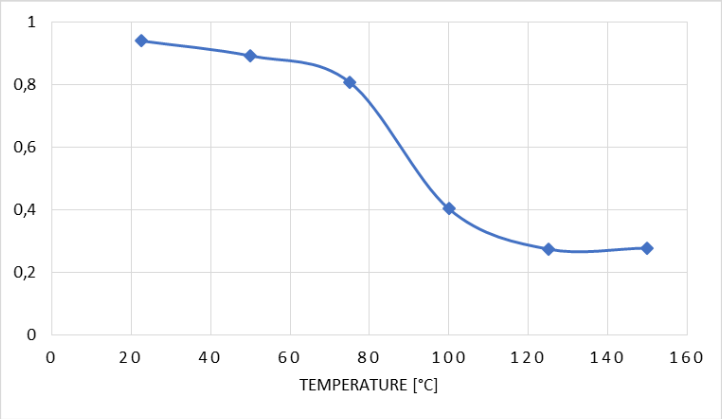

Figure 5. Experimental longitudinal modulus of elasticity.

As the temperature increases, the longitudinal modulus of elasticity has been reduced moderately in two regions: as it approaches Tg, previously identified as 98 ° C, and right after moving away from that region. At the point of attachment of the glass transition temperature, the initial stiffness of the composite is significantly reduced, which may have been motivated by a possible thermal deterioration of the matrix. An inflection point of the graph derives from this region of temperatures.

The specimen matrix has crosslinks that resist the possible movements caused by the temperature change, thus, as it had been heated, it was degraded permanently and before the fiber. In this case, the fiber is parallel throughout the specimen; it was responsible for withstanding the high temperatures. Thus, it influenced that lower stiffness drops outside the region of the glass transition temperature.

Next, the thermal analysis had been performed on the transverse modulus of elasticity E_2 with the specimen demonstrated in Figure 4 (bottom), whose information has presented in Table 4. Specimen data to calculate the transverse modulus of elasticity.

Table 4. Specimen data to calculate the transverse modulus of elasticity.

Mass (g)	5.5682 ± 0.001
Length (mm)	101.02 ± 0.11
Width (mm)	16.61 ± 0.23
Tickness (mm)	2.51 ± 0.09

The impulse excitation had been also performed every time the furnace reached a certain temperature, generating the graph demonstrated in Figure 6. The graph resulting from this test has also been normalized. It can be observed how the temperature had a negative influence on the elastic modality E_2 of the composite material. Analogous to the mode of longitudinal elasticity, as the temperature increases, the transverse modulus of elasticity also decreases. However, at this point a difference in the value drop has been noticed, compared to Figure 5.

Near of the glass transition temperature, 98 ° C, it is expected that the fall would be expressive in the modulus of elasticity due to the possible thermal deterioration of the matrix, but since the beginning of the temperature change, the transverse modulus of elasticity decreased significantly and only after Tg this reduction has slowed down.

As previously mentioned the matrix specimen is thermoset and degraded permanently as the temperature has raised and before the fiber. In this case, this fiber is perpendicular to the extension of the body and consequently, the matrix was responsible for withstanding the high temperatures. Thus, it has influenced this stiffness drop from the beginning of the test.

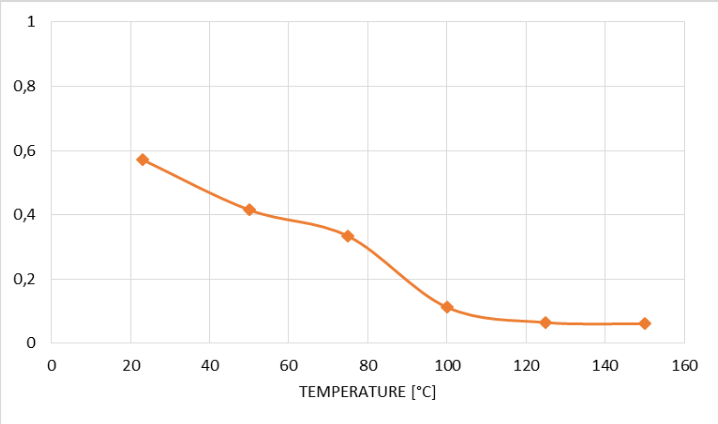

Figure 6. Experimental transverse modulus of elasticity.

5.3 Comparison of the theorical and exsperimental moduli of elasticity

It is important to mention that in the literature of origin of Figure 1, Vasielev and Morozov (2001), the value of the glass transition temperature of the material used wasn't presented, but it is possible to explain that it would be close to 200 ° C. The evident drop in the modulus of longitudinal elasticity E_1 observed when the temperature approaches this value has done this interpretation. Both of curves start at point 1 because the value of the real modulus of elasticity approached with accuracy the theoretical modulus of elasticity.

At the end of the impulse excitation technique, it was possible to present both modulus of elasticity in the same graph, Figure 7. By observing the two curves, it can be stated that the longitudinal modulus of elasticity suffered a softer fall outside the region of the Tg than the transverse modulus of elasticity, like the case described in the literature, Figure 1.

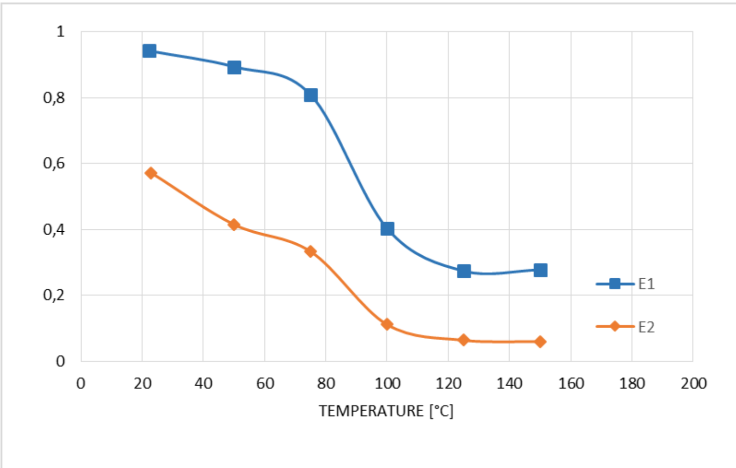

Figure 7. Experimental curves of the thermal effects in both moduli of elasticity.

6. CONCLUSIONS

As the composite materials can be included in several structural projects, there is the possibility that the environment in which it has been inserted presents adverse conditions that can cause the degradation of its mechanical rigidity. The present paper represents a preliminary study of the temperature in the longitudinal and transverse modulus of a carbon-epoxy laminate.

The thermal effects are more significant in the transverse modulus of elasticity, which the stiffness modulus is reduced 41.67 % from the room temperature to 75 °C. While for the longitudinal modulus of elasticity, the reduction was softer, 14.12 % of the original value in the same temperature range. The value 75 °C was chosen as a reference because after this point, the temperature would approach the glass transition temperature, when both stiffness modulus would suffer a sharper reduction caused by the degradation of the polymer.

The reason for the difference of modulus of elasticity decrease could have been the cut orientation of the specimen in the relation of the fiber material. For the longitudinal modulus of elasticity, the fiber material was parallel throughout the length of the specimen, so it had better withstand the thermal effect and showed a softer stiffness reduction. For the transverse modulus of elasticity, the fiber material was perpendicular to the specimen. Thus, the matrix material was responsible for supporting the possible thermal degradation and ended up not supporting with the same intensity as the fiber material, presenting a sudden reduction.

7. ACKNOWLEDGEMENTS

The authors would like to acknowledge the Univesidade Federal da Integração Latino-Americana (UNILA) for lending us the Sonelastic equipment to carry out this paper.

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