MECHANICAL AND ELECTRICAL CHARACTERIZATION OF GRAPHITE/EPOXY COMPOSITES

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Abstract. *There is a growing demand for polymeric composite materials in industrial applications, such as, for instance, bipolar plates for polymer electrolyte fuel cells in automobile applications. This kind of composite can replace with lower cost and greater efficiency metallic components. The basic requirement is that the composite must possess adequate electrical and mechanical properties for the particular application. Tensile and conductivity tests were performed in different composites. The goal of the present paper is to relate the macroscopic properties (elastic modulus, yield strength, tensile strength, fracture strength and electrical conductivity) of graphite/epoxy composites with the weight percentages of graphite powder. The study has demonstrated the potential of the use of epoxy/graphite powder composites and from the initial study, it is expected a semiconductor behavior for a graphite weight fraction around 50 wt.%.*

Keywords*: graphite/epoxy composites, mechanical properties, electrical properties.*

1. INTRODUCTION

Intrinsically (or inherently) conducting polymers (ICPs) are organic polymers that conduct electricity (Shirakawa *et all.* 1977, Liu *et all.* 2003). Such compounds may have metallic conductivity or can be semiconductors. Conductive polymers are generally not plastics, i.e., they are not thermoformable. But, like insulating polymers, they are organic materials. They can offer high electrical conductivity but do not show mechanical properties as other commercially used polymers do.

One way of classifying solid materials is according to the ease with which they conduct an electric current; within this classification scheme there are three groupings: conductors, semiconductors, and insulators. Metals are good conductors, typically having conductivities on the order of 10^7 $(\Omega\text{-m})^{-1}$. At the other extreme are materials with very low conductivities, ranging between 10^{-10} and 10^{-20} $(\Omega\text{-m})^{-1}$ these are electrical insulators. Materials with intermediate conductivities, generally from 10^{-6} to 10^4 $(\Omega\text{-m})^{-1}$ are termed semiconductors. (Calllister).

An interesting alternative to the ICPs may be the addition of conductive filler into non conducting polymers. Such composites combine low cost, reasonable conductivity, processability and good mechanical strength. One of the most promising applications of such kind of composite are in fuel cells (Lee *et all*. 2007).

From an economic and technical point of view flexible graphite is found to be one of the best candidates for the use as bipolar plate material for polymer electrolyte fuel cells. The bipolar plate cost is a considerable part of the cost of a polymer electrolyte fuel cell stack. Especially for automobile applications, it is necessary to strongly reduce this cost. Nevertheless, the application of polymer electrolyte fuel cell (PEMFC) to the electrical vehicles is still restricted by high material cost and complicated manufacturing process. A combination of a minimum mechanical strength with a reasonable conductivity is a basic requirement. Generally, carbon nanotubes, carbon blacks or carbon fibers are added to the matrix.

The present paper is a first step in a research program aiming at using graphite powder to enhance the electrical conductivity of the epoxy resin. The mechanical and electrical properties of the composite with different weight percentages (0, 5, 10 and 15 wt. %) of graphite powder have been investigated.

The goal is to study the effect of adding graphite powder in the epoxy matrix, analyzing both the mechanical behavior and electrical properties.

2. EXPERIMENTAL PROCEDURES

2.1 Material

Graphite/epoxy composites with respectively 5, 10 and 15 wt.% powder graphite were considered in the present study. Epoxy resin has several advantages, including exceptional combination properties such as hardness, chemical resistance, high heat distortion temperature, thermal stability and weather properties. Powder graphite is usually incorporated into this resin to increase electrical conductivity, both at ambient and high temperatures. The composites were manufactured in the Laboratory of Pipeline Testing LED-LMTA at the Universidade Federal do Rio the Janeiro (UFF) and the procedure to manufacture graphite/epoxy composites consists in mixing the graphite powder and the base resin, which consists in a diglycidyl ether bisphenol A, with a spatula for approximate 15 min. After homogenization, the aliphatic amina hardener was added to start the polymerization process. Then, the produced composite is pored into the mold. The tensile specimens were prepared according to ASTM D 638.

2.2 Mechanical Testing

Tensile tests were performed following the recommendations of ASTM D 638 Standard. A prescribed stroke speed of 5mm/min was adopted. These tests were conducted until the specimen fracture using a servo-electric test Universal machine (Shimadzu-100kN). Five specimens were tested for each fraction of powder graphite. The procedure adopted as a criterion for determining the Proportional limit establish as Proportional limit the value of stress to corresponded to the point where a straight line cuts the abscissa axis at 0.02% strain, tangent to stress-strain curves.

2.3 Conductivity Testing

The electrical conductivities of the different specimens were measured by capacitance(C) and dissipation energy factor(D) using model 4284A LCR meter (Inductance (L), Capacitance (C), and Resistance (R)) 20Hz to 1MHz, in this work we used 1000Hz. The device under test (DUT) is subjected to an AC voltage source. The meter detects the voltage over, and the current through the DUT. From the ratio of these the meter can determine the magnitude of the impedance. The phase angle between the voltage and current is also detected and between that and the impedance magnitude the DUT can be represented as an L and R or a C and R. The meter must assume either a parallel or a series model for these two elements (Agilent. 2000).

Capacitance is measured in Farads and the basic definition of a Farad is a capacitor of a size such that one Ampere flowing into it for one Second will change the terminal voltage by one Volt. Capacitance is that property of a system of conductors and dielectrics which permits the storage of electricity when potential difference exists between the conductors. Its value is expressed as the ratio of a quantity of electricity to a potential difference. A capacitance value is always positive.(Sens and Ueti, 2009)

Dissipation Factor is the tangent of the loss angle of the insulating material. A measure of the power factor (or losses) of a capacitor, given as D.F. = 6.28 fRC X 100%, where R is the equivalent series resistance (ESR) of the capacitor, f is the frequency (Hz.), and C is capacitance (Farads). Dissipation Factor varies with frequency and temperature. (Eroglu, 2010)

The conductivity tests were performed at CEPEL (Centro de Pesquisas de Energia Elétrica)-Eletrobrás.

3. EXPERIMENTAL RESULTS AND DISCUSSION

3.1 Tensile Tests

The experimental stress-strain curves for different graphite fractions are shown in fig. 1 and 2. In Table 1 are presented the average elastic modulus, the proportional limit, the ultimate tensile strength and the tensile fracture strength.

It can be seen that comparing the tensile tests response of the Resin epoxy and of the Composites (resin epoxy with powder graphite) that there was a variation with the powder graphite addictions, the fracture strength was reduced and the ductility too, but there is a small increase in the elastic modulus. It can observe that between 5, 10 and 15 wt.% of graphite fraction, the mechanical properties variation is smaller, it means, the mechanical tests results showed that the graphite addition decrease the strength limit, but the difference behavior between the composites with the graphite fraction are smaller, however there are a small increase in the stiffness until 1% of strain. The results show also with the graphite added a reduction of the material stiffness occurs when the strain exceeds a value of approximately 2.0%, Fig.1.

The yield strength is not well defined in this composite, it don’t have an evident transition between elastic and plastic region both before and after graphite added.

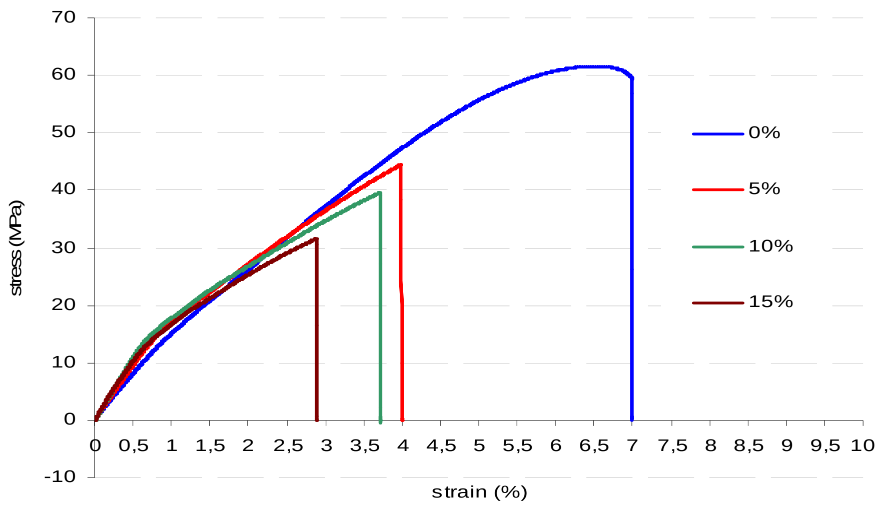

Figure 1. Tensile stress-strain curves for 0, 5, 10 and 15% wt. graphite fraction until fracture.

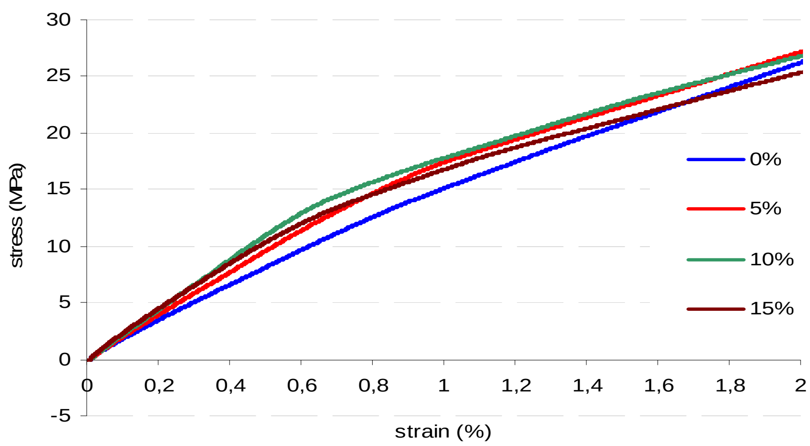

Figure 2. Tensile stress-strain curves for 0, 5, 10 and 15% wt. graphite fraction until 2% of strain.

In general, to determine Yield stress in polymeric materials is not obvious. The plateau region of instability with constant stress does not appear in the tensile tests curves. Moreover, using the stress value associated with 0.2% strain, usually applied to metals is unrealistic for the polymers. Thus, it was adopted as a criterion for determining the Proportional limit, the procedure in which establish as Proportional limit the value of stress to corresponded to the point where a straight line cuts the abscissa axis at 0.02% strain, tangent to stress-strain curves.

Table 1. Experimental results for tensile composite properties.
Average results of 5 specimens.

Properties	0 wt.% Graphite	5 wt.% graphite	10 wt.% graphite	15 wt.% graphite
Elastic Modulus (GPa)(1)	1.6 ±0.3	2.0 ±0.2	2.2 ±0.3	2.2 ±0.2
Proportional limit (MPa)(1)	6.0 ±1.0	7.0 ±0.8	10.0 ± 0.5	8.3 ±0.5
Ultimate Tensile Strength (MPa)(1)	61.6 ±1.9	44.5 ±1.1	39.2 ±1.2	31.5 ±0.9
Tensile Fracture Strength (MPa)(1)	59.3 ±1.2	44.5 ±1.1	39.2 ±1.2	31.5 ±0.9

(1): measured at room temperature

The next Figures 3 and 4 shows the experimental Parameters results with the trend line and curve equation. Fig. 3 shows that Ultimate Tensile Strength decreases with graphite increase, nearly, in the linear way; it shows the mechanical behavior trend with the powder graphite addition.

The Elastic Modulus increased lightly with graphite, this increase was around 20% with 5% of graphite, as shown in table 1 and in the Fig.4, tending to a constant value, in the polynomial way, with this equation is possible also forecast the material behavior.

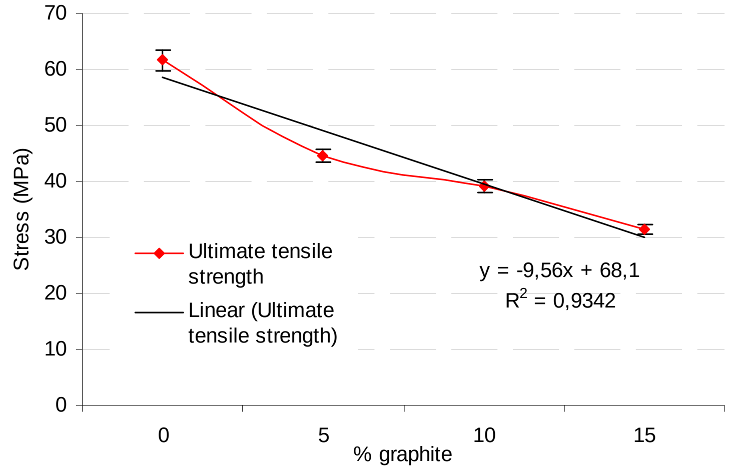

Figure 3. Ultimate Tensile Strength versus % graphite, with the linear trend line and curve equation.

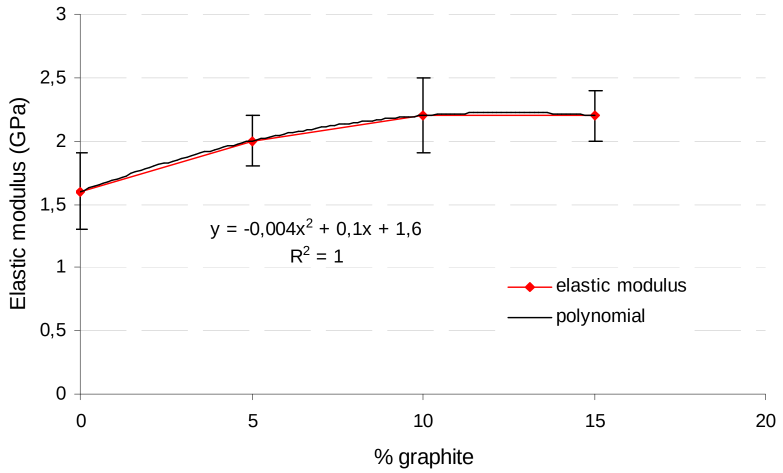

Figure 4. Elastic Modulus (GPa) versus % graphite, with the polynomial trend line and curve equation.

3.2 Conductivity Test

The electrical conductivity tests results, for different graphite fractions, are presented in Tab. 2 and Fig.5, the results average. It can be observed that the capacitance and the product capacitance(C) x "Energy dissipation factor"(D) (C.D) increases with the graphite fraction, showing that there is influence of the powder graphite, modifying the conductivity of the resin, it means, decreasing resistivity. The curve is adjusted to a polynomial curve showing the trend of the conductivity to rise with the graphite addition and it can help forecast that electrical behavior is not linear, but will increase faster in high graphite fraction.

Table 2. Experimental results of the composite conductivity properties.
Average results of 5 specimens.

% de grafite	C= Capacitance(1) (pF)	D=Energy Dissipation Factor(1) (%)	C.D (pF. %)
0	0.159±0,003	0.125±0,001	0.01988±0.000534
5	0.190±0.002	0.105±0.0003	0.01995±0.000267
10	0.280±0.004	0.081±0.001	0.02268±0.000604
15	0.400±0.005	0.067±0.001	0.02660±0.000735

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pF = picofarad = 10^{-12}	C.D= capacitance x dissipation factor

(1): Measured at room temperature

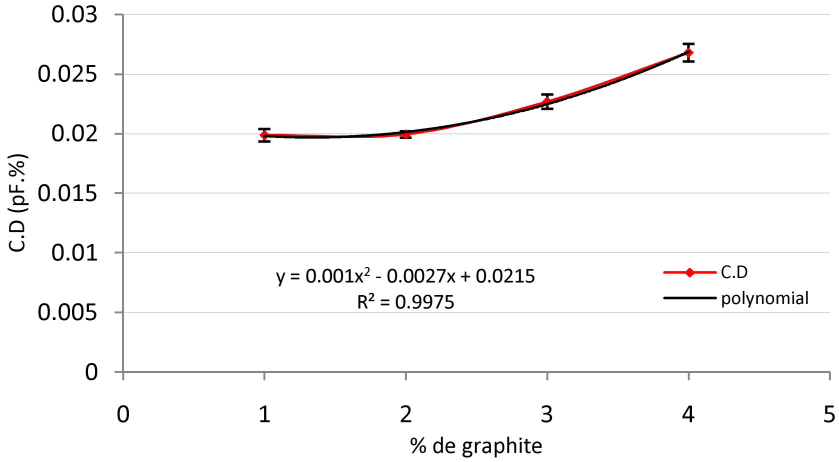

Figure 5. The product of (C) Capacitance and (D) Energy dissipation factor versus % graphite curves, with the polynomial trend line and curve equation.

4. CONCLUSION

This study is a first step of a research program aiming at using graphite powder to enhance the electrical conductivity of the epoxy resin. The mechanical and electrical properties of the composite with different weight percentages (0, 5, 10 and 15 wt.%) of graphite powder have been investigated. Although these percentages are still not high enough to produce a semiconductor composite, the study has demonstrated the potential of the use of epoxy/graphite powder composites. Reasonably small quantity addition of graphite powder can modify the mechanical and electrical behavior of a polymer matrix. The main challenge is that the mechanical strength of the epoxy matrix strongly decreases with the initial addiction of graphite, but the conductivity increases very slowly. The conductivity of a semiconductor is in a range of 10^{-4} to 10 S/cm. From the initial study, it is expected a semiconductor behavior for a graphite weight fraction around 50 wt.% .

5. ACKNOWLEDGEMENTS

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