Evaluation of Electrical, Mechanical and Wear Behavior of Laminated Epoxy/Carbon Fiber Composite with Different Fillers

Gean Vitor Salmoria[1*], Loise Silva[2], Rodrigo Acácio Paggi[3]

Federal University of Santa Catarina – Department of Mechanical Engineering, NIMMA Laboratory
*Corresponding Author

Received: 01 April 2022/ Revised: 11 April 2022/ Accepted: 18 April 2022/ Published: 30-04-2022

Abstract— *Epoxy resin reinforced with carbon fibers are widely used in applications where requirements of lightness and high mechanical strength are needed. Composites containing fillers with different properties can improve important characteristics of components. In aerospace applications, the use of low removal rate due to the impact of suspense particles must be satisfied in order to reduce maintenance costs of components. Since carbon fibers have low electrical resistivity and are in the range of semiconductors, these composites can be used as radar absorbing materials (RAM). The addition of conductive fillers may increase this intrinsic property of the composite, and also improve the mechanical and wear properties of the laminate. Results showed that the addition of micronized graphite (MG), multi-walled carbon nanotubes (MWCNTs) and short steel fibers (SSF) reduce the weight loss on abrasion test, but reduce values of flexural module (except for the addition of 5% of MG). In terms of electrical properties, fillers showed tendency to increase the electrical conductivity mainly for reduce fraction of fillers added, showing that higher contents can produce a non-homogeneous structure since that the mechanical and wear properties were affect in the same order.*

Keywords— Composites, carbon fiber, wear resistance, mechanical properties, conductive filler.

I. INTRODUCTION

Manufacturing techniques and development of composites have attracted increasing interest mainly due to its applications in the aerospace industry, such as military or civil aviation. Composites based on epoxy resin reinforced with directional carbon fiber had presented excellent corrosion and mechanical properties (mainly the high specific strength), allow greater freedom in order to obtain structural parts and are still promising to reduce maintenance and operation costs of aircraft. According to Librantz (2006) [1] the substitution of aluminum for polymer composites can result in a reduction of up to 30% on the final weight and 25% in acquisition costs of structural parts.

Use of fillers can improve mechanical strength and abrasive wear. A frequent question when reinforcing or adding fillers in composite materials is on the improvement of other interesting properties such as mechanical and wear abrasion strength, essential for building aircraft. Very pronounced effect on aircraft crashes due to dust particles, sand, ice that generate significant cost in terms of maintaining the areas exposed to these events. Although the carbon fibers possess sliding characteristics, their structure is different from carbon black or graphite. In this case, the incorporation of these materials on composite laminate can improve the wear characteristics around the failure mechanisms, like a fiber thinning, fiber-matrix debonding and fiber pull-out and low cycle fatigue [2]. The author also refer that the carbon fiber provides less wear resistance and higher coefficient of friction than glass fiber. According to Takeichi (2008)[3] the reinforcement with carbon black decreases linearly the friction coefficient of PTFE composites. Nanometric particles (around 25 to 38nm) were used in this work. The 10% mass of carbon black was the best concentration for the highest wear resistance.

It is the knowledge that the process variables affect the wear and friction of samples. Wan et al (2006) [4] evaluated the wear rate and coefficient of friction of carbon fabric reinforced epoxy resin composites. The results showed that fiber volume fraction and testing conditions (load and velocity) affected the wear more significantly than the friction. It was also found that fiber–matrix bonding had an impact on the friction and wear of the 3-D composites. Furthermore, the specific wear rate decreased with the increase in the product load and velocity. On the other hand, the addition of different kinds of fillers with sliding characteristics is less studied by the authors. Besides providing slip characteristics, the added particles may have the role of retaining wear or tearing of the material due to its greater mechanical strength when compared to the polymer matrix.

Given the characteristics of conductive carbon fibers used in composites, these materials are also intended for electromagnetic shielding. In general, this class of materials and filers used for this purpose are known as radar absorbing materials (RAM), which promote the conversion of electromagnetic radiation by heat or by reflection [5]. There are a wide variety of electronic devices that require protection against electromagnetic waves provided by telecommunications companies, for example. The characteristics of higher electrical conductivity in the primary structural parts of an airplane are destined for dissipation of electrical discharges received by the aircraft. Can be also useful to return of electrical current from the generator. In this sense, these devices that perform essential functions during flight need to be isolated by conductive boxes which dissipate the incident waves or even omitted [6]. The absorption is given basically by two distinct categories of absorbers: the dielectric and magnetic. Conductive fillers are used to control only the complex permittivity and can be added in smaller proportions compared with the magnetically fillers absorbing [7] because the characteristics of lower density.

The carbon fibers are promising in this kind of application; they reflect the incident waves due to its high electrical conductivity [8]. Among the fillers with dielectrical absorption, the addition of carbon particles, graphite or metal sprayed into polymer matrices are the materials most commonly used for this purpose [5]. In the work of Kim et al (2009)[7], composite laminates of fiberglass sealed by epoxy resin with addition of carbon nanotubes have been suggested as suitable for absorption of electromagnetic waves. However, a layer of composite carbon fiber/carbon nanotubes intending in this case the reflection of the incident waves. For the structural and physical characteristics, the carbon nanotubes can reach the percolation threshold of the material with more modest concentrations which is interesting due to lower variation change of other material properties, especially mechanical.

The use of alternative fillers such as short steel fibers can be beneficial to composite in terms of the high conductivity observed for most metals and infer better mechanical properties with relative cost often less than some synthetic fillers. Some authors like Mamunya et al (2002) [9] evaluated thermal and electrical properties of composites with cooper and nickel powders and obtained improvement on both properties. Studies with low cost and no-powder fillers like short steel fiber are still poor and become interesting since those benefits can be very useful. This work aims to investigate the maintenance of the mechanical properties and improved electrical properties and wear resistance for composite laminates based on carbon fiber and epoxy resin with the addition of conductive fillers, including: micronized graphite (MG), multi-walled carbon nanotubes (MWCNTs) and short steel fibers (SSF).

II. MATERIALS AND METHODS

2.1 Materials

The epoxy resin used in this study was EPOCAST 52-A of Huntsman. Carbon fibers of type "plain wave" in the form of woven oriented alternately, with 0 and 900, respectively. The average thickness of the blanket was 0.095 mm.

In this study we used natural crystalline micronized graphite (MG) of Nacional de Grafite company, with diameter of 5 ± 2 micrometers and purity level exceeding 95%. Multi-walled carbon nanotubes (MWCNTs) from MER corp. company with an average diameter of 140nm and length of 7μm. The short steel fibers (SSF) were provided by Bombril company with aspect ratio of approximately 10.

2.2 Sample fabrication

Samples of carbon fiber and epoxy resin were obtained by manual lamination with final dimensions of 100 x 100 mm. The same orientation of the fibers was maintained during the overlap of all layers. To warranty precise control to physical dimensions, the thickness was maintained at a value close to 1 mm with the aid of a spacer obtained through techniques of rapid prototyping. Overlapping layers occurred on a hard flat surface previously covered with a layer of mold release agent in order to facilitate the extraction of the part after cure. The proportion of resin/hardener used was 2:1, mixed with speed and time previously determined. The conductive fillers were firstly mixed on the hardener because the lower viscosity. Figure 1 shows the experimental procedure of mixture.

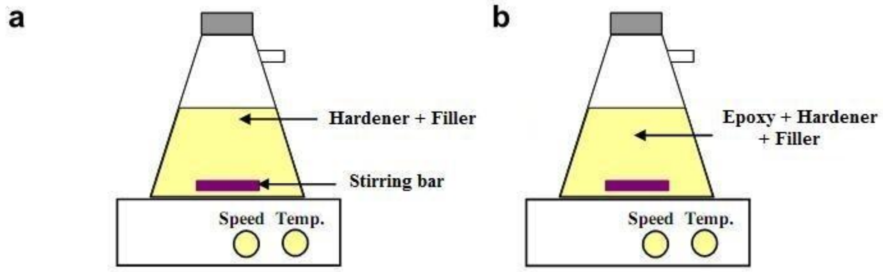

FIGURE 1: Manufacturing process of composites based on carbon fabric, epoxy resin and conductive fillers, showing the prior mixing with hardener (a) and pos-mixing adding the resin (b) (Adaptated from Kim, 2009)[10].

Samples were obtained with five layers of woven carbon fiber soaked with about 1.85 g of resin for each layer. The procedure of cure was conducted in two steps: 1) 24 hours at room temperature under the action of a standard weight and 2) Thermal curing at 60, 85 and 155 °C for one hour at each level.

2.3 Electrical properties

To obtain the electrical conductivity values of samples of epoxy resin with and without fillers reinforced with bi-directional carbon fiber was used a two-probe test. With this method was possible to obtain a direct measure of electrical resistivity knowing the voltage (V) and current (A) that flows through the sample under effect of a dc electrical field. It was necessary in this case the knowledge of geometric dimensions in order to warranty safe results [11]. To measures was used an electrometer Keithley Instruments Model 6517, with a source current DC voltage source Keithley Model 224.

Voltages (V) measures were recorded by applying an electrical current that could vary in the range of nano to milliamps. The dimensions (length, width and thickness) were used to calculate the initial resistivity and then the electrical conductivity of the material. The dimensions set for the test were squares with 10 mm side and a thickness close to 1 mm.

Figure 2 shows a simplified sketch of measuring method used for electrical conductivity acquisition. The current is applied across the sample and the voltage is detected by the equipment. A low pressure was applied on the extremes in order to warranty a good contact between components.

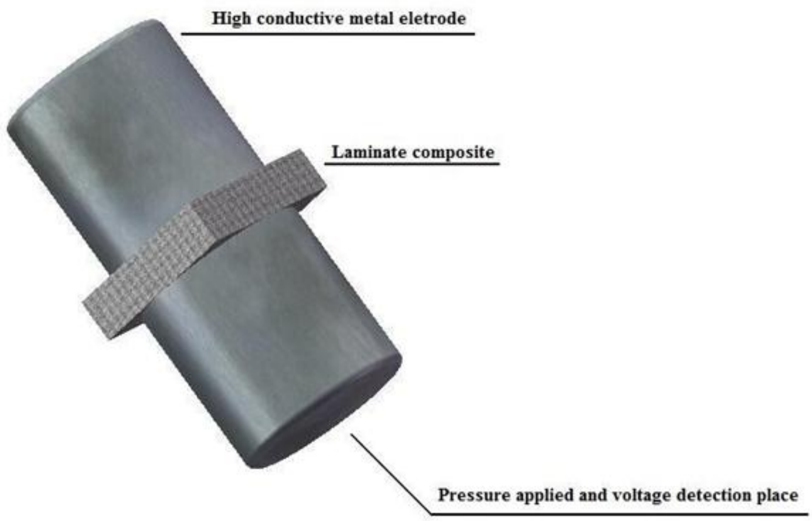

FIGURE 1: Sketch of two probe measure method

The values of electrical conductivity (σ) were obtained using firstly the second law of Ohm represented by electrical resistivity (ρ) (Ω.cm) of material calculated using the equation 1. This equation considers that electrical characteristics are dependent of geometric dimensions of sample. The σ values discussed in the text were obtained by the inverse of resistivity measures (2) given in S.cm-1.

$$\rho = RA/L \quad (1)$$

Where R is the resistance (Ω), A is the area of section on direction of current (cm2) and L is the length (cm) between two points analyzed.

$$\sigma = 1/\rho \quad (2)$$

2.4 Mechanical tests and wear resistance

Mechanical properties of laminated composites with and without fillers were tested in bending mode using a DMA Q800 equipment from TA instruments. Flexural modulus was the main property analyzed. The tests were conducted at controlled force mode with single cantilever clamp, the loading rate used was 2N/min at room temperature.

The abrasive wear strength of each composition was evaluated using the equipment model TABER 3155. The mass loss of the material was evaluated of each 1000 cycles, with a total of 10,000 cycles at the end of the test.

2.5 Morphology

The main objectives of the morphological analysis were the observation of the fracture and the adhesion between fiber and resin, therefore the test pieces were made by the cryogenic fracture of the composite material. The method used to obtain the test pieces was the immersion of the material in liquid nitrogen for 30 seconds, so the material would become considerably brittle, and consecutive application of bending solicitation until the complete fracture of the material.

Afterwards, morphology observations were made on the JEOL JSM-6390LV Scanning Electron Microscope, with magnifications between 70 and 600X, at the 11kV accelerating voltage. The fracture surfaces of the test pieces were observed to analyze the type and reason of fracture, and the regions where were located pull out of the fiber, to analyze the adhesion between the materials.

III. RESULTS AND DISCUSSION

3.1 Electrical behavior

Figure 3 provides a comparison between the electrical conductivities for the composites with different fillers evaluated in this work. At higher concentrations, the conductivity values decreased in all cases. The addition of MG was more favorable in terms of improvements in the electrical characteristics when added small concentrations of filler. In the case of MWCNTs, all fractions had reduced levels compared to composite without adding any filler. Results obtained by Oh et al. (2004) [12] before manufacturing and testing epoxy composites with the introduction of many contents of carbon black (5 to 20%wt) also identified that addiction of lower contents of fillers is responsible for better mechanical and electrical properties to the construction of a radar absorbing structure (RAS). The content indicated was 5 and 7% wt, performing a similar result obtained in this work considering the similarity of fillers.

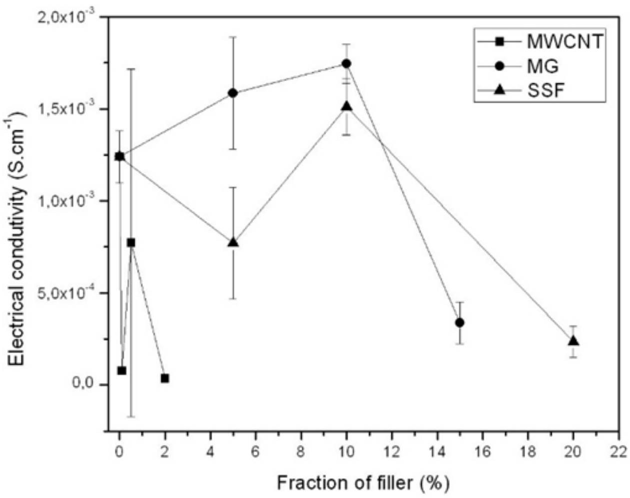

FIGURE 3: Electrical conductivity values for composites with different fillers and contents.

Graphite and carbon black are important fillers when conductive or electromagnetic/radio frequency shielding applications are necessary. Azim et al (2006) [13] evaluated the addition of these fillers to obtain organic-based coatings for referred applications. The authors determined that high quantities of fillers are necessary to obtain minimum resistivity in order of 2x10-5 ΩM. The amount indicated was approximately 40%wt. The results determined in this work revealed that conductivities values are in the semiconducting range and the order of magnitude was not significantly modified when comparing the fillers added. In this case, the carbon fibers can be providing the major conducting capacity. Since that the content of MG added is considerably very low, the behavior of improve electrical properties was proved and major changes can be observed just with introduction of bigger contents of conductive fillers, especially MG.

3.2 Mechanical properties

Figure 4 shows a comparison for the composites with MG added in different proportions. The curves have remained with very low linear distortion due to the rigidity of the material. With 5% by weight of MG has been verified an increase in material stiffness for the material without filler. With the introduction of 10 and 15% in weight it was observed a small reduction in the values of flexural modulus.

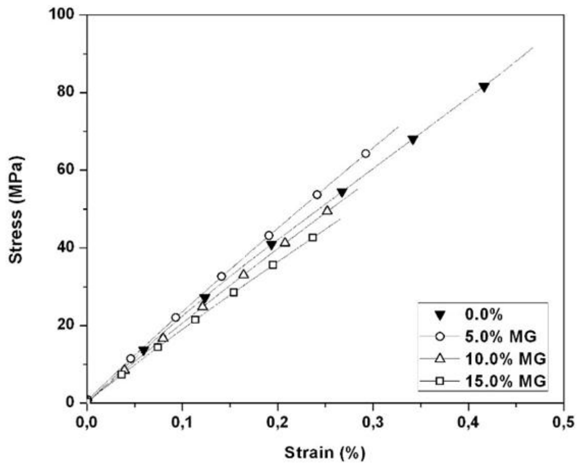

FIGURE 4: Stress X strain curves for different fractions of micronized graphite (MG) and material without conductive fillers.

Figure 5 shows a comparison for the composites with MWCNTs added in different proportions. The addition of growing MWCNTs contents in the laminate composite provided a gradual reduction in the values of flexural modulus. The smaller size characteristic of MWCNTs and consequently the difficult to disperse this material consist on a possible reason of drop on mechanical properties because of the agglomerate’s formation. Bigger contents of MWCNTs are subject more easily to this effect. Zhou et. al (2008) [14] obtained an improvement of 15-20% with the addition of 2%wt of CNf. Yet, increments were acquired by a high-intensity ultrasonic liquid processor to disperse the fillers homogeneously. The specimens were manufactured in a vacuum-assisted resin transfer molding consisting of a more favorable processing to obtain composites without structural defects.

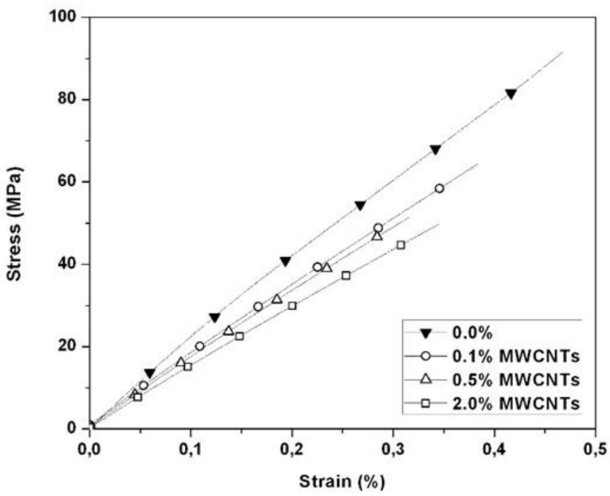

FIGURE 5: Stress X strain curves for different fractions of MWCNTs and material without conductive fillers.

Figure 6 presents a comparison for the composite with added SSF in different proportions. With the incorporation of 5% by weight of SSF the curve and values of flexural modulus remained virtually unchanged, on the other hand the addition of 10 and 20% caused a reduction in stiffness of the material. The difficult to mix and disperse high quantities of filler is a responsible fact to cause this behavior.

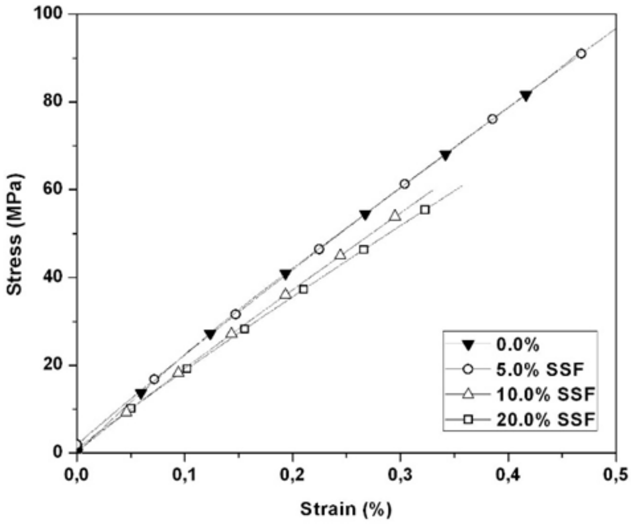

FIGURE 6: Stress X strain curves for different fractions of short steel fiber (SSF) and material without conductive fillers.

Figure 7 presents a compilation of flexural modulus values for all the compositions evaluated. In general, only the addition of 5%wt of MG produced an increase in the values of the module. Another interesting feature was the correlation of increase in the percentage of fillers with the gradual reduction of material stiffness. One of the likely causes for this effect was the difficulty of dispersion found in these cases, which can cause the formation of clusters that reduce the mechanical properties by constituting themselves centers of defects generator.

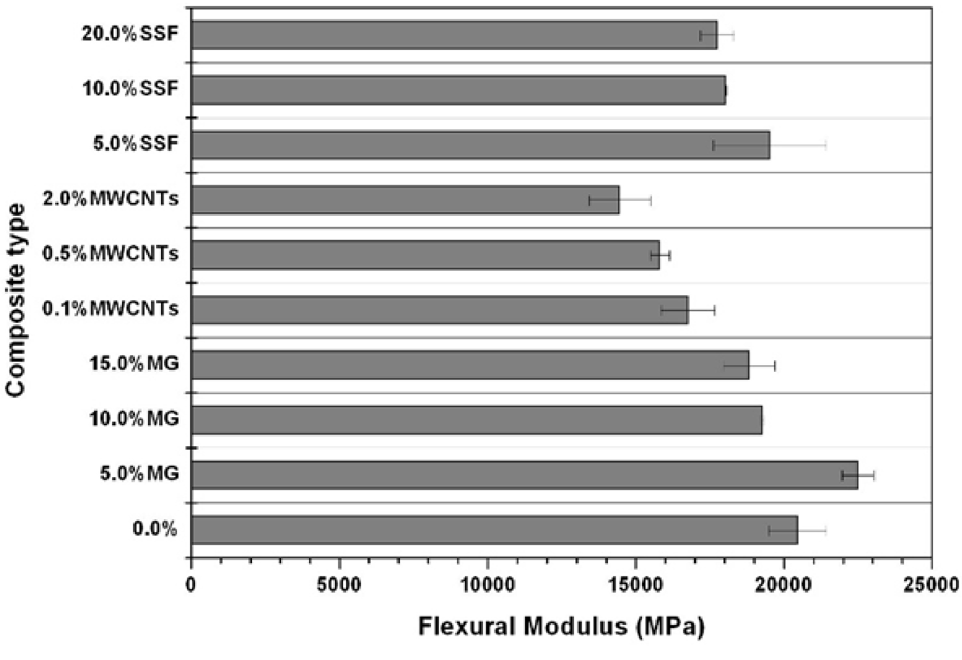

FIGURE 7: Summary of flexural modulus values obtained for composites with and without addiction of conductive fillers.

There is a concern around the authors about the difficult on introduce and mainly disperse the nano-scaled fillers. Work produced by Fiedler (2006) [15] evaluated the characteristics and properties generated by filler of different sizes given more attention for CNTs as nano fillers in polymers, but also the limitations and challenges one has to face dealing with nanoparticles. A necessity of a proper dispersion as well as a possibility of orientating CNTs was shown, in order to attain the best possible properties. These limitations were verified in this work since that the MWCNTs do not presented their expected potential. The same incorporation method practiced for all fillers show that the nano fillers are more suitable to agglomeration comparing to the others.

3.3 Morphology

Figure 8 shows both laminate carbon fiber composite without filler incorporation and the same composite with different fractions of MG. The addition of MG did not change the level of porosity on the resin matrix, showing that the mixture step was successfully developed for all contents. Considering that the specimens were laminated without vacuum-assisted system, some residual closures were expected, as verified on the SEM analysis. It was clearly verified the layered structure of pure resin and resin plus carbon fibers, indicating that the resin flow during the curing step created an intermediate layer located between two fibers woven. On higher magnifications it is possible to see the pull-out of carbon fibers, representing a satisfactory moliability by the resin. It is also revealed places where delamination was more pronounced, like on the composite with 5.0% wt of MG (fig. 8d). By analysis of the fracture behavior, the start of crack was observed on fig. 8b, evidencing that the contact place between fiber and resin is critical for failures.

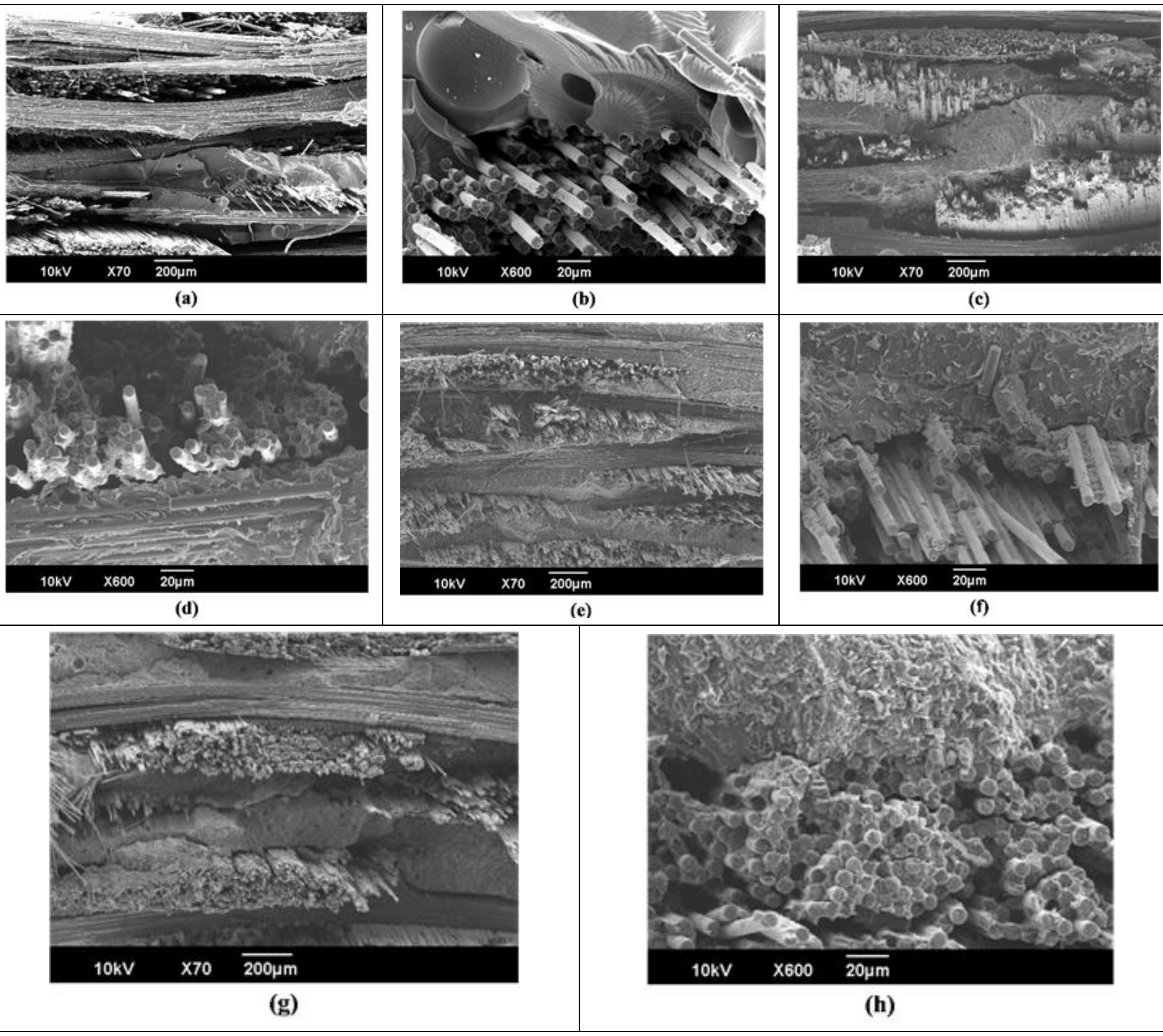

FIGURE 2: Microstructure comparison between laminate (CF) without fibers (a and b) and with addiction of 5.0%wt MG (c and d), 10.0%wt MG (e and f) and 15.0%wt MG (g and h) under different magnifications

Figure 9 shows the comparison between laminate carbon fiber composite without filler incorporation and the same composite with different fractions of MWCNTs. The layered structure obtained presented more uniformity when added 0.1 and 0.5%wt of MWCNTs, in contrast with the composites containing 2.0% of MWCNTs, which shows the difficulty in the mixture step. The delamination was more pronounced with higher fractions of filler, both in between matrix-fiber and fiber-fiber. The porosity distribution was practically unaltered.

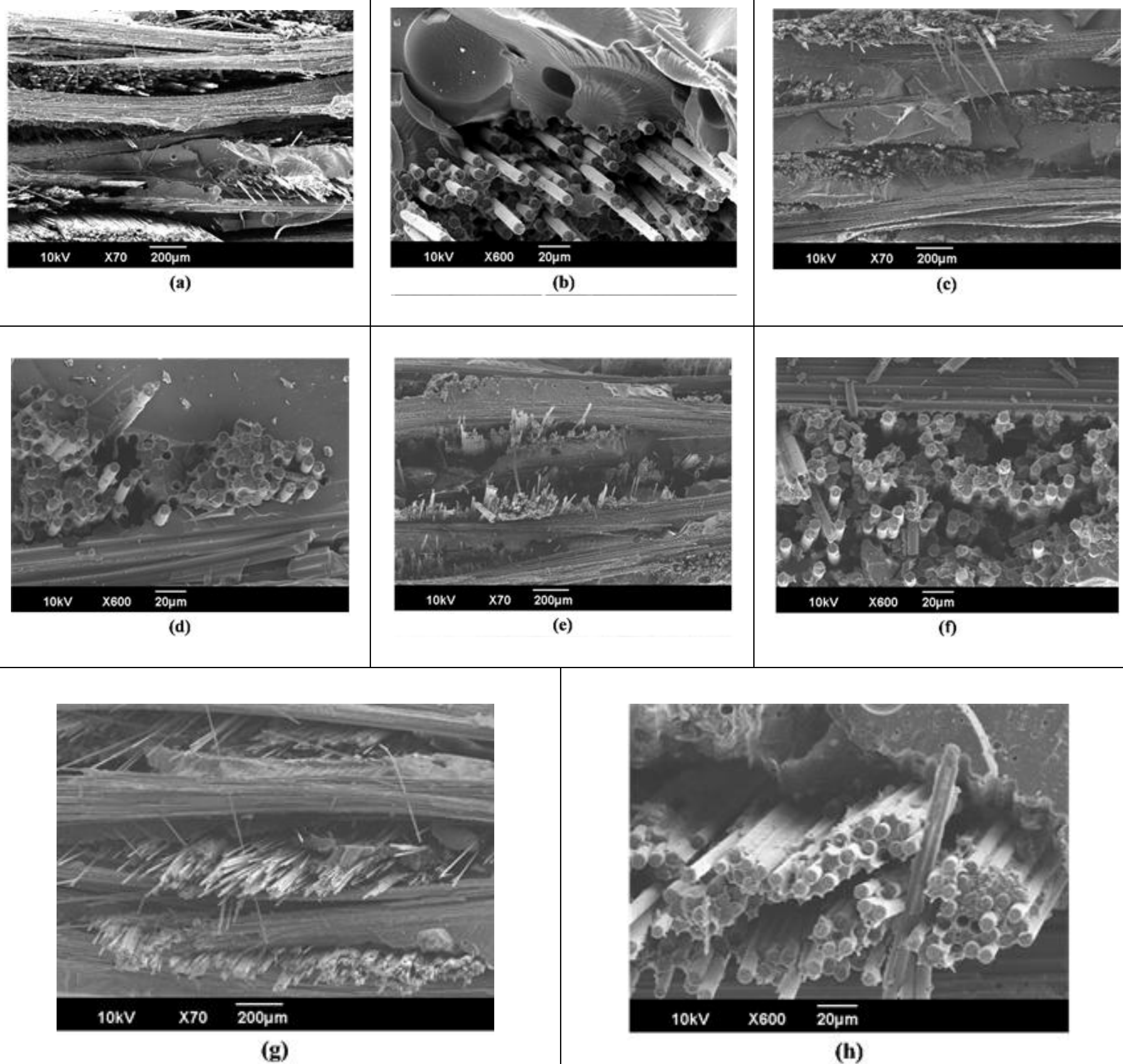

FIGURE 3: Microstructure comparison between laminate (CF) without fibers (a and b) and with addiction of 0.1%wt MWCNTs (c and d), 0.5%wt MWCNTs (e and f) and 2.0%wt MWCNTs (g and h) under different magnifications

Figure 10 compares laminate carbon fiber composite without filler incorporation and the same composite with different fractions of SSF. It was not verified great changes on uniformity and porosity across the sectioned specimens. Increasing the fractions of SFF added on the matrix, the adhesion between fibers and resin became poor, mainly with the incorporation of 20.0% wt SSF, shown in fig. 10h.

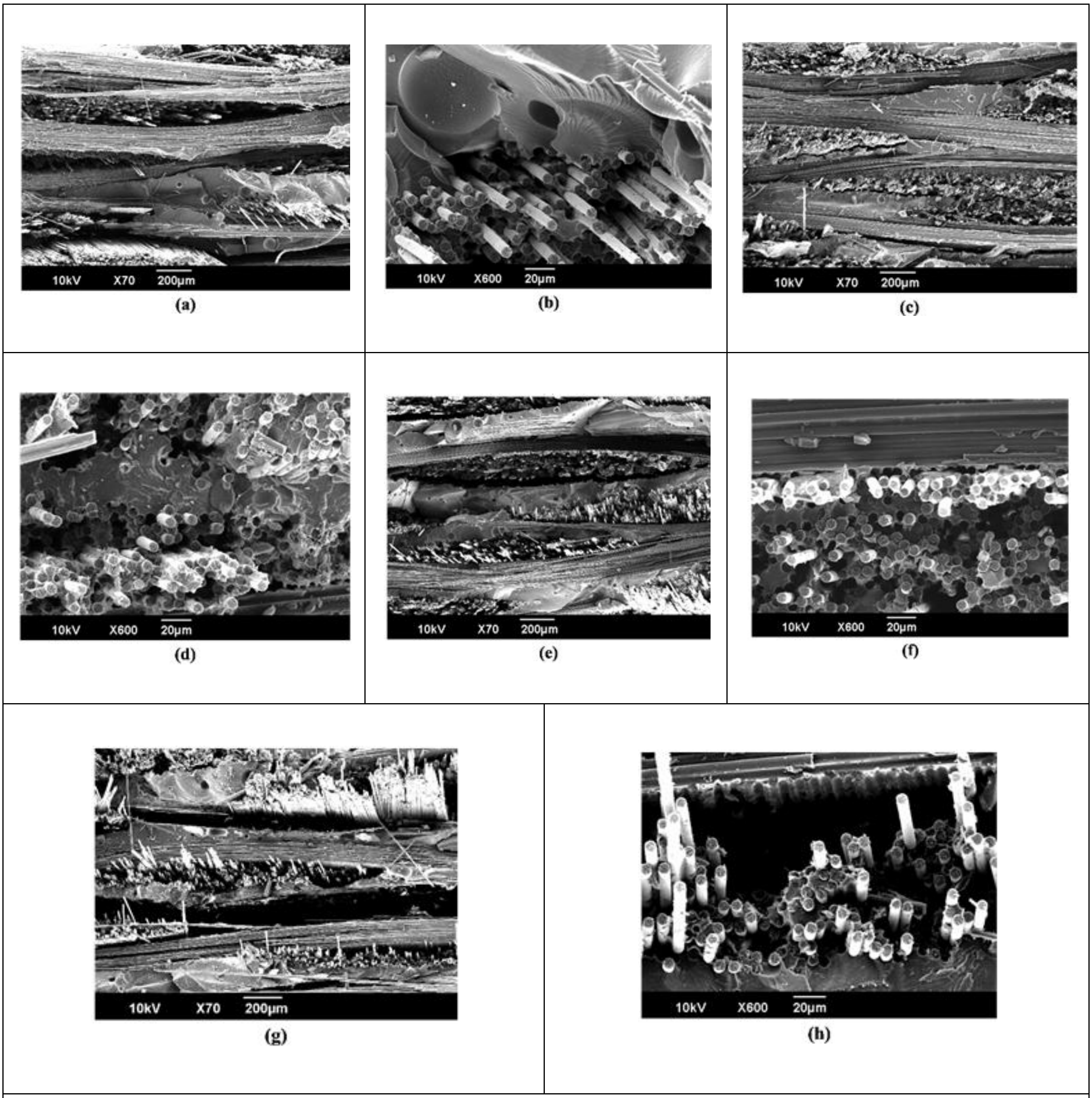

FIGURE 10: Microstructure comparison between laminate (CF) without fibers (a and b) and with addiction of 5.0%wt SSF (c and d), 10.0%wt SSF (e and f) and 20.0%wt SSF (g and h) under different magnifications

3.4 Wear behavior

Figure 11 shows the respective weight losses for the composites with and without addition of MG. For all ratios of fillers, the weight loss at the end of 10,000 cycles was approximately 0.005 g, amount considerably less than their loss to the composite without fillers, which was about 0.030 g. The crystalline structure, characteristic of graphite, that present character of slip and therefore self-lubricating, can be the explanation for the reduction in weight loss. Since the wear behavior has not changed with the increase in the percentage of the fillers, it appears that the addition of 5% by weight already promotes a saturation property. It was possible to notice that the change of weight loss behavior was verified before 3,000 cycles, indicating that the initial time is responsible for accommodating the wheels over the sample and removing the residual mold release agent. This phenomenon was observed in all experiments realized.

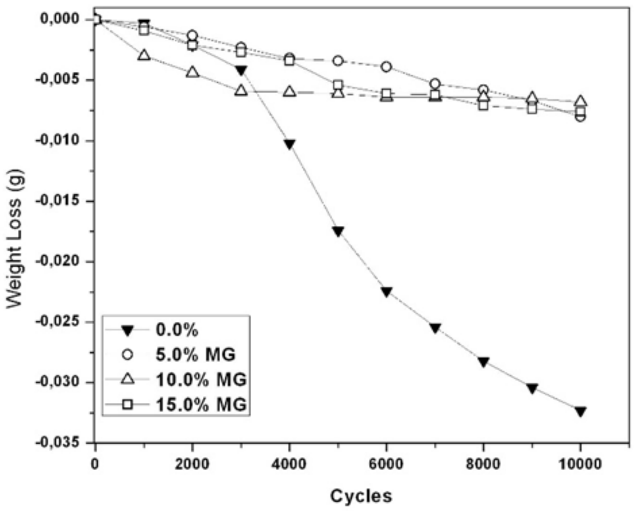

FIGURE 4: Comparison of mass loss for different concentrations of micronized graphite (MG) in relation to the composite without filler

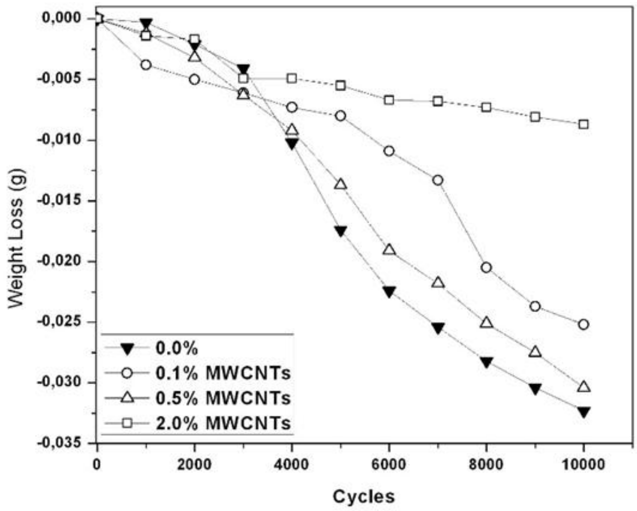

FIGURE 5: Comparison of mass loss for different concentrations of MWCNTs in relation to the composite without filler

Figure 12 shows the corresponding weight loss for the composites with and without the addition of MWCNTs. The loss with addition of 0.1 and 0.5% by weight does not change significantly the values of weight loss comparing with the composite without added charges. Only with the addition of 2.0% by weight, the values were close to those obtained with the 5.0% MG addition with maximum loss of 0.005 g. The crystalline nature of MWCNTs can explain the results if compared with addiction of MG.

Figure 13 shows the corresponding weight loss for the composites with and without addition of SSF. The addition of 5% of SSF reduces significantly the weight loss, from 0.030 to about 0.010 g. Only at 10 and 20% weight the losses were near the samples with addition of 5.0%MG.

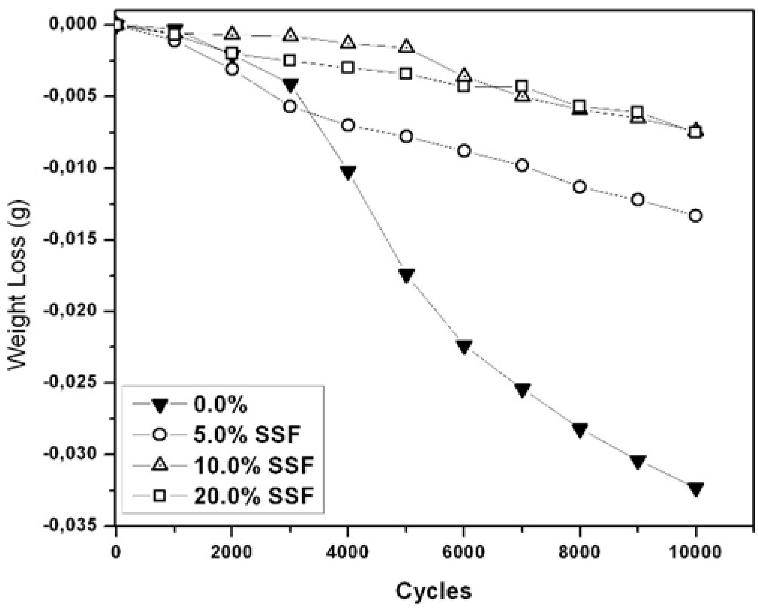

FIGURE 13: Comparison of mass loss for different concentrations of short steel fiber (SSF) in relation to the composite without filler.

The general analysis of the results of wear strength and their weight loss, the minimum values of 0.005 g were obtained with different fillers and concentrations, as follows: 10 and 20% of SSF, 2.0% of MWCNTs and from 5.0% of MG. In this sense the graphite has better wear characteristics with lower amounts of filler related to the cost of material.

Research developed by Chen et al (2007) [16] studied different mixture methods and surface treatments on carbon nanotubes. Among other results identified, acid treatment and coupling agent are not necessary because the as-received MWCNTs are already responsible for good results for tribological properties. In this case, the tribological results verified in this work are not correlated with the dispersion level of MWCNTs. SSF and MG generated lower loss weight because of structural or physical properties.

IV. CONCLUSION

Considering the effect of adding fillers for improving mechanical and electrical properties, the percentage of 5%wt of MG composite produced the best combined properties. In all three cases, the addition of high concentrations of filler caused a decrease in electrical conductivity and flexural modulus values, indicating a poor dispersion in the resin matrix and likely increasing the distance between the layers of carbon fiber, expected like most responsible for conduction. The percentage of MWCNTs evaluated at this work did not present a significant change in the electrical conductivity.

Morphological analyzes showed that the increasing of filler fraction made the adhesion more difficult, resulting in problems of delamination. The fillers added cannot be seen by electron scanning microscopic, attributing a successfully mixture method, therefore the bundles of fillers were not verified on the cross section of cryogenic fractured specimens.

The wear strength for weight loss had similar results for the addition of MG and SSF, with a mean value of 0.005 g. With the addition of MWCNTs, occurred a larger loss of mass of the order of 0.030 g. The difference in the chemical structure between the graphite and MWCNTs may explain the changes on properties analyzed. The MG material has sliding characteristics and MWCNTs, with their crystalline structure, does not have functionally slip planes.

ACKNOWLEDGEMENTS

The authors would like to thank the Brazilian Space Agency (AEB) and CNPq for the financial support.

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