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EFFECT OF GRAPHENE CONTENT ON THE MECHANICAL PROPERTIES OF GRAPHENE REINFORCED ALUMINUM MATRIX COMPOSITES 27TH COBEM

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Abstract. *The objective of this work is to investigate the effects of the graphene content on the mechanical properties of graphene reinforced aluminum matrix composites. Samples with five distinct volume fractions of graphene reinforcement are considered: 0.0% (pure aluminum), 0.3%, 0.5%, 0.7% and 1%. The composite specimens are produced using powder metallurgy, where the graphene is mixed with 99.5% wt. purity aluminum powder using a vortex mixer. The composite samples are compacted in a mold producing cylindrical specimens, with dimensions of 10mm in diameter by 10mm in height, which are taken to furnace for sintering. The microstructure of the composite material is analyzed using a scanning electron microscope (SEM), and the mechanical characterization of the composite material is done by means of compression tests which are conducted using a compression machine. Therefore, it will be possible to assess the influence of the graphene content on the material strength behavior, based on the experimental results, considering the different graphene volume fractions. The results are expected to contribute to understanding and predicting the behavior of this metal matrix composite material, widely employed in various engineering applications, such as, automotive, military and aerospace activities.*

Keywords: *Graphene, composite materials, powder metallurgy, mechanical properties.*

1. INTRODUCTION

Lightweight materials with high mechanical strength are in high demand in many engineering fields, particularly in the automotive and aerospace sectors. Aluminum is widely used in these areas due to its versatility, easy of application, low density, and great ductility. In order to enhance the properties of this material and use it in more advanced applications, various techniques are adopted, and one of them is incorporating graphene as reinforcement, which shows promising results in improving the mechanical properties of aluminum and its alloys.

However, there are still some challenges in producing this metal matrix composite, so many authors present different ways of mixing the materials and trying to extract the maximum performance from them. Numerous researchers have conducted studies on metal matrix composites, notably employing aluminum as the matrix, with the addition of graphene or carbon reinforcements. Through powder metallurgy, Shin *et al.* (2014) applied carbon nanotubes and few-layer graphene as reinforcement to aluminum, using ball milling and hot extrusion as the manufacturing method for material blending. The advantage of this approach is the ability to control and visualize the direction of reinforcement. Some other authors have used graphene nanoplatelets (GNP) as reinforcement. Lazarova *et al.* (2022a) produced aluminum samples reinforced with graphene nanoplatelets using the ball milling method for mixing, at a speed of 700 rpm for 10 minutes, with graphene fractions ranging from 0 to 0.7% by weight of the samples.

In a subsequent study, Lazarova *et al.* (2022b) fabricated samples with a weight fraction ranging from 0.1% to 1.1%, mixing for 30 minutes. Zheng *et al.* (2020) mixed the materials in a similar manner, using ball milling at a speed of 300 rpm for 10 hours. Khanna *et al.* (2022) carried out the mixing using a stainless steel ball mill. This is one of the most commonly applied techniques for blending the components, and in the majority of results presented by the authors, the efficiency of reinforcement in improving the mechanical properties of the material is positive. Authors such as Bhadauria *et al.* (2018) used an ultrasonicator to mix aluminum and GNP immersed in acetone, as another method of blending. Rashad *et al.* (2015) also used an ultrasonicator with ethanol. Afterward, the material was added to a mechanical shaker at 2000 rpm for 1 hour, filtered, and dried at 80°C for 12 hours, followed by hot extrusion.

Various characterization techniques are applied by the authors, including X-ray diffraction, used to analyze the materials composition and the presence of graphene. Often, due to the small amount added to aluminum, the peaks of this material may not be visible. Scanning electron microscopy is used for microstructure analysis (SEM), to view the presence

of pores and impurities. Transmission electron microscopy allows the analysis of graphene dispersion, interfacial relationships between the matrix and the reinforcement, as well as visualization of the graphene nanoplatelets' shape.

To better identify the properties of the composite material and predict its behavior in applications, the material is characterized along with simulations and mechanical tests. After determining the material and reinforcement to be used, as well as the manufacturing method, a series of analyses and tests are initiated. Cylindrical specimens are produced and their surfaces prepared for analysis using scanning electron microscopy (SEM), hardness tests, and compression tests, which are of particular interest for this investigation. Before the mechanical compression test, the specimens top and bottom surfaces are sanded, polished, and lubricated to reduce the barrel-shaped effect during the test and achieve more accurate and precise results.

Stress-strain curves are usually determined through tensile mechanical tests, but this test has limited usefulness due to the necking phenomenon that occurs in the sample, making it useful only for lower strain rates. On the other hand, the mechanical compression test of a short cylinder, for example, is a better and more suitable test for obtaining yield stress values, as it allows for higher deformations, even exceeding 2.0, especially when dealing with ductile materials.

According to Dieter (1988), in an axial compression test, a cylinder with an initial diameter D_0 and initial height h_0 could be compressed to a height h and expanded to a diameter D following the law of constant volume:

$$D_0^2 h_0 = D^2 h \quad (1)$$

During the deformation process, we can observe that the cylindrical test specimen begins to take on a barrel-shaped form. This behavior is justified because frictional forces oppose the flow of metal from the center to the extremities, and this resistance is more significant in the regions in contact with the base of the testing equipment. Meanwhile, the metal located in the central part of the sample can flow radially without restriction. This creates an undeformed region near the contact surface, as shown in the figure below:

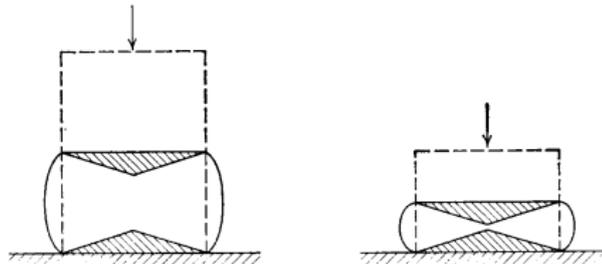


Figure 1. Undeformed regions (hatched) due to friction at the ends of the specimen in the compression test. Dieter (1988).

In mechanical tests, there is friction between the specimen and the surfaces of the equipment or forming tools, and typically, in the analysis of test results, this frictional force is neglected. However, the obtained result is then not entirely realistic.

2. MATERIALS AND METHODS

The materials used for the fabrication of the aluminum matrix composite with graphene reinforcement were sourced from the materials available in the university metallurgy laboratory. Specifically, aluminum powder with a purity of 99.5% and graphene nanoplatelets (GNP) from the Alfa Aesar brand were employed, according to Figure 2, (a) and (b). At Figure 3., we can observe the microscopy of the aluminum powder used:

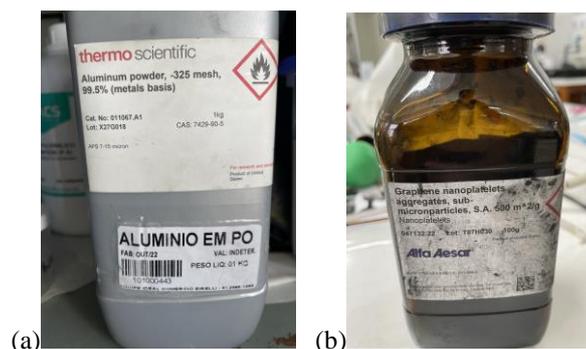


Figure 2. Materials used in the fabrication of the samples.

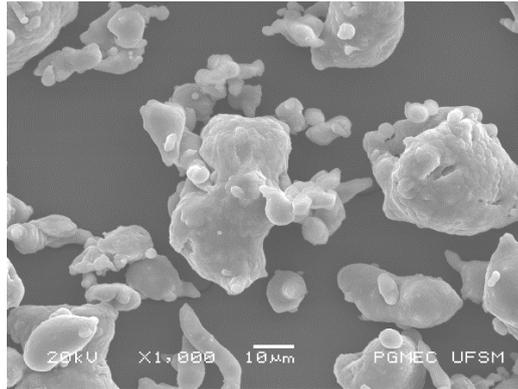


Figure 3. SEM images from pure Al powder.

After the materials were selected, the process of manufacturing cylindrical specimens is initiated using powder metallurgy. The process followed a sequence: weighing the quantities, mixing, compacting, sintering, and surface preparation.

First, the materials were weighed using a precision balance, and graphene was added to the aluminum in various proportions, as described in Table 1.:

Table 1. Graphene fractions in relation to the weight of the samples.

Graphene fraction wt. %	Al Powder (g)	GNPs (g)
0	2.1	-
0.3	2.0937	0.0063
0.5	2.0895	0.0105
0.7	2.0853	0.0147
1	2.079	0.021

Subsequent to adding the reinforcement to the aluminum, the powder was placed in cryogenic tubes with a capacity of 5ml and mixed in a vortex shaker, Figure 4, for 4 hours at 540rpm to ensure the most homogeneous material mixture possible.

Following the mixing period, the material was compacted using a mold for the production of cylindrical samples in a manual hydraulic press, as shown in Figure 5. They were pressed for 10 minutes with a load of 285MPa. After compaction, the samples were sintered in a furnace, gradually ramping up the temperature to 650°C. The heating rate was 2°C per minute until reaching 500°C, followed by 10°C per minute until reaching 650°C, in a vacuum atmosphere inside the furnace. The samples were kept inside the furnace until they returned to room temperature. Ten samples were produced for each graphene portion, totaling 50 units. Figure 6 displays the appearance of the samples after sintering.



Figure 4. Vortex mixer and cryogenic tubes.

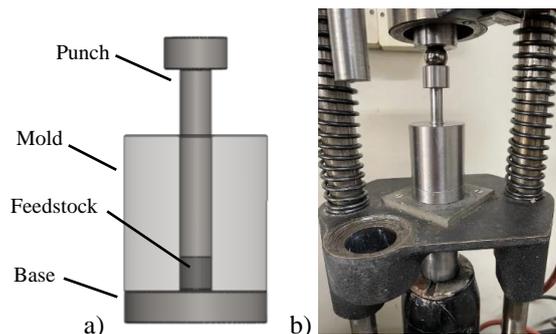


Figure 5. a) Sample compression scheme. b) Mold positioned in the hydraulic press.



Figure 6. Cylindrical specimens of graphene-reinforced aluminum composite.

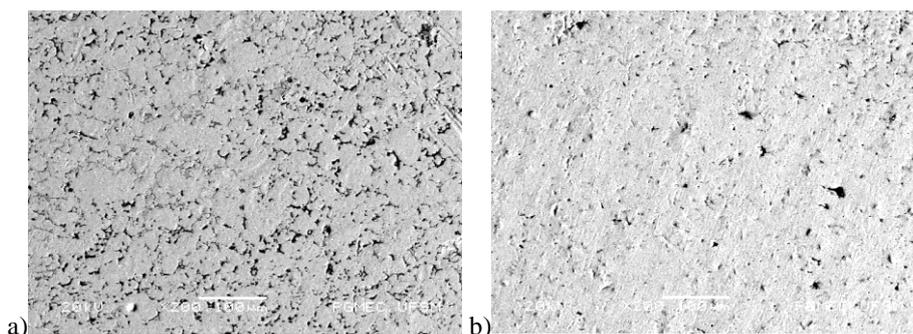
The Scanning Electron Microscopy (SEM) Jeol JSM6360, was used to obtain detailed information about the morphology of the aluminum-graphene composite, its interactions, and cluster visualization. Alongside the microscope analysis, energy-dispersive X-ray spectroscopy (EDS) was conducted using the Bruker Nano Compact equipment. An approximate magnification of 500x was employed for visualization, and subsequent element mapping of the sample. Additionally, a compression test, in a universal testing machine, Instron – 34TM-50-SA 50kN, was performed, speed 1 mm/min, displacement of 5.5mm, to determine important mechanical properties of this material, such as compressive strength and stress-strain curves. These properties are essential for understanding the structural behavior and applicability of the composite in different scenarios. The Vickers microhardness test was conducted using a SHIMADZU HMV-2 microhardness tester, with a pre-load of 4.9N for 15 seconds. Five measurements were taken on the surface of each sample using a 40x magnification lens, following a line between the edges and the center of the diameter of each sample.

3. RESULTS AND DISCUSSION

Through the acquired SEM images, ranging from 100x to 4000x magnification, the aluminum powder was initially analyzed (Fig. 3) to observe the shape and size of the grains. Subsequently, the analysis of the composites was conducted, revealing that the samples sintered and displayed varying levels of porosity. There were also contaminants present, likely originating from the abrasion with sandpaper during preparation, which can be observed in Figure 7, with the addition of 0.3% of GNP and Fig. 5b with 0.7% of GNP. Additionally, EDS analysis showed carbon distributed within the aluminum matrix, and in samples with a higher graphene content, such as 0.7% and 1%, carbon agglomerations were evident, possibly due to less efficient mixing, as shown in Figure 8. Such agglomerations are undesirable and can negatively impact the composite's mechanical performance.

For the pure aluminum sample, sintering was observed, but with significant non-uniformities and porosity. Contaminants from the preparation process were also detected, (Fig. 7 a).

In samples with 0.5% of reinforcement, a uniform distribution of porosity and carbon was visible, along with the presence of aluminum, carbon, and contaminants on the surface. Using 0.7%, carbon agglomerations were noticeable in specific regions, as seen in Figure 8. The samples with 1% also exhibited carbon agglomerations and some discontinuities on the surface, resulting from the sintering process.



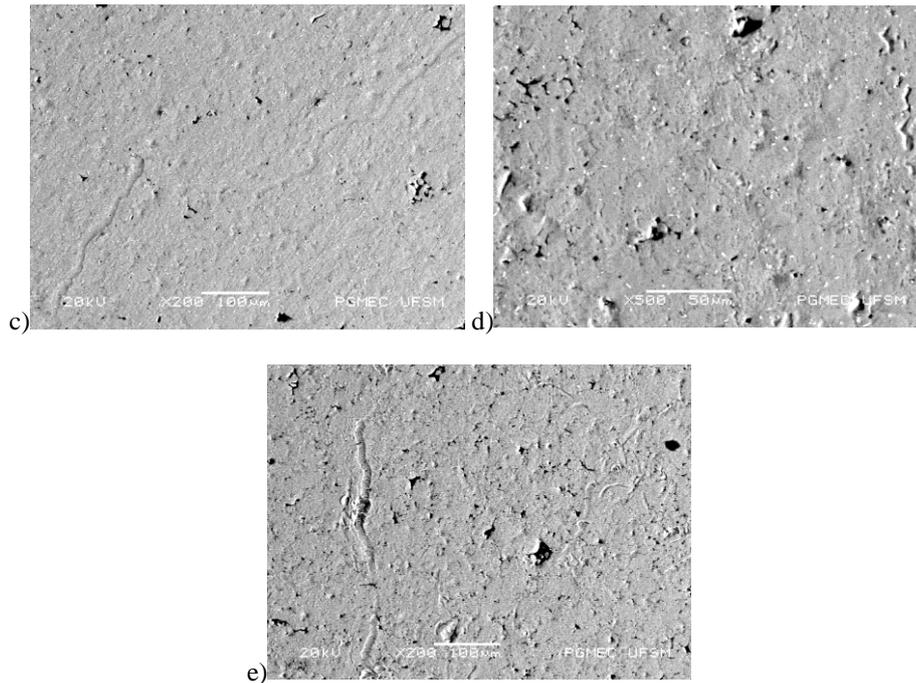


Figure 7. SEM images of the samples, a) Pure Al; b) 0.3 wt.% graphene; c) 0.5 wt.% graphene; d) 0.7 wt.% graphene; e) 1 wt.% graphene.

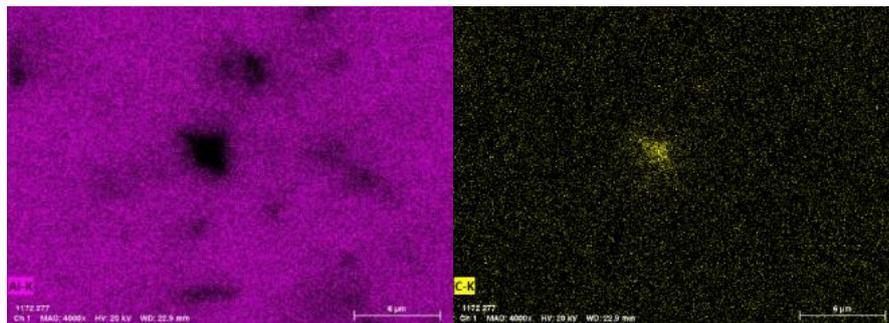


Figure 8. EDS result of the sample with 0,7wt.%.

In order to reduce the barrel shape effect, graphite grease was used as lubricant in this test, applied before carrying out the test on the upper and lower surface of the specimen. The format of samples after the test can be seen in figure 6:



Figure 9. Samples after the compression test.

Using the compression testing machine, it was possible to conduct the test on the cylindrical specimens, which were then compressed to a displacement of 5.5mm. The equipment provided us with load-displacement data, which were then analyzed to obtain the stress-strain curve, shown in figure 10. We can observe that the samples with graphene reinforcement exhibited better performance in compression strength compared to the pure aluminum sample. Through these results, it was evident that the reinforcement contributed to enhancing the material's resistance, even though the mixing method is not yet ideal.

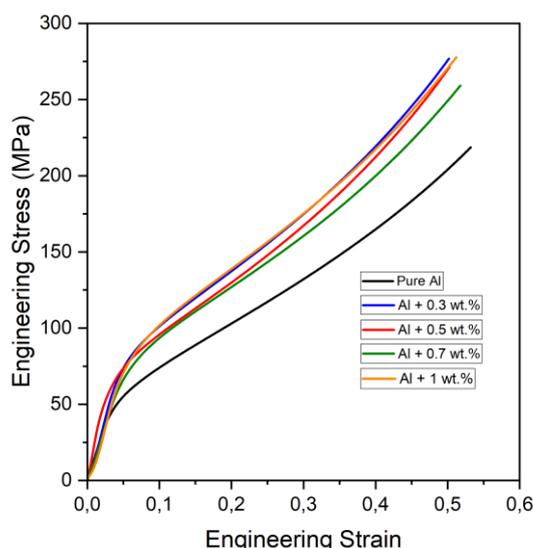


Figure 10. Stress-strains curves of the tested materials.

As a result of the Vickers microhardness tests, we can observe that the manufacturing method used may not have been the most effective, leading to a decrease in mechanical performance in some quantities of graphene due to possible agglomerations and poor reinforcement distribution. In the tested specimens, we found that samples with 0.5% graphene showed better results compared to pure aluminum, while samples with 1% exhibited lower microhardness values. The test was conducted on 5 samples for each reinforcement amount, and the average results are shown in Table 2.:

Table 2. Vickers microhardness results.

Graphene fraction wt. (%)	HV
0	24,23
0.3	23,15
0.5	25,72
0.7	23,67
1	22,5

4. CONCLUSIONS

In this study, a available method for material mixing in the laboratory was chosen, and cylindrical composite specimens were produced for characterization and mechanical testing. It was observed that the samples with graphene reinforcement exhibited an increase in compression strength compared to those made of pure Al. However, it is evident that the chosen mixing method is not yet ideal, as some results, especially mechanicals, are still significantly lower compared to those of other researchers. In microhardness testing, we obtained a better material response with the addition of 0.5% graphene. Another sample preparation method could be employed to improve the visualization of results through SEM (Scanning Electron Microscopy). The use of graphite grease as a lubricant was crucial for achieving uniform deformation during the compression test.

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6. RESPONSIBILITY NOTICE

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