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# CHARACTERIZATION OF GRAPHITE NANOSHEETS BY SCANNING ELECTRONIC MICROSCOPY (SEM) AND TRANSMISSION ELECTRON MICROSCOPE (TEM)

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## INTRODUCTION

Graphite is considered a highly prospective filler material for future polymer composites, due to its high aspect ratio and specially for its good electric and thermal conductivity and raised resistance.

Graphite is constituted by thousands of graphene sheet layers, where the carbon atoms are firmly packed in a two-dimensional net of successive hexagons. The graphene sheets can be separated by means of chemical and physical treatment. Exfoliated graphite can be produced from chemical treatment with oxidant agent intercalate. After the intercalation stage the sample suffers a sudden vaporization or decomposition of species, resulting in a perpendicular direction expansion of the graphene layers. Additionally ultrasonic treatment results in graphite nanosheets [1, 2, 3].

The objective of this paper is to study the morphology modification from the natural flake graphite until the graphite nanosheets, by SEM and TEM.

## EXPERIMENTAL PROCEDURE

The graphite used in this study was natural flake graphite (NG) and it was exfoliated by a process that is subdivided in intercalation and expansion, because the sheets were bonded on the hexagonal plane and needed to be separated down to a nanometer scale.

In the intercalation process, a mixture of concentrated sulfuric acid (H<sub>2</sub>SO<sub>4</sub>) and nitric acid (HNO<sub>3</sub>) (4:1, v/v) was mixed with the NG, for 24 hours, under appropriate stirring conditions. After that it was washed with distilled water until neutralization and then dried in a furnace above 110°C for 3 hours, to remove the remaining water by evaporation, resulting in the intercalated graphite (IG).

The dried particles of IG were treated at 1000°C for 30 s, thereby obtaining expanded graphite (EG). The graphite nanosheets (NG) were obtained by ultrasonic treatment in an acetone bath for 9 hours, filtered and dry in a furnace above 100°C for 3 hours [4].

## RESULTS AND DISCUSSION

In Figure 1 it is possible to observe the natural graphite subdivided in added of compact graphite sheets in the vertical direction of the flake. After the intercalation process it was possible to observe that the graphite flakes presenting innumerable sheets of graphene had been separate. In this stage, acid mixture (H<sub>2</sub>SO<sub>4</sub>/HNO<sub>3</sub>) is intercalated entering the layers of the graphite, separating, in part, the graphene sheets, resulting in graphite intercalated (IG), Figure 2.

At such a high temperature, intercalates (trapped between graphite layers) decompose and force the graphite layers to separate randomly [3]. The thermal shock provokes an increase in the perpendicular dimension to the carbon layers of each particle of the intercalated graphite that is transformed very fast into expanded graphite, Figure 3. After the expansion, ultrasonic treatment was employed to break down the worm-like structure resulting in individual graphite nanosheets (GN) that are less than 60 nm, as measured from SEM images, Figure 4. Figure 5 shows TEM images of graphite nanosheets in scale of 10nm and the parallel lines, which are the cross-sections of the graphite layers.

## CONCLUSION

The methodology used for the graphite nanosheets preparation proved to be a satisfactory technique. SEM images showed that part of the graphite was integrated and part expanded. The individual graphite nanosheets (GN) were approximately 60nm thick.

## ACKNOWLEDGEMENTS

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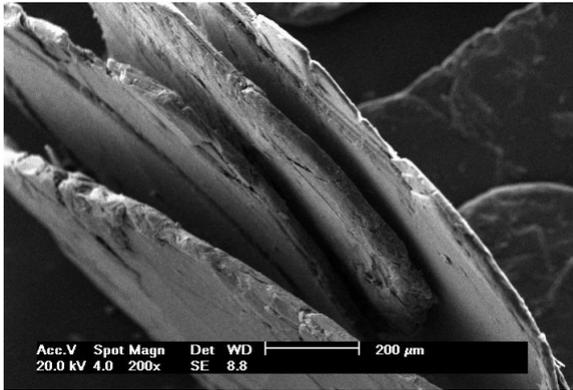


Figure 2 – SEM image in SE mode – Intercalated graphite (IG).

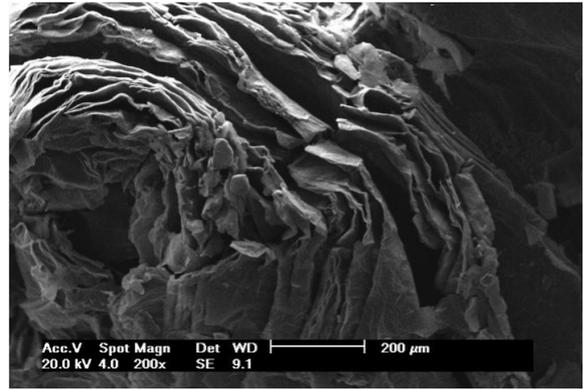


Figure 2 – SEM image in SE mode – Intercalated graphite (IG).

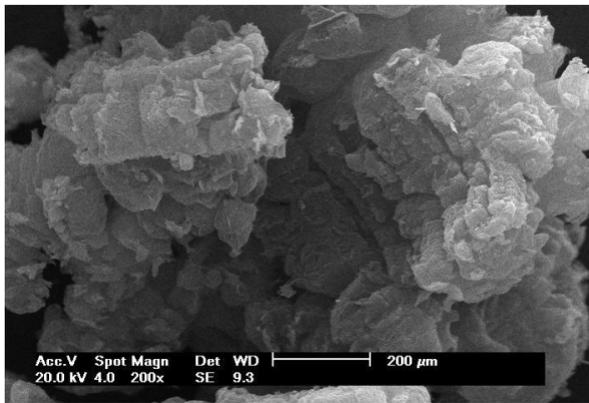


Figure 3 – SEM image in SE mode – Expanded graphite (EG).

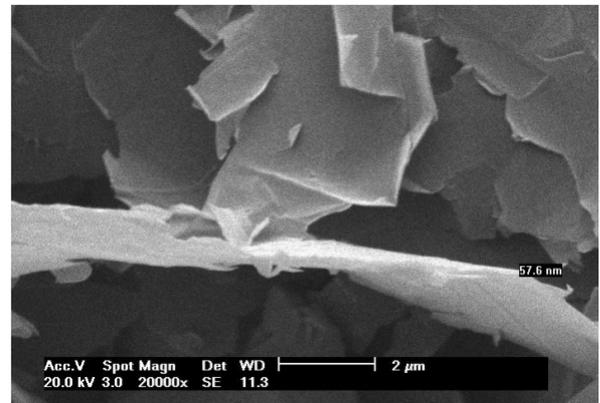


Figure 4 – SEM image in SE mode, graphite sheets of nanometers in thickness < 60nm.

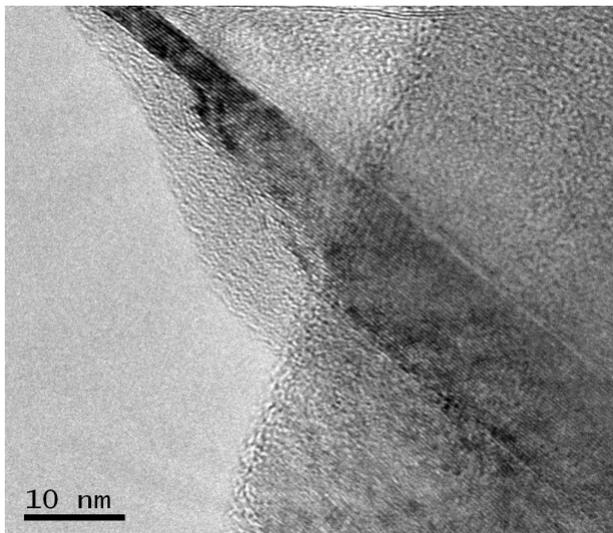


Figure 5 – TEM image of graphite nanosheets (GN), in scale bar 10 nm.