

## Removal of MgO impurity crystals by mechanical milling exfoliation of graphene obtained by CO<sub>2</sub> atmosphere synthesis method.

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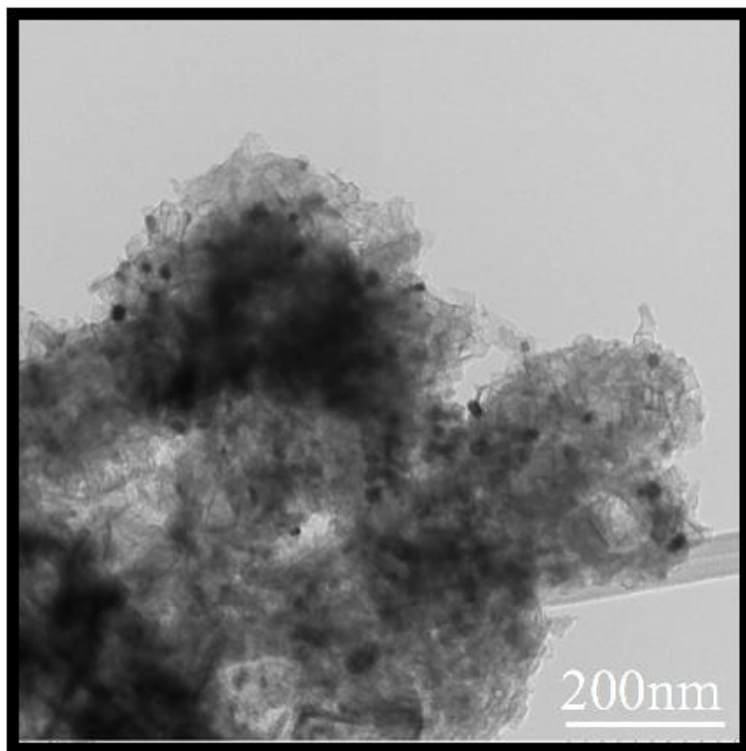
Nowadays graphene has a great area of interest due to its electric, mechanical and thermal properties compared to other materials, it has been reported that graphene (GPN) has a high fracture resistance of 125 GPa which makes it an excellent option as a reinforcer [1]. Graphene can be classified depending on the number of layers in its structure, considered monolayer (1 layer), few layers (i.e., 2–10 layers) [2].

Graphene can be obtained by using the CO<sub>2</sub> atmosphere method [3]. This method consists of burning metallic Mg inside a CO<sub>2</sub> atmosphere obtaining C (graphene) and MgO as a product of the reaction, then MgCl<sub>2</sub> and HCl as waste after cleaning and obtaining the graphene [4]. It makes this method more environment friendly compared to the Brodie method, Staudenmaier method, and Hummers method, which require the use of oxidants such as concentrated sulfuric acid, nitric acid, and potassium permanganate which later become toxic wastes.

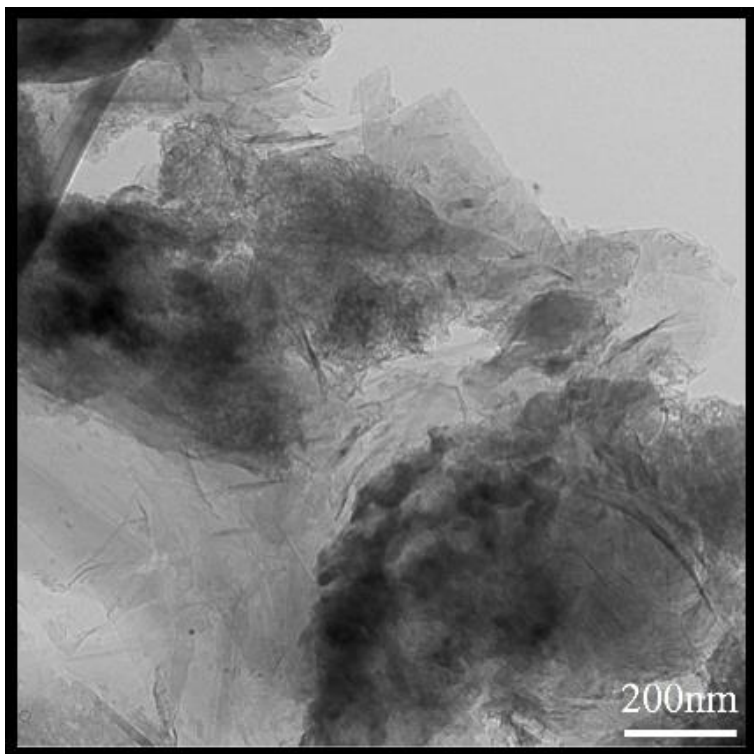
After analyzing by TEM the graphene obtained, in figure 1 can be seen the MgO crystals remain inside the graphene structure. These crystals are visibly wrapped inside the graphene sheets hence it is not possible to dissolve these MgO crystals due to the lack of interaction between the MgO crystals and the HCl. A TGA test was performed to analyze the inorganic reduces in the graphene which in this case are the MgO crystals.

A new way to approach the wrapped MgO crystal is by exfoliating the graphene sheets by mechanical milling, this helps us to expose these crystals and make them interact with the HCl to its subsequent removal. By milling 1g of graphene for 1h in a Spex mill, then cleaning the graphene milled again with the HCl solution to finally remove as much as possible the MgO crystals exposed in the mechanical milling.

After the mechanical milling, we obtained an incredible reduction of the MgO crystals from a 23%wt to a 3%wt after analyzing the inorganic residues obtained by a TGA test, and it can be seen by TEM images in figure 2 less density of the MgO crystals in the graphene compared to figure 1.



**Figure 1.** TEM of Graphene not mechanical milled.



**Figure 2.** TEM of Graphene mechanical milled 30min.

## References

- [1] Jingyue Wang, Zhiqiang Li, Genlian Fan, Huanhuan Pan, Zhixin Chenb, Di Zhanga, Reinforcement with graphene nanosheets in aluminum matrix composites, *Scripta Materialia* 66 (2012) 594–597.
- [2] Wick P, Louw-Gaume AE, Kucki M, Krug HF, Kostarelos K, Fadeel B, et al. Classification framework for graphene-based materials. *Angew Chem Int Ed* 2014;53(30):7714–8.
- [3] Juan Zhang “Synthesis of graphene from dry ice in flames and its application in supercapacitors”, *Chemical Physics Letters* 591 (2014) 78–81.
- [4] Amartya Chakrabarti “Conversion of carbon dioxide to few-layer grapheme”, *J. Mater. Chem.*, 2011, 21, 9491.