APRIL 20-21, 2021
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Fundamental Research Insights
GO 2021

Few-layered mesoporous graphene obtained through high energy dry ball-milling.

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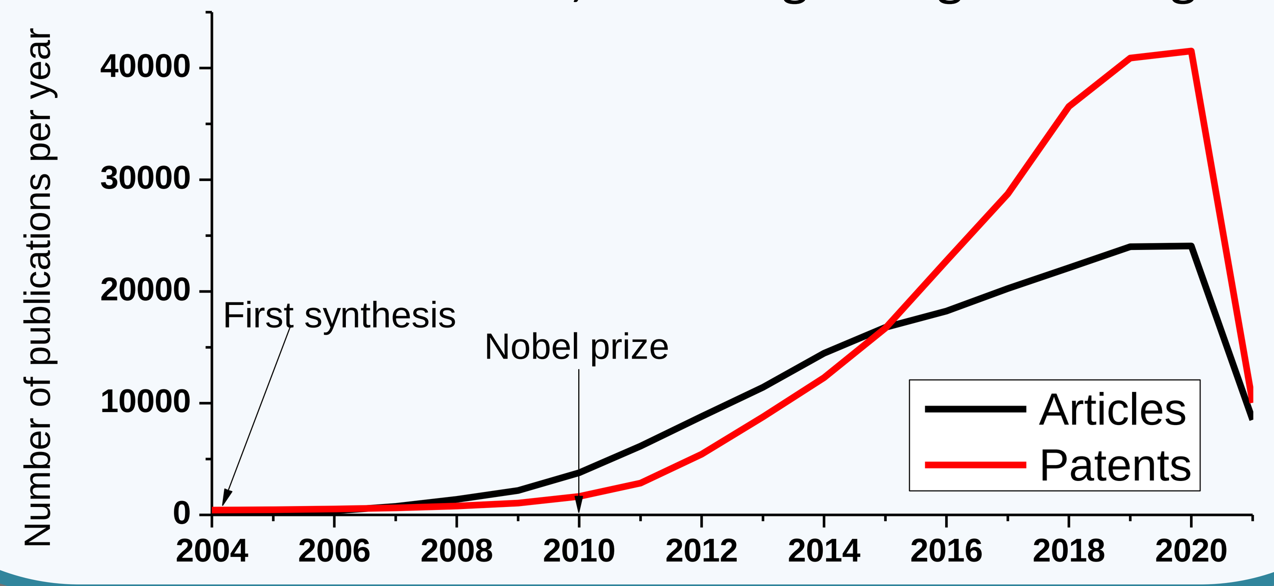
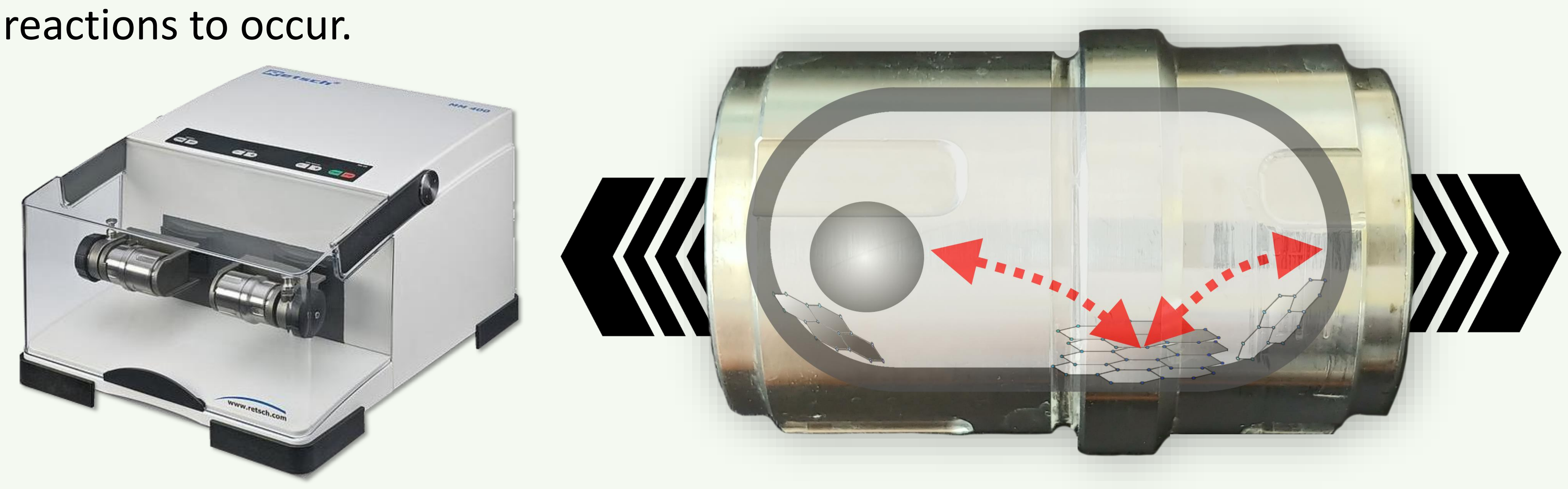
Graphene and graphene-based materials (GBMs) attract enormous attention, with tens of thousands of documents being published each year and an inexhaustible increment in interest. In the last few years, the focus on graphene has shifted from research towards industrialization, with product standardization and large-scale production still being obstacles to exploit these material's full potential [1].
To overcome those obstacles, in this work, we developed a high energy dry ball-milling method. After comprehensive characterization, the obtained material is described as few-layered mesoporous graphene (FLMG). Finally, we propose applications for this material, including NO2 gas sensing.

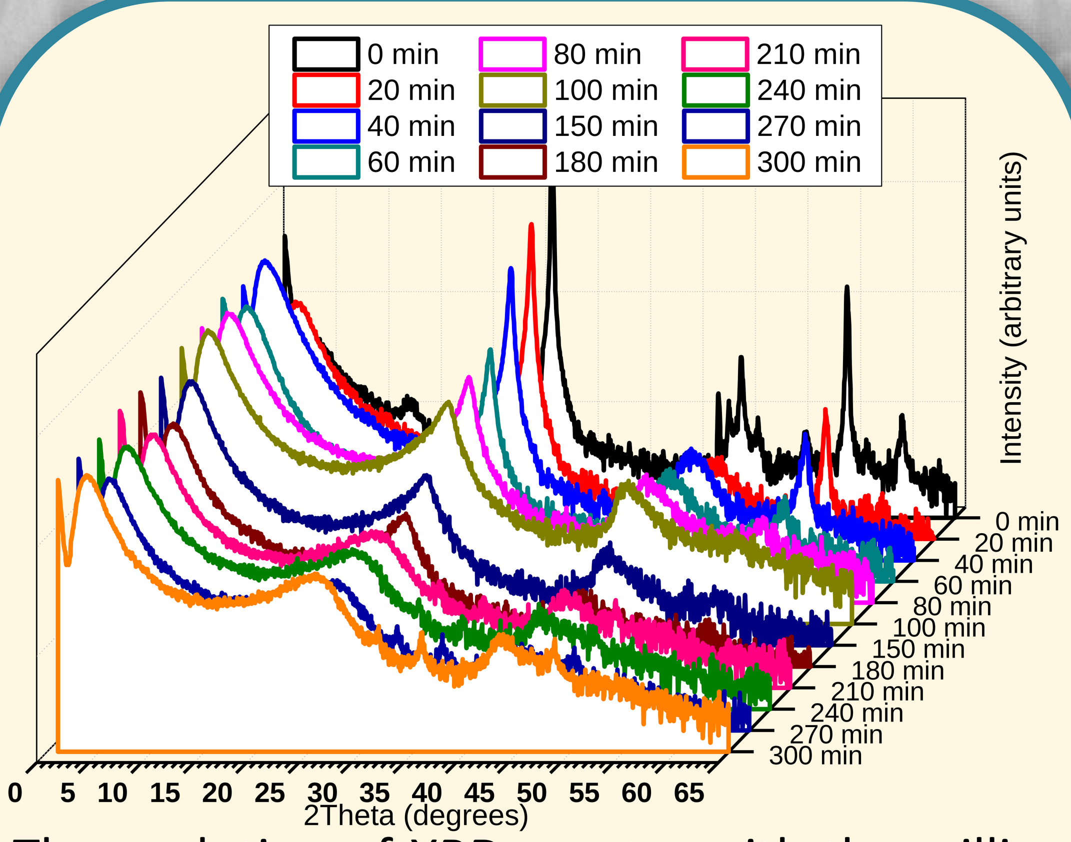


The high energy ball-milling is a well-known technique for the synthesis of nanomaterials and mechanical alloying. During the milling, energy is transferred to the materials inside the milling jar, allowing some physical and chemical reactions to occur.

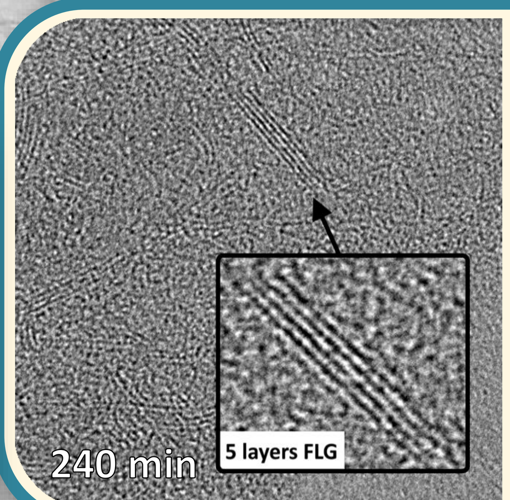


We used graphite as a precursor with no added elements (neither liquid medium nor additives), thus requiring no additional treatments. Graphite suffers exfoliation and defects formation during the process.

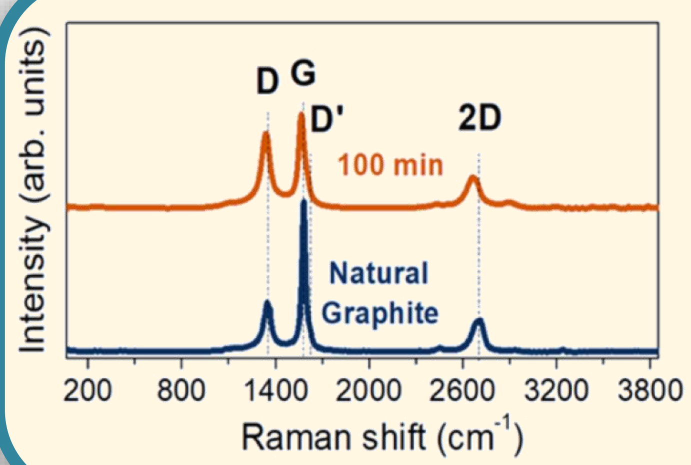
This approach makes the high energy dry ball-milling a one-pot, low-cost, and environmentally-friendly technique to get few-layered mesoporous graphene. A detailed description of the method is provided elsewhere [2].



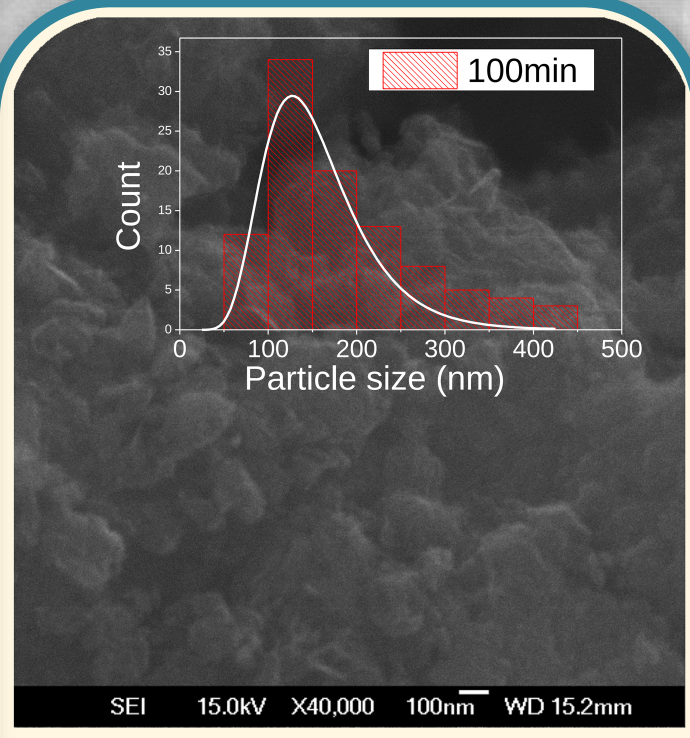
The evolution of XRD spectra with the milling time show the decrease of 26.5° [002] maximum, associated with the exfoliation process of graphite.
The absence of 11.5° maximum evidence no oxidation of graphene (supported by UV-vis absorbance).
Low angle reflections (2.5°-4.5°) are related to the existence of mesoporous structures with no preferential order and limited organization.



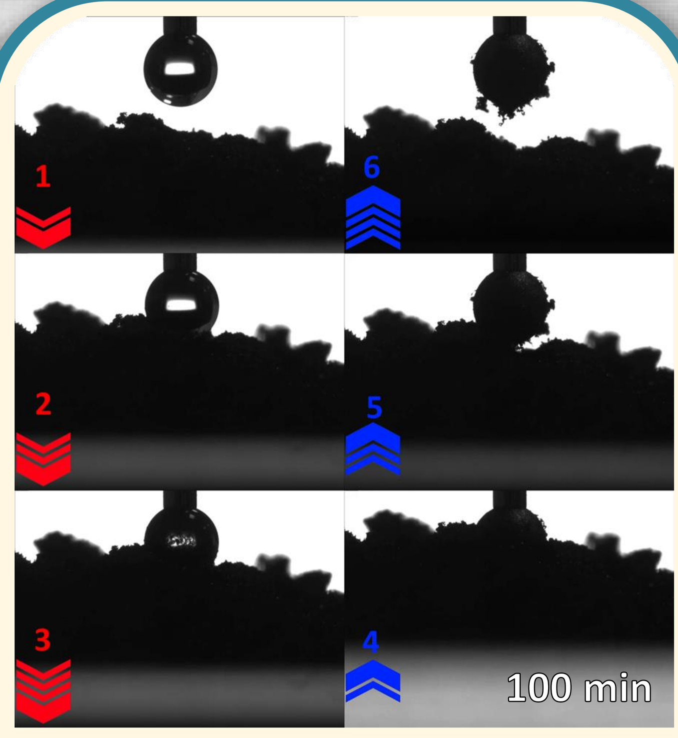
Particles are composed of crystalline domains 3-10 layers stacking, in essence, few-layered graphene.



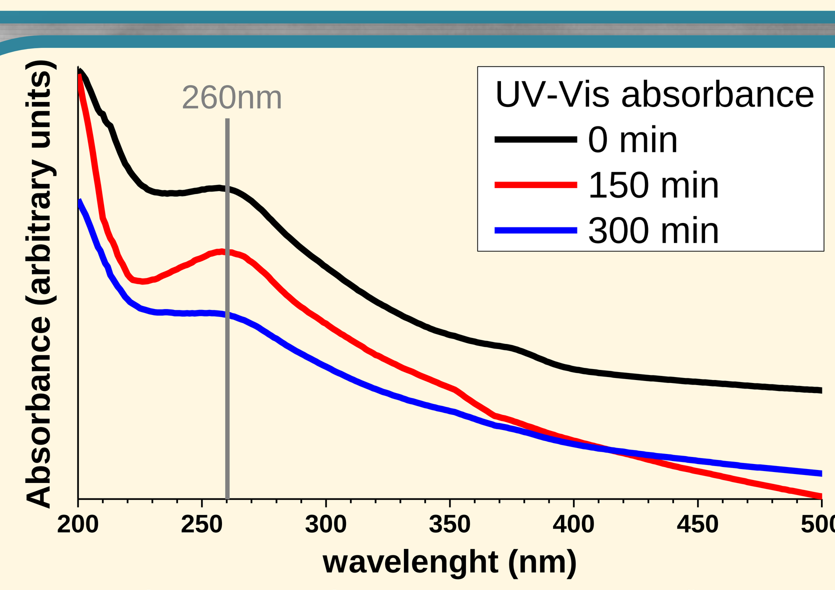
The Amp(D)/Amp(D') ratio seen with confocal Raman microscopy for the 100min sample indicates vacancy-type defects with noticeable signs of agglomeration, which would favour the sp3 hybridization.



Milled particles are less platelet-shaped than the original graphite and show evidences of aggregation.

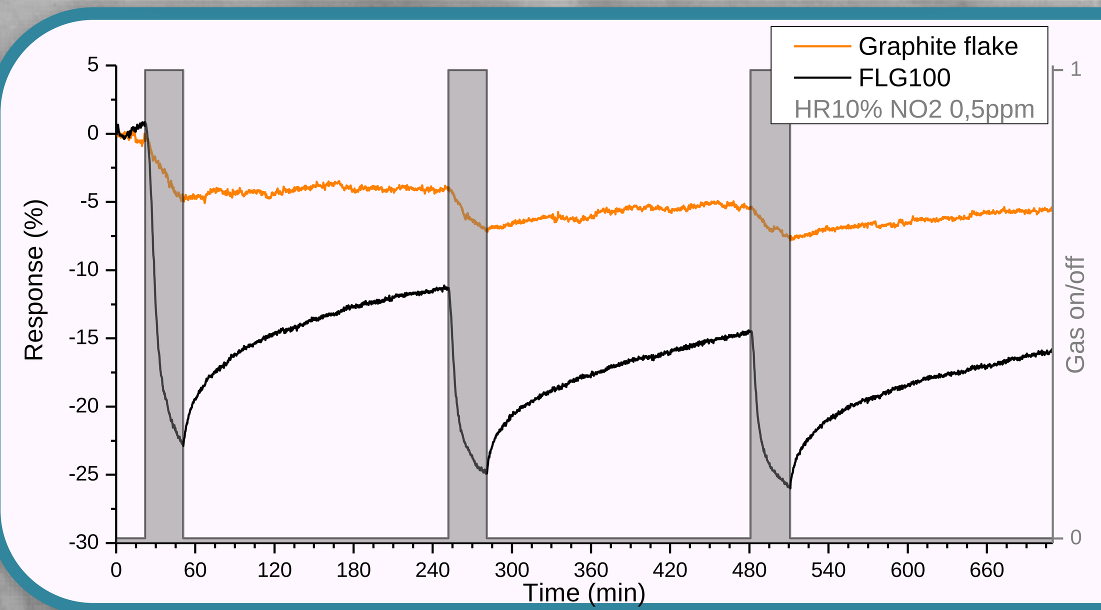


Superhydrophobicity is confirmed by the sessile drop method.
(video at online version of ref. [3]).



UV-vis abs. reach a maximum for 260nm, corresponding with electron transitions in non oxidized C=C bonds, even at highest milling times.

D. Matatagui et al. provides further characterization analysis [3].



This high-energy dry ball-milling method satisfies the requirements for large-scale GBMs production, being more cost-effective and greener than some of its alternatives.

Furthermore, the obtained FLMG is reproducible and valuable. High specific surfaces and defects on graphene-based materials are desired for various applications such as gas sensing, where FLMG demonstrated excellent performance compared to standard graphite [3].

Future work involves more research on applications for this material, especially in large-quantity demanding sectors such as energy storage or polymer-based composites.

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REFERENCES

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[2] P. Marín, E. Navarro, J. Lopez-Sanchez, A. Peña, M.C. Horrillo, D. Matatagui. Obtención a gran escala en un solo paso... Patent no. ES 2 779 151 - B2 (2020)
[3] D. Matatagui et al. Sensors and Actuators: B. Chemical 335 (2021) 129657

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