Nanoscale Comparison of graphite exfoliation by supramolecular, chemical and electrochemical methods

Zhenyuan Xia[1], Emanuele Treossi[1], Vincenzo Palermo[1]

[1]Istituto per la Sintesi Organica e la Fotoreattività-Consiglio Nazionale delle Ricerche (ISOF-CNR), via Gobetti 101, 40129 Bologna, Italy.
xia@isof.cnr.it

Being a single-atom-thin carbon sheet, graphene has been intensively studied due to its impressive mechanical, thermal, optical and electronic properties. Meanwhile, Nature has provided us with large amounts of graphene sheets in high quality, stacked into graphite mineral; all we have to do is finding a way to exfoliate the single sheets in kilogram-scale yield and high quality.

Graphite exfoliation can be simply achieved in different ways, such as chemical reduction of graphene oxide (GO) [1], exfoliation by extended sonication with organic solvent or surfactants [2], and electrochemistry approach [3]. While all these techniques yield graphite exfoliation, the exfoliation mechanism and the quality/size of the obtained graphene are greatly different. The exfoliation in organic solvents yields high quality sheets, but the mechanism of graphene dispersion in these solvents is still not clear. On the other side, the mechanism of chemical and electrochemical exfoliation involves gas production and subsequent large scale mechanical exfoliation, which have been previously studied [4].

In this work, we compare the nanoscale exfoliation process of graphite into graphene performed by sonication-assisted exfoliation in a widely used organic solvent (*N*,*N*-dimethylformamide, or DMF) with more disruptive exfoliation by oxidation (using a modified Hummers method) or by electrochemical oxidation.

Differently from previous work, we focused our attention not only on the exfoliated sheets, but on the bulk material that's left behind after the exfoliation process. Watching at the similarities and differences in surface morphology etched by the exfoliation process, we shall have information on the mechanism behind it.

For this, highly oriented pyrolytic graphite (HOPG) samples have been exfoliated using the aforementioned approaches, and then characterized by optical microscopy (OM), Atomic Force Microscopy (AFM) (in Fig.1), Scanning Electron Microscope (SEM), and X-rays Diffraction (XRD), monitoring the effects of different exfoliation techniques at the nanoscale.

References

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Figures

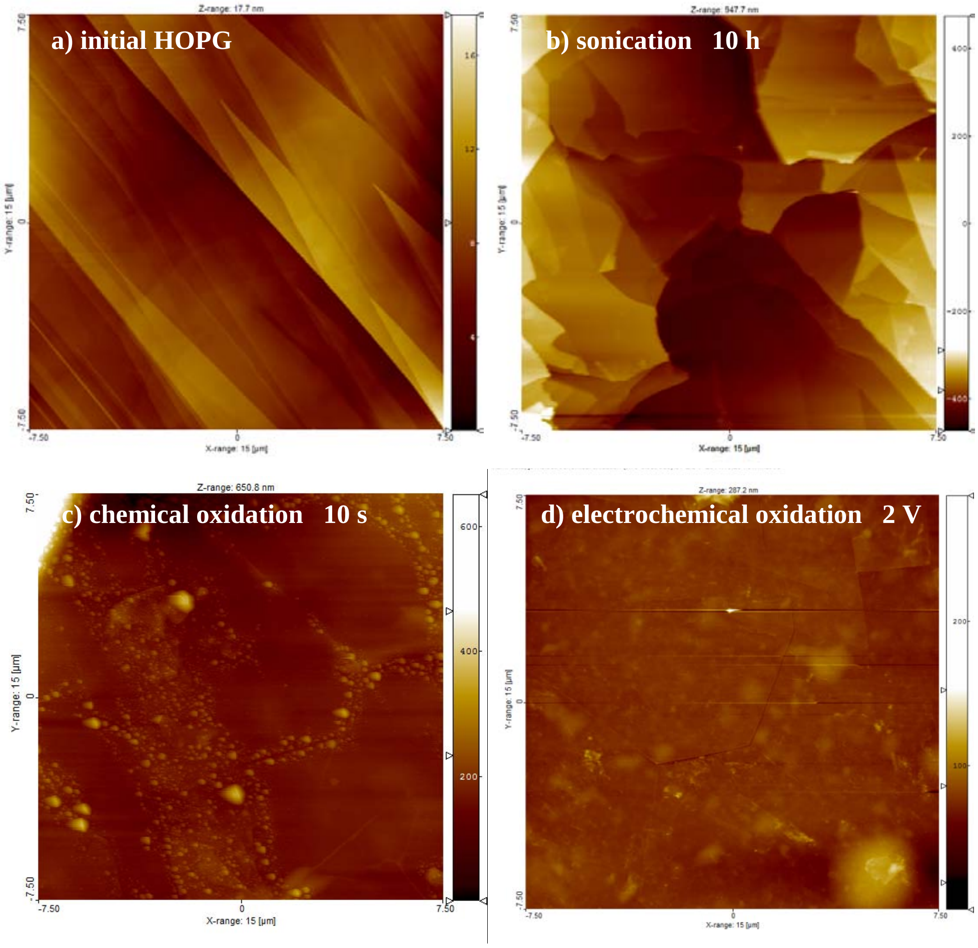

Fig. 1. AFM images of a 15 × 15 μm region of basal planes HOPG prepared under different conditions: a) initial HOPG; b) HOPG after 10 hours of sonication in DMF;; c) HOPG after 10 second of chemical oxidation by a modified Hummers method; d) HOPG anode after 30 min of electrochemical oxidation by applying 2 V in 0.5 M H_2SO_4.