

Enhanced Exfoliation Technique for the Separation of Graphene Nanosheets

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Abstract— Graphene sheets are carbon-based materials which combine exceptional electron conductivity, mechanical strength and optical transparency. Graphene nanosheets were fabricated by an enhanced, safer and mild technique in a shortened processing time. Samples were characterized by SEM, XRD, TGA, AFM and Raman Spectroscopy.

Keywords: Graphite Oxide, Graphene nanosheets, Exfoliation

Introduction

Graphene has attracted great interest due to the unique electronic, thermal, and mechanical properties, resulting from its 2D structure, and to its potential applications like microchips, chemical sensing instruments, biosensors, energy storage devices and other innovations. The first graphene sheets were obtained by extracting monolayer from the three-dimensional graphite using a technique called micromechanical cleavage in 2004 [1].

Graphite is described as a three-dimensional crystal built from graphene layers. There are several chemical treatments by the use of graphite oxide (GO) to separate graphene layers from graphite. Treating graphite with strong acids forms GO. GO is an oxygen-rich derivative of graphite and a layered material capable of undergoing intercalation by one-dimensional expansion along its *c*-axis. Brodie [2] produced the first GO samples by repeated treatment of Ceylon graphite with an oxidation mixture consisting of potassium chlorate and fuming nitric acid in 1850s. The rich surface chemistry of GO allows for covalent modification by reactions at hydroxy and epoxy groups. The chemical reduction of exfoliated GO with reducing agents like hydrazine hydrate and hydroquinone is a potential way in large scale production of graphene [3].

The purpose of this work is threefold: (i) to tailor the characteristic properties of graphene oxide papers at different oxidation times, (ii) to optimize reactant ratios during oxidation process, and (iii) to reduce the number of graphene layers in graphite.

Experimental

The chemical treatment for the separation of graphene nanosheets was conducted in three steps as follows: intercalation and oxidation of raw graphite, thermal exfoliation and chemical reduction. After each step, sonication process was performed for the homogenous dispersion in water about 1 hr at room temperature. Graphite oxidation was conducted by using potassium dichromate/sulfuric acid as oxidant and acetic anhydride as intercalating agent with different reaction times from 50 min up to 10 days. GO samples were exfoliated thermally under an argon atmosphere at different expanding temperatures and different expanding times in a tube furnace. GO and expanded GO samples were chemically reduced by refluxing with hydroquinone in water in order to obtain graphene nanosheets.

Results and Discussion

There are two chemical methods of producing the graphene nanosheets: (1) “graphite oxidation, sonication, chemical reductions” and (2) graphite oxidation, sonication, thermally expansion, and chemical reduction. In the present study, the characteristics of samples were tailored by comparing of these two ways with various characterization techniques to find the best way for the fabrication of graphene nanosheets.

The average number of layers for pristine graphite flake, GO, expanded GO, reduced expanded GO and reduced GO were calculated with the application of Debye-Scherrer equations from XRD patterns as 86, 79, 30, 37 and 9, respectively. In order to verify these layer numbers, average layer numbers were also calculated from the thickness value from AFM divided by interplanar spacing obtained from XRD patterns as 89, 17, 25, 17 and 11, respectively.

Both the reaction procedures with expansion and without expansion causes the formation of graphene nanosheets (Figure 1a and 1b).

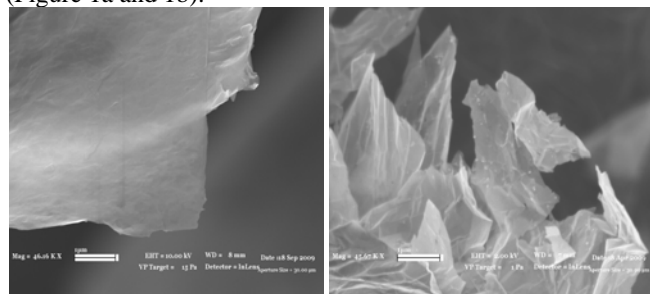


Figure 1. SEM images of reduced graphene oxide sheets via inlens detector (a) after oxidation process and (b) after expansion process.

Conclusions

An improved, safer and mild method was proposed for the exfoliation of graphene like sheets from graphite. After detailed characterization, the process time can be reduced by following the method: graphite oxidation, ultrasonic treatment and chemical reduction of GO samples.

References

- [1] K. S. Novoselov, A. K. Geim, S. V. Morozov, D. Jiang, Y. Zhang, S. V. Dubonos, I. V. Grigorieva, A. A. Firsov, *Science* **306**, 666 (2004).
- [2] B.C. Brodie, *Philos. Trans R Soc London* **149**: 249 (1859).
- [3] G. Wang et al. *J. Phys. Chem. C* **112**, 8192–8195(2008).