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Graphene From Two Chemical Routes: Synthesis and Characterization

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Abstract – Two different chemical routes have been applied to produce graphite oxide (GO) from graphite and pure hydrazine was used to convert GO paper into solution-stable graphene. Graphite oxide produced by Hummers' method yielded large (~10 µm) single sheets, while GO produced by Brodie's method yielded mainly nano-sized single- and few-layered graphene (FLG). In-plane electrical resistance has been measured by building resistor device and out-of-plane resistance has been measured by conductive atomic force microscopy. The products were characterized by X-ray diffractometry, optical microscopy, scanning electron microscopy and AFM.

Graphene or FLG have been produced by a number of different methods, usually time consuming and having low yield. For instance, highly oriented pyrolytic graphite can be peeled off using adhesive tape and transferred to Si substrate. However, single sheets are few and need to be found among a myriad of multi-layers flakes. SiC can be reduced to yield graphene, but large samples need elevated temperature. These methods are difficult to scale up to achieve mass production of graphene or FLG, already demonstrated through solution-based route [1]. This work presents synthesis and characterization of solution-based graphene with different morphologies, using two routes to obtain the precursor graphite oxide (GO).

Graphite was oxidized using (i) a modified Hummers' method (sulfuric acid/potassium permanganate) [2] and (ii) a modified Brodie's method (fuming nitric acid/sodium chlorate) [3]. GO made using (i) was dried to produce a GO paper, then processed in pure hydrazine to yield solution-stable graphene (H-graphene). The same reduction procedure was applied to GO (ii) to produce B-graphene. Diluted solutions of both products were spin coated onto SiO_2/Si substrates and samples were analyzed by SEM, AFM. Out-of-plane resistance was measured by conductive AFM and resistor devices were built to measure in-plane resistance.

Figure 1a shows large graphene sheet typical for H-graphene samples. The average step height is 0,83 nm, which comprises a single sheet. On the other hand, B-graphene samples (Figure 1b) presented nano-sized graphene with step height of 1.15 nm, which comprises graphene or few-layer chemically-reduced graphite. Figure 2a shows AFM contact mode of a H-graphene and the respective electric current map is presented in Figure 2b.Out-of plane resistance measured this way to H-graphene was 13.4 MΩ, while resistor device yielded 55 kΩ for in-plane resistance.

Different chemical routes are applicable to synthesis of chemically converted graphene. Different morphologies can be achieved by varying oxidizing agents and purification procedure, which may be suitable for different applications. High electrical resistance shown by chemically converted graphene is a great drawback of this procedure and efforts are required to either lower the oxidation degree while maintaining the solubility or enhance the reduction in order to eliminate defects introduced during the oxidation step.

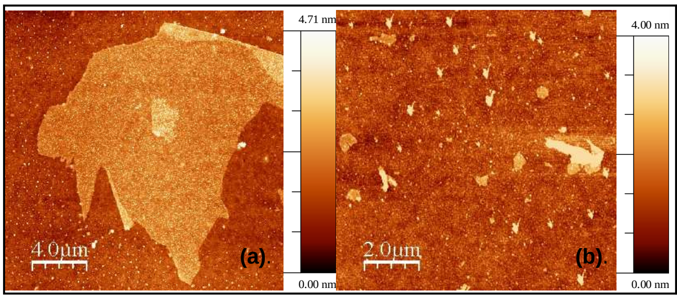

Figure 1: AFM tapping mode image of H-graphene (a) and B-graphene (b) over SiO_2/Si substrate.

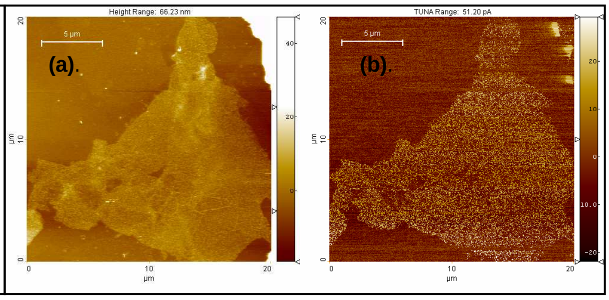

Figure 2: AFM contact mode image of H-graphene partially overlapped by gold pad (a) and the electric current map (b).

References

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