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Electrical behavior of amide functionalized graphene oxide and graphene oxide films annealed at different temperatures

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ABSTRACT

Films of graphene oxide (GO) and amide functionalized graphene oxides (AGOs) were deposited on SiO_2/Si(100) by spin coating and were thermally annealed at different temperatures. Sheet resistance of GO and AGOs films was measured using four probe resistivity method. GO an insulator at room temperature, exhibits decrease in sheet resistance with increase in annealing temperature. However, AGOs' low sheet resistance (250.43 Ω) at room temperature further decreases to 39.26 Ω after annealing at 800 °C. It was observed that the sheet resistance of GO was more than AGOs up to 700 °C, but effect was reversed after annealing at higher temperature. At higher annealing temperatures the oxygen functionality reduces in GO and sheet resistance decreases. Sheet resistance was found to be annealing time dependent. Longer duration of annealing at a particular temperature results in decrease of sheet resistance.

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1. Introduction

The remarkable electronic, thermal and mechanical properties of graphene have made it an important material for electronic [1], optoelectronic [2], capacitor and sensing applications [3,4].

The techniques used so far for the preparation of thin films of graphene are mechanical exfoliation, graphitization of SiC [5], chemical vapor deposition [6] and chemical preparation process *via* graphene oxide (GO) [7,8]. The mechanical exfoliation method used to prepare high quality graphene thin films is unsuitable for large scale applications due to its low productivity. Thus, large-scale preparation of graphene thin films has been considered as rate-limiting step in the applications of graphene. Among other methods, chemical preparation method is considered to be an efficient approach but at the cost of film quality as compared to mechanical exfoliation. The thermal and chemical reductions methods have been employed so far to reduce GO. For chemical reduction different reducing agents such as hydrogen sulphide [9], hydrazine [10,11], sodium borohydride [12], dimethylhydrazine [13] and hydroquinone [14] are widely used. But as the field effect mobility of reduced GO (rGO) is smaller than that of exfoliated graphene [15–17] and reducing agents used for reduction are hazardous. Moreover, poor dispersibility and processibility of chemically reduced GO in various solvents make them incompatible for most of electrical and other applications. Thus in the present work thermal reduction has been preferred as it avoids the hazardous reducing agents.

Recently, much effort has been made to prepare organic soluble graphene by incorporating organic moieties onto oxygen-containing groups of GO to enhance its electrical properties. Park et al. in 2013 observed enhancement in the electrical conductivity of GO by iodo functionalization [18]. Punckt et al. [19] found that functionalized graphene has wide range of electrical properties depending on exfoliation and reduction method. More studies were done to understand the electrical behavior of functionalized GO using polyvinyl alcohol [20], alkylated graphene [21], amines [22–25], silanes [26], 2-amino-4, 6-didodecylamino-1,3,5-triazine [27] etc.

In the present work covalently grafted amide functionalized graphene oxides (AGOs) were synthesized by chemical method and then reduced thermally to yield reduced amide functionalized graphene oxide (rAGO). Thus from chemical as well as thermal modification, it is expected that electrical behavior of GO can be modified for its use in electronic and sensor applications.

2. Experimental details

2.1. Materials

Graphite powder (purity 99.99%), sodium nitrate ($NaNO_3$), sulphuric acid, potassium permanganate ($KMnO_4$), hydrochloric acid (HCl) and hydrogen peroxide (H_2O_2) were purchased from Rankem RFCL Pvt. Ltd., India. Aniline, 2-aminothiazole, 2-aminopyrimidine, hydroxybenzotriazole (HOBt), *N,N′*-dicyclohexylcarbodiimide (DCC), dimethyl sulfoxide (DMSO) and dimethylformamide (DMF) were purchased from Karl Fisher Co., Ltd. Sodium hydroxide (NaOH), ammonium

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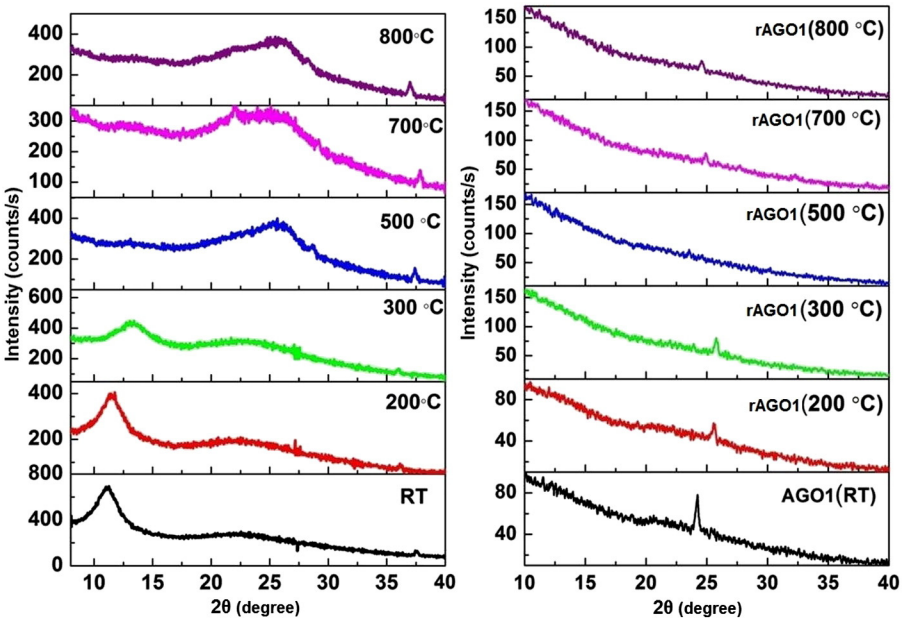

Fig. 1. (a) XRD of GO and thermally reduced thin films of GO. (b) XRD of AGO1 and thermally reduced thin films of AGO1 carried out at different temperatures.

hydroxide (NH_4OH), hydrofluoric acid (HF) and tetrahydrofuran (THF) were purchased from LOBA Chemie Pvt. Ltd., Mumbai, India.

2.2. Preparations of GO

GO is a water-soluble nanomaterial prepared using Hummers method through graphite powder. First, graphite (2 g) and $NaNO_3$ (1 g) were mixed in H_2SO_4 (46 ml) under stirring in ice bath. $KMnO_4$ (6 g) was slowly added to the above placed mixture [28]. The reaction mixture was then stirred for 30 min at 40 °C to form a thick paste. Subsequently, 80 ml water added to the formed paste, followed by another 90 min stirring at 90 °C. Successively, 200 ml water and 6 ml H_2O_2 were added in the above mixture. The solution was then filtered and washed with de-ionized water (DI) water. The filtered paste was dissolved in 100 ml DI water and ultrasonicated for 1 h. GO powder was obtained by centrifugation of solution for 20 min at 4000 rpm and drying it at room temperature.

2.3. Preparations of AGOs

The AGOs were prepared using aniline, 2-aminothiazole and 2-aminopyrimidine named as AGO1, AGO2 and AGO3, respectively. GO (0.3 g) was dispersed in 30 ml DMF by ultrasonication for 60 min at room temperature. Then, NaOH (0.3 g, 7.5 mmol) was added and resulting solution was stirred for 60 min at room temperature. Subsequently, aniline (0.3 g, 3.2 mmol), HOBt (3.2 mmol) followed by DCC (3.2 mmol) was added to the above reaction mixture and stirred for 24 h at room temperature. AGO powder collected by centrifugation

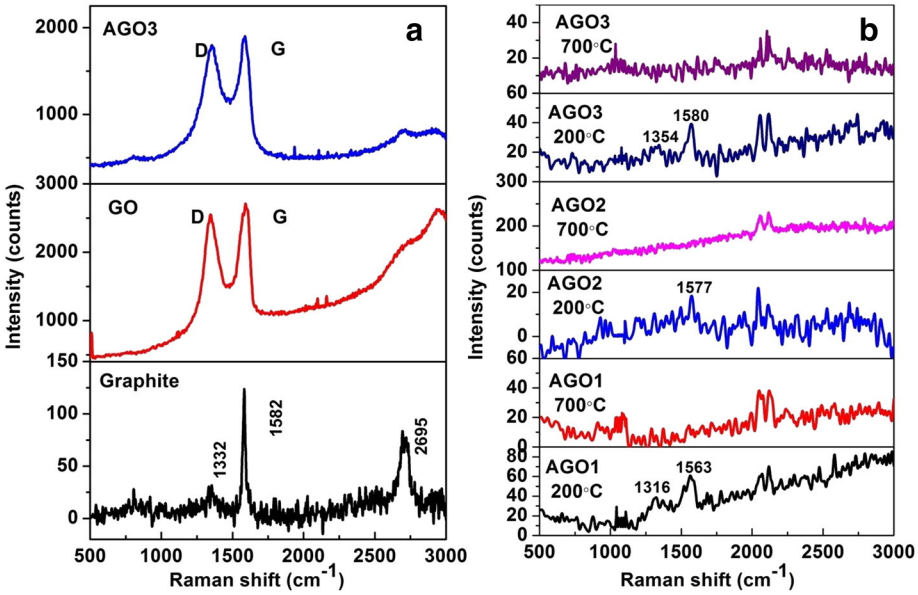

Fig. 2. Raman spectra of a) GO and AGO3, b) AGO1, AGO2 and AGO3 films annealed at 200 °C and 700 °C.

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