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Synthesis of Graphene via Exfoliation of Graphite by Electrochemical Method

Sunny Goswami*

ECE Department, Ambala College of Engineering and Applied Research Ambala, Haryana, India

***Corresponding author:** Sunny Goswami, ECE Department, Ambala College of Engineering and Applied Research Ambala, Haryana, India,
E-Mail: Sunnygoswami560@gmail.com

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Abstract

Graphene has become a promising material for many different applications, such as nano electronic devices, physical, chemical and biochemical sensors, transparent conductive films, clean energy scavenging and storage devices and nano composite formulation. Recently gas sensing as a critical application intelligent system, is receiving increasing attention in both industry and academia. Sensing application using graphene sheets as transducer have experienced surge of activities in recent years, especially for gas sensing platforms and electro chemical sensing, because of high electrical conductivity and high surface area of graphene. Furthermore, proper functionalization of graphene enables enhanced selectivity. In this work we investigate the synthesis of graphene by exfoliation of graphite by electrochemical method. It is the thinnest material in the universe and strongest ever measured. The characterization of the graphene is done with XRD.

Keywords: Graphene; XRD; Electrochemical exfoliation; Spectroscopy

Introduction

The electrochemical setup that is used to exfoliate graphite normally contains the following elements: a graphite working electrode, counter electrode, reference electrode, electrolyte, and power supply. Typically, highly orientated pyrolytic graphite (HOPG), graphite powders, graphite rods, graphite foil, or graphite flakes are used as the working electrode. In order to provide a conducting surface, graphite flakes can be adhered to conductive carbon tapes to form the working electrode. They can also be adhered to a tungsten wire by a silver pad Pt wire, mesh, plate, or rods or graphite are most frequently used as counter electrodes. The experimental setup is usually arranged. The working electrode and counter electrode are immersed in the electrolyte with a certain distance kept between them. A positive or negative voltage is applied to the graphite working electrode depending on the exfoliation mechanism desired. Apart from this common setup, Liu et al. used two pencil cores as graphite sources, using graphite as both the anode and cathode. An alternating bias between +10 V and -10 V was applied to the two pencil electrodes to exfoliate them. Although the setup was more efficient and had higher exfoliation rate than using one graphite electrode, the product was expected to be more inhomogeneous with broader thickness and size distribution. Abdelkader et al. reported a novel setup for a continuous electrochemical exfoliation process in which 0.5-2 gm of few-layer graphene can be produced per hour. In their setup, the graphite electrode was inserted slowly from the bottom of the cell with only the graphite in contact with the electrolyte being exfoliated. The well-exfoliated few-layer graphene sheets floated to the top of the electrolyte and flowed out of the cell, whereas the partially exfoliated graphite would remain at the bottom and be further exfoliated. Presently, the main challenge lies in the effective and uninterrupted electrical delivery to each graphene

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layer in the graphite, and is a pressing need to develop a commercially scalable and controllable setup. Graphene is a mono atomic two-dimensional structure of carbon atoms. It is an allotrope of carbon and it can be considered as the building block of many carbon allotropes. Many layers of graphene can be stacked together to form. Raman Spectra Raman spectroscopy provides information about molecular vibrations that can be used for sample identification and quantization. The technique involves shining a monochromatic light source (i.e. laser) on a sample and detecting the scattered light. The majority of the scattered light is of the same frequency as the excitation source. Raman spectroscopy can be used for both qualitative and quantitative applications.

Electrochemical Process

The electrochemical setup that is used to exfoliate graphite normally contains the following elements: a graphite electrode, Pt wire, electrolyte, and power supply. Typically, highly orientated pyrolytic graphite (HOPG), graphite rods, graphite flakes are used as the working electrode. In order to provide a conducting surface, graphite flakes can be adhered to conductive carbon tapes to form the working electrode. The experimental setup is usually. The graphite electrode and Pt wire are immersed into the electrolyte with a certain distance kept between them. Used one pencil cores as graphite sources, using graphite as cathode and pt wire as an anode. An alternating bias between +10 V and -10 V was applied to the pencil electrodes and pt wire to exfoliate them. The product was expected to be more in homogeneous with broader thickness and size distribution for continuous electrochemical exfoliation process in which 0.5-2 gm of few-layer graphene can be produced per hour. The well-exfoliated few-layer graphene sheets floated to the top of the electrolyte and flowed out of the cell, whereas the partially exfoliated graphite would remain at the bottom and be further exfoliated.

Raman Spectra

Raman spectroscopy provides information about molecular vibrations that can be used for sample identification and quantization. The technique involves shining a monochromatic light source (i.e. laser) on a sample and detecting the scattered light. The majority of the scattered light is of the same frequency as the excitation source. Raman spectroscopy can be used for both qualitative and quantitative applications. The spectra are very specific, and chemical identifications can be performed by using search algorithms against digital databases. As in infrared spectroscopy, band areas are proportional to concentration, making Raman amenable to quantitative analysis. In fact, because Raman bands are inherently sharper than their infrared counterparts, isolated bands are often present in the spectrum for more straightforward quantitative analysis (**FIG. 1**).

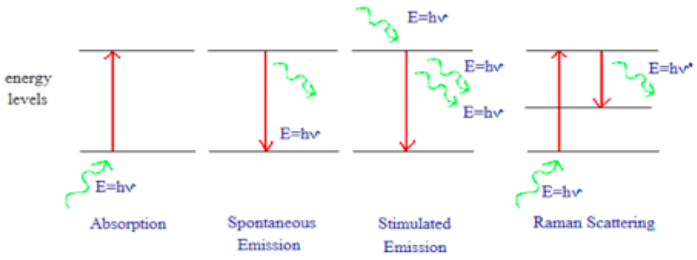

FIG. 1. **Basic of Raman spectroscopy.**

The process of graphene synthesis

The various materials used as per requirement and the method employed to synthesis the graphene has been discussed. The procedure is expressed in detail in section and an outlook of the process followed is presented in the form of the flow chart (**FIG. 2**).

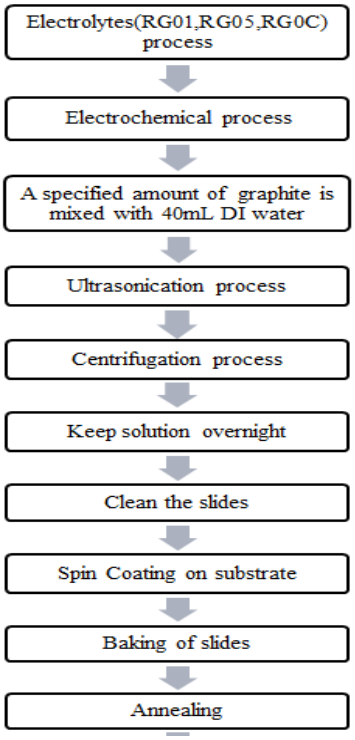

FIG. 2. **The process of graphene synthesis.**

Result and Discussion

FIG. 3 shows the XRD results of the Sample, G0-C. The peaks in the graph shown the crystalline nature of the sample and the **TABLE 1** below shows the grain size calculations.

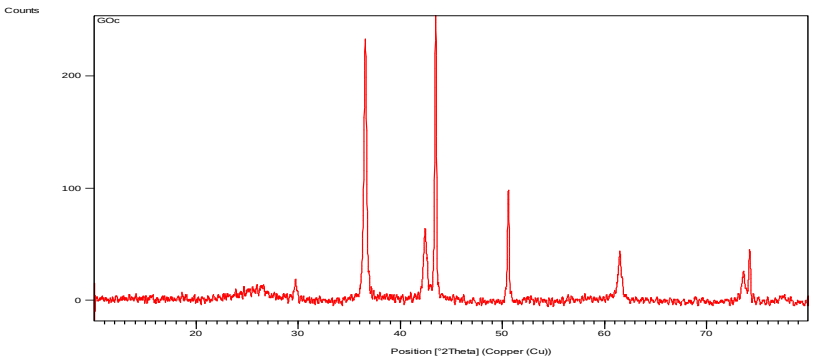

FIG. 3. **XRD results of a sample of GO-C.**

TABLE 1. **Peak list of sample 5: GO-C.**

S. No	Pos. [°2Th]	FWHM [°2Th]	Rel. Int. [%]	D (grain size in nm)
1.	26.5053	0.384	4.79	23.118127
2.	36.592	0.384	93.85	25.766887
3.	43.4395	0.384	100.00	28.492259
4.	50.5699	0.384	37.77	32.572929

According to the result of GO-C is best than the other samples and the best grain size is 28.492259. Results of all the samples are good but Sample 5: GOC is best than the other samples [1-8].

Conclusion

The main advantage of electrochemical methods is in utilizing a relatively clean physiochemical process to not only exfoliate graphite but to also produce high value-added functional graphene. In fact, the typically-used chemical oxidant for graphite oxide production, potassium permanganate, commercially produced via electrolytic oxidation of potassium manganate. Hence, in theory, the direct electrolytic oxidation of graphite could be a more cost-effective method without creating the need to remove spent chemical oxidant. Currently, there are a greater number of studies focused on the electrochemical graphite exfoliation. However, the former is still not a fully optimized and controllable process, as evident in the typical production of few-layered graphene instead of complete exfoliation to single-layered graphene. In addition, the process has to be scalable and cost-effective in order to break the bottlenecks in graphene production. The XRD results obtained confirm the exfoliation of graphite. Formation of graphene is due to an increase in the distance between the layers of graphene as a result of intercalation. So we conclude after the result of all samples that result of GO-C is best and grain size is 28.492259 nm.

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