



Short Review Paper

Synthesis of Graphene Oxide by Hummer's Method and its Physical Applications

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Abstract

Graphene is newly invented material which is very important in condensed matter physics and material science. Graphene has high crystal and electronics properties. Graphene is a two dimensional crystal made up of only carbon atoms. Graphene does not requires any proof of its importance because of its electronic spectrum, grapheme is emerging as a new standard of "relativistic" condensed matter physics. It is 100 times stronger than steel. This review deals with the synthesis methods, characterizations and applications.

Keywords: Graphene, Hummer's method, Physical applications.

Introduction

Graphene is the alternative path to graphene oxide (GO) which is recently invented¹. It is hydrophilic because of the high affinity to water molecules, and can be easily dissolved in solvents and water. It is also used in microelectronics². There are number of applications of graphene such as hydrogen storage and solar cell³, paper like materials⁴, conductive films which are transparent⁵, electro mechanical devices⁶, polymer composite⁷ and in the biological field that is in biomedicine⁸ etc. Graphene is the two-dimensional form of carbon. Graphene is the best-studied carbon allotrope⁹. Due to its high thermal conductivity graphene has been considered as an excellent suitable catalytic support. It has good mechanical strength, electrical conductivity, and large specific surface area (SSA). We can use graphene based metal and metal oxide in electro catalysis, and in carbon-carbon bond formation.

Structure of Graphene

Graphene has been discovered in 2004. It is a two dimensional crystal material as shown in Figure-1. It is regarded as the elementary structure of carbon-based materials¹⁰.

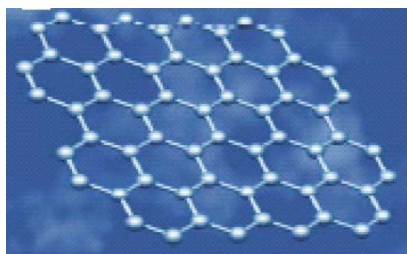


Figure-1
Structure of graphene

The thickness of the graphene is 0.34 nm. Graphene have larger specific surface area¹¹. The thermal conductivity of the graphene is outstanding as well as high speed electron mobility (15000 cm²/(V.s)) at room temperature. The mechanical stress of the graphene is 1060 gpa¹², while the density is low. Graphene was first published in October 2004, the amount of papers relevant to graphene was more. Graphene also has many special characteristics, such as bipolar super current, absence of Anderson localization, and anomalous quantum Hall effect¹³. The unique characteristics of the graphene provides a new platform for research. We can observe many phenomena in graphene. Graphene is found to be the strongest material ever measured. It is found that the graphene is harder than diamond in density and 100 times stronger than that of the best steels¹⁴.

Review of Literature

Brodie reported graphene firstly by in 1859¹⁵. By adding KClO₃ in a single addition to a slurry of graphite in fuming HNO₃ the oxidation process was performed. The C/H/O ratio of the oxidation product was determined to be 2.19/1.00/0.80, which is the typical composition of graphite oxide¹⁶. In 1998 Staudenmaier *et al.*¹⁷ found and modified the Brodie method by using HNO₃ and concentrated H₂SO₄ as the oxidizing agents. Over a period of one week in the procedure the KClO₃ was added slowly and carefully. Hummers *et al.* In 1958 found that graphene can be prepared using Hummer's method¹⁸ "improved Hummers method"¹⁹ was reported by Marcano *et al.* In 2010.

Methods for synthesis of Graphene

There are various methods for synthesis of graphene as follows.

Mechanical exfoliation method: This is the simplest method known and can be employed in any laboratory to produce graphene sheets from few micrometers to 0.1mm. The materials required for preparing graphene sheets are scotch tape and thick graphite bar or sheet having high purity. To detach a layer of graphene from the graphite bar the scotch tape has been repeatedly used. After a layer of graphene is attached to the tap it is transferred mechanically on to the substrate like silicon, glass etc. The main disadvantage of this method is it is time consuming and does not produce uniform quality graphene films. The graphene layer developed by this method are very thick²⁰.

Chemical exfoliation method: Mechanical exfoliation method and chemical exfoliation method are similar to each other except that here the graphite is immersed in mixture sulfuric acid and nitric acid in order to increase the spacing between different layers graphene present in the graphite material, so that these can be extracted easily by adding layers of atoms between graphite layers. Facilitates easy peel off graphene layers and process is termed as intercalation. Other technique such as ultrasonic heating and acid treatment are generally used to produce nano ribbons also known as thin flat graphene wires²⁰.

Epitaxial growth on sic: The other way to prepare graphene is to heat silicon carbide (sic) to high temperature generally more than 1100°C. By this process epitaxial graphene can be produce. Carbon atoms are left to form graphene the sublimation of the Si atom occurs from the surface in this method. The face of silicon or carbon terminated for graphene formation²⁰.

Hummer's Method: Hummer's method is widely used method for preparation of graphene. This method have several advantages that are we can produce graphene in the laboratory easily and the quantity of the output product is more. In Hummer's method first graphite powder is mixed with acid and graphite oxide is formed. Then graphite oxide is converted into graphene oxide using sonication. In a typical experiment 2g of graphite powder is added to 46 ml of sulfuric acid in an ice bath. Then, 6 grams of potassium permanganate is added gradually over a period of 30 minutes with constant stirring in a round bottom flask. The mixture is stirred for 2 hours approximately and 100 ml of water is added. Finally, 280 ml of hydrogen peroxide is added and the reaction is stopped. The mixture is filtered and washed with HCL and dried. This gives the graphite oxide, sonicating graphite oxide in distilled water gives graphene oxide which is converted to reduced graphene oxide (RGO) by using hydrazine hydrate. Many properties of RGO are similar to graphene but due to some defects it do not produce hexagonal graphene's structure. The reaction involved in the process is exothermic in nature and releasing lot of energy therefore, process needs to handle carefully²⁰.

Electrochemical Exfoliation method: Now a days electrochemical method is widely used to produce graphene directly from graphite source. In a typical experiment setup,

silver wire is used as a cathod and graphite rod is used as an anode both anode and cathode are immersed in the electrolytic solution of sulfuric acid so that the minimum distance between two is 5.0 cm. A positive voltage of +10 is applied between anode and cathode. After few minutes, the graphene nanosheets starts accumulating in the electrolyte solution from the graphite rod. The graphene nanosheets from the electrolyte solution is filtered and used for further study²⁰.

Applications

Graphene have a large range of applications which are discussed below.

Electronic devices: Graphene-based polymer composites can be used as electrodes for organic solar cells²¹ and liquid crystal devices it is possible because of the high electrical properties²² of graphene.

Sensors: Due to the large specific area and low noise, graphene is the capable candidate to detect a variety of molecules such as gases to biomolecules²³.

Energy storage: As graphene have a large range of applications, energy storage is one of the applications of graphene.

Biomedical applications: There are many applications of graphene. One such application is in the biomedical. In 2008 the first report on biomedical application of graphene was found²². Some applications in biomedical field are given below.

Drug delivery: Because of strong interaction exists between hydrophobic drugs and aromatic regions of graphene sheets graphene can be for drug delivery. For intracellular drug delivery Wen *et al.* Found redox-responsive PEG mechanism in pegylated nano graphene oxide²⁴.

Gene delivery: For the treatment of inborn and acquired diseases the gene therapy is a great tool.

Cancer therapy: For the treatment of cancer graphene and graphene-based polymer nanocomposites has been used. Negligible in vitro toxicity to various cell lines exhibited by PEG functionalized GO (NGO-PEG) reported Lin *et al.*²². Markovic *et al.* Found that graphene-mediated photothermal killing of cancer cells involve mitochondrial membrane depolarization and oxidative stress, resulting in the necrotic cell death²².

Conclusion

Graphene is transparent, thin, flexible, and mechanically strong, conductor. The conductivity of graphene can be increased by chemical doping. Graphene have high mobility by which material can be used for high frequency applications in electronics. Now a days it is possible to make large sheets of

graphene. In satellites and aircraft Grapheme based composite materials could also be used.

References

1. Stankovich et.al. (2006). Graphene-based composite materials. *nature*, 422, 282-286.
2. Eda G. and Fanchini G. et.al. Large-area ultrathin films of reduce graphene oxide As a transparent and flexible electronic material. *Nature nanotechnology*, 3, 270-274.
3. Wang X., Zhi L and Mullen K. (2007). Transparent, conductive graphene electrodes for Dyesensitized solar cells. *Nano letters*, 8, 323-327.
4. Dikin D.A., Stankovich S., Zimney E.J. and Piner R.D. et al. (2007). Preparation and characterization Of graphene oxide paper. *nature*, 448, 457-460.
5. Park S., Mohanty N. and Sui D. et.al. (2010). Flexible, magnetic and electrically conductive graphene /Fe₃O₄ Paper and its application for magnetic controlled switches. *J. Phys. Chem. C*, 114(41), 17465- 174771.
6. Bunch J.S., van der Zande A.M., Verbridge S.S. and Frank I.W. et.al. (2007). Electromechanical Resonators from Graphene sheets. *Science*, 315(5811), 490-493.
7. Eda G. and Chhowalla M. (2009). Graphene-based Composite Thin Films for Electronics. 9:814-818.
8. Mohanty N. and Berry V. (2008). Graphene-based singal-bacterium resolution biodevice and DNA transistor: Interfacing graphene derivative with nanoscale and microscale Biocomponents. *Nano Letters*, 8, 4469-4476.
9. Katsnelson Mikhail (2007). Graphene: carbon in two dimensions. *Materials Today*, 10(1-2), 20-27.
10. Geim A.K. and Novoselov K.S. (2007). The rise of grapheme. *Nature materials*, 3, 183-191.
11. Chae H.K., Siberio-Pérez D.Y. and Kim J. et.al. (2004). A route to high surface area, porosity and Inclusion of large molecules in crystals. *Nature*, (6974), 523-527.
12. Schadler L.S. and Giannaris S.C. et.al. (1998). Load transfer in carbon nanotube epoxy composites. *Applied Physics Letters*, 26, 3842-3484.
13. Cao KE and Zimao Y. (2008). Outstanding Properties and Applications Potentials of grapheme(j). *Materials Review*, China Academic Journal Electronic Publishing House, www.cnki.net
14. Liang-Xu DONG and Qiang CHEN (2010). Properties, synthesis, and characterization of grapheme. *Sci.China*, 1, 45-51.
15. Brodie B.C. (1859). On the atomic weight of graphite. *Philos. Trans. R. Soc. Lond.*, 149, 249-259.
16. Young R.J, Kinloch I.A. and Igong et.al. (2012). The mechanics of graphenenanocomposites: A Review. *Composites Science and Technology*, 72(12), 1459-1476.
17. Staudenmaier L.V. (1898). Verfahren zur darstellung der graphitsäure. *Ber.Dtsch. Chem. Ges.*, 31, 1481-1487.
18. Hummers W.S. and Offeman R.E. (1958). Preparation of graphitic oxide. *J. Am. Chem. Soc.*, 80, 1339-1340.
19. Marcano D.C., Kosynkin D.V., Berlin J.M., Sinitiskii A. and Sun Z. et.al. (2010). Improved Syntesis of graphene oxide. *ACS Nano*, 4, 4806-4814. Oxidenanoribbons from Multiwalled carbon nanotubes. *ACS Nano*, 4, 2059-2069.
20. Singh Randhir and kumar Dinesh et.al. (2015). Grphene :potential material for nanoelectronics applications. *IJPAP*, 53(8), 501-513
21. Li W., Xu Z., Chen L., Shan M. and xtian et al. (2014). A facile method to produce grapheme Oxide-g-poly (L-lactic acid) as a promising reinforcement for PLLA nanocomposites. *Chem. Eng. J.*, 237, 291-299.
22. Das Tapan K and Prusty Smita (2013). Graphene-Based Polymer Composites and Their Applications. *Polymer-Plastic Technology and Engineering*, 52(4), 319-331.
23. Yong Y.C. and Dong X.C. et.al. (2012). Macroporous and monolithic anode based on polyaniline Hybridizedthree-dimensional graphene for high-performance microbial fuel cells. *ACS Nano*, 6, 2394-2400.
24. Park T.G, Jeong J.H and Kim S.W. (2006). Current status of polymeric gene delivery Systems. *Adv. Drug Deliv. Rev*, 58(4), 467-486.