Journal of Composites and Compounds 1 (2019) 22-26

Available online at www.jourcc.com

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Journal of Composites and Compounds

Preparation of graphene nanolayers through surfactant-assisted pure shear milling method

Asghar Kazemzadeh[a], Mohammad Ali Meshkat[b], Houman Kazemzadeh[c], Mostafa Moradi[d], Reza Bahrami[e]*

Rasul Pouriamanesh[e]

[a] *Department of Semiconductors, Materials and Energy Research Center, Karaj, Iran*
[b] *Department of Nanomaterial, Materials and Energy Research Center, Karaj, Iran*
[c] *Tehran University of Medical Science, Tehran, Iran*
[d] *Department of Materials Science & Engineering, Sharif University of Technology, Tehran, Iran*
[e] *Department of Materials and Metallurgical Engineering, Amirkabir University of Technology, Tehran, Iran*

ABSTRACT

In this study, graphite powder was used to prepare few-layer graphene sheets through shear milling. During the process, graphite was well dispersed in double distilled water as a lubricant and sodium dodecylsulfate (SDS), followed by shaking and milling under low energy. The exerted sheer force led to the continuous delamination of graphene flakes. The microstructural investigation was performed by SEM. In addition, the energy-dispersive X-ray spectroscopy (EDS) analysis was performed to determine distinct levels of carbon in different fragments of graphite. The ultrathin multilayer structure of graphite was successfully obtained using the surfactant of SDS, which can lead to the production of molecularly thin sheets by mechanical peeling. Moreover, it was found that this synthesis method has some advantages, including cost-effectiveness and ease in performance for producing many graphene nanolayers.

Peer review under responsibility of JCC Research Group.

ARTICLE INFORMATION

Article history:
Received 10 July 2019
Received in revised form 21 November 2019
Accepted 27 December 2019

Keywords:
Ultrathin
Multilayer Structure
Graphene nanolayer
Sodium-dodecylsulfate

1. Introduction

These days, nanomaterials, due to their highly attractive properties, have been broadly used in various types of applications, such as life sciences, the environment, information technology, etc. [1-5]. There are various methods to produce nano-sized materials, including sol-gel [6-9], ball-mill [10-13], co-precipitation [5], self-propagating high-temperature synthesis [14, 15], electrospinning [4, 16], etc.

Recently, the application of graphene has been gained increasing attention by researchers because of its unique properties [17-21]. Graphene, a single layer of graphite, is a basic block of sp^2-bonded carbon for graphitic materials such as graphite [22-25], fullerene [26-28] and carbon nanotubes [29-38], which has both unique mechanical and physical properties making it a promising material for applications in nanotechnology [39-44]. A stable graphene sheet was discovered by Novoselov and Geim (2004) for the first time [45].

The value of the thermal conductivity of graphene is even more than diamond, and it has a high potential in increasing the convective heat transferring ability of a nanofluid [46-48]. The usage of graphene into the lubricant improves its potential for heat removal from a system, because of the significant role of graphene in increasing the amount of thermal conductivity. Recently, graphene has been introduced as the thinnest solid lubricant by some researchers with a mechanism of superlubricating, which could be confirmed by atomic force microscopy (AFM) [49, 50]. Graphene has been introduced by Bermen et al. [51], as a promising candidate for using in steel lubricants. The tribological properties of graphene have been investigated, and the results have shown excellent properties of graphene compared to the nanoparticle of graphite or carbon nanotube (CNT) [52].

It can be used in many applications such as solar cells [53, 54], hydrogen storage [7, 55], sensors [56-58], detectors [59], transistors [60] and other electronic devices in many applications due to its good electrical property [19, 21, 61-68], which makes it a good candidate to be used in lots of composites [69, 70]. Therefore, recently, many investigations have been focused on graphene and graphene nanolayer by numerous physicists, chemists, and material scientists [71].

Producing graphene at a large scale with uniform thickness has been one of the most important subjects for many researchers and scientists [72, 73]. Many controlled synthesis routes, viz. chemical vapor deposition (CVD), laser reduction, and wet chemical routes have been proposed to prepare a large amount of graphene [63, 74-78], with suitable physicochemical properties [79-81]. However, these processes should

* *Corresponding author: Asghar Kazemzadeh; E-mail: ASG642001@Yahoo.com*
DOR: 20.1001.1.26765837.2019.1.1.4.3
https://doi.org/10.29252/jcc.1.1.4

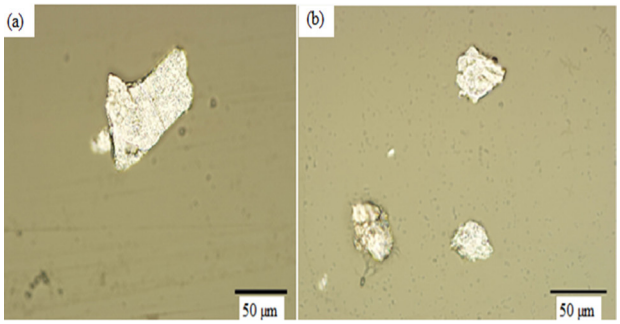

Fig. 1. Optical microscopy images of graphite particles after 60 hours of shear milling (a) with SDS and (b) without SDS.

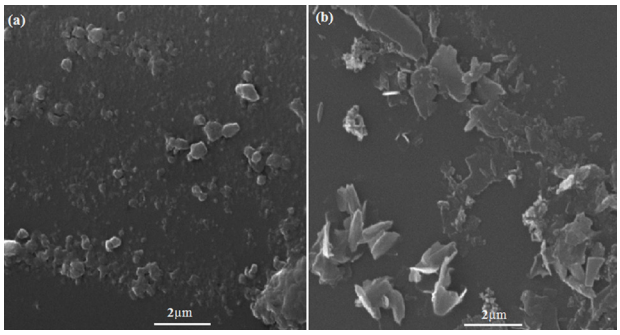

Fig. 2. SEM micrographs of graphite particles after 60 hours milling (a) without SDS and (b) with SDS.

be low cost and simple e.g. mechanical exfoliation of graphite, producing a large area graphene sheet.

Several methods have been used for producing layered materials, like graphene, with no unwanted defect or functionalization. Meanwhile, mechanical exfoliation is the best option among these methods that is very simple procedure.

The delamination occurs via applied shear and compressive forces on the particles, in which the shear force should overcome the van der walls force existing between the sheets. Accordingly, there are several reports for preparing graphite nano-sheets or graphene by graphite exfoliation through three roll milling or shear ball milling [77, 82, 83].

However, all these studies have used graphene powders in the form of solid additive, in which the obtained suspension had low stability. Meanwhile, commercial fluids require high stability for a long time [84]. To overcome this limitation, functionalized graphene sheets were prepared e.g., aromatic or long alkyl chain functionalized graphene sheets dispersed in non-polar oil medium [85, 86]. Zhang et al. [87] used poly-alphaolefin-9 (PAO) and dispersed oleic acid-modified graphene sheets in it. Although long-term storage stability is not achieved, graphene showed good wear resistance.

One of the major challenges in the graphene research field is how to count the number of atomic layers. Although optical microscopy can show graphene with certain layers on a silicon substrate, it is difficult to identify the number of layers of graphene accurately [62]. Therefore, although there are many reports about graphene nanolayers, insufficient reports have been issued about the preparation of the nanolayers via shear milling procedure.

In order to apply effective exfoliation, the stirred-media bead milling system can be utilized that has more shear forces other than impact one. Peukert et al. [75] used this method to prepare scalable graphene in a water medium along with an ionic surfactant.

Thus, in this paper, the synthesis of nanolayers via shear milling procedure is presented instead of the ball milling process to achieve nanolayer graphene flakes and stable dispersion of graphene in the medium. Scanning electron microscope (SEM) and optical microscopy (OM) were also used to characterize thin flakes on Si substrate. To provide well-dispersed graphite in water, sodium dodecyl sulfate (SDS) was used as the surfactant.

2. Experiment Procedure

High purity graphite powder with an average particle size of 200 µm and SDS were purchased from Merck Co. (Germany). Two SDS-containing and SDS-free mixtures with 0.25 gr graphite and 200 ml deionized water were separately prepared and a flat alumina disc with 2cm diameter and 4mm thickness was put in the flask of each mixture. The flasks were shaken with orbital flask shaker for 60 hours at a speed of 380 rpm to keep the disc flat. Then, the samples were spin-coated on the Si substrate and dried at 80 °C for 2 h.

Raman spectra analysis was done with SEKI 750 Raman analyzer to investigate the sample in argon ion laser (514.5 nm line). The JEOL 2100 electron microscope was used for TEM analysis of samples on a carbon-coated copper grid. The optical microscopy (OM) and SEM (model: LEO 1450 VP) images of the samples were captured, and data of energy-dispersive EDX (model X-MAX) for different areas were collected.

3. Results and Discussion

Since graphite and graphene flakes are hydrophobic and tend to agglomerate in water, an ionic surfactant like SDS can hinder the coagulation and restacking of particles. The resulting suspension of graphite with SDS is stable in water to carry out the rest of the process. During delamination in the stirring media mill, the transferred energy from the grinding media causes sheets to be fractured, and mostly small fragments of graphite can be present in the ultimate suspension. During milling, SDS is adsorbed on the surface of the particles and creates a strong repulsive force that prevents the agglomeration and restacking of delaminated sheets. The optical microscopy results, which are presented in Fig. 1, show that the particle size of graphite flakes decreased to less than 50 µm in both mixtures, after mechanical treatment for 60 h. Fig. 2 represents the SEM images of graphite fragments on the surface of the silicon substrate. The presence of SDS leads to a significant change in the shape of particles after shear milling for 60 h (Fig. 2). It is worth noting that water allows the graphene planes to slip easily and SDS prevents the agglomeration of the particles and keeps the layers apart from each other. Applying low energy through shear milling of the graphite particles induces shear on graphene layers without causing high crystal defects [63].

As can be seen in Fig. 2(b), the particles obtained after 60 hours of milling have flak-like shape. Fig. 3 illustrates the SEM micrographs of thin flaky graphite. The accurate thicknesses of the thin sheets are difficult to estimate. Three areas are highlighted via arrows on in Fig. 3(a).

Energy-dispersive X-ray analysis (EDX) of point 2 is shown in Fig. 3(b), which contains C, O, and Si as well as a negligible amount of Na element. This proves the presence of carbon and silicon existing in the graphene and substrate, respectively. Fig. 4 shows various intensities of carbon at mentioned areas (Fig. 3(a)) after deposition on a silicon substrate. It is evident that the minimum intensity of C level corresponds to the layer with a minimum contrast (point 1), which refers to the very thin layers of graphene.

The transmission electron microscope (TEM) image of nanolayers is shown in Fig. 4. It can be seen that several dark layers are superimposed on each other. This denotes that several graphene nanolayers are stacked.

In order to distinguish the graphene from graphite and determine the number of layers, Raman spectroscopy was performed. D-band about

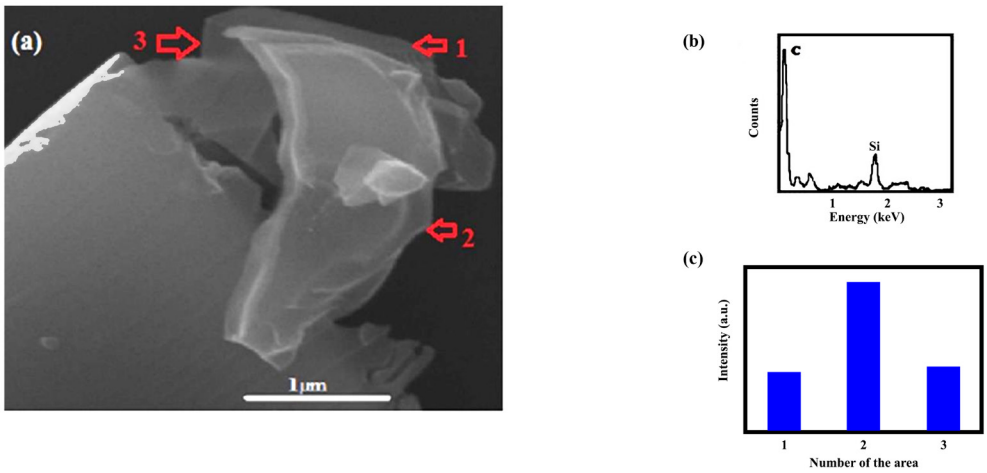

Fig. 3. (a) SEM micrographs of thin flaky graphite, (b) EDX spectrum of point 2, and (c) carbon intensity at each point .

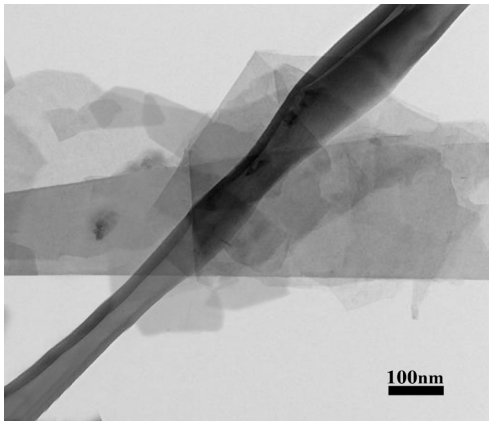

Fig. 4. TEM micrograph of graphene nanolayers.

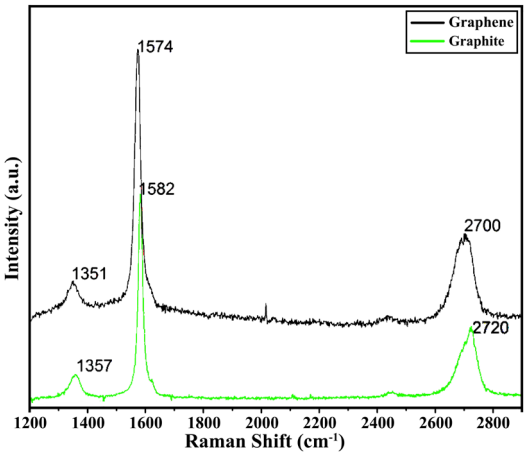

Fig. 5. Raman spectra of dispersion after being processed.

1350 cm^{-1}, G-band about 1580 cm^{-1} and 2D-band about 2700 cm^{-1} correspond to graphitic materials. Fig. 4 illustrates the Raman spectra of the materials. The G-band at 1581 and 1573 cm^{-1}, and D-band at 1356 and 1352 cm^{-1} for graphite and graphene samples, respectively, prove almost the graphitic nature of the samples. In addition, the ordered and increased density of sp2 carbon is clear for the milled sample. It is reported that the 2D symmetry, as well as its full width at half maximum (FWHM), can distinguish the graphene and bulk graphite [88]. Furthermore, the 2720 cm^{-1} is ascribed to a 2D peak for graphite that depicts the formation of few layers as well as the delamination of a few layers or monolayered graphene.

Besides, a single 2D peak was found in many places in the diluted film. On the other hand, the graphitic nature of the peak was also found in some places. Thus, it can be concluded that both multilayers and single-layered graphene sheets are formed.

4. Conclusions

Multilayer graphene sheets were synthesized by simple surfactant-assisted pure shear milling at ambient temperature. Water and SDS would accelerate the peeling-off process, and the SDS ionic surfactant hinders the agglomeration of graphite flakes. Optical microscopy showed the size reduction caused by mechanical energy. In addition, SEM and TEM images showed that the grinding media transferred enough energy to overcome the Van der Waals forces among the graphene sheets. The thickness of the achieved multilayer graphene sheet was estimated to be nano-sized.

Acknowledgment

The author is grateful to the materials and energy research center for financial support and fellowship.

Conflict of Interest

All authors declare no conflicts of interest in this paper.

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