# Exfoliated Graphite/Graphite Nanosheet-Epoxy Nanocomposites Revealed from the View Point of Synthesis Bottleneck

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**Abstract.** *This brief work illustrates the comparison between exfoliated graphite and graphite nanosheet reinforced polymeric nanocomposite. It involves exfoliation of natural graphite to form exfoliated graphite by intercalation and thermal exposure. Highly porous exfoliated graphite was mixed with epoxy to synthesize nanocomposite with the help of high shear-strain rate by three-roll mill. The velocity difference between roll induces high shear-strain rate in the rolls gap for better mixing. It creates a shear stress in the viscous mixture to disperse the exfoliated graphite in the epoxy and tear apart exfoliated graphite simultaneously so as to get graphite nanosheets. The multiple passes of this mixture at changing roll gap eventually assist in mixing and tearing apart of exfoliated graphite. Addition of hardener to this mixture, followed by oven curing and room temperature curing leads to polymer based nanocomposite. During this work, EG-epoxy and GNS-epoxy nanocomposites were synthesized and evaluated by scanning electron micrography studies. Finally, this paper illustrates the issues related to synthesis of exfoliated graphite/graphite nanosheets-epoxy nanocomposite. These issues are related to (a) Intercalation, (b) Exfoliation, and (c) Mixing. Each issue is discussed and illustrated.*

## Keywords:

Exfoliated graphite, graphite nanosheets, nanocomposite, natural graphite, three roll mill

## 1. Introduction

Natural Graphite (NG) is one polymorph of the carbon. Analogous to carbon nanotube, which is a graphene in the form of seamless tube, graphite is a close stacking of graphene owed to the covalently bonded $SP^2$ hybridized carbon which imparts high strength to graphite. However, the weakly bonded graphene sheets can slide with respect to each other under normal loading condition, yielding slipperiness (softness) to graphite [1]. Composite materials are engineered and tailor-made materials obtained by combining two or more materials to acquire desired combination of properties for specific applications. A nanocomposite, on the other hand, is a composite system involving reinforcement having first, second, or third dimension(s) less than 100 nm [2-3]. It is strange that, the graphite, a softest mineral and a very slippery lubricant is a high strength component (reinforcement) in composites.

This paper illustrates the exfoliation of NG to form a worm shaped EG by the process of intercalation through a chemical route. Highly porous EG was mixed with epoxy to synthesize nanocomposite. The mixing of EG with an epoxy is very critical for the success of nanocomposite synthesis. Hence, a high shear-strain rate mixing was employed for nanocomposite synthesis. EG tear apart in the form of small pieces during mixing. Thus graphite nanosheets (GNS) or a bunch of GNS's can be obtained. In this paper, three-roll mill was used for mixing in which the velocity difference between roll induces high shear-strain rate in the roll gap. This shear-strain rate induces a shear stress in the viscous mixture to disperse EG in epoxy matrix. The multiple passes of this mixture eventually tear apart EG successively and helps in proper dispersion of EG/GNS in the matrix. Thus graphite nanosheet-epoxy nanocomposite was obtained. Similarly, EG-epoxy nanocomposite was also synthesized during this brief study. Finally, this paper illustrates the issues related to EG/GNS-epoxy nanocomposite with the aid of scanning electron micrographs. These issues are classified as issues related to (a) Intercalation, (b) Exfoliation, (c) Mixing, and (d) Nanocomposite synthesis. Each issue is discussed and illustrated in detail.

## 2. Synthesis of Exfoliated Graphite (EG)

When immersed in the solution of acid, NG forms an intercalation compound (IC) in which the host molecule/atom (acid) gets "sandwiched" between the available spaces in graphite layers. Most extensive work in graphite intercalation compounds (GICs) was reported by Dresselhaus et. al. [4]. Common intercalates are $H_2SO_4$, $FeCl_3$, K-THF, formic acid, etc. [4]. More than 100 reagents can be intercalated into graphite to form GICs [5]. Some other technique used for exfoliation of NG includes dry process which involves direct reaction of gaseous $SO_3$ with NG [6], and microwave irradiation of GIC (prepared by gaseous $SO_3$ intercalation through direct reaction) [7]. A review on exfoliation of graphite has given by Chung [8]. Typical exfoliation process employed during this work involves following steps: (a) Chemical treatment of NG with concentrated sulfuric acid and nitric acid, (b) Intercalation of $H_2SO_4$ into NG to form GIC, (d) Washing of GIC with water to maintain a pH value of solution between 5~6, (e) Oven drying of washed GIC, and (f) Thermal shock to dried GIC at 1000 °C for 10 seconds. The exfoliation process flow is schematically illustrated in Figure 1. It was reported [2] that exfoliation increases volume of GIC by 200-300 times leading to EG. The EG is highly porous material with extremely low density with worm like shape.

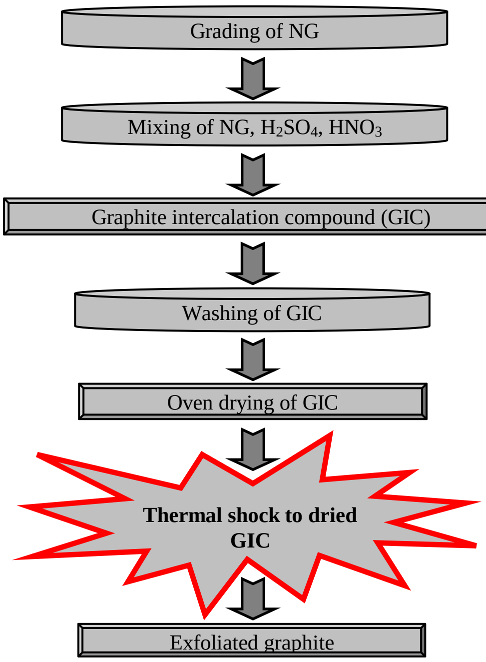
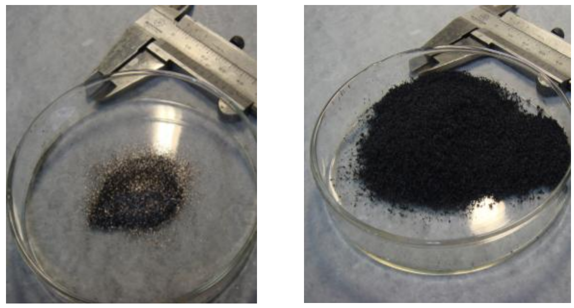



**Fig. 1** Process flow chart for exfoliated graphite (EG) synthesis

Flakes of NG as supplied by supplier were passed through sieve to get definite sized NG. During chemical treatment, NG was carefully mixed with a solution of 98 % sulfuric acid and 65 % nitric acid for 15 min. at room temperature. During the reaction, $SO_3$ molecules intercalate between graphite/graphene layers to form an intermediate compound termed as GIC. It was followed by filtering of GIC to remove unwanted sulfuric acid and nitric acid. The filtered GIC was then washed with pure water so as to reduce its pH to 5 ~ 6. The washed GIC was then dried in oven which was maintained at 100 ºC for removing unwanted water. Finally, GIC was exfoliated by subjecting it to 900 - 1000 ºC in an oven for a short duration of 10 seconds. Thermal shock thus induced to GIC exfoliates (pop up) GIC in the similar fashion as that of "Popcorn". The intercalate assist to built up excessively high pressure in GIC to exfoliate it in an explosive manner by rapid expansion. As mentioned above, exfoliation increases volume of GIC by 300 times. A volumetric comparison of 38.96 gm of NG and corresponding EG is shown in Figure 2. The EG is highly porous, layered material with extremely low density, and a worm like shape.



Natural graphite (NG) Exfoliated graphite (EG)

**Fig. 2** Volumetric comparison of 38.96 gm of NG and corresponding EG

## 3. Synthesis of Graphite Nanosheet (GNS) Epoxy Nanocomposite

For the preparation GNS-epoxy nanocomposite, first EG has to be converted to GNS. As the graphene layers in the EG are folded and opened, are weakly connected by Van der Waal forces, and are highly porous in nature with elliptical pores, hence can be separated by using various dispersion techniques. A high-speed mixing [9], sonication [10-12], and high shear strain rate [13, 14] are common dispersion techniques. A paper by Kuilla et. al. [16] reviews recent advances in modification of graphene and fabrication of graphene-based polymer nanocomposites owing to the ability to produce a dramatic improvement in properties of composite at very low reinforcement. The potential of using EG/GNS (nanoplatelets), as reinforcement in polypropylene (PP) that can produce multifunctional polymer composites was explored in [17]. Additionally, the properties of PP based nanocomposite where evaluated and compared with PAN based carbon fibre nanocomposites. In their work, Inagaki et. al. [18] calculated pore structure, distributions of area, length of major and minor axes of ellipsoidal pores, and aspect ratio of ellipsoidal pores by conducting micrography analysis of four EG samples (two-industrial samples and two-laboratory-made samples) with the aid of image processing. It was concluded that duration of thermal shock during exfoliation controls the degree of exfoliation.

In this work, high shear strain rate method was adopted to shear apart group of nanosheets from worm shaped EG while, simultaneously it was mixed with epoxy. The separated/dispersed pieces of EG were normally below 50 nm thickness, hence are termed as GNS. Figure 3 shows the arrangement for mixing of EG and epoxy with the help of 3-roll mill.

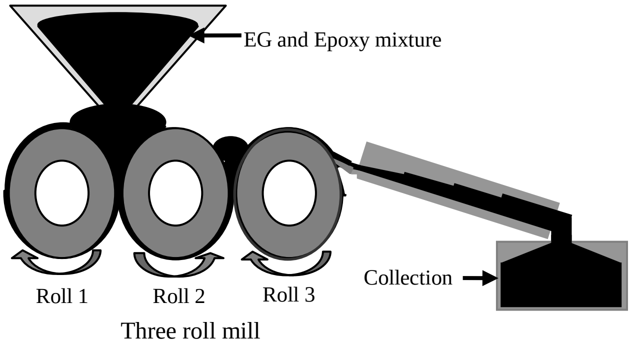



**Fig. 3** Schematic of nanocomposite mixing in 3-roll mill

As shown in Figure 3, EG was slowly mixed with epoxy in a definite proportion before pouring over the trough between roll 1 and roll 2. In 3-roll mill rolls are moving in opposite direction at variable speed. Normally, 1–5 % of EG by weight can be added for uniform mixing. The epoxy (matrix) is a thermosetting polymer which is having low viscosity and good wetting ability. A definite gap has been maintained between rolls; however this gap can also be varied as per requirement. Owing to opposite direction of rotation of rolls 1 and 2, EG-epoxy mixture passing through corresponding gap will be carried by both rolls. Initially, the mixture was accumulated above the trough between roll 1 and 2 in the form of a cylindrical segregation/feed. A pair of Teflon guide was used to confine the segregation/feed over a defined section of rolls and prevent it from flowing away towards the roll ends. The segregation became smaller and smaller with the time when the mixture gradually passed through the gap and finally being collected at the end of roll 3. The amount of mixture carried by roll 1 joins original feed, whereas the some portion of mixture carried by roll 2 circulates back to original feed and the rest of the feed gets transferred to roll 3 at gap between rolls 2 and 3. Owing to variable speed of rolls, they provide a high shear-strain rate at the roll gaps to tear apart the GNS from the mixture of EG and epoxy. It was simultaneously dispersed properly into the epoxy. The shear-strain rate at the roll gap is directly proportional to velocity difference of adjacent rolls and inversely proportional to the gap between rolls [2-3, 15]. Finally, the mixture was collected at the other side of the roll 3 using stainless steel scraper blade (called as apron). During this work, gap between rolls was set to 50, 30, 20, 10, and 5 μm. Initially, gap was set to 50 μm and mixture was poured over the trough between rolls 1 and 2. This procedure is repeated 4 times to ensure tearing of EG and its mixing with epoxy without changing the roll gap. The whole procedure is repeated for all above mentioned gaps so as to achieve successful tearing of EGs to GNSs.

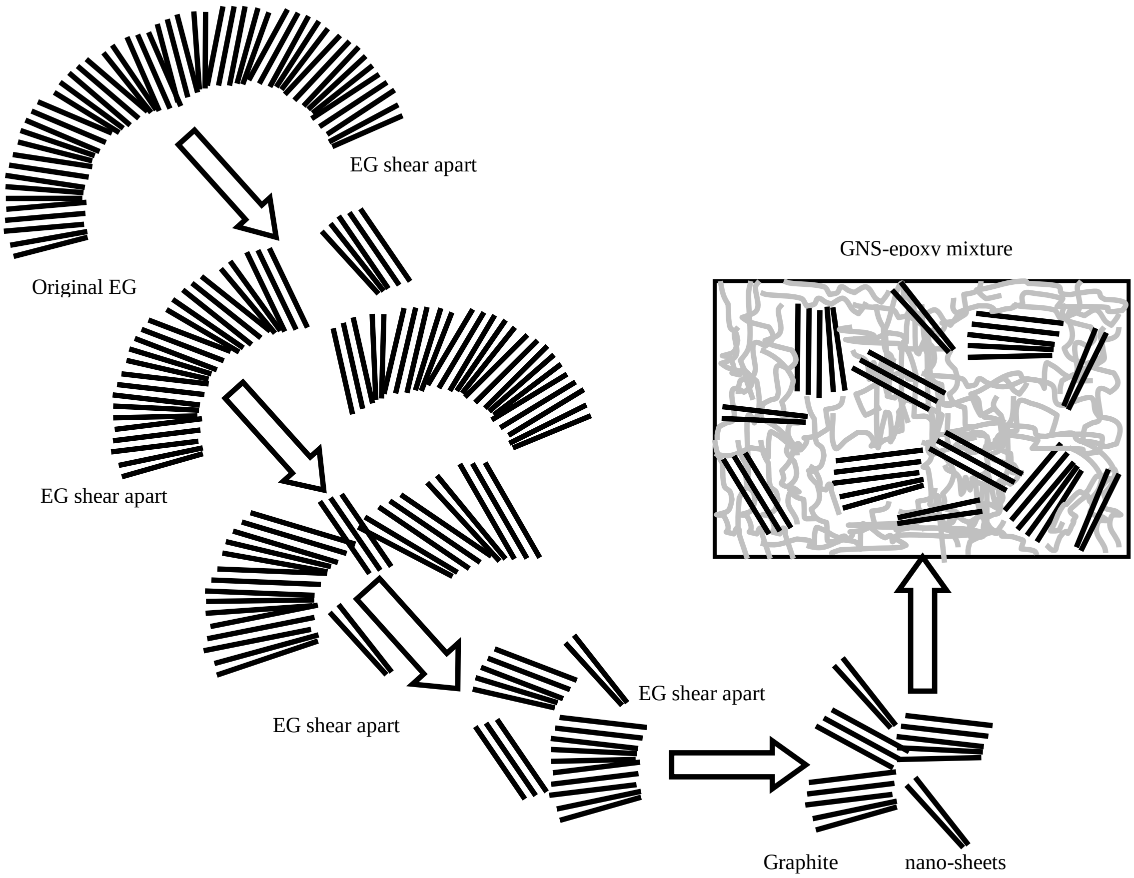



**Fig. 4** Schematic illustration of mechanism of EG tearing during mixing

The mechanism of tearing of EG during its passage through 3-roll mill and mixing with epoxy is illustrated in Figure 4. As seen in the Figure 4, the passage of EG at first roll gap bisect semicircular EG with some small segment of group of GNSs. During successive passages of broken EG throughout repetitive mixing, more fragmentation of EG occurs, which finally leads to formation of GNSs. While separated apart from the original EGs, newly formed GNSs were mixed uniformly in the epoxy matrix. It is to be noted that EGs are very porous. The individual nanosheets are joined (linked) together with each other by folded (sometimes curved) nanosheets. Thus one cannot avoid the possibility of crushing/closing of GNS or fragmented EG.

Finally, an epoxy-EG/GNS mixture was added in stoichiometric proportion with hardener/activator and mixed with hand stirring. During mixing, an epoxy monomer reacts with hardener monomers forming a covalent bond which ultimately results in highly cross linked polymer. It was then cured in an oven at 80 - 100 ºC for 24 hours. The curing does not harden the GNS-epoxy mixture, but help in escaping certain gases and air bubbles formed during chemical reaction between epoxy and hardener. Thus avoids the pore formation in the nanocomposite. Finally, the cured mixture was poured in a prepared mold and kept in rotation machine at 10 rpm for 70 min. at room temperature for final curing. Thus, the block of nanocomposite was prepared and sliced to definite sizes using diamond cutter for microscopic examination. Similarly, EG-epoxy nanocomposite was also prepared by above mentioned procedure. Typical nanocomposite block is shown in Figure 5.

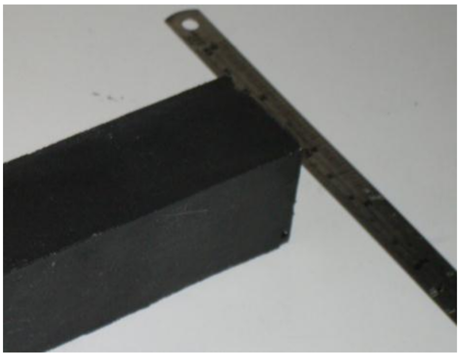
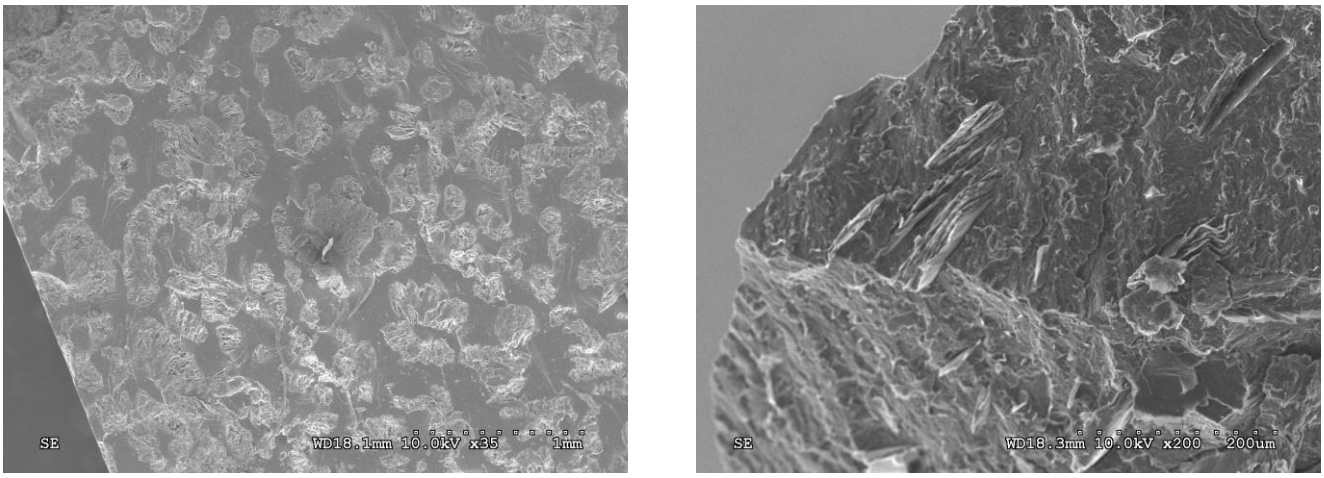


**Fig. 5** Typical nanocomposite block

## 4. Micrography of Nanocomposite

The microscopic study of nanocomposite was carried out using scanning electron microscope (SEM). The polished specimen of EG-epoxy nanocomposite and fractured specimen of GNS-epoxy nanocomposite were observed under SEM for comparison (Figures 6 (a) and (b)). As seen in Figure 6 (a), a worm shaped EG is properly dispersed in an epoxy matrix. It is also observed that the full EGs are rare in the matrix. Variable sized fragments are seen everywhere, even few graphene layered fragments are also seen which may be created during mixing of EG with epoxy matrix while passing through 3 roll mill gaps. The orientation of fractured EGs is also random. On the other hand, small flakes of GNS in epoxy are noticed inside fractured specimen of nanocomposite (Figure 6 (b)). The random orientations of these GNS reveal that there is uniform distribution of reinforcement in the matrix. The pull-out of some GNS is also observed which is revealed from the pull-out gaps and the overhanging GNS.



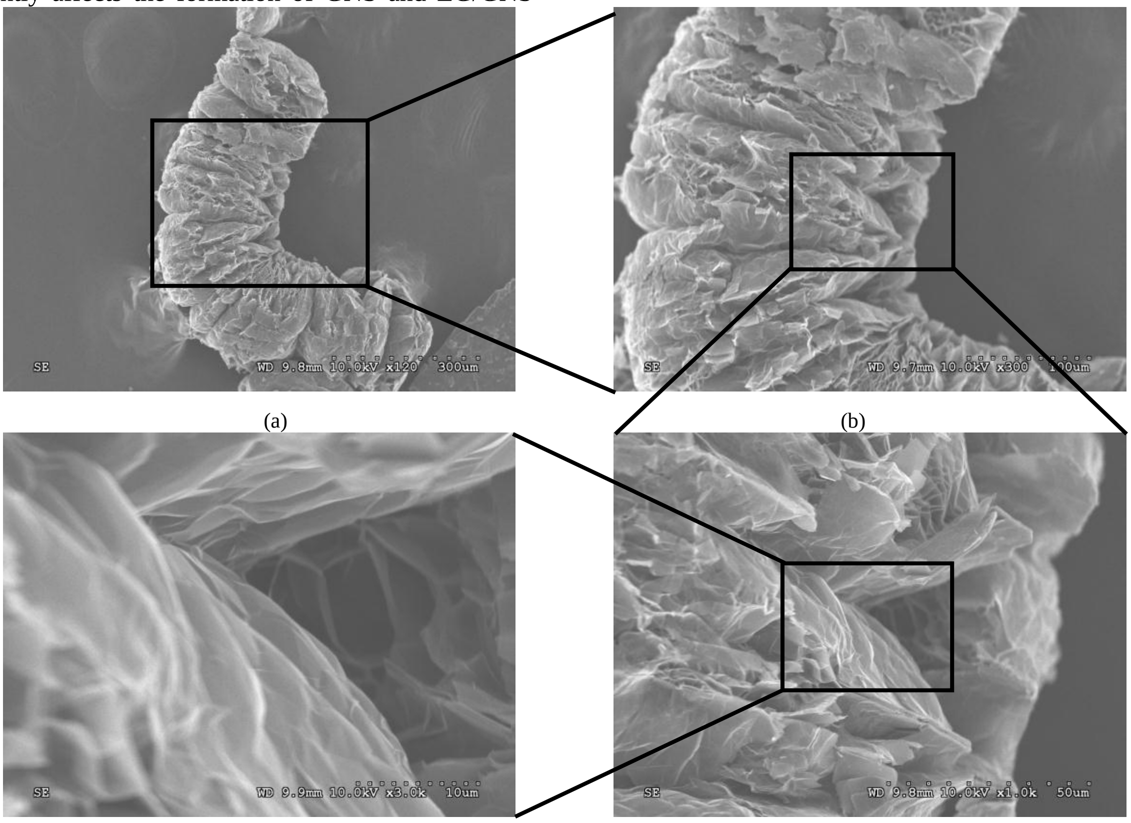

(a) (b)

**Fig. 6** SEM micrograph of (a) Polished EG-epoxy nanocomposite and (b) Fractured GNS-Epoxy nanocomposite

## 5. Results and Discussions

Issues during EG/GNS-epoxy nanocomposite synthesis can be credited to various process steps (Figure 1). For convenience they can be sub grouped as issues related to the exfoliation of NG and synthesis of EG/GNS-epoxy nanocomposite. In particular, the issues associated to exfoliation of NG can be related to (a) Time of intercalation, (b) pH value of GIC, (c) Drying of GIC, (d) Temperature during exfoliation, and (e) Duration of exfoliation (thermal shock). On contrary, the issues related to the EG/GNS-epoxy nanocomposite synthesis can be related to (a) Mixing of EG/GNS with epoxy, (b) Separation of EG to GNS during mixing, (c) Mixing of hardener to mixture of EG/GNS-epoxy, (d) Curing of composite, (e) Air bubble removal during curing, (f) Bonding between GNS and epoxy, (g) Dispersion of GNS in epoxy, and (i) Orientation of GNS in matrix. Some of these issues were discussed earlier [2]. This work attempts to explain some aforementioned issues with related micrographs.

The effect of issues related to exfoliation have detrimental effect on the synthesis of EG from NG, which subsequently affects the formation of GNS and EG/GNS-epoxy nanocomposite. As mentioned previously, intercalate induces explosive forces to expand NG, improper intercalation due to inappropriate intercalation time, pH of GIC, and drying of GIC affects exfoliation drastically which leads to the improper EG. In an ideal situation, a fully expandable EG is expected where some graphene layers completely separate apart from each other and some graphene layers form a bridge between separated graphene layers. It is presented in Figures 7 (a)-(d). A worm shaped EG is clearly seen in Figure 7 (a). Close look at Figures 7 (b)-(d) reveals expanded, curved, and folded graphene layers during exfoliation. One can observe ideally expected exfoliation (volume expansion) of NG in Figures 7 (a)-(d).

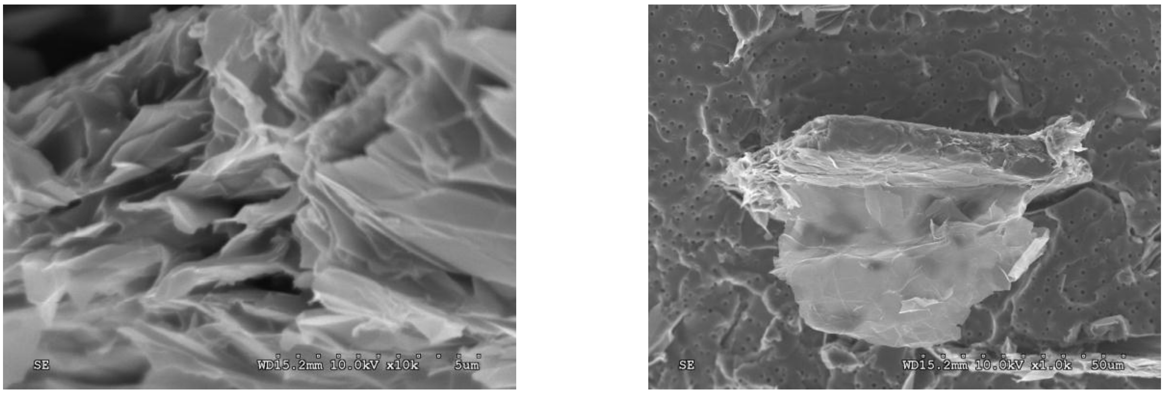



(a) (b)

(c) (d)

**Fig. 7** SEM micrograph showing fully exfoliated graphite with grapheme layers

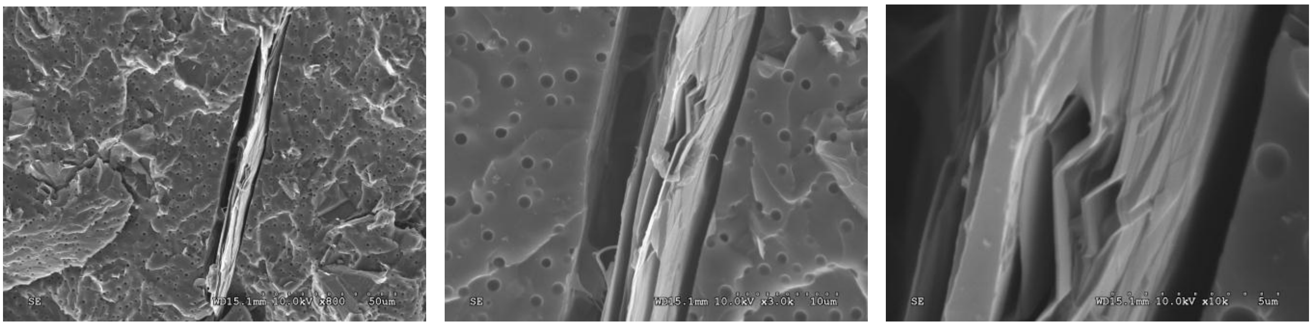
Properly exfoliated but improperly connected EG is shown in Figure 8 (a) exhibiting random arrangement of graphene and improper stacking. Improperly exfoliated NG is shown in Figure 8 (b) showing closely stacked graphene layers at certain regions. Improper exfoliation is due to improper intercalation. The lower portion of Figure 8 (b) shows that a group of graphene layers are slightly separated from improperly exfoliated NG. These improperly exfoliated EG cannot be separated further when they are passed through 3-roll mill; however, 3-roll mill may compress such improperly exfoliated NG and also severely affects formation of GNS. This leads to non-availability of matrix infiltration during composite fabrication. It is found that properly intercalated NG may suffer improper exfoliation due to temperature of furnace and duration of exposure of NG at elevated temperature. Lower temperature than preferred and lesser time than favored would also affect exfoliation.




(a) (b)

**Fig. 8** SEM micrographs showing (a) fully EG without proper branching/connections, and (b) unexfoliated/partially-exfoliated graphite

Improper intercalation and subsequent thermal shock may leads to partial exfoliation of some part of NG (Figures 9 (a)-(c)). An improper bonding between GNS and epoxy is also observed. Additionally, pores (diameter ~ 1 µm) are also seen dispersed randomly in matrix owing to evaluation of gases due to epoxy-hardener reaction. The pores are also evolved as a result of improper oven curing. These pores cannot be completely removed in nanocomposites however careful selection of epoxy-hardener system can eliminate or reduce this problem. Additionally, one can change to resin system with lower viscosity, can also apply proper vacuum so as to reduce pores formation. Another significant problem that occurs during nanocomposite synthesis is improper wetting of the reinforcement by matrix and insufficient infiltration of matrix inside reinforcement. GNS/EG-epoxy nanocomposite illustrated in Figures 9 (a)-(c). One can clearly observe the gap on both side of GNS embedded in the matrix. The wide gap towards left side of reinforcement may be due to fracture of some portion of GNS from original group followed by pull out due to applied load. On contrary, the gap on the right side may be due to improper wetting of reinforcement by matrix during mixing. Another observation reveals that the GNS (group of GNS) reinforcement after initial fragmentation may be compressed while passing through roll gaps or NG may be partially exfoliated. One can easily observe layered nature of EG/GNS and typical 120 º folding of graphene.

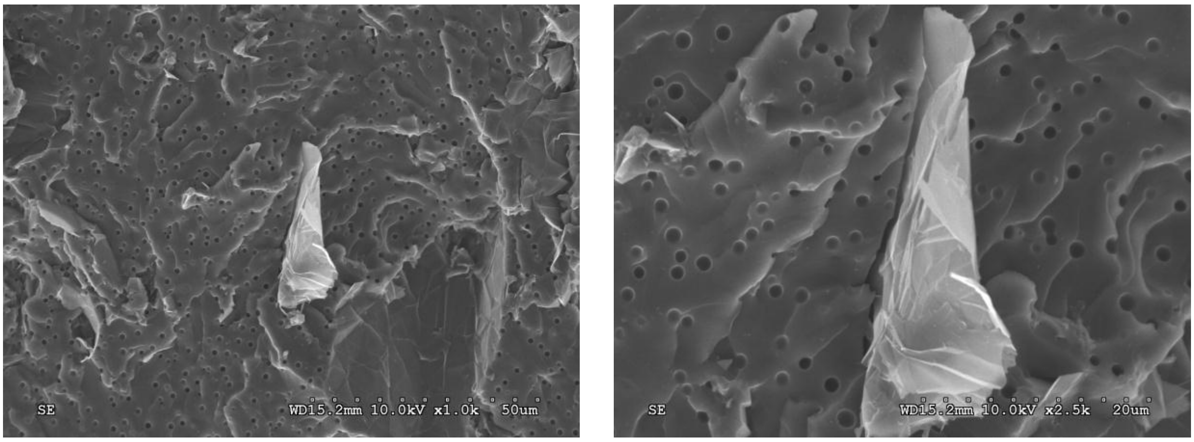



(a) (b) (c)

**Fig. 9** SEM micrographs showing the partial exfoliation of some portion of NG

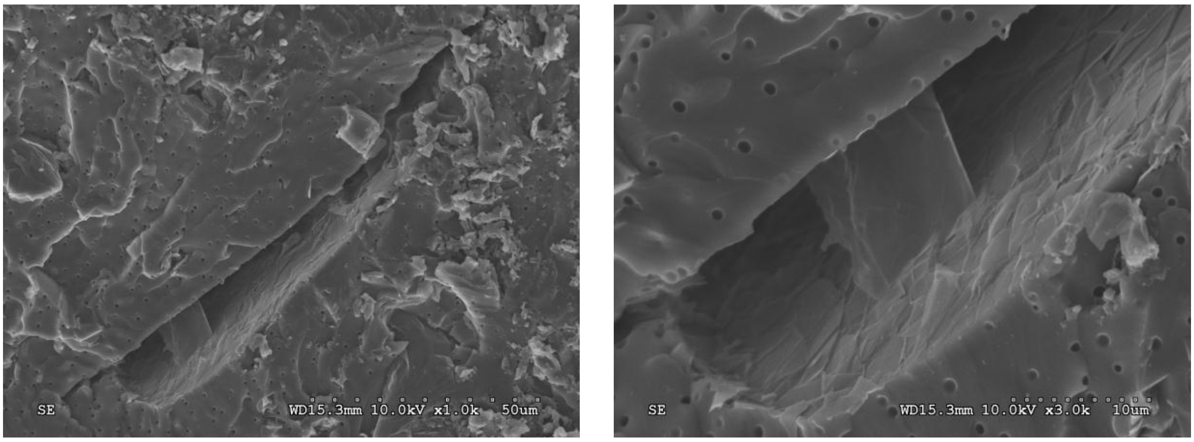
As explained earlier, second major issue is related to nanocomposite synthesis. The main issue is mixing and dispersion of reinforcements in matrix for effective load transfer. When the nanoscale materials are used as an additive in the composite, the dispersion would be perhaps the most important issue. The GNS often get together in the form of agglomerates and does not allow an infiltration of epoxy for consolidation. Without the surrounding matrix for load transfer, the GNS acts more like a void rather than a reinforcement. It is harder for GNS to be completely wetted by epoxy due to larger surfaces and much longer flowing paths. It is important to select a proper initial roll gap, so as to tear apart EG into GNS effectively. Figure 6 (a) illustrates the uniform dispersion of EG and GNS in matrix, however, one can observe preferred orientation of GNS in Figure 6 (b) resulted due to mixing/stirring of EG/GNS-epoxy mixture with hardener. The preferred orientation may induce direction dependent nanocomposite properties.

In the Figures 10 (a) and (b), one can observe nearly perfect nanosheet with only few graphene layers. Half portion of the nanosheet is trapped inside the nanocomposite; however the rest of nanosheet is pulled from the other fractured portion of the nanocomposite. It is also evident that the forces responsible for pull out of nanosheet can induce localized folding and partial fracture of nanosheets. One can observe pull out region in Figures 11 (a) and (b) where a portion of nanosheet is visible, which clearly forms bridge between two opposite faces of pull out region. Such events are very common when nanocomposites are subjected to external load till failure. The pours appeared in these micrographs detriments the properties of nanocomposite.




(a) (b)

**Fig. 10** SEM micrograph showing isolated perfect GNS




(a) (b)

**Fig. 11** SEM micrograph showing pull in region with retained GNS

## Conclusion

Based on the above discussion, it is concluded that the success of nanocomposite synthesis depends on the properties enhancement of matrix due to addition of reinforcement. It was concluded that issues during EG/GNS-epoxy nanocomposite synthesis can be attributed to the issues related to the intercalation of NG, to the issues related to the exfoliation of NG, and issues related to the synthesis of EG/GNS-epoxy nanocomposite. Improper exfoliation, improper mixing, improper matrix infiltration in reinforcement, improper wetting may be some issues. However, folding of graphene, crushing of graphene, compression of some or complete EG are other issues related to synthesis of nanocomposite. The orientation of reinforcement (EG, GNS) and evolution of pours are also other issues which hinders the properties of nanocomposite It is also found that the exfoliation of NG to EG and conversion of EG to GNS are going to have profound effect on the synthesized nanocomposite.

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