

Physical Properties Variation of Graphene and Multi Wall Carbon Nanotubes by Planetary Ball Mill and Heat Treatment

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Abstract. The article reports the experimental study of graphene, multiwall carbon nanotubes and their composite where samples are made by applying dry grinding and wet grinding as well as heat treatment processes. The investigation information is recorded and compared by SEM analysis, EDS spectrum and density measurement which give the informative evidence. And finally the article tries to find the basic difference in physical properties of these samples.

1. Introduction

The rising cost of indium and high temperature processing ¹⁾ and brittleness of ITO (indium tin oxide) ²⁾ engendered thinking alternatives which are GN (graphene) ³⁾ as well as CNTs (carbon nanotubes) ⁴⁾. And, it is the extensive concern to evaluate which one is better one by comparing among GN, CNTs and their composite in various applications, for example, in heat sink applications as coolant ⁵⁾. Within a few decades there are several researches have been done in this field and are still going on. And the outcomes are not only positive but also informative, alluring and practically applicable.

GN, two dimensional extended honeycomb network of sp² hybridized carbon atoms ⁶⁾, high electron mobility ⁷⁾, excellent mechanical, chemical and thermal properties ⁸⁾, has been used broadly in super capacitors ⁹⁾, optoelectronic devices ¹⁰⁾ as well as in several high performance sensors ¹¹⁾. And, CNTs, one of the lightest ¹²⁾, strongest ¹³⁾, stiffest ¹³⁾, electrically ¹⁴⁾ and thermally ¹⁵⁾ conductive nanoparticles, is also applied almost in same fields as that of GN. Now a days, metal or metal nanowires ¹⁶⁾ and

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hybrids of these ¹⁷⁾ are also used for making composites with GN and CNTs for getting better performance.

This paper studies the comparison among GN, MWCNTs and their composite i.e. GN-MWCNTs and tries to report as follows:

- SEM reports give the surface analysis information (Section 3.1) by which roughness and porosity are revealed.
- EDS spectrum gives the information of the presence of carbon as well as other particles (Section 3.2) which also confirms the presence of any contamination.
- Density measurement informs the amount of particles within a given area (Section 3.3) of each sample.

It should be noted that the procedure and the results of this paper are only for comparison and do not suggest the optimum conditions.

2. Experimental Details

2.1. Materials

Raw MWCNTs with a $\sim 20\text{nm}$ diameter, $\sim 5\mu\text{m}$ length (Carbon Nanomaterial Technology Co., Ltd, South Korea) and graphene nanopowder with 8nm (average flake thickness) flakes, average particle size $\sim 550\text{ nm}$, specific surface area $100\text{ m}^2/\text{g}$, and 99.9% purity (graphene supermarket) are used in this experimental study.

2.2. Grinding process

A planetary ball mill machine (HPM-700) (Haji Engineering, Korea) is used for grinding where MWCNT, GN and their composite are ground for one hour with 400rpm according to the experimental approach.

2.3. Density measurement

A gas Pycnometer (AccuPyc1340, micromeritics) is used for measuring density of 0.10g of GN, MWCNTs and GN-MWCNTs composite samples.

2.4. Furnace

An electric box furnace (Dongwon scientific Co.) is used for applying thermal treatment of the samples at 90°C for a day.

2.5. Samples



Figure 1. a) Dry ground GN, b) Wet ground and heat treated GN, c) Dry ground MWCNT, d) Wet ground and heat treated MWCNT, e) Dry ground GN-MWCNT, f) Wet ground and heat treated GN-MWCNT(T)

Total six samples are made where GN, MWCNTs and GN-MWCNTs composite are three main samples. And each sample is prepared in two ways: one is dry grinding and another one is wet grinding and furnace under some given parameters as mentioned above.

3. Experimental results and discussion

3.1. SEM results

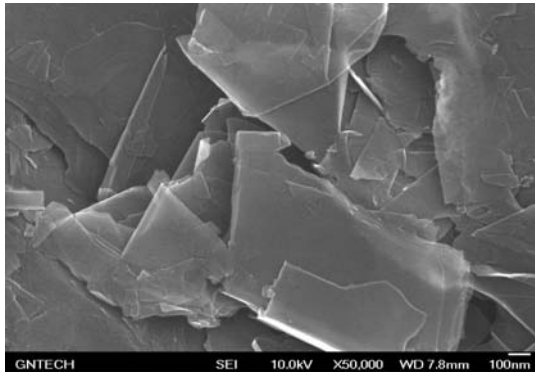


Figure 2. SEM image of dry grinding of GN.

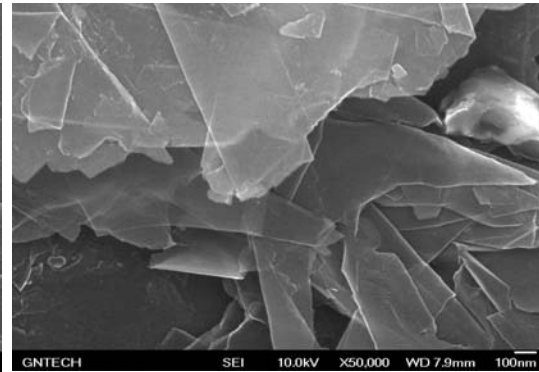


Figure 3. SEM image of wet grinding and heat treatment of GN.

From Fig. 2. and Fig. 3, it has seen that there is no eloquent information of substantial difference between without and with heat treatment of ground GN. So, in this experiment heat treatment has not been effective for GN samples.

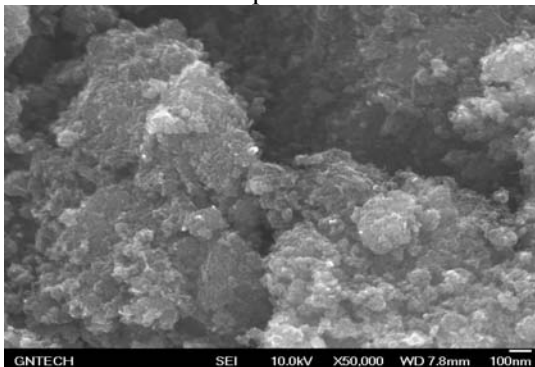


Figure 4. SEM image of dry grinding of MWCNTs.

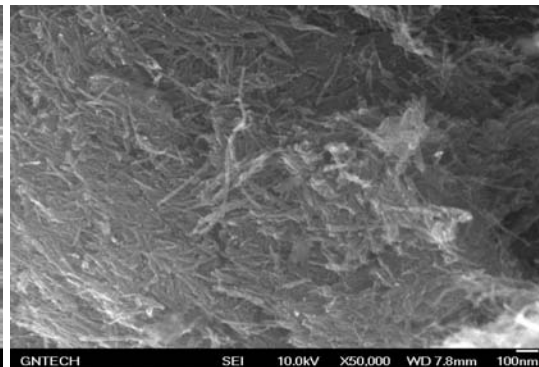


Figure 5. SEM image of wet grinding and heat treatment of MWCNTs.

Without heat treating the surface of ground MWCNTs has been rough, whereas, heat treatment (Fig.5.) has ameliorated the surface.

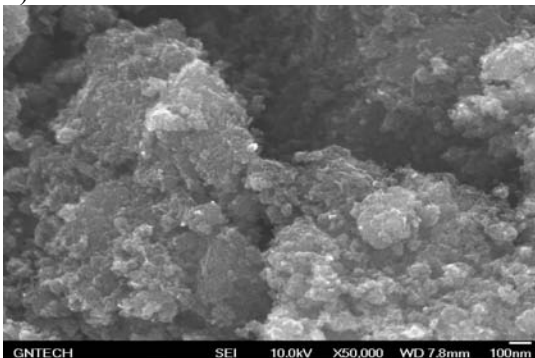


Figure 6. SEM image of dry grinding of GN-MWCNTs.

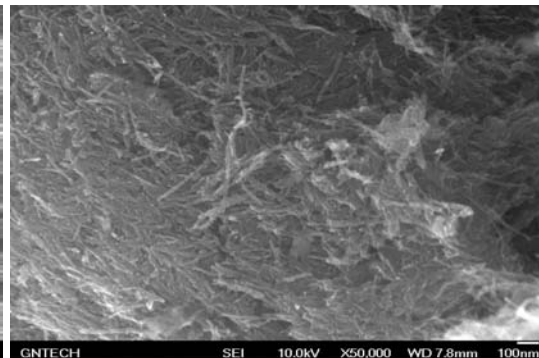


Figure 7. SEM image of wet grinding and heat treatment of GN-MWCNTs.

Fig. 6. and Fig. 7. have revealed that same as ground MWCNTs, heat treatment has upgraded the surface of GN-MWCNTs incomparably.

3.2. EDS spectrum

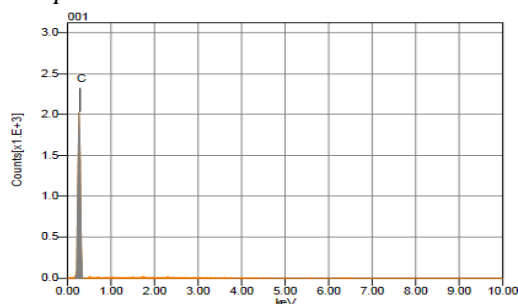


Figure 8. SEM image of dry grinding of GN-MWCNTs.

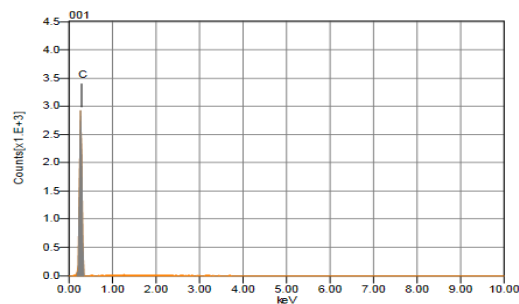


Figure 9. EDS spectrum of wet grinding and heat treatment of GN.

Table 1: Dry grinding of GN.

Chemical	mass%	Atom%
C	100.00	100.00
Total	100.00	100.00

Table 2: Wet grinding and heat treatment of GN.

Chemical	mass%	Atom%
C	100.00	100.00
Total	100.00	100.00

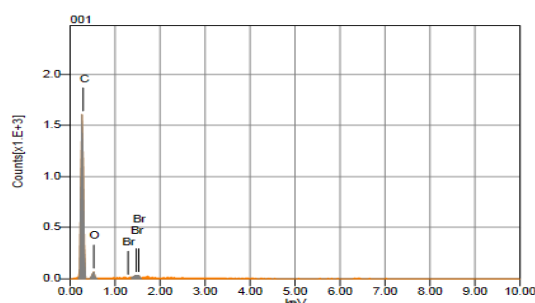


Figure 10. EDS spectrum of dry grinding of MWCNTs.

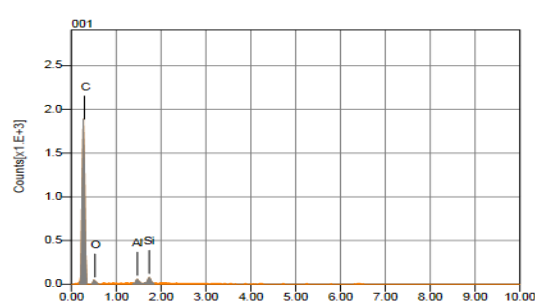


Figure 11. EDS spectrum of wet grinding and heat treatment of MWCNTs.

Table 3: Dry grinding of MWCNTs.

Chemical	mass%	Atom%
C	88.47	93.06
O	8.10	6.40
Br	3.43	0.54
Total	100.00	100.00

Table 4: Wet grinding and heat treatment of MWCNTs.

Chemical	mass%	Atom%
C	91.45	94.23
O	5.96	4.61
Al	1.11	0.51
Si	1.48	0.65
Total	100.00	100.00

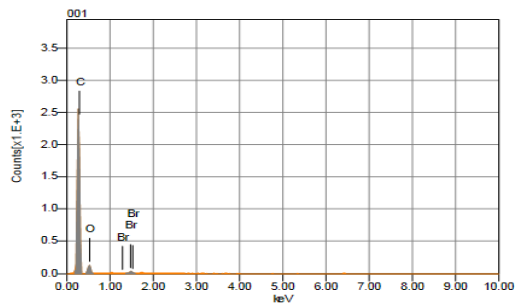


Figure 12. EDS spectrum of dry grinding of GN-MWCNTs.

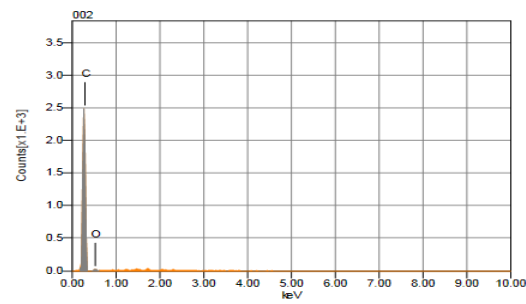


Figure 13. EDS spectrum of wet grinding and heat treatment of GN-MWCNTs.

Table 5: Dry grinding of GN-MWCNTs.

Chemical	mass%	Atom%
C	86.15	90.74
O	11.17	8.84
Br	2.68	0.42
Total	100.00	100.00

Table 6: Wet grinding and heat treatment of GN-MWCNTs.

Chemical	mass%	Atom%
C	97.13	97.83
O	2.87	2.17
Total	100.00	100.00

3.3. Density

For without heat treatment, the densities of GN, MWCNTs and GN-MWCNTs samples are 1.45166 g/cm³, 1.97942 g/cm³ and 1.46752 g/cm³ respectively and for with heat treatment, the values are 1.83476 g/cm³, 2.42982 g/cm³ and 1.71582 g/cm³ respectively (Fig: 14). In both cases, density of MWCNTs is higher than other two. And heat treatment has given better density. The adherence problem of making GN sample has occurred severely than other that of other samples. And because of this problem the sample of GN has not been collected properly after dry grinding. There may be a side effect of this on the density of GN sample which is the lowest value of this density measurement.

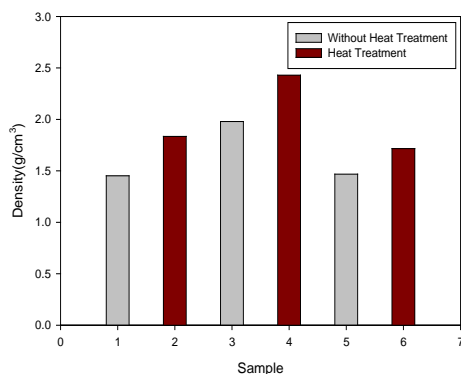


Figure. 14. Density of sample1: dry grinding of GN, sample 2: wet grinding and heat treatment of GN, sample 3: dry grinding of MWCNTs, sample 4: wet grinding and heat treatment of MWCNTs, sample 5: dry grinding of GN-MWCNTs, sample 6: wet grinding and heat treatment of GN-MWCNTs.

4. Conclusions

At first, the surface analysis was done with the help of SEM. After that, EDS spectrums were analyzed in order to analyze the surfaces broadly. And finally the values of the density of the six samples were measured. After analyzing these data, the key conclusions can be digested as follows:

- There is no significant change or improvement by applying heat treatment of ground GN.
- Heat treatment is effective for decreasing the surface roughness of MWCNTs as well as GN-MWCNTs.
- The amount of carbon in these samples increased noticeably by heat treatment.
- There was an adherent of particles to grinding media, i.e., vial and balls during dry grinding which was substantially reduced by wet grinding process.
- Wet grinding and heat treatment approaches represented the considerably higher density than only dry grinding process. And for the dry grinding and the wet grinding as well as heat treatment processes, the density of MWCNTs is noticeably higher than others.

Acknowledgment

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