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SYNTHESIS OF GRAPHENE VIA ARC DISCHARGE AND ITS CHARACTERIZATION: A COMPARATIVE APPROACH†

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Herein, few layer graphene was synthesize using two arc discharge chambers of different volumes to ascertain the influence of chamber size on the quality and yield of graphene. In both arc discharge chambers (A and B), graphite rods were ignited at arc current of 200 A and pressure of 500 Torr to produce vaporized carbon atoms which were deposited on the chamber wall. The synthesized graphene was characterized using the combined effect of UV spectroscopy, X-ray diffraction, Raman spectroscopy, scanning electron spectroscopy and transmission electron spectroscopy. It was observed that, an increase in the chamber size led to an increase in the number of graphene layers (4 – 6 layers) and an increase in the crystalline size D (9.6 – 17.4 nm) as revealed by XRD results. Raman analysis shows lower value of I_D/I_G of 0.62 indicating the presence of lower defect in chamber A as compared to the I_D/I_G value of 0.93 observed in chamber B. A graphene yield of 0.96 g was obtained from chamber A while 0.67 g of the same product was obtained from chamber B. The fabricated arc discharge systems suggest that a larger chamber size could promote a better yield of graphene on an industrial scale. Hence, the research is relevant to the development of larger amounts of quality FLG for industrial device applications.
Keywords: *Few-layer graphene; Arc discharge; Plasma; Chamber size; Spectroscopy*
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INTRODUCTION

Graphene is the basic structural component of carbon allotropes counting charcoal, graphite, carbon nanotubes and fullerenes [1] and has attracted a considerable attention due to its unique electronic and optoelectronic properties [2]. In many respects, graphene is considered to be an alternative to silicon device applications owing to its exceptional band structure (relatively zero bandgap) characteristics, outlining its improved optical properties and electrical competences. Besides, graphene is becoming increasingly used in the manufacture of lightweight and strong body parts for vehicles [3]. However, reproducible and stable techniques to yield larger amount and high-quality crystallite graphene at an industrial scale to compete the already commercialized silicon-based devices hold out to be the most worrisome impediment to producing graphene. Graphene is currently being studied for several applications such as transparent electrodes, graphene field-effect transistors, paper-based ultra-capacitors, composites, sensors, and integration with modern tools in neuroscience [4].

Graphene was first discovered and recognized through mechanical exfoliation process [5]. Though the exfoliation practice yielded high quality of graphene for research drives, it is not a sustainable technique for the industrialization of graphene based devices [1]. Towards such goal, it is believed that chemical processes such as Chemical Vapor Deposition (CVD) is more suitable as reported by researchers [6, 7], other techniques for graphene synthesis include; plasma-enhanced chemical vapor deposition (PECVD) [8], micromechanical cleavage [9], reduction of graphene oxide [10], epitaxial growth on silicon carbide [11], and arc discharge method [12].

Among all these methods, the arc discharge technique seems to be relatively cheaper and easy to operate. The arc discharge method of graphene synthesis requires some process conditions such as; high temperature, the influx of vaporized carbon, buffer gas, and in some cases a catalyst could be used [13]. These process conditions are allowed to interact in a confined volume of an arc discharge chamber, resulting in the growth of nanomaterial. Several studies on these parameters have been carried out with much emphasis on the features of the synthesized products [7]. Li et al. [14] proved a technique in which petroleum asphalt is utilized as a precursor in a water arc discharge system. To date, many authors have studied the effect of gas pressure, gas temperature, arc current and thermal gradient of growth zone on the characteristic of graphene synthesize via the arc discharge approach [15, 16]. However, the influence of the arc discharge chamber size on the products obtained has been far less studied, mainly when considering experimental works.

In view of the success of the arc discharge method in the production of graphene, further study of this technique for large-scale synthesis of few layer graphene (FLG) on an industrial scale is a subject of concern [17]. Herein, we report on the effect of chamber size on the quality and yield of FLG produced via the arc discharge method. It was observed that by exploring larger chamber size, optimal temperature gradient could be achieved due to the larger surface area of the chamber as it cools gradually, resulting in better yield as compared to the smaller chamber [18]. Though graphene is known as an excellent electronic material, synthesizing single layer of it has been less explored. However, the majority of these methods of graphene synthesis categorized with low output and large-scale production are yet to be achieved [19].

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This paper therefore, presents how increase in arc chamber size could facilitate graphene synthesis to an industrial scale. Hence, the arc discharge approach still remains one of the most viable techniques for synthesizing FLG in bulk amounts for industrial use.

EXPERIMENTAL PROCEDURE

The two-arc discharge systems A (450 mm×100 mm) and B (300 mm×60 mm) were set up as shown in Figure 1. The two electrodes in the arc discharge chamber were set up such that they are about 1 to 2 mm apart. The mechanical feed-through enables movement of the movable electrode controlled from outside the stainless chamber. The chamber was evacuated to a pressure of 10^{-2} Torr. Argon gas was then introduced into the chamber from the argon gas cylinder to a pressure of 500 Torr. Both electrodes were connected to a high current of 200 A using a high current source. The mechanical drive was used to create an initial contact and immediate separation of the electrodes to initiate the arc discharge leading to evaporation of the graphite electrodes. The inlet and outlet were used to circulate water through the wall of the chamber. Arc discharge of the electrodes leads to an influx of vaporizes carbon deposited on the chamber wall. The deposited carbon soot sample was carefully collected for analysis.

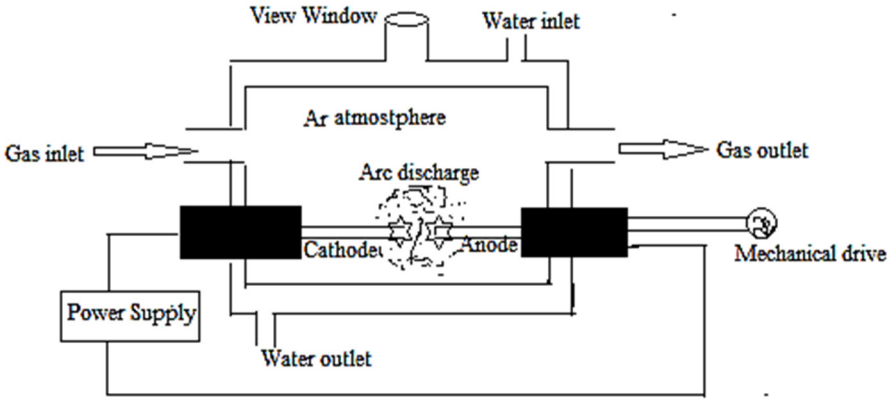

Figure 1. Schematic diagram of arc discharge system

To analyze the synthesized graphene nanoparticles, the following measuring instruments were used; UV-visible (UV-vis) spectroscope (Infinite F200 M; TECAN, Ltd., Männedorf, Switzerland), Rint Ultima X-ray diffraction (XRD) (Rigaku Co., Tokyo, Japan). Raman spectroscope (NRS-7100, JASCO, Tokyo, Japan), Scanning electron microscope (SEM) (JSM-6510LV, JEOL, Ltd, Japan) and Transmission electron microscope (TEM) (JEM-2100F, JEOL, Ltd., Tokyo, Japan).

RESULTS AND DISCUSSION

Figure 2 shows the UV-vis absorbance spectra of the synthesized graphene nanoparticles for Arc chambers A and B. Samples collected from arc chambers A and B respectively, revealed maximum absorption peaks at 251 nm and 258 nm with a continuous fall that stretches beyond 600 nm.

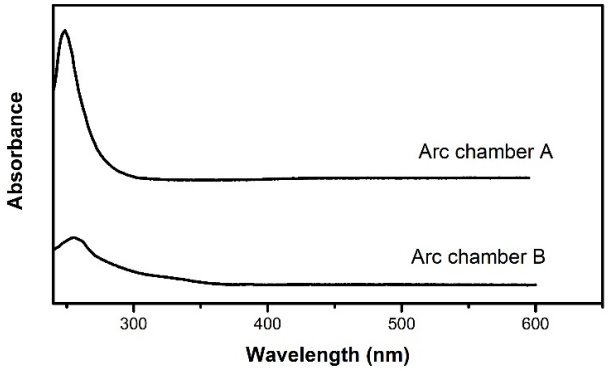

Figure 2. UV-vis spectra of graphene synthesized using chamber A and B

This characteristic is typical of graphene and is consistent with the optical property of graphene and other graphene nanoparticle composites [20]. It can be seen that the absorption peak of synthesized graphene in Arc chamber B shifted to a higher wavelength with a decrease in absorbance. This low absorbance is due to the increase in crystallite size (D) of the synthesized graphene using the smaller Arc chamber [2]. In the case with the Arc Chamber A, the larger chamber, a higher absorption (Gaussian) peak at the wavelength of $\lambda = 251$ nm was observed. The high absorption is due to the increased in Chamber size which causes a decreased in D, thus producing fewer layers of graphene as compared to

chamber B. Interestingly, graphene has another origin called graphene quantum dots (GQDs). Thus far, the high quality synthesized graphene nanoparticles in Arc Chamber A is of D <10 nm, making it a good candidate for applications in GQDs and graphene-based photoelectric devices [2, 21]. Similarly, this graphene material will enhance electroluminescence due to its smaller D and high optical absorption in the UV region in agreement with previous works [2, 20]. Details of the number of layers and D of graphene synthesized using arc chamber A and B are presented in Table 1.

Table 1. Crystalline size (D), interlayer spacing (d) and number of layers of graphene synthesized using arc chamber A and B

Arc Chambers	FWHM (β)	2θ (0)	D (nm)	d-spacing (Å)	No of layers
A	0.8858	26.7	9.623	3.37206	4
B	0.4904	26.6	17.379	3.36081	6

Figure 3 shows XRD patterns of the samples synthesized with the different Arc discharge chamber sizes (A and B). The XRD spectra show an intense peak of (002) plane of diffraction at 2θ = 26.7° and 26.6° for arc chambers A and B respectively.

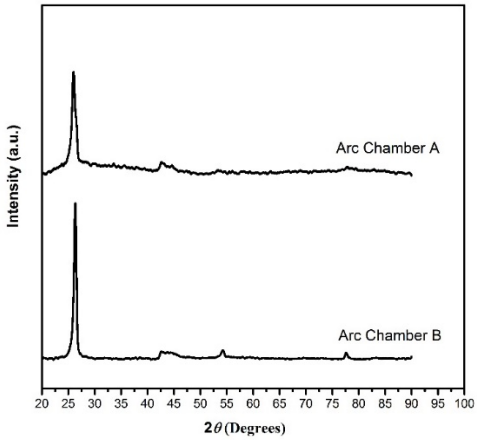

Figure 3. XRD patterns of graphene synthesized using chamber A and B

This signifies the characteristic of a well-ordered crystal structure of carbon materials [22]. The interlayer spacing (d) ~ 3.37 Å and 3.36 Å as obtained for arc chamber A and chamber B are slightly higher than that of graphite (3.34 Å). The diffraction peak of pure graphite originates around 26.3^0, matching to the highly ordered d of 0.34 nm sideways the (002) direction [23]. The significant difference in diffraction pattern at 2θ recorded by XRD in both Chambers could be ascribed to the loose nature of FLG [24]. The much defect in Arc Chamber B implies that decrease in chamber size increases D and d, hence much defects could be recorded whereas increase in chamber size produced FLG with less defect thereby yielding higher quality graphene. The D and d of the synthesized FLG were calculated using the Debye-Scherer equation (1) [22]:

$$D = \frac{0.94\lambda}{B\cos\theta} \tag{1}$$

where λ is the X-ray wavelength in nanometer (nm), β is the peak width of the diffraction peak profile at half maximum height in radians, θ is the scattering angle in radians. It was observed that the peak intensity was higher for the smaller arc chamber, implying an increase in D. The number of layers of the FLG synthesized at various arc chamber sizes was calculated using the Scherer equation (2) [25]:

$$N = \frac{D}{d} + 1 \tag{2}$$

where all symbols have their usual meaning. This clearly shows that graphene synthesized in a larger arc chamber is of fewer layers and more crystalline compared to that synthesized using a smaller arc chamber as shown in Table 1.

The Raman spectroscopy primarily provides evidence concerning the uniformity of graphene carbon skeleton [26]. Presented in Figure 4 is the Raman spectrograms of the synthesized graphene using arc chambers A and B. The spectroscopy revealed three major bands, the D band ~1348 /cm, band G~1576 /cm and band 2D~2690 /cm indicating the presence of graphene signature with the absence of graphite shoulder [13]. This result is similar to previous work of other researchers [8, 27]. The D band is related to the level of defects present in graphene [26]. D and 2D bands are both characteristics of sp^2 hybridized carbons. In pure form, graphene has well-ordered structure screening intense graphitic bands G at 1600 /cm which arise from the scattering of the E1g phonon sp^2 hybridized carbon [28]. The intensity of D peak in Arc chamber B increases gradually together with the number of holes in the structure or sp^2 new centers created by covalent bonds activated by defects band 2D~2690 /cm which informs about the value of cumulative load of defects

and number of layers of the synthesized graphene plane [23, 26]. In the incidence of <1% of the structural defects, the breakdown of the D band can be employed to confirm the quality of the synthesized graphene [26].

The number of defects of the graphene-based nanomaterial can be revealed by the ratio of intensity of D band and the G band (I_D/I_G), while the I_{2D}/I_G ratio could be used to distinguish the number of synthesized graphene layers of the FLG. Therefore, when the ratio I_D/I_G is greater than 1, it depicts the protuberant defective state of the synthesized graphene nanomaterial [20, 29]. Comparatively, as presented in Table 2, the increase in I_D/I_G and I_{2D}/I_G in Arc Chamber B are caused by increased number of defects on the surface of graphene [8, 20, 27, 29].

Thus from Table 2, the I_D/I_G ratio is in values of 0.62 and 0.93 corresponding to the Arc chamber A and B respectively. Since Arc chamber B produces the highest value of 0.93 of I_D/I_G ratio, indicating that there are more defects in the smaller chamber B unlike the fabricated Arc chamber A with the larger volume. However, in both chambers the quality of graphene nanoparticles produced are high since both have I_D/I_G values that are relatively less than 1 [20, 29]. Nevertheless, it has been reported that the number of synthesized graphene layers could increase from 3 – 7 when the I_{2D}/I_G is ~0.5. Previous report also estimated a FWHM of 68 cm^{-1} and I_{2D}/I_G of ~0.6 for 3 – 5 layers [30]. These values are closely related to the ones obtained in Table 2. This is also in agreement with the number of layers obtained from the XRD result presented in Table 1.

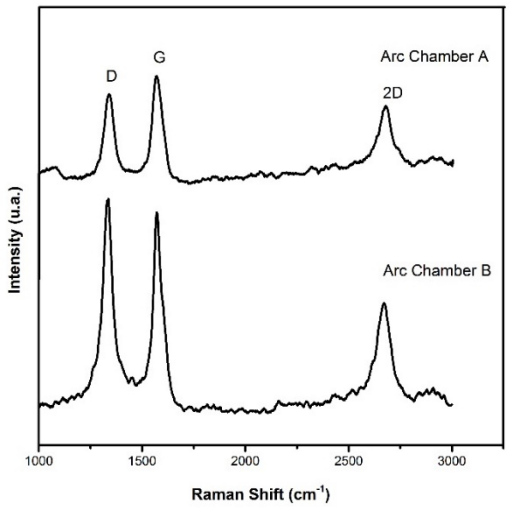

Figure 4. Raman spectrograms of graphene synthesized using chamber A and B

Table 2. Intensity ratios and full width at half maximum (FWHM) of graphene synthesized using arc chamber A and B

Arc Chamber	I_D/I_G	I_{2D}/I_G	FWHM(2D)
A	0.62	0.68	78.46
B	0.93	0.76	77.408

The SEM images provided the surface morphology of the synthesized graphene nanoparticles as shown in Figure 5a and b. The SEM images of the synthesized FLG in both Arc chambers are similar, revealing the presence of a large number of FLG flakes in agreement with previous studies [25]. Nucleation of graphene domain with an average size of 5 μm was observed in the SEM micrograph.

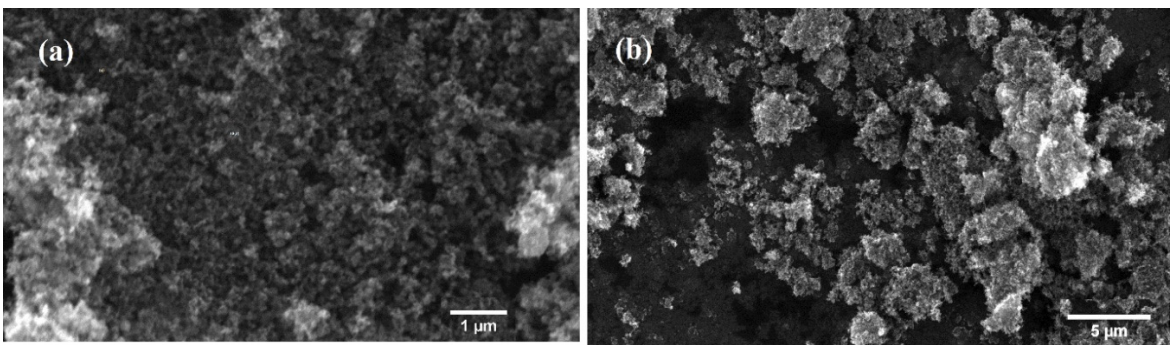

Figure 5. SEM micrographs of synthesized graphene via (a) Arc chamber A and (b) Arc chamber B

The structure of the synthesized graphene in Arc discharge chambers A and B was examined by the TEM images as shown in Figure 6a and b. The micrographs show a large area of FLG graphene sheet with a size of 100 – 200 nm. It was observed that the surfaces of the FLG sheets in both chambers displayed are quite uniformly distributed and with

wrinkles [22]. This is due to the defects caused by each chamber during synthesis and the variation in the number of graphene layers for each chamber as shown in Table 1. However, the graphene obtains from chamber A, in Figure 6a, reveals high level of transparency than the synthesized graphene in chamber B (see Figure 6b). The variation in transparency could be ascribed to changes in the lattice parameters and the number of layers of graphene in both arc discharge chamber [30].

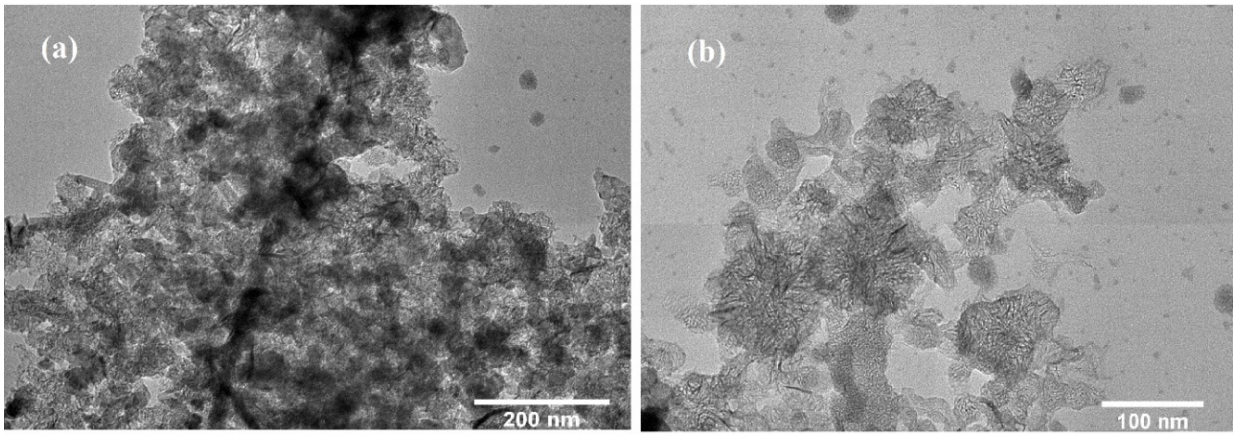

Figure 6. TEM micrographs of synthesized graphene via (a) Arc chamber A and (b) Arc chamber B

CONCLUSION

The research work here presented has comparatively investigated the amount and the quality of graphene synthesized in two arc discharge chambers of different sizes (volume). The graphene was synthesized from the graphite electrodes via arc discharge chamber technique. The fabricated larger arc discharge chamber of dimension 450 mm×100 mm hereafter tag Arc chamber A gave a better amount of graphene yield of 0.96 g when compared to the 0.67 g yield obtained from an arc discharge chamber B of smaller dimension 300 mm×60 mm diameter. The UV spectroscopy result revealed that the absorption peak of synthesized graphene in Arc chamber B shifted to a higher wavelength with a decrease in absorbance. The UV-vis spectrum of both chambers shows Gaussian absorption peaks around the wavelength of 251 – 258 nm. From the XRD results, the synthesized graphene samples on average, showed a $2\theta = 26.65°$ peak with (002) orientation that is characteristic of graphene. The synthesized graphene sample in Arc chamber A is of 4 layers with thickness of 9.62 nm compared to the synthesized nanoparticles in Arc chamber B having 6-layer graphene flake of greater thickness. These results are supported with the layer numbers obtained from the Raman spectroscopy results. The ratio ID/IG confirmed the FLG with the UV-vis spectrum revealing the graphene signature. The average size of flakes observed in the SEM micrographs was 5 µm. The TEM micrographs show graphene nanostructure with thickness variation. Our results show the viability of large-scale production of quality graphene using the arc discharge approach, which may open up more opportunities in the wide range of applications of FLG, comprising anti-corrosion coatings, composites, super-capacitors, energy storage, and transparent conductive films.

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СИНТЕЗ ГРАФЕНУ ДУГОВИМ РОЗРЯДОМ ТА ЙОГО ХАРАКТЕРИСТИКА: ПОРІВНЯЛЬНИЙ ПІДХІД

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Синтезовано кілька шарів графену з використанням двох дугових розрядних камер різного об'єму, щоб з'ясувати вплив розміру камери на якість і вихід графену. В обох камерах дугового розряду (A і B) графітові стрижні запалювали при струмі дуги 200 A і тиску 500 торр, щоб отримати випаровані атоми вуглецю, які осідали на стінці камери. Синтезований графен був охарактеризований за допомогою комбінованого ефекту УФ-спектроскопії, дифракції рентгенівських променів, спектроскопії раманівського розсіювання, скануючої електронної спектроскопії та електронної спектроскопії на просвічування. Було помічено, що збільшення розміру камери призвело до збільшення кількості графенових шарів (4-6 шарів) і збільшення кристалічного розміру D (9,6-17,4 нм), як показали результати XRD. Раманівський аналіз показує нижче значення ID/IG 0,62, що вказує на наявність меншого дефекту в камері A порівняно зі значенням ID/IG 0,93, що спостерігається в камері B. Вихід графену 0,96 г було отримано з камери A, тоді як 0,67 г той самий продукт було отримано з камери B. Виготовлені системи дугового розряду припускають, що більший розмір камери може сприяти кращому виходу графену в промислових масштабах. Отже, дослідження актуальне для розробки більшої кількості якісного FLG для застосування в промислових пристроях.

Ключові слова: *кількашаровий графен; дуговий розряд; плазма; розмір камери; спектроскопія*