Post-cutting surface evaluation of the Cu foil substrate grown with single-layer graphene

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Abstract. A large-sized graphene purchased for research purposes can be cost effective as its usage is not limited to one experimental study. It can be cut into many smaller pieces for other experimental studies. In this study, Cu foil substrate grown with single-layer graphene via chemical vapor deposition (CVD) with a size of 2” × 2” was cut into small pieces with a size of 10 × 10 mm$^2$ using a pair of high-quality stainless-steel scissors. The surface quality of the cut substrate was evaluated using three microscopes with different types of illumination sources. In the post-cut substrate, a wrinkle and roughness at the edge of the substrate surface were observed, which can be attributed to the uneven surface contact and force distribution during the handling and cutting process using a tweezer and a pair of scissors. However, the wrinkle in the substrate is not the main reason for the degraded quality of graphene grown on the substrate. Meanwhile, the roughness at the edge area of the cut substrate may be reduced by controlling the speed and angle of the cutting forces. The information obtained from this preliminary study of sample preparation is important for future work in the synthesis of graphene nanocomposites for its application in nanofluids.

Keywords: Graphene, foil substrate, microscope and surface analysis.

1. Introduction
Graphene is a monolayer-thick hexagonal lattice with sp$^2$ hybridization [1]. Owing to its extraordinary properties, such as large surface area [2], ultra-thinness, and superior mechanical strength [3], it has ubiquitously attracted interest from researchers in numerous technological fields [4-7]. Currently, the synthesis of graphene is not only limited to laboratory scale, but the global market is also developing with a lot of graphene manufacturers driven by the rising demand from industry and research fields [8]. Various types of graphene are available in the global market, such as chemical vapor deposition (CVD) graphene, graphene nano powder, graphene solution, graphene oxide, and reduced graphene oxide.

In an experimental study requiring repetitive experiment, buying many pieces of small-sized CVD graphene is expensive. To solve this problem, a large-sized CVD graphene can be purchased as it is cost effective, and its use is not only limited to one purpose, but it can also be used for various experimental studies. A large-sized CVD graphene can be divided into small pieces. However, it should be noted that the cutting process might change the substrate (foil) surface and edge properties, especially at the cut area. Thus, prior to using the cut CVD graphene for further experimental studies, it is important to
evaluate its surface quality and edge condition. This can be performed by using a light microscope, laser microscope, and electron microscope.

Microscope is an instrument used to observe larger and more detailed images of objects from biological specimens [9], material structure [10], and many others that the human eye cannot see [11]. It comes in different types; each microscope has distinct features and is significant for different uses in various fields.

In previous studies [12-13], the foil substrate was cut for sample preparation. However, considerable attention has not been given to the quality of the cut sample prior to use for further experimental work.

Taking this into consideration, three different types of microscope, i.e., light microscope, laser microscope, and electron microscope, were used in this study to evaluate the quality of the substrate surface and edge condition after the cutting process. Light microscope, laser microscope, and electron microscope have different wavelengths, thereby giving different image resolutions of the cut sample substrate. Among the three, electron microscope gives higher image resolution as it has shorter wavelength than the light and laser microscopes. With these microscopes, an insight regarding the quality of the cut substrate, which will be used for the next experimental study related to the synthesis of nanofluid that consists of graphene, can be obtained.

2. Experimental Details
In this study, a copper (Cu) foil substrate grown with single-layer graphene (Graphene Supermarket, Calverton, NY) with a size of 2" × 2” was used. Single-layer graphene was grown on the Cu foil substrate via the CVD method, where both sides of the Cu foil were exposed to the gaseous during the CVD process. The Cu foil substrate was then cut into small pieces with a size of 10 × 10 mm² using a pair of high-quality stainless-steel scissors. Thereafter, imaging was performed using three types of microscopes with different illumination sources to analyze the post-cutting substrate surface and edge condition. The microscopes used were optical microscope (Nikon SMZ745) and digital microscope (Olympus DSX510) that use visible and light-emitting diode (LED) light as the illumination sources, respectively; 3D laser scanning microscope (Olympus LEXT OLS4100) that uses a semiconductor laser with a short wavelength of 405 nm; and field emission scanning electron microscope (FESEM) (JEOL JSM-7800F) that uses electrons as an illumination source. Observation was performed on the same cut substrate in the following order: (i) optical/digital microscopes, (ii) laser scanning microscope, and (iii) scanning electron microscope to minimize the damage to the substrate during the imaging process.

![Figure 1.](image)

Figure 1. (a) Original substrate size, (b) post-cut substrate size, (c) stainless-steel scissors used for cutting
3. Results and Discussion

Figure 2 presents the images of the front and rear sides of the surface of the cut substrate observed using digital microscope and optical microscope, respectively. The front side of graphene grown on the substrate has a better quality than the rear side. From the results, the line texture of the Cu foil substrate can be observed in both the images, although that obtained by optical microscope was not clearly visualized as the observation was done by the naked eye. This was also a consequence of the depth of field becoming shallower as the magnification increased while zooming with optical microscope. The single-layer graphene on the Cu foil substrate could not be observed since its optical density is about 2.5% [14]. FESEM observation was then performed at a high magnification on both sides of the Cu foil substrate, which then confirmed the line texture of the Cu foil substrate (Figure 3).

![Figure 2](image1.png)

**Figure 2.** (a) Image of the front-side surface taken by the digital microscope and (b) image of the rear-side surface taken by the optical microscope

![Figure 3](image2.png)

**Figure 3.** FESEM images of the Cu foil substrate at the front-side and rear-side surfaces

In addition to the line texture of Cu foil substrate, wrinkle could also be observed on the cut substrate. Further analysis was then conducted to obtain further detail on the emergence of the wrinkle using a 3D laser scanning microscope. Line profiling at three designated areas on the cut Cu foil substrate was performed to obtain morphological information on each area. Figure 4 presents the line profiles of each designated area at the front and rear sides of the cut Cu foil substrate. Based on the results, nonuniformity of the surface can be observed in the areas where the wrinkles emerged, mostly close to the edge of the substrate. This nonuniformity can be considered to be caused by the uneven surface contact and force distribution during the cutting and handling processes using a pair of scissors and a tweezer. However,
the wrinkle that emerged at this stage did not affect the presence of the grown graphene layer on the foil substrate [14]. The values of the surface roughness, $R_a$, and RMS surface roughness, $R_q$, are presented in Figure 5.

**Figure 4.** Line profiles at three designated areas on front side and rear side of the Cu foil surface

**Figure 5.** Surface roughness and RMS surface roughness versus line profiles at three designated areas for front and rear side of the Cu foil substrate
Thereafter, high-resolution imaging using FESEM was performed to evaluate the edge condition of the Cu foil substrate. The results presented in Figure 6 indicate that the edges around the cut area exhibited a local surface deformation, which then caused the roughness on the cutting surface. In addition, creases were observed near the cut edge [Figure 6 (a) and (b)], which can be explained by the following principle. The cutting process caused a fracture or shear to the cut material. The portion that was cut away is called chip. The friction between the scissors and the Cu foil substrate that has developed edge roughness during the cutting process was attributed to the existence of cutting force, as presented in Figure 7. During the cutting process, six forces were applied, i.e., cutting force, thrust force, shear force, normal to shear force, friction force, and normal to friction force [15]. The forces produced from the cutting process caused the chip to break and generated residual stresses that contributed to the roughness at the cut area [16]. Although cutting the substrate using a pair of scissors produced edge roughness, this method is still recommended compared with cutting the substrate using a diamond cutter or knife. This is because this method can prevent damage to the graphene layer due to its direct contact with the surface. However, the roughness caused by the contact between the substrate and scissors may be reduced by controlling the speed and the angle of the cutting forces.

Figure 6. FESEM images of cut edge area viewed from (a), (b) top view and (c), (d) cross-section view
In this study, the substrate (foil) was cut using a pair of high-quality stainless-steel scissors for simplicity. A large-sized CVD graphene was cut into small pieces for use in various experiments to reduce the research cost. It was then followed by surface observation analysis of the cut substrate surface using three microscopes with different illumination sources. This was performed to evaluate its quality before being used for the next experimental study related to the synthesis of nanofluid consisting of graphene.

In the post-cut substrate, a wrinkle at the substrate surface and surface roughness at the cut area of the substrate edges were observed, which are attributed to the uneven surface contact and force distribution during the handling and cutting processes using tweezer and a pair of scissors. However, this substrate wrinkle is not the main reason for the degraded quality of graphene grown on the substrate. Meanwhile, the roughness at the edge area of the cut substrate may be reduced by controlling the speed and angle of the cutting forces. The information obtained from this preliminary study on sample preparation is important for future work in the synthesis of graphene nanocomposites for its application in nanofluids. In addition, this study provides useful insights regarding the evaluation of the substrate surface obtained by post-cutting process of a large-sized CVD graphene substrate to a smaller one, which can be used for repetitive experiments or various research purposes.

5. References
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