Observing graphene and graphite by nitrogen ion beam microscopy

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Introduction

The recent development of a gas field ion source (GFIS) based microscope is used for helium and, more recently, neo ion beam microscopy. With the former, nanometer scale milling of suspended graphene has been demonstrated [1]. The latter is being developed to increase the milling rate, thus bridging the large gap between helium GFIS-focused ion beam (FIB) and gallium FIB. The GFIS-FIB based nanofabrication system, recently developed by us, uses nitrogen or helium source gas [2]. During nanofabrication on exfoliated graphene devices, we observed different visibilities of the graphene on silicon dioxide in imaging mode when using nitrogen or helium source gas.

Method

Graphene was exfoliated from Kish graphite onto the silicon/SiO_2 samples (300 nm) with pre-patterned Ti/Au markers for easy location of graphene and graphite flakes. Then, graphene was patterned (O_2 RIE, PMMA etch mask) and contacted (Ti/Au lift-off using PMMA resist). The commercial sample with transferred CVD graphene was processed with a similar PMMA based fabrication process to pattern Ti/Au structures on it, but CVD graphene was partially peeled during lift-off. The nitrogen GFIS-FIB uses an acceleration voltage of 25 kV and secondary electron (SE) images (800x800 pixels) are acquired using an Everhart-Thornley detector with an 8 bit analog to digital converter (ADC).

Results and Discussion

SE images of an up to 50 nm thick exfoliated graphite flake are shown for nitrogen and helium ion beam in Fig. 1a and b, respectively. Different imaging ion doses were used to limit the damage to structures on the sample. As can be seen, the graphite flake clearly visible in Fig. 1b is barely visible in Fig 1a. The SE image of CVD graphene on a very similar sample is shown in Fig 1c, with good visibility.
The exact reason for the low visibility of exfoliated graphene and graphite on SiO_2 is not yet understood, but we will discuss several effects of relevance. This includes fundamental principles that cannot be influenced by the experiment such as stopping power, ion velocity, energy distribution of SE, SE escape depth and charging. Furthermore, we will discuss experiment related effects, such as imaging dose, PMMA contamination and the effect of increasing the ADC resolution.

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References

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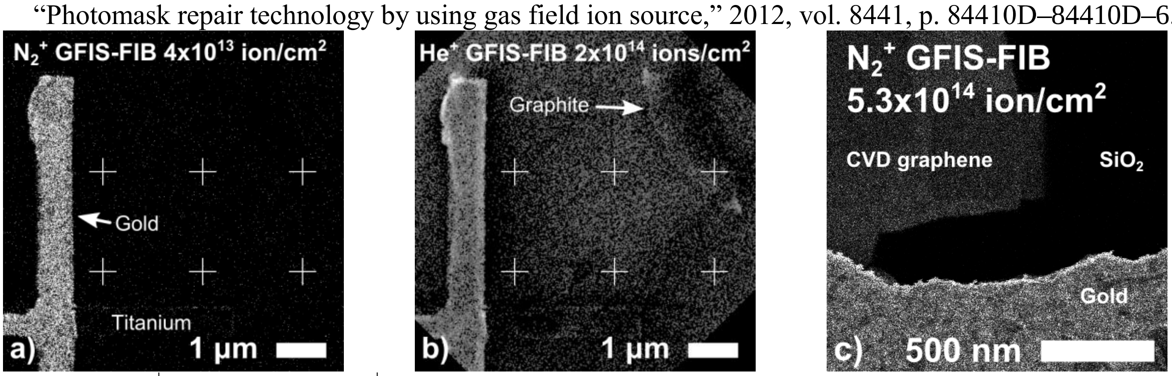

Fig.1. (a+b) N_2^+ GFIS-FIB and He^+ GFIS-FIB secondary electron micrographs showing identical location on 300 nm SiO2/Si sample with exfoliated graphite and graphene. The up to 50 nm thick graphite flake is barely visible by nitrogen ion beam. White crosses are added as guide to the eye. (c) N_2^+ GFIS-FIB secondary electron micrograph showing CVD monolayer graphene and gold structures on SiO_2. The edge of graphene is clearly recognizable.

