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Facile Fabrication of Graphene-Sealed Microwell Liquid Cell for Liquid Electron Microscopy

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Liquid-phase transmission electron microscopy (LP-TEM) is a promising tool to observe directly growth dynamics of nanoparticles or biomaterials in liquid media [1-2]. The LP-TEM technique inevitably needs a liquid cell to contain intactly liquid inside high-vacuum TEM. Thus, the liquid cell should not only provide high-contrast imaging, but also stably trap liquid under electron beam irradiation. While both stable liquid confinement and atomic resolution imaging have been achieved with recently developed liquid cells composed of covering graphene membranes on patterned windows [3-5], the liquid cells suffer from both complicate fabrication process and small viewing areas for TEM observation. In this work, we introduce a conventional patterning technique to fabricate liquid cell with graphene-sealed, micrometer-sized wells and viewing areas.

As shown in figure 1(a), fabrication process starts with SiN deposition on the silicon wafer by low-pressure chemical vapor deposition (LPCVD). Holy microwells are patterned on the top SiN film by using optical lithography and reactive ion etching, followed by rectangular window patterning on the backside using optical lithography and wet etching. Figure 1(b) shows that optical microscopy image of patterned windows on Si wafer. A single chip with 2 mm by 2 mm dimensions is designed for the application to conventional TEM holders. CVD-grown graphene is directly transferred onto the backside window [1]. In figure 1(c), TEM image and electron diffraction pattern show that transferred graphene perfectly covers all micrometer-sized holes. Finally, we trap aqueous buffer solution with 150 mM NaCl as a target solution in the mircowells and cover them with another graphene membrane. In figure 2(a), TEM image of a fabricated liquid cell shows leak-proof confinement of solution in the high-vacuum condition. Upon the electron beam irradiation, in figure 2(b), we observe that salts are precipitated by radiolysis of solution [6]. Electron diffraction pattern in figure 2(c) indicates that precipitates are NaCl crystals.

In summary, we develop a facile fabrication process of graphene-coated liquid cells with large viewing areas. The fabrication process includes conventional patterning and direct graphene transfer. The graphene-sealed mirowell liquid cells will be likely used for the study of nano- or bio-science [7].

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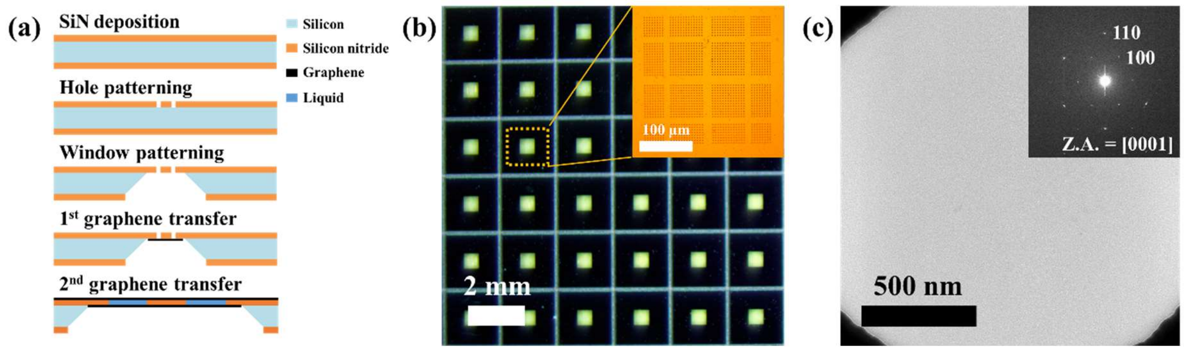

Figure 1. (a) The schematics of fabrication process of graphene-sealed microwell liquid cells. (b) Optical microscopy images of rectangular windows on Si wafer and holy microwells at a single chip (inset). (c) Bright-field TEM image and diffraction pattern (inset) of transferred graphene.

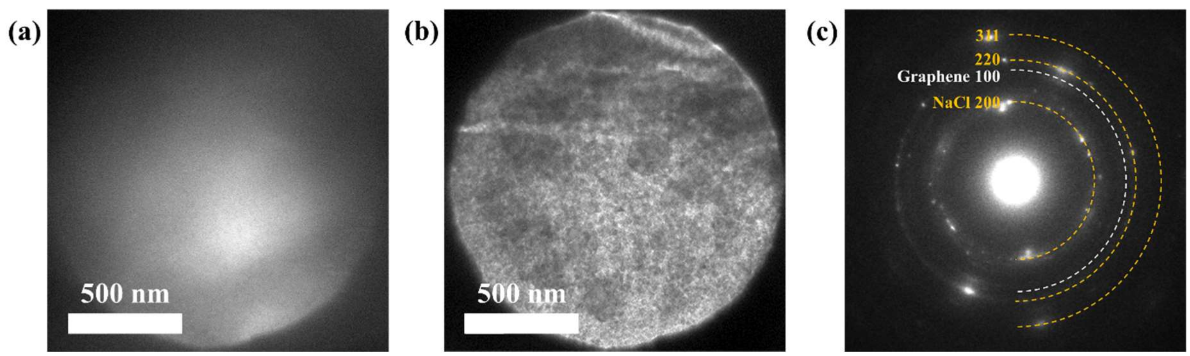

Figure 2. (a) TEM image of trapped buffer solution in the liquid cell. (b) Bright-field TEM image and (c) diffraction pattern of precipitated NaCl crystals under electron beam irradiation.