Approaches to achieving ultra-high-vacuum in 60 kV ultra-high-resolution STEM microscope

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Single-layered materials such as graphene, BN are susceptive to knock on damage. To reduce this effect, (scanning) transmission electron microscopy (STEM/TEM) at low acceleration voltage is applied. We have developed low-kV STEM/TEM instruments such as Triple C#1 (CFEG and Delta correctors, 15-60 kV) [1] and TripleC#2 (Monochromator FEG, and Delta correctors, 15-60 kV) [2] to observe layered materials with less damage. Even using with lower acceleration voltage such as 15 to 60 kV, it was found that layered materials are etched by electron irradiation with gas atoms remained in a microscope column. To reduce the etching effect, base pressure in the column should be at the range of the ultra-high-vacuum (UHV). We report approaches to achieve UHV in the low-kV Cs-corrected STEM instrument (TripleC#3)

Figure 1 (a) and (b) shows photos of the TripleC#3. An UHV stage chamber and UHV condenser-mini-lens (CM) chamber are installed on the ARM200F-based microscope, which is evacuated with a TMP-SCRP dry system for rough pumping. The UHV chambers of the microscope are differentially evacuated by sputter-ion-pumps (SIP 150 L/s for stage, and SIP 75 L/s for CM) and Titanium-sublimation pumps (TSP 400 L/s). CM chamber, stage chamber, and objective lens were sealed with metal O-rings to reduce vapor permeability. Stage and CM chambers are connected to the SIPs with metal gasket. Rubber O-rings are used for a side-entry goniometer sealing to keep compatibility of specimen holders. The microscope has a higher-order aberration corrector (Delta corrector) to compensate the six-fold astigmatism as well as spherical aberration. The electron source is CFEG. To suppress the disturbances caused by the temperature and atmospheric pressure fluctuation, the microscope is covered with an enclosure chassis.

The pressure of the stage chamber was measured by a Bayard-Alpert gauge to 8 x 10^{-8} Pa without liquid nitrogen trap. After a specimen holder was inserted with liquid nitrogen trap, the vacuum pressure was measured to 4 x 10^{-7} Pa. This indicates that pre-evacuation chamber should be improved to achieve the UHV in stage chamber. Figure 1 (c) shows a TEM image of Si[110], without Cs-corrector for image forming system, taken at 60kV. Its Fourier transform (FT) exhibits information of 76 pm as shown in Figure 1 (d). With the correction of the six-fold astigmatism in addition to spherical aberration, flat contrast area reached to 71 mrad in semi-angle. Figure 2 (a) and (b) show an ADF-STEM image taken at 60kV and its FT of the image of gold particle. The lattice fringes and FT spots of 118 pm clearly seen. An ADF-STEM image of Si[110] shows 136-pm-seperated Si-Si dumbbells and its FT shows spot of 105 pm as shown in Figure 2 (c) and (d). Thus, mechanical stability of the UHV column and UHV-evacuation-pipe is high enough to achieve atomic resolution STEM imaging.

References
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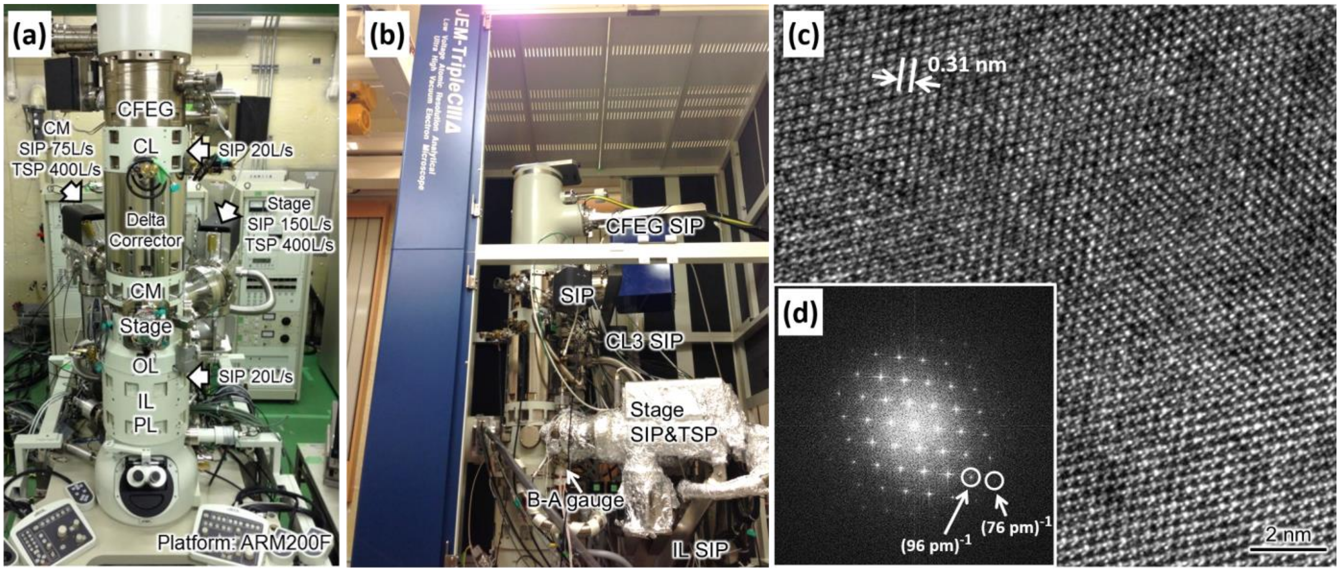

Figure 1. (a) and (b) Photos of low-kV UHV STEM instrument (TripleC#3). (c) shows TEM image (without Cs corrector) of Si[110] crystal taken at 60 kV. (d) shows Fourier transform (FT) of the TEM image.

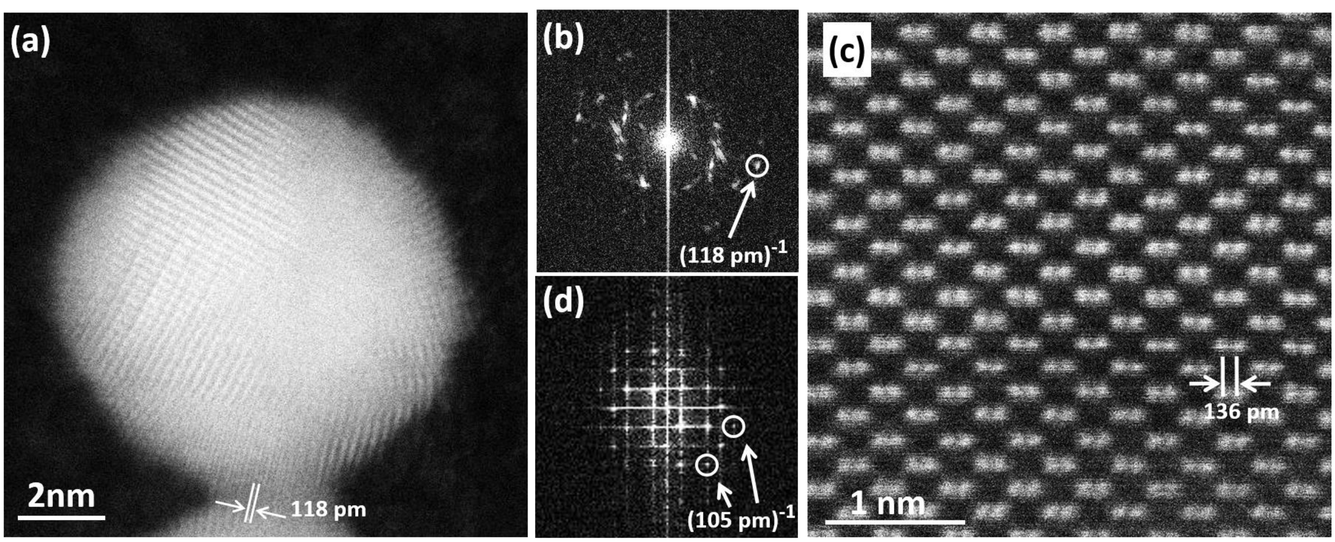

Figure 2. (a) ADF-STEM image of gold particle and (b) its FT. (c) ADF-STEM image of Si[110] crystal and (d) its FT. These images were taken at 60 kV with convergence semi-angle of 35 mrad, pixel time of 32μs/pix, and the pressure of stage chamber of 4 x 10^{-7} Pa.