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Focus Ion Beam for Cross-Sectional TEM Specimen Preparation of Nanomaterials

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Focus ion beam (FIB) together with an OMNI-probe for specimen in-situ lift-out becomes very popular in preparing cross-sectional TEM specimens. As compared with conventional TEM specimen preparation techniques, FIB not only saves specimen preparation time, but also enables researchers to make a specimen from a specific position in certain crystallographic orientation easily. However, ion beam damage is a big concern, which is especially true for high resolution electron microscopy of nanomaterials. This paper will address this challenge together with sample charging and Pt diffusion.

Ion beam damage could start from Pt deposition, the first step of cross-sectional TEM specimen preparation by FIB. Pt deposited layer is used to minimize curtain effect and protect specimen from damage, however, when a high energy ion beam is used for depositing the Pt layer, the top surface of the specimen could be damaged, leading to an amorphous layer. Nanomaterials are materials with their dimensions on the nanometer scale at least in one dimension, such as ultra-thin films and an embedded layer of nanoparticles. The ion beam damage may amorphorize interesting nanomaterails permanently (Fig. 1a). Carrying out electron-beam Pt deposition before ion-beam deposition and adjusting ion beam voltage may be helpful, but the simplest solution to protect nanomaterials is to coat them with a 40-100 nm carbon layer. With the carbon layer, high quality HRTEM images can be readily obtained (Fig. 1b).

Sample charging could be another issue in TEM specimen preparation. Fig. 2 shows a layer of nanoparticles embedded in an insulating SiO_2 film. The charging effect of SiO_2 film makes sample observation and e-beam Pt deposition difficult. A carbon layer coating can protect the nanoparticles, meanwhile, it solves the charging problem.

Pt particles may diffuse into interesting specimen area if they are directly deposited onto sample surface (Fig. 1a). These particles could be observed in carbon layer 30 nm away from Pt/carbon interface (Fig. 2). Therefore, a 40-100 nm thick carbon layer is suitable to deal with Pt diffusion related issue.

Effects of FIB parameters on TEM specimen quality are also discussed.

In conclusion, high quality cross-sectional TEM specimen of nanomaterials can be achieved by FIB together with carbon coating and suitable FIB parameters.

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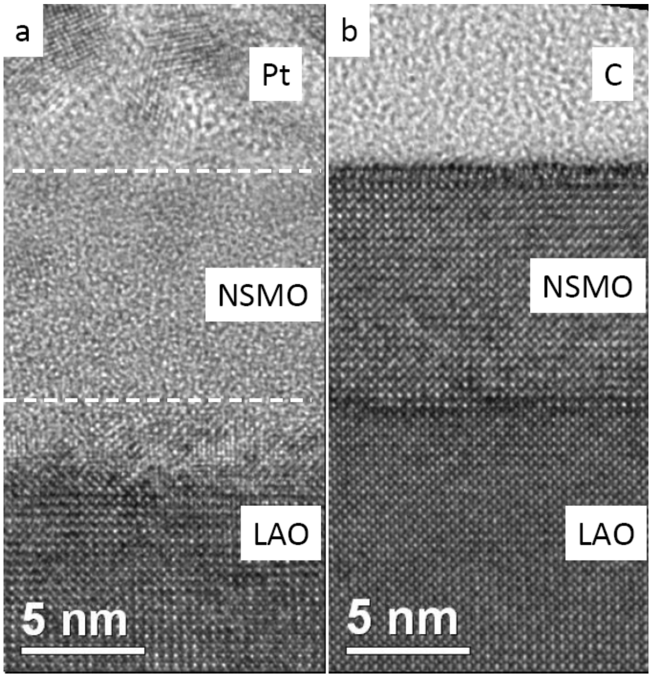

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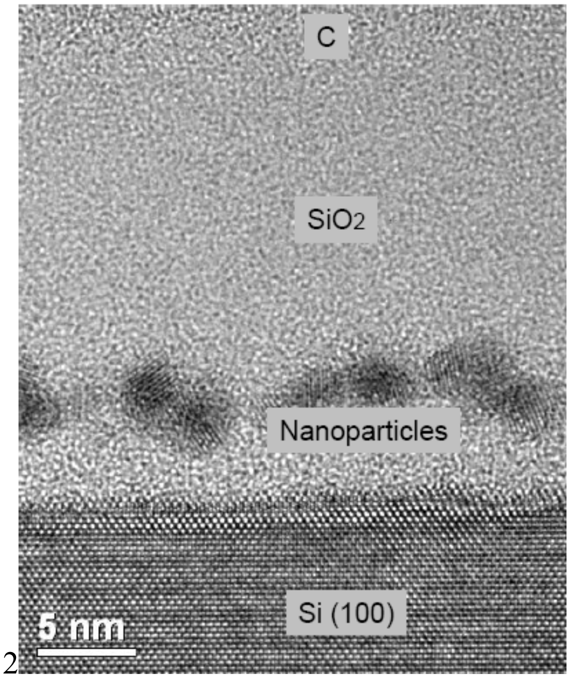

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Fig.1 NdSrMnO thin film TEM specimen prepared by FIB technique. a) Ion beam induced damage in the thin film and $LaALO_3$ substrate. b) High quality HRTEM image of the film protected by carbon layer.

Fig. 2 Nanoparticles showing lattice fringes are embedded in an insulating SiO_2 thin film covered by a carbon layer.

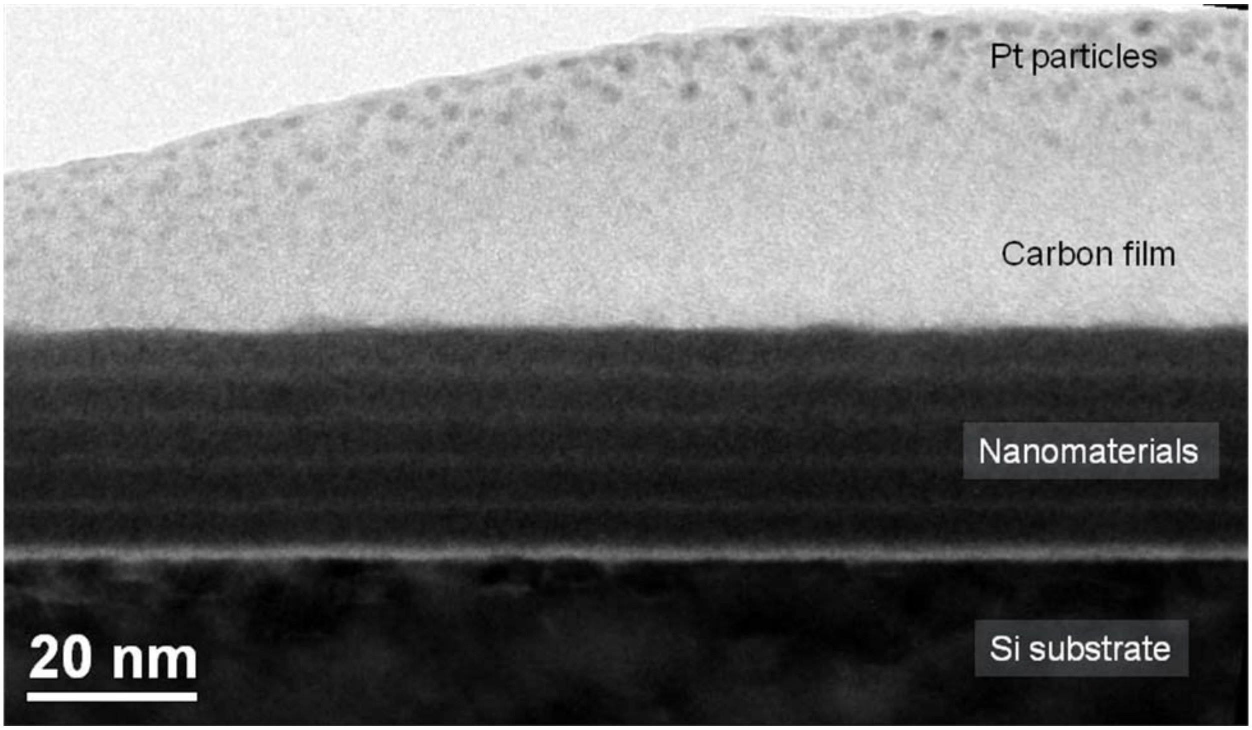

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Fig. 3 Pt partcles diffuse into the carbon protection layer for about 30 nm.