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A method for 3 dimensional structural and compositional imaging of nano-materials

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In the fields of electronic devices and nano-materials, demands for three dimensional (3D) imaging are increasing rapidly. Recently, we have developed a method for multi-directional observation of a specific site at atomic level resolution [1], [2]. In this method, the specimen is prepared using the FB-2100 focused ion beam (FIB) system equipped with a FIB micro-sampling system [3], [4] and a FIB-scanning transmission electron microscopy (STEM) / transmission electron microscopy (TEM) compatible specimen rotation holder. This technique allows milling of the specimen and STEM/TEM observation to be carried out alternately so that 3D imaging of a specific site can be performed at the optimized specimen thickness. The specimen is FIB milled to a pillar shape and mounted on the tip of a needle stub which is held in the rotation mechanism of the specimen holder. The specimen holder allows 360 ° rotation and ±20 ° tilting of a pillar shaped specimen in the narrow-gapped high resolution pole-piece of a TEM or STEM. With this technique several sets of X-ray maps can be obtained using different tilt and rotation series, and 3D elemental distributions can be extracted with very high precision [5]. High resolution STEM/TEM images such as crystal lattice fringes can be observed from various directions. Since the reduction of FIB damage is one of the important issues for high resolution image observation, a low energy Ar ion milling technique was used in the final stage of a sample preparation. A newly developed GENTLE MILL HI low energy Ar ion milling system operated at 200V was used for the reduction of the FIB damage. The Ar ion milling system has been modified to permit the direct insertion of the FIB-STEM/TEM compatible specimen rotation holder. Figure 1 shows a TEM image of the Si (110) plane (a) and the corresponding electron diffraction pattern (b) observed after the specimen was FIB milled at 40kV. A TEM image and the corresponding electron diffraction pattern taken after the specimen was Ar ion milled at 200V are shown in Fig.1c and Fig.1d, respectively. The microscope used for the high resolution TEM observation was a Hitachi H-9500 300kV high resolution TEM. After the Ar ion milling, the thickness of the amorphous layer was reduced from 28nm to 1.2nm and the contrast of the halo-ring in the diffraction pattern was weakened. Figure 2 shows high resolution TEM images (a-c) of a pillar shaped Si single crystal specimen and corresponding diffraction pattern (d-f). The crystal lattice fringes of the Si (110), (100), (1-10) planes are clearly observed in the pillar shaped specimen of 120nm x 150nm. In addition, the low energy Ar ion milling improves quality of both high resolution TEM images and 3 dimensional elemental maps.

References

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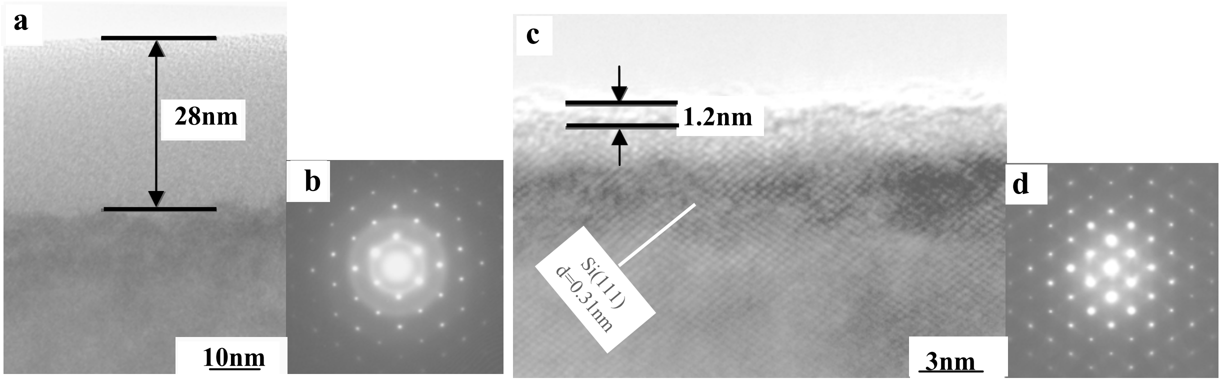

Fig.1 TEM image of the Si (110) plane (a) and the corresponding electron diffraction pattern (b) observed after the specimen was FIB milled at 40kV. A TEM image (c) and the corresponding electron diffraction pattern (d) taken after Ar ion milling the sample at 200V.

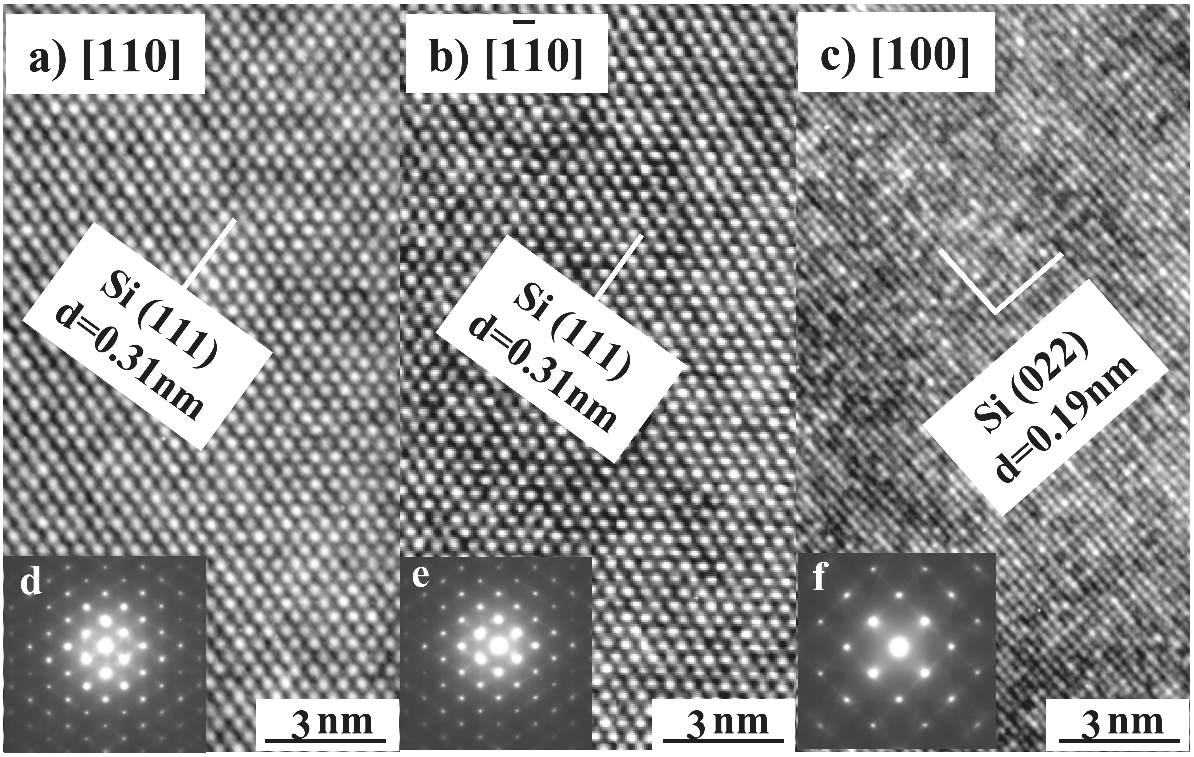

Fig.2 High resolution TEM images(a-c) and electron diffraction patterns (d-f) observed from three directions.Specimen : Si-device, Accelerating voltage : 300kV