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Meeting-report

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Electron energy loss spectrometry (EELS) analysis can offer not only elemental identification and compositions, but also insight on chemical bonding status such as bonding types through near edge features and bonding distances with coordination numbers through extended edge features appear in spectra. The bonding-related information obtainable by EELS analysis is similar to that generated by X-ray absorption spectroscopy (XAS) in synchrotrons available at several Department of Energy National Labs. In fact, an EELS system mounted on the latest aberration-corrected scanning transmission electron microscope (STEM) has benefits over the synchrotron-based XAS approaches. For example, electrons have much higher interactions with matters than X-ray, so that energy-loss signal generation is more efficient. Furthermore, analytical spatial resolution in the STEM-EELS can be as low as sub-Å levels in ab aberration-corrected STEM, whereas achievable resolution can be ~20 nm in the latest synchrotron-based XAS system. Due to the limitation in energy-loss ranges (~3,000 eV) in a typical EELS system, unfortunately, EELS information recorded from lower energy-loss edges is hardly compared with XAS data obtained through relatively higher energy edges. Therefore, we have developed a new EELS system acquirable for higher electron energy-loss signals (aiming to expand the acquirable energy-loss range from conventional ~3,000 eV to 13,000 eV) and installed to an existing aberration-corrected S/TEM JEOL JEM-ARM200CF together with a new scanning generator called MDP at Lehigh. The system development and installation were performed in collaboration with JEOL (USA, Germany and Japan) and CEOS (Germany). In this study, we applied further system optimization to acquire higher energy EELS signals.

The new EELS system is based on a CEOS Energy Filtering and Imaging Device (CEFID) [1] with two electron detectors: a CMOS-based CCD camera TVIPS TemCam-XF416 and a highly sensitive hybrid-pixel electron detector Dectris ELA. EELS signals can be acquired by either CEOS Panta Rhei software or newly developed JEOL FEMTUS software. To achieve the high energy-loss signals properly, several hardware optimizations are essential. In the spectrometer, (1) the spectrometer optics including multipole lenses before and after the liner tube were optimized and (2) an energy-selective beam blocker was inserted in the spectrometer to reduce the unexpected electron reflections. Furthermore, (3) the post objective-lens optics of the microscope was also optimized for the higher energy-loss detection by tuning the focal point of the projector lens [2]. The optimization of post-objective lens setting, especially the intermediate lens focus, is essential to obtain energy-loss illumination parallel to the dispersion direction on the electron detector without shrinking in a certain energy-loss range. This intermediate lens optimization works well up to ~10 keV energy-loss range. To achieve energy-loss range beyond 10 keV, (4) further post objective-lens tuning was required. By applying the additional lens tuning, it was possible to record high energy-loss signals beyond 10 keV without changing the accelerating voltage.

Figure 1(a) shows a 2D distribution of energy-loss electrons at the energy-dispersion plane in the 2,000-eV view mode with the spectrometer energy-shift of 10.5 keV. This energy-loss image was acquired at 200 keV without changing the accelerating voltage. It is confirmed that the energy-loss illumination on the electron detector is still parallel to the dispersion direction. For this measurement, a commercial thin film called MAG*I*CAL^{TM} has been used, which consists of an ion-milled cross-sectional multilayers of Si/Si-Ge alloy, fabricated by a molecular-beam epitaxy technique [3]. Figure 1(b) shows an EELS spectrum integrated vertically to the dispersion direction. The Ge K edge is clearly visible over 11-keV energy-loss range, which was taken from one of Si-Ge layers with ~10 nm width as shown in Fig. 1(c). It should be noted that the Ge composition in this Si-Ge layer is ~19 at%. An EELS spectrum-imaging (SI) dataset was acquired from a green box region shown in Fig. 1(c). A high-angle annular dark-field (HAADF) image acquired together with the SI data is shown in Fig. 1(d) with the Ge-K edge map extracted from the SI dataset (Fig. 1e). The Si-Ge layers with 10-nm width are clearly resolved in the Ge-K edge map, which is already beyond the spatial resolution achievable in any synchrotron-based system [4].

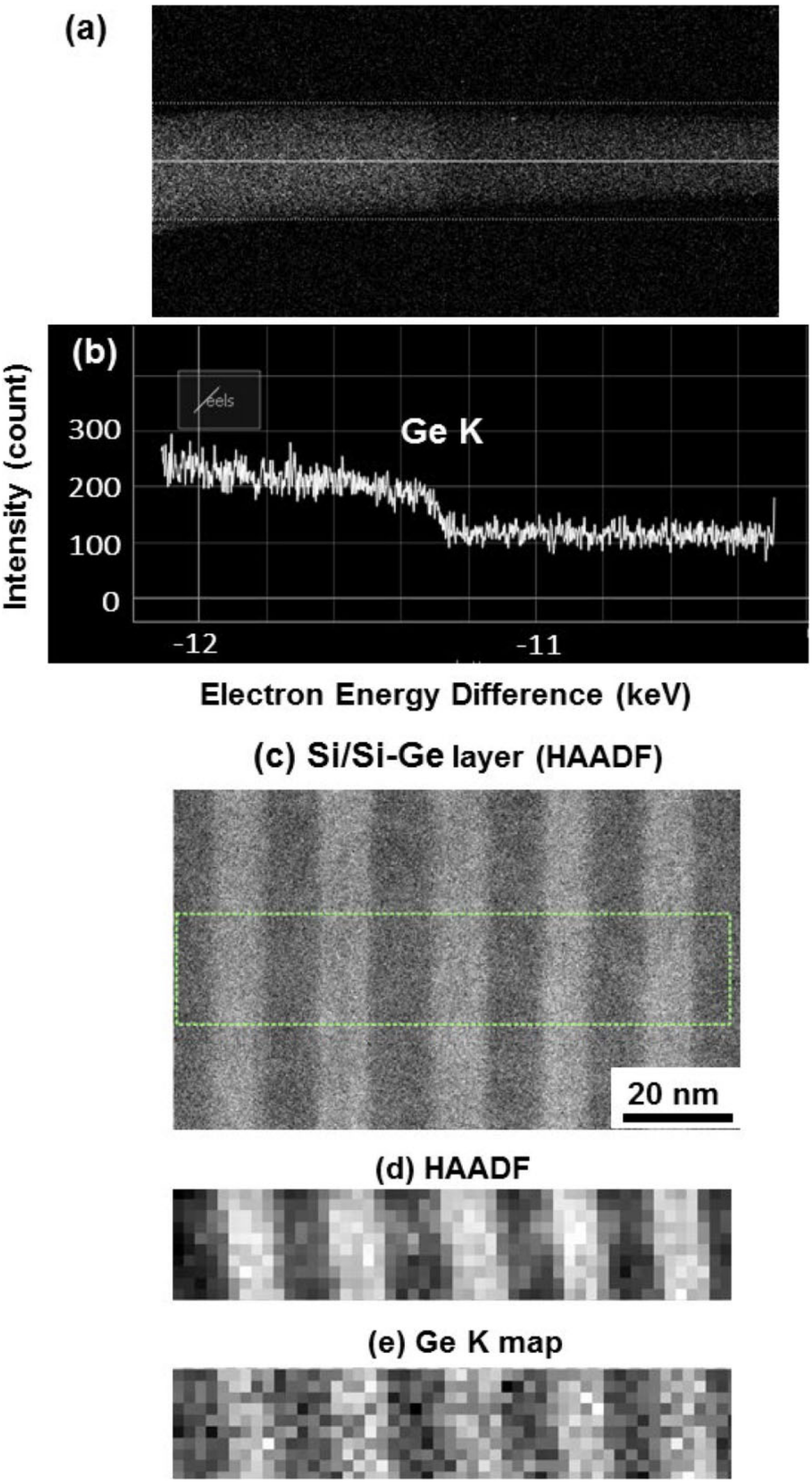

Figure 1: (a) A 2D-dispersed energy-loss distribution at the dispersion plane in the 2,000-eV view mode, taken by the CEFID system with the energy shift of ~10.5 keV after the system optimization. (b) An EELS spectrum showing the Ge K edge over 11 keV, obtained by integration of the energy-loss image shown in (a) vertically to the dispersion direction. (c) A HAADF-STEM image showing Si/Si-Ge layers in the MAG*I*CAL^{TM} thin specimen. (d) A HAADF-STEM image obtained together with an EELS-SI dataset from a green boxed region shown in (c). (e) A Ge-K edge map extracted from the EELS-SI dataset.

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