

Quantitative Li Mapping in Al alloys by Sub-eV Resolution Energy-Filtering Transmission Electron Microscopy (EFTEM) in the Aberration-Corrected, Monochromated TEAM0.5 Instrument

M.D. Rossell¹, M. Watanabe², R. Erni¹, V. Radmilovic¹ and U. Dahmen¹

¹ National Center for Electron Microscopy, Lawrence Berkeley National Laboratory, Berkeley, CA94720, USA.

² Dept. of Materials Science & Engineering, Lehigh University, Bethlehem, PA18015, USA.

Elemental/compositional mapping is especially suitable for characterization of nano-scale features of materials, such as fine precipitates and interfaces/boundaries, in (scanning) transmission electron microscopy ((S)TEM) to reveal two dimensional fluctuations in composition in vicinity of the fine features. Besides X-ray and energy-loss electron imaging in STEM, such elemental distributions can also be obtained by an EFTEM method in the TEM mode. The EFTEM approach is useful to obtain maps from large fields of view with a relatively short acquisition time. In conventional instruments, however, the EFTEM method is inferior to the STEM-based energy-loss approach in both spatial and energy resolution. The poor spatial and energy resolution in EFTEM can be improved if an EFTEM image is acquired by a sub-eV energy-slit with a monochromated illumination in an image-corrected system. The high spatial/energy resolution EFTEM technique is now applicable using the aberration-corrected, monochromated TEAM instrument installed at NCEM.

Unfortunately, the sub-eV EFTEM acquisition may be limited since electrons with same energy-loss range are hardly well separated in the sub-eV range, which is called non-isochromaticity (NIC) of the filter. It is essential to correct the filter NIC for sub-eV EFTEM imaging. In this study, NIC values were determined at single pixels by carefully measuring zero-loss peak position in an EFTEM spectrum-image acquired around the zero-loss peak. Then, the measured NIC values were used to correct the NIC. The result of the NIC correction is compared with the pre-corrected filtered image in Fig. 1. By the NIC correction, the energy deviation is significantly corrected at least in terms of the peak positions of extracted spectra.

The sub-eV EFTEM method with the NIC correction was applied to quantify Li composition in vicinity of complex core/shell nano-precipitates in Al-Li-Sc alloys. EFTEM spectrum images (SIs) were acquired around Al plasmon-peak range (12-17 eV) with a 0.1 eV step using the aberration-corrected, monochromated TEAM0.5 microscope. After the NIC correction, the spatial drift correction [1] and multivariate statistical analysis [2] were applied to the acquired EFTEM spectrum images. In Al-Li alloy, the Li composition can be obtained by measuring the plasmon peak position [3]. Figure 2 shows maps of (a) Al plasmon peak position and (b) Li composition determined from the sub-eV EFTEM spectrum image. The accuracy of the Li composition is related to the accurate measurement of the Al plasmon peak position, which requires the NIC correction. The sub-eV EFTEM acquisition under monochromated illumination in image-corrected instrument can provide atomic level resolution [4].

References

- [1] B. Schaffer et al. *Ultramicrosc.* 102 (2004), 27.
 [2] M. Watanabe *et al.*, *Proc. Inter. Conf. on Solid-Solid Phase Transformations in Inorganic Materials 2005*, vol. 2, 431 (2005).
 [3] D.B. Williams and J. Edington, *Phil. Mag.* 30 (1974), 1147.
 [4] This research was supported by the Scientific User Facilities Division under Contract # DE-AC02-05CH11231 and the TEAM project under Contract # DE-AC03-76SF00098 of the Office of Basic Energy Sciences, U.S. Department of Energy.

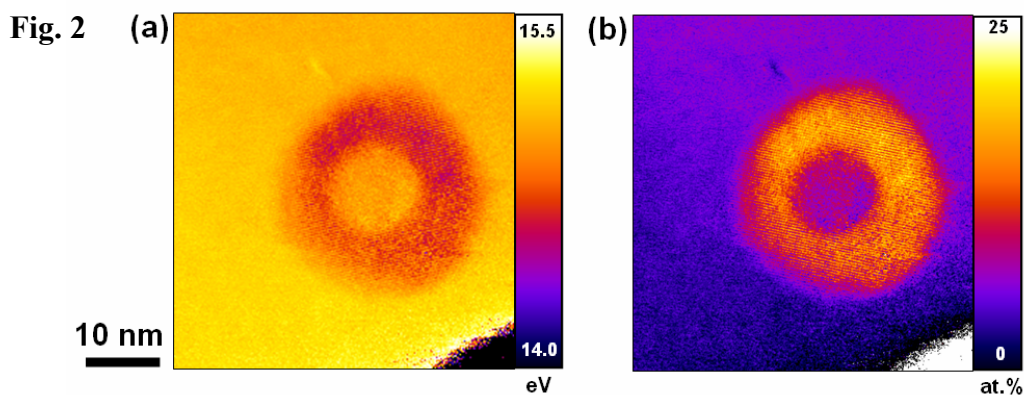
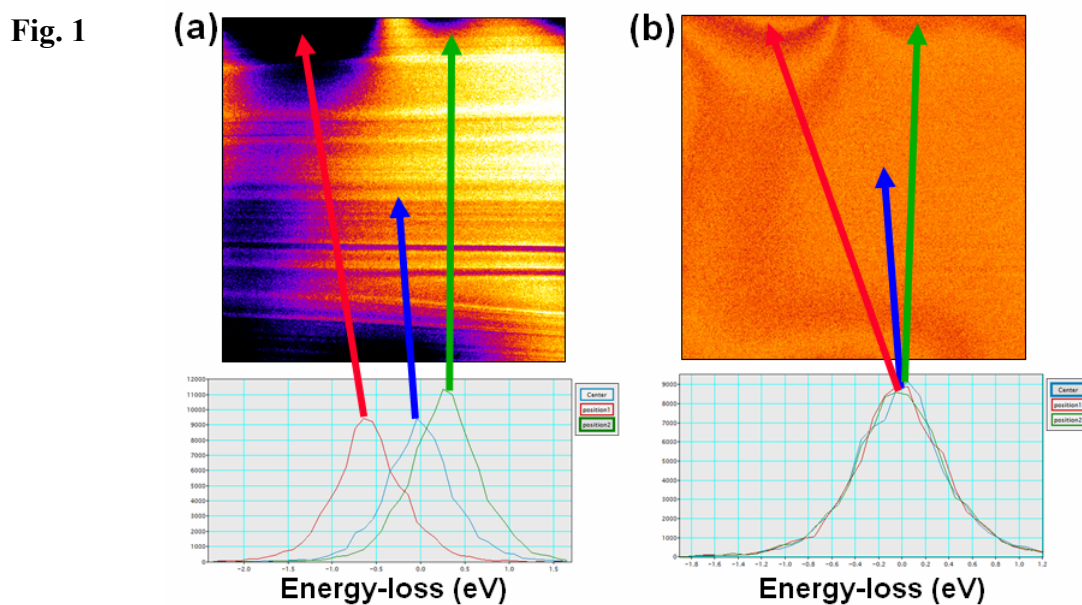


Figure 1: Comparison of the zero-loss peak images obtained with a 0.1 eV energy slit: (a) before and (b) after the NIC correction.

Figure 2: (a) A Plasmon-peak position map around a core/shell precipitates in an Al-Sc-Li alloy after the NIC correction. (b) A quantitative Li composition map determined from the Plasmon peak shift.