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High Spatial and Energy Resolution EELS using a Monochromated STEM

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The ability to directly correlate electron energy loss spectroscopy (EELS) with Z-contrast images in the scanning transmission electron microscope (STEM) provides a powerful tool to characterize nanoscale materials. The incorporation of a monochromator into the FEI Tecnai F20 UT at the National Center for Electron Microscopy (NCEM) extends the power of these techniques by increasing the sensitivity of core-loss spectra to small changes in fine-structure and by allowing the low-loss region of the spectrum to be analyzed in detail. Here, the configuration of the monochromated Tecnai will be discussed and examples of spectra obtained utilizing the highest spatial and energy resolution will be presented [1].

As an example of the use of monochromator at the highest energy resolution, a series of CdSe quantum dots (QDs) have been analyzed [2]. Using the monochromator, valence EELS overcomes the spatial limitations of other methods and allows the optical properties of individual QDs to be assessed. From these measurements, the properties of a particular sized QD can be accurately quantified and the size distribution can be estimated. Figure 1a shows a typical low-loss spectrum for bulk CdSe. The CdSe QDs probed by EELS were dispersed on a Si_3N_4 film, with the band-gap measurements shown in Figure 1b. Although the overall size dependence of the band gap is in agreement with theoretical values [3] and optical absorption measurements [4], this first local VEELS study of single QDs reveals significantly higher band-gap values. This disagreement between the results can be attributed to the impact of the particle-size distribution on the ensemble optical measurements and illustrates the extra level of sensitivity to the properties of nanoparticles that can be achieved by EELS.

As an example of the use of EELS at the highest spatial resolution, intergranular phases in Si_3N_4 have been analyzed for five sintering additive types, La_2O_3, Sm_2O_3, Er_2O_3, Yb_2O_3 and Lu_2O_3 (Figure 2) [5]. Precise EELS measurements to identify specific atomic bonding configurations were performed on the Sm sample with a 1.4Å probe size and are shown in Figure 3. There is no difference between the Sm N_{45} edge spectra at A and B, suggesting that the atomic environment and bonding characteristics are the same for the Sm atoms at both locations. For the Si L_{23} edge, a double peak (102eV and 103.5eV) appears in the spectrum taken from atom position A. The first peak can be identified with a Si-N bond while the second peak is associated with a Si-O bond. This indicates that the environment around the terminating Si atoms at A contains oxygen, while the atomic environment of the Si atoms at B appears to involve only nitrogen [6].

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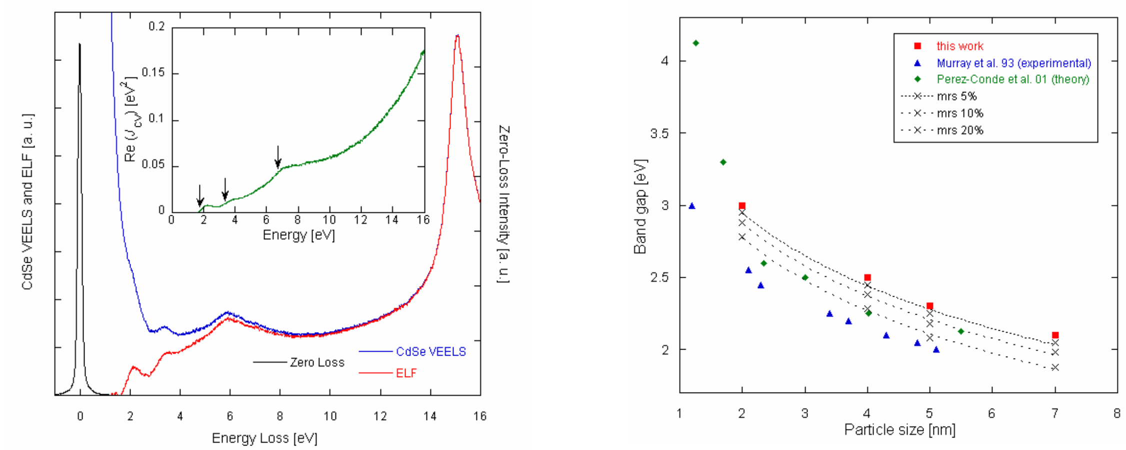

FIG 1. (a) VEEL spectrum bulk CdSe (blue), with the corresponding zero-loss peak (black) and the energy-loss function *ELF* (red). The inset shows the real part of the interband transition strength J_{CV} (b) Size dependence of the band gap of CdSe quantum dots.

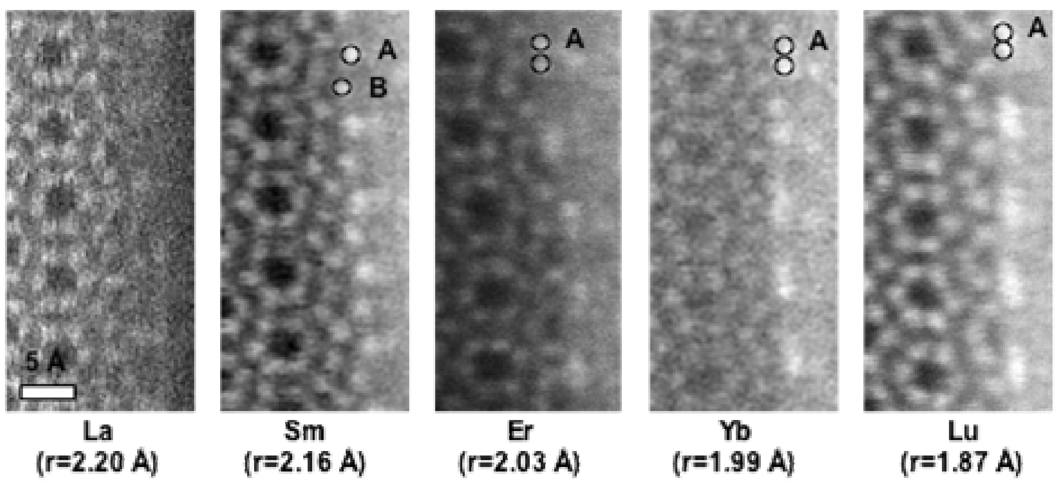

FIG 2. Z-contrast images along the [0001] zone axis showing the attachment of the rare-earth atoms at the Si_3N_4 prismatic plane facing the amorphous intergranular phase.

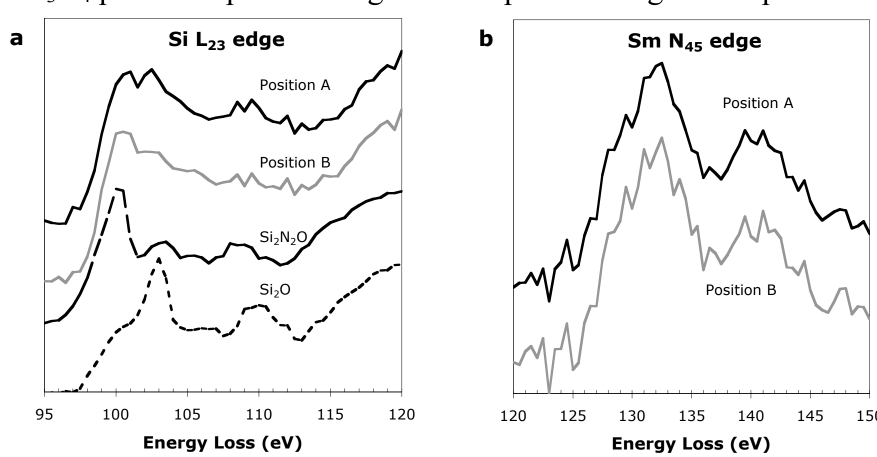

FIG 3. EELS measurements from Si_3N_4 containing Sm. The results for (A) the Si L_{23} and (B) the Sm N_{45} edges identify the individual bonding characteristics at positions A and B.