

Simultaneous High-Speed DualEELS and EDS Acquisition at Atomic Level

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The modern STEM is capable of providing a wealth of information, but without fast, efficient detectors that wealth would be lost. Over the last few years, EELS systems have become faster, more efficient and capable of acquiring large energy and dynamic ranges while EDS detectors have greatly increased collection angles and counting rates. These advances would be meaningless without the improved brightness of modern electron sources and the aberration correction of the electron probe. In this paper, we show how advances in instrumentation and software control allow EELS and EDS signals to be acquired at high-speed with minimal acquisition dead time. We will present data at atomic level over a range of conditions that show the complementary nature of the techniques and highlight the strength of each.

To achieve efficient joint EELS / EDS data acquisition, we have coupled together the STEM scan system, EELS detector and EDS detector via a single clock mastered off the EELS detector to ensure all the systems are in exact synchrony. To ensure data fidelity, the native detector applications are used to acquire the data into buffers and the data is transferred to the final application (in this case DigitalMicrograph) in blocks while the CPU is idle.

To illustrate these advances, data were acquired at IBM San Jose, CA, USA using a probe corrected 200 kV STEM equipped with C-FEGJeol ARM STEM operating at 200 kV equipped with a C-FEG source. A GIF Quantum ER EELS system equipped with DualEELS capability was used for the acquisition of energy-loss data and a large area (~1 sr). EDS detector was used to acquire the EDS data. EELS data were acquired in DualEELS mode and the low-loss (0 eV to 500 eV) and core-loss (400 eV to 900 eV) EELS spectra were acquired nearly simultaneously with 10 μ s transition time between exposures. Having the low-loss spectrum allows the possibility of removing the effects of plural scattering which change the shape of the near edge structure and also the correction for the effects of energy drift. In this way, the true chemical shift of the edge threshold can be observed and accurately measured. The spectrometer was set up for an energy resolution analysis with a dispersion of 0.25 eV / channel. With these conditions, the energy resolution measured was 0.75 eV with 500 eV energy range in the EELS spectrum. By looking at the shape of the near edge structure in the EELS spectrum, it is possible to extract information on the oxidation state, coordination state and much more. In addition, optical information present in the low-loss region is also available since the EELS data were taken in DualEELS mode.

The sample analyzed is a SrTiO_x / LaFeO_x crystal and the data acquisition was carried out across the interface. EELS data were used to collect chemical information and generate elemental maps of the O, Ti, Fe and La. In the EELS spectrum, the main signals for the Sr are the M_{4,5} at 130 eV and the SrL_{2,3} at 1940 eV. The M-edges have a poor peak-to-background ratio due their low energy while the L-edges are out of the energy range for this experiment (0 eV to 900 eV). Since the EDS data were acquired simultaneously with the EELS data and long dwell times were used, the EDS signal can be used to

generate a Sr elemental map that can be ultimately combined with those obtained by EELS. Since the EDS signal does not provide chemical information, only composition can be studied for Sr at the experimental conditions used in this paper. Chemical information present in the EELS spectrum is available only for O, La, Fe and Ti. Figure 1 shows a colorized elemental map of Ti, Fe and La obtained using EELS and using EDS. The color map suggests intermixing between Fe and Ti. Such intermixing can be confirmed and further investigated using EELS by looking at the fine structure of the edges across each atomic column. Figures 2a-c show EELS spectra of Ti L, O K and Fe L edges extracted from the selected regions in Figure 1. Changes can be observed in every spectrum although they are pretty remarkable in the case of the O and Fe in Figures 2b,c with the latter showing a chemical shift towards higher energy moving into the LaFeOx. This is a clear indication of a change in the oxidation state. The low-loss spectra across the same selected regions are shown in Figure 2d. Although, the low-loss electrons are more delocalized compared to those in the core-loss region, minor changes can be also observed. The interface was also analyzed using STEM diffraction where diffraction patterns are taken point by point across the interface along with EDS spectra. This gives a further insight into the structure across the interface and information about strain and crystal distortion can now be revealed.

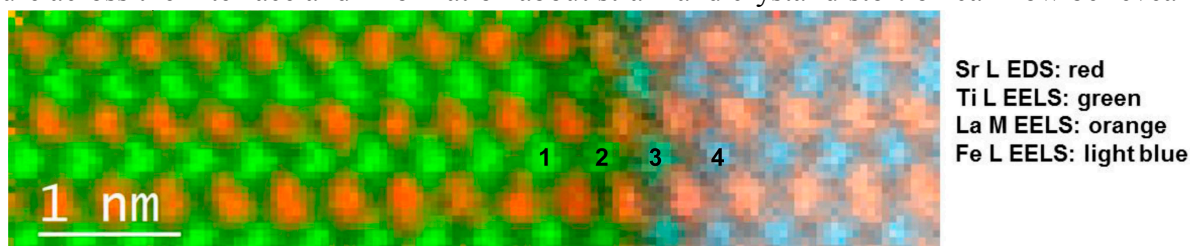
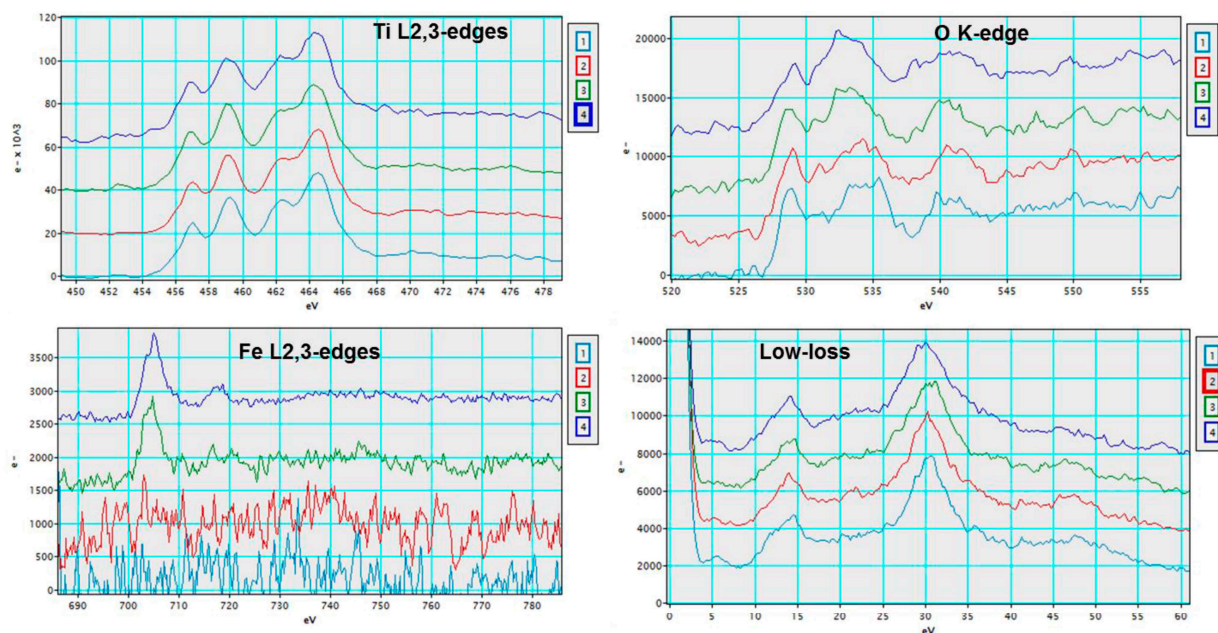


Figure 1: Color maps of Ti L at 456 eV in green, La M at 832 eV in orange and Fe L at 708 eV in light blue obtained using EELS and Sr L at 1.81keV in red obtained using EDS. The numbers 1 - 4 correspond to the selected region where the EELS spectra in Figures 2a-d were extracted. There appears some elemental intermixing at the interface



Figures 2: EELS spectra extracted from the selected region in Figure 1; a) Ti L_{2,3}-edges at 456 eV; b) OK-edge at 532 eV; c) Fe L_{2,3}-edges at 708 eV; d) Low-loss region