Advanced in EELS Instrumentation and Analysis: High-Speed Spectroscopy, with Extended Energy and Dynamic Range

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The acquisition of high-quality EELS data in the transmission electron microscope (TEM) presents many challenges not experienced by most TEM acquisition modes. The central challenges are dose efficiency and dynamic range. For EELS, the range of intensities of interest in a single spectrum can often span 6 to 7 orders of magnitude making recording problematic. Since the spectrum is recorded in parallel, EELS acquisition can be very dose efficient but only if the acquisition device can be read out quickly and efficiently.

To address these issues, we have developed a next generation post-column energy filter, the GIF Quantum, which excels at energy filtered imaging but also incorporates several new features that allow the optimal collection of energy-loss spectra generated by the high-brightness electron sources currently available. Key features of the GIF Quantum include a new CCD camera design that achieves high spectra readout rates (1kHz) with very little overhead, and a system of electrostatic deflectors that allows the nearly simultaneous (<10µs delay) recording of dual energy-loss ranges with microsecond exposure control. These deflectors enable the optimized acquisition of both high-energy core-loss electrons together with the zero-loss and low-loss electron signal.

In this talk, I will present details and advantages afforded by these new developments and show application data collected under optimized conditions from different materials. As example in this abstract I show in Figure 1 the elemental distribution map of the Sr $L_{2,3}$-edges at 1940 eV in red, Ti $L_{2,3}$-edges at 456 eV in green, Fe $L_{2,3}$-edges at 708 eV in amber, La $M_{4,5}$-edges at 832 eV in blue and Cu $L_{2,3}$-edges at 931 eV in purple. Such map was acquired in just about 4 minutes using an exposure time per spectrum of 5ms and this proves the high sensitivity and speed of the camera fitted to the GIF Quantum. It is particularly interesting to see the diffusion of Cu into the Fe atomic column at the interface.

In Figure 2a, I show the elemental distribution map of Ce and Zr. These maps in addition to only compositional information they deliver chemical information. The different oxidation state of Ce is chemically and atomically resolved. The substrate seems to contain only CeO_2 with Ce in the oxidation state 4+ whereas the interface with the Yttria-Stabilized Zirconia (YSZ) seems to contain Ce_2O_3 with the Ce in the oxidation state of 3+. The Ce chemical maps were obtained by means of multiple linear least square (MLLS) using the Ce $M_{4,5}$-edges at 883 eV reference spectra with the Ce in the oxidation state 3+ and 4+ respectively. Differences in the shape of the Ce M spectrum can be easily observed easily resolved even with an energy resolution of 2 – 3 eV. This is shown in Figure 2b that reports the background removed Ce $M_{4,5}$-edges spectra extracted from the region across the interface and in the bulk respectively. The chemical maps shown in Figure 2a were acquired in less

than 2 minutes using an exposure time per spectrum of 20 ms and a beam current of 125 pA. Again, this proves the sensitivity and speed of the GIF Quantum camera and also the power of EELS capable to acquire compositional information as well as chemical.

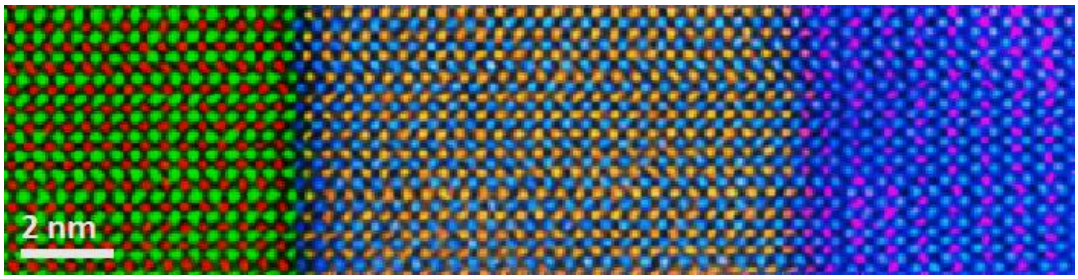
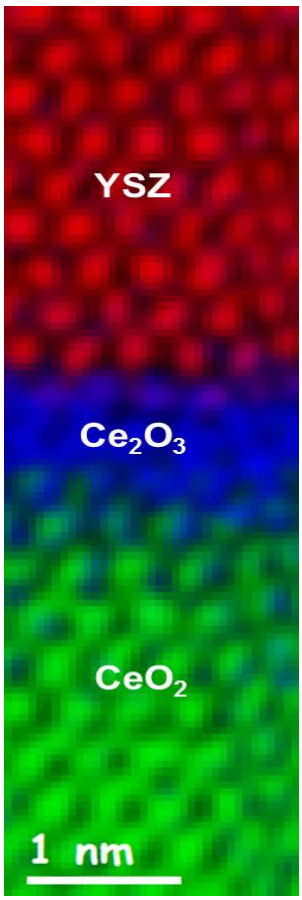
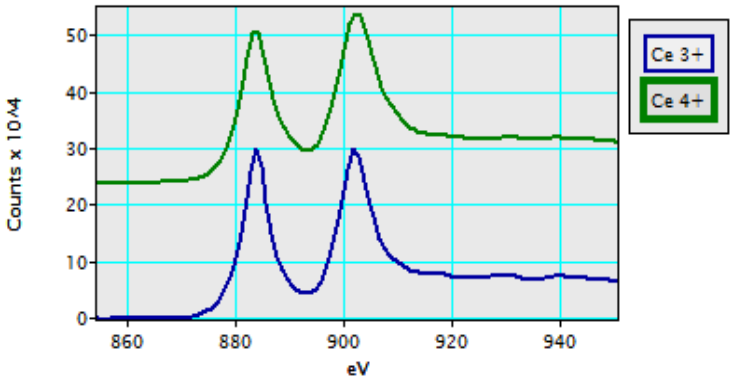


Figure 1: EELS colorozed maps of Ti L at 456 eV in green, La M at 832 eV in blue and Fe L at 708 eV in amber, Sr L at 1940 eV and Cu L at 931 eV in purple. Particular thanks go to Drs Phil Rice and Teya Topuria at IBM Almaden, San Jose CA, USA for providing access to the microscope installation, the TEM specimen and assistance in setting up the microscope for the experiment.





Figures 2: a) Atomic EELS colorized elemental maps of Zr $L_{2,3}$-edges at 2222eV in red and the Ce $M_{4,5}$-edges at 883 eV in green and blue for the Ce 4+ and Ce 3+ respectively. The interface YSZ / Ce_2O_3 look fairly nice and flat unlike the interface Ce_2O_3 / CeO_2 that looks quite rough. The different chemistry of the CeO_x is spatially resolved at atomic level; b) background removed Ce $M_{4,5}$-edges spectra extracted from the areas in the sample where Ce is in the 3+ and 4+ oxidation state respectively. The shape is different showing the different chemistry. Particular thanks go to Prof Peter A. Van Aken at Max Plank Institute in Stuttgart Germany for providing access to the microscope installation and the TEM specimen used for the experiment.