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Comparative ELNES Measurements on Selected Transition Metal Oxides on a New High Energy-Resolution Spectrometer / Monochromator TEM

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Although it is well known that EELS near edge fine structures (ELNES) are a powerful tool for studying chemical bonding at the nanometer level, important applications were prohibited due to the limitation of the initial energy width of the source, which is typically 0.6 eV for Schottky emitters. If the energy resolution can be improved towards the 0.1 eV regime, near edge fine structure details can be revealed not previously observable by EELS in common instruments.

To address this, FEI / Gatan has constructed a monochromized 200 kV (S)TEM which aims at the 0.1 eV energy resolution level for EELS. The improved energy resolution is due to several factors: (a) The energy spread of a Schottky field emitter is reduced by a Wien filter monochromator positioned directly after the field emission gun. (b) The 200 kV high tension tank was improved by adding mechanical as well as electrical damping elements [1], and (c) a high resolution energy filter delivers more stable electronics as well as improved electron optics [2]. The electron optics of the spectrometer as installed at the FELMI now comprises additional multipol lenses in front of the magnetic prism, which eliminate 3rd and some of the 4th order spectral aberrations. The electron optical performance of the spectrometer can be represented by its isochromatic surface as displayed in fig. 1. It shows the relationship between the spectrometer collection angle (implicitly expressed by the size of the indicated apertures) and the focussing error of the spectrometer. The filter can now accept maximum scattering angles, which are about 70% larger compared to conventional post-column filters without compromising energy resolution. Larger spectrometer entrance apertures in combination with further reduced camera lengths result in a system with considerably improved collection efficiency for EELS measurements, giving spectra of higher quality especially on dose sensitive samples.

The main objective of this study is to check the performance of the new spectrometer on a monochromized and unmonochromized 200kV FEG (S)TEM with a well characterized sample and to compare these results with previous experiments. For this purpose, vanadium oxide (V2O5) was chosen for its challenging fine-structure at the V 2p(3/2) and V 2p(1/2) peaks and for its known sensitivity for reduction under the electron beam [3,4].

V2O5 has an orthorhombic structure, with vanadium being surrounded by six oxygen atoms, forming a distorted octahedron. The L3 and L2 peaks are further split into doublet features as a consequence of an octahedral ligand-field splitting, which divide the d-state into t2g and eg orbitals. Although the L3 and L2-edge of TiO2 (the neighbouring d0 oxide) show well-resolved t2g-eg features, the energy separation for V2O5does nothave the same value and in fact is only in the order of the energy resolution of a typical FEG TEM at around 0.8 eV [5]. Experimental spectra of V2O5 taken on different instruments are displayed in fig. 2. While the basic shape of the V L2,3 edge comes out clearly on all instruments, most fine structure details are only observable with field-emission instruments, where the monochromized TEM exhibits a significantly better separation of the spectral features. The paper intends to demonstrate the usefulness of the new spectrometer/monochromator combination for ELNES measurements applied to technologically relevant transition metal oxides.

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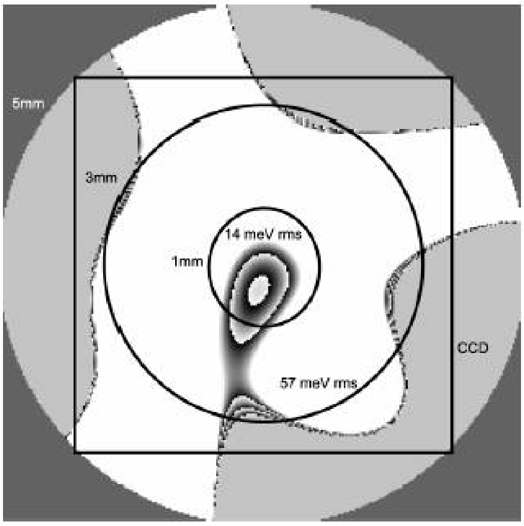

Fig. 1: Extrapolated experimental isochromatic surface of the high resolution EELS spectrometer at 200 kV. Apertures are drawn as circles. Measurements indicate that for a practic al total energy resolution of 0.3 eV on a monochromated microscope, a 3 mm aperture can still be used.

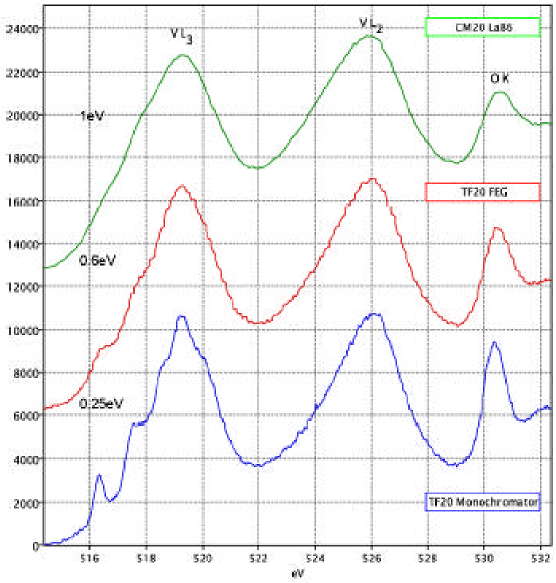

Fig. 2: Experimental EELS spectra from V2O5 recorded on a FEI CM20/ LaB6 / GIF200 (top), a T20F FEG with a high-resolution spectrometer (middle) and a T20F / monochromator (bottom). The t2g-eg ligand field splitting of 0.8 eV being clearly revealed only on the monochromated microscope.

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