ELNES Investigation of different niobium oxide phases

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Purely stoichiometric diniobium pentoxide (Nb₂O₅) is an attractive material for dielectric applications in high capacity solid electrolyte capacitors. However, besides the desired Nb₂O₅ there is a variety of other niobium oxide phases with different electrical properties [1]. Therefore, the determination of the local stoichiometry within the nanoscale dielectric oxide layers in capacitor structures is essential to understand failure mechanisms. EELS and ELNES investigations with high spatial resolution are well suited for this task, but reference spectra for niobium oxide phases with known stoichiometry are not available up to now. Their measurement is the aim of the present work.

Niobium monoxide (NbO) and diniobium pentoxide (Nb₂O₅) were investigated. The structure of the two stoichiometric oxides was checked by selected area electron diffraction. EELS reference spectra of the Nb-M₄,₅, Nb-M₂,₃ and O-K edges were recorded, using a JEOL JEM-3000F field emission transmission electron microscope equipped with a Gatan Imaging Filter. After background subtraction, multiple scattering was taken into account by Fourier-ratio deconvolution.

Figure 1 shows representative EELS reference spectra. NbO and Nb₂O₅ can be clearly distinguished by the fine structure of the Nb-M₄,₅ (Fig. 1.a), Nb-M₂,₃ (Fig. 1.b) and O-K edges (Fig. 1.c). The two oxides can also be distinguished in a quantitative manner by measuring the intensities under the Nb-M₂,₃ edges (I_M₂,₃) and the O-K edge (I_K) and by calculating their ratio I_M₂,₃/I_K. Moreover, k-factors can be derived, which can be later used to characterize niobium oxide phases with unknown stoichiometry.

![Fig. 1 Energy loss reference spectra of the Nb-M₄,₅ (a), Nb-M₂,₃ (b) edges and of the O-K edge (c) recorded from stoichiometric NbO and Nb₂O₅.](image)

References

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