ELECTRON ENERGY LOSS SPECTROMETRY IN THE TEM: CHEMICAL INFORMATION AT THE NANOMETER SCALE

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Progress in materials science and nanotechnology are closely connected with the availability of instruments that allow to study the morphology, geometric structure and chemical composition of materials at the nanometer scale. Modern analytical transmission electron microscopes (TEM) certainly fulfil this task in an unsurpassed way. Observation over a large range of magnifications, dark and bright field imaging, electron diffraction, tomography and holography are some of the imaging techniques that can be used in a well equipped analytical TEM. In addition to the imaging capabilities, there is the spectroscopic part and most analytical instruments are equipped with energy dispersive detectors for the detection of characteristic X-rays or electron energy losses caused in inelastic interactions between the probing electrons and the sample material. Especially electron energy loss spectrometry (EELS) is used for the characterization of inelastic scattering processes over a wide energy range, including plasmon and valence band excitations as well as core shell excitations. The spectra can be recorded at high special resolution and provide information about the elemental composition and electronic structure of a material. Due to the high energy resolution, EELS is very sensitive to even slight changes in the electronic structure.

The analytical power obtained by combination of EELS and TEM will be demonstrated for the case of vanadium oxides. Vanadium is able to adapt a wide range of oxidation states ranging from II+ to V+. This makes it an interesting material not only in the field of catalysis but also in energy storage, where vanadium oxides are used for the intercalation of Li ions. The shape and fine structure of the vanadium L and oxygen K edges change in function of the vanadium oxidation state and the geometric arrangement of the oxygen atoms coordinating the vanadium [1] (Fig. 1). This can be used for the identification of the oxidation state of a vanadium oxide species as shown for example in Fig. 2 for the case of a vanadium oxide cluster dispersed on the surface of a multi walled carbon nanotube.

Taking vanadium phosphorous oxides as an example it will further be shown that by the assistance of band structure calculations based on density functional theory and simulated EELS spectra, contributions from differently coordinated atoms to the observed spectrum can be identified [2].

References:

[1] C. Hébert, M. Willinger, D.S. Su, P. Pongratz, P. Schattschndeider, R. Schlögl, Eur. Phys. J. B. **28** (2002) 407
[2] M.-G. Willinger, D. S. Su, R. Schlögl, Phys Rev B **71** (2005) 155118.

Figures:

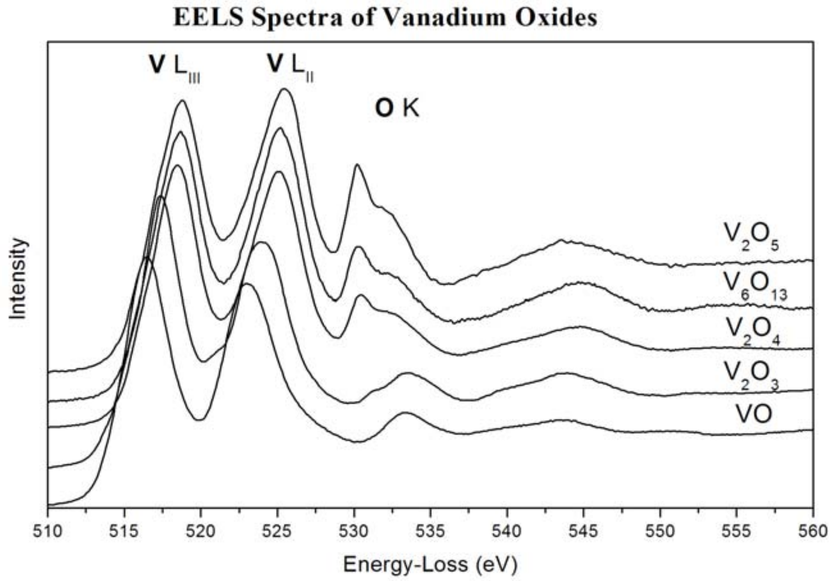

Fig. 1. Vanadium L$_{III}$, L$_{II}$ and oxygen K ionization edges recorded for different binary vanadium oxides. Clearly visible are the shift in position and the changes in the intensity ratio of the vanadium L edges as a function of the vanadium oxidation state. The structure of the oxygen K ionization edge also changes drastically when going from V_2O_5 to VO.

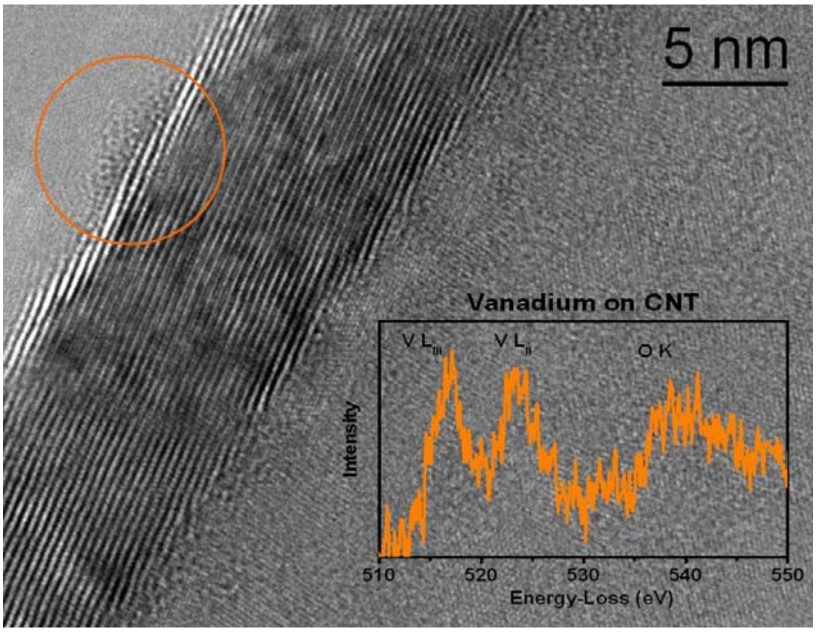

Fig. 2. Although the spectrum recorded from the region indicated by the circle is very noisy, the energetic position of the ionization edges allows the assignment of a V^{III+} oxidation state for the vanadium oxide cluster on the surface of the carbon nanotube.