V_2O_5 thin film electrodes in rechargeable Li-ion batteries – Sample characterization by EELS

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V_2O_5 is a promising candidate as intercalation compound for rechargeable Li-ion batteries due to its orthorhombic layered crystal structure which allows reversible Li^+ intercalation processes [1], [2].

Thin films (300nm in thickness) were prepared by ion-beam sputtering from a V_2O_5 powder target. Silicon wafers were used as substrate for XRD measurements; alternatively glass substrates were chosen for conductivity investigations. Different sputter parameters, such as oxygen partial pressure and substrate temperature were combined to create the desired structure. It was recorded by XRD that the required layered structure can only be achieved by adding oxygen during sputtering and annealing the sample under ambient atmosphere afterwards.

EELS investigations were carried out to determine the V-oxidation state depending on the preparation conditions quantitatively. Since the typical edges (VL_3, VL_2 and O_K) appear in an energy loss interval of about 30 eV, quantification based on the typical L_3/L_2 ratio method [3] becomes difficult. Therefore the chemical shift in the different edge positions depending on the preparation parameters was investigated in detail [4], see also figures 1 and 2.

To quantitatively evaluate the oxidation states these results are compared to well defined powder materials (VO_2, V_2O_5). It becomes obvious that the desired stoichiometry (Vanadium valency: +5) is hard to obtain using the ion-beam sputtering technique.

These results are closely related to the dc-conductivity measurements. An increase in oxygen content during sputtering leads to a decrease in conductivity and annealed samples show the lowest values in σ. This behaviour is related to crystallization processes and can be explained by a semiconducting model based on defects which are responsible for conductivity.

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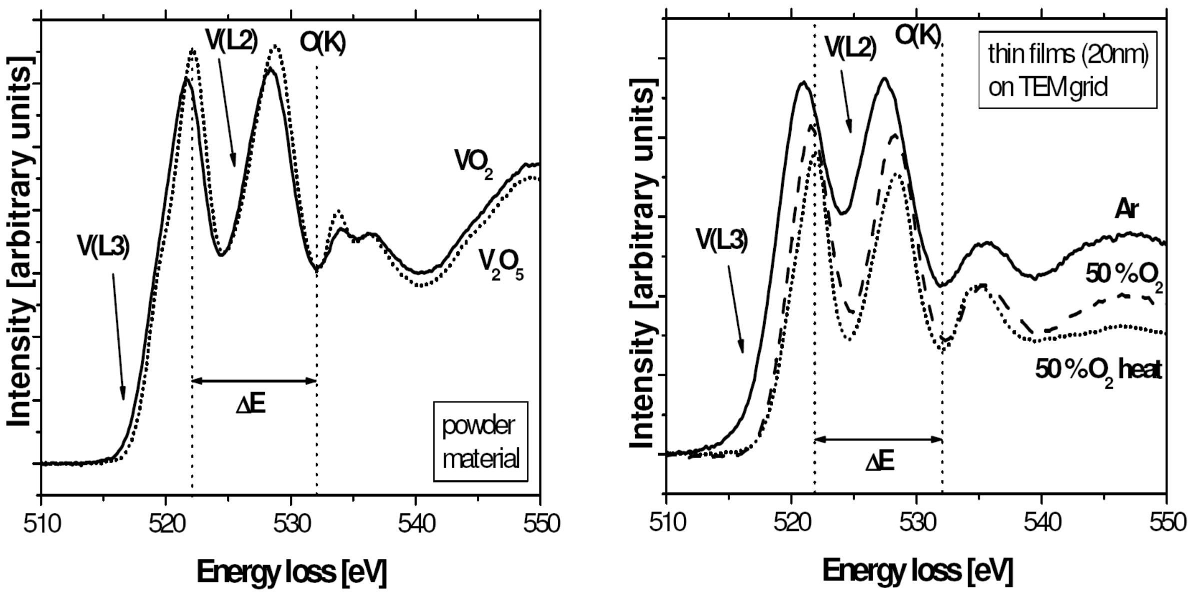

Figure 1. Left: EELS measurements for VO_2 and V_2O_5 powder materials (reference data).
Right: EELS results for sputtered thin films (20 nm on a TEM grid). Different sputter parameters were chosen.
Solid: Ar-sputtering; Dashed: 50% oxygen content during sputtering; Dotted: 50% oxygen and post-annealing in air (400°C for 3 days).
The chemical shift depending on the sputter parameters becomes apparent.

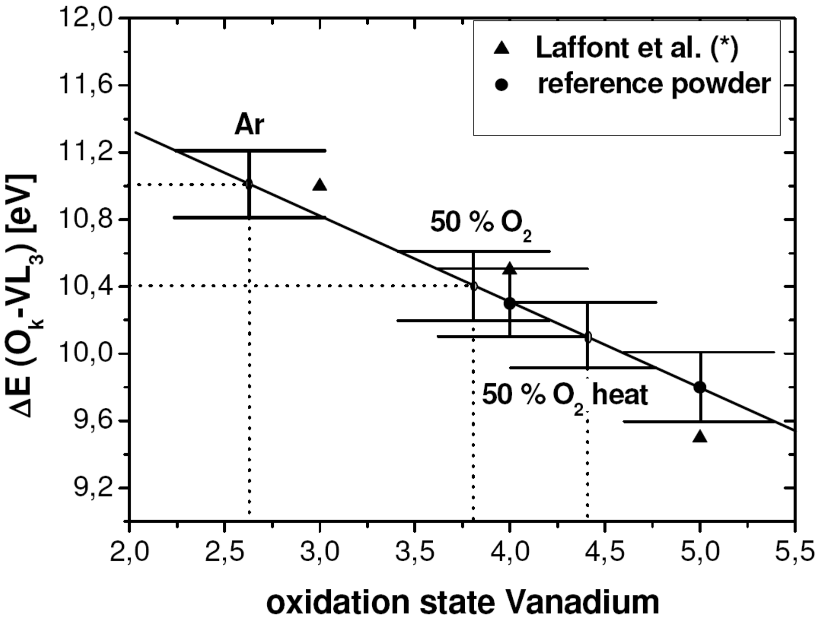

Figure 2. Energy shift plotted against the V-oxidation state depending on different preparation parameters to quantify the composition. Powder materials used for calibration: VO_2 (+ 4) and V_2O_5 (+ 5).

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