In situ TEM measurements to study structural changes of VO_2 nanowires

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Some vanadium oxide compounds are known to undergo a metal-insulator transition (MIT). The transition temperature of MIT effects varies for different oxygen contents. In particular, VO_2 is of special interest for use in device applications due to a metal-insulator transition at a moderate temperature of about 68°C [1]. At the transition temperature the resistivity of the material changes abruptly by several orders of magnitude. This effect is accompanied by a structural phase transition [2] as well as a change in optical properties [3].

The vanadium oxide nanowires were grown by the vapor-transport method on (0001) quartz substrates in the temperature range from 650°C to 850°C [4]. TEM studies were carried out to confirm that the nanowires are indeed composed of single-crystalline VO_2, as well as to show the structural phase transition from the monoclinic phase at room temperature to the rutile high temperature phase. For TEM investigations the material was mounted on holey carbon film on standard Cu-grids. The in situ cooling-heating experiment was done with the Gatan cooling holder which is cooled with liquid nitrogen.

Figure 1a shows a VO_2 nanowire of about 50 nm in diameter, the EDX spectrum in Figure 1b confirms the composition of V and O, Cu is from the Copper-grid.

The CBED pattern in figure 2a was taken at -165°C. It matches with ICSD CC: 74705 data for monoclinic VO_2 (a=0,57529 nm, b=0,45263 nm, c=0,53825 nm, b=122,602°). The zone axis is [0 -1 1]. Then the sample was heated up slowly. At +72°C we observed a sharp phase transition. The monoclinic structure changed into the tetragonal high temperature phase (ICSD CC: 4110: a=0,45546 nm, c=0,28514nm), the zone axis is [-1 1 1] (figure 2b). After the sample was cooled to room temperature, the same CBED pattern as in figure 2a appeared again (figure 2c). These results show that the phase transition processes are fully reversible.

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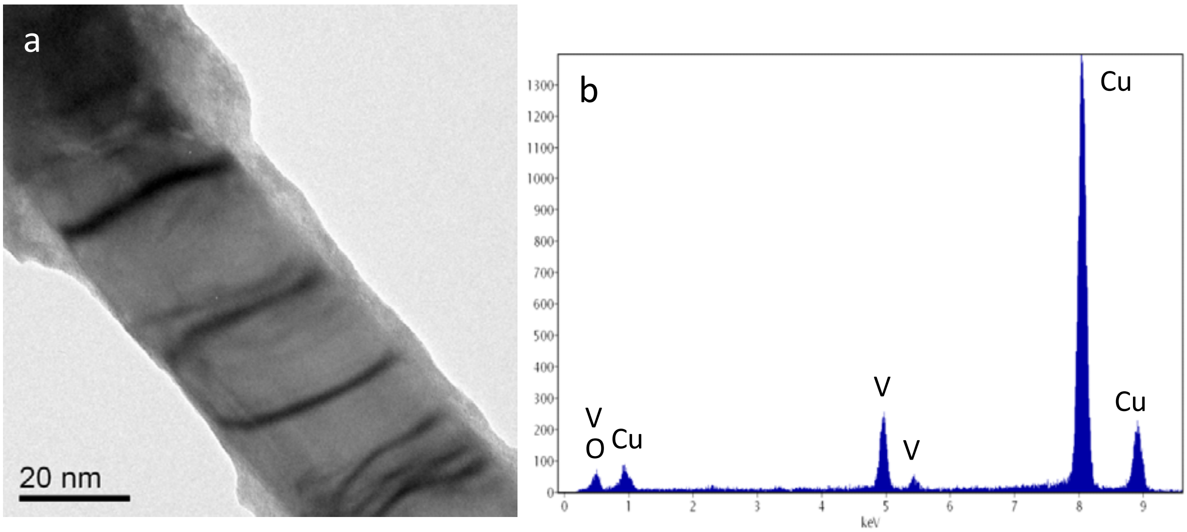

Figure 1. a) TEM bright field image from a VO_2 nanowire, b) EDX spectrum from the area shown in a).

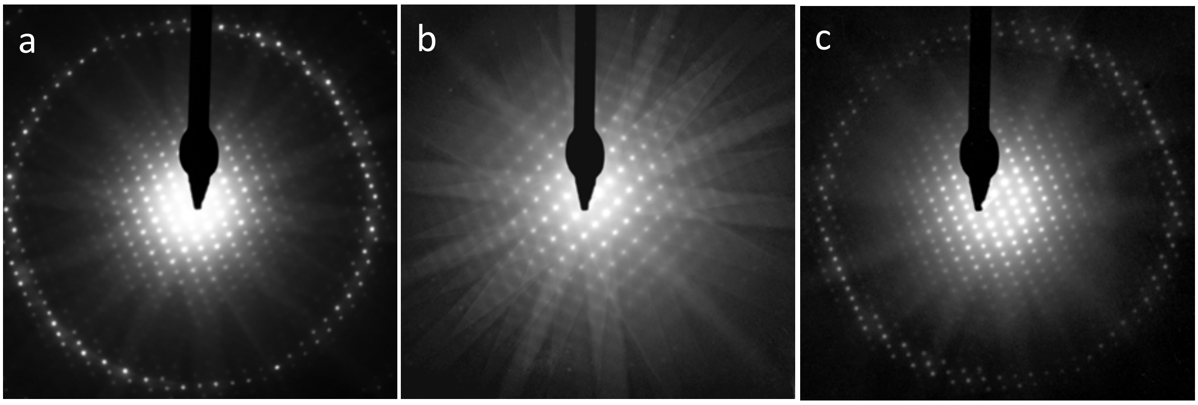

Figure 2. Series of CBED patterns from area in Figure 1a at different temperatures.
a) The diffraction pattern was taken at -165°C and shows the monoclinic low temperature phase, b) after heating to +75°C the pattern shows the tetragonal high temperature phase, c) shows again the monoclinic structure after cooling down to +43°C.

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