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Simultaneous Structural and Electrical Analysis of Vanadium Dioxide Using *In Situ* TEM

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Vanadium dioxide (VO_2), a correlated electron material, has received significant attentions due to its metal-insulator transition (MIT) at ~ 67 °C [1]. This transition is associated with structural phase transition from the monoclinic (M1), an insulating phase, to rutile (R), a metallic phase. This metal-insulator transision is accompanied by a noticeable resistivity, optical transparency and magnetic changes. These distinctive properties have inspired many applications such as thermo/electrochromics, Mott transistors, memristors, thermal actuators, gas sensors, strain sensors and temperature sensors. Recent efforts focus on controlling of phase transition and domain structures in finite size VO_2, which results in different material properties and play a critical role in device applications.

We investigated the M→R phase transition of individual VO_2 nanowires (NWs) using *in situ* TEM. We first manipulated individual NWs between two electrical electrodes using an *ex situ* lift out tool [2] as shown in Figure 1a. We then heated at 5 °C/s from room temperature (RT) to 130 °C, and measured the electrical behavior simultaneously to correlate the evolution of resistance during the phase transition. Figure 1b shows three distinct regimes of resistance change collected during the simultaneous heating and electrical *in situ* TEM experiments. In the first regime, heating from RT to 50 °C, the NW transitioned to a combination of M1 and M2 phases, and the resistance drops significantly from ~50k Ohm to ~14k Ohm. In the second regime, heating from 50 °C to 100 °C, the insulator phase transitioned mostly to the metal phase, and the resistance changed from ~14k Ohm to ~4k Ohm. In the final regime, the NW completely transitioned to the metal phase.

In situ diffraction patterns (DP) and dark field TEM images confirmed the phase transition from monoclinic (101) to tetragonal (101). Figures 2a-c represent evolution of DP of the same region shown in Figures 2d-f while the sample was heated from RT to 60 °C and 100 °C. The red dotted line indicates the reaction front motion through the NW as the M→R transition occurs. We further investigated types and distributions of different intermediate phases during the M→R using weak-beam dark-field technique. Additionally, motion of domain walls and lattice constant measurements were further investigated via high-resolution *in situ* TEM imaging. We successfully observed, indexed and characterized the initial, intermediate and final phases of the VO_2 transition, and correlated the phases to the electrical properties. These results will help lead to future development and application of phase change materials in various fields of electronics and nanodevices.

References:

[1] V. Eyert, Ann Phys (Berlin) 11 (2002), p. 650.
[2] L. A. Giannuzzi et al., Microsc Microanal 21 (2015), p. 1034

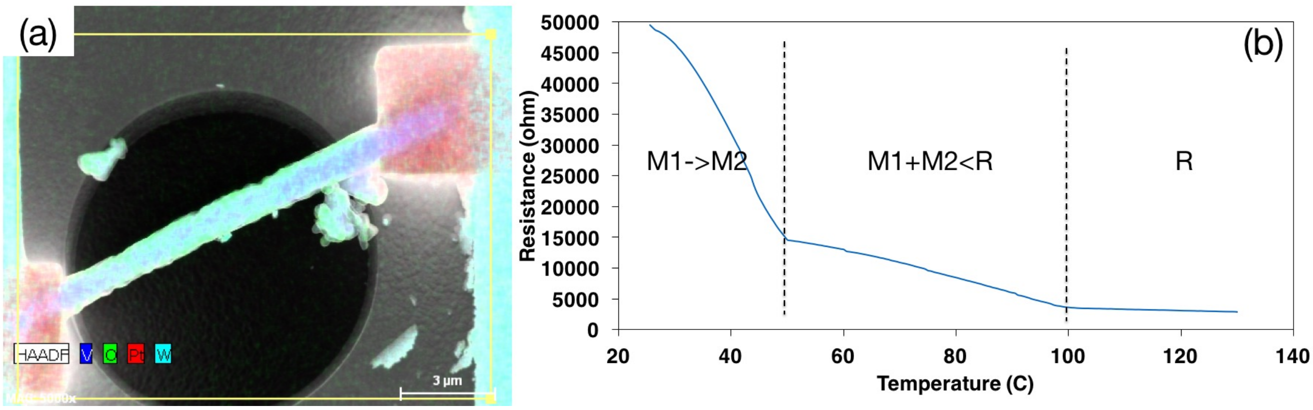

Figure 1. (a) EDS map of an individual VO_2 NW connected with Pt pads to W electrical electrodes of a MEMS E-chip device. (b) Plot of evolution of resistance as a function of temperature where clear distinction between regimes of phase transition is visible.

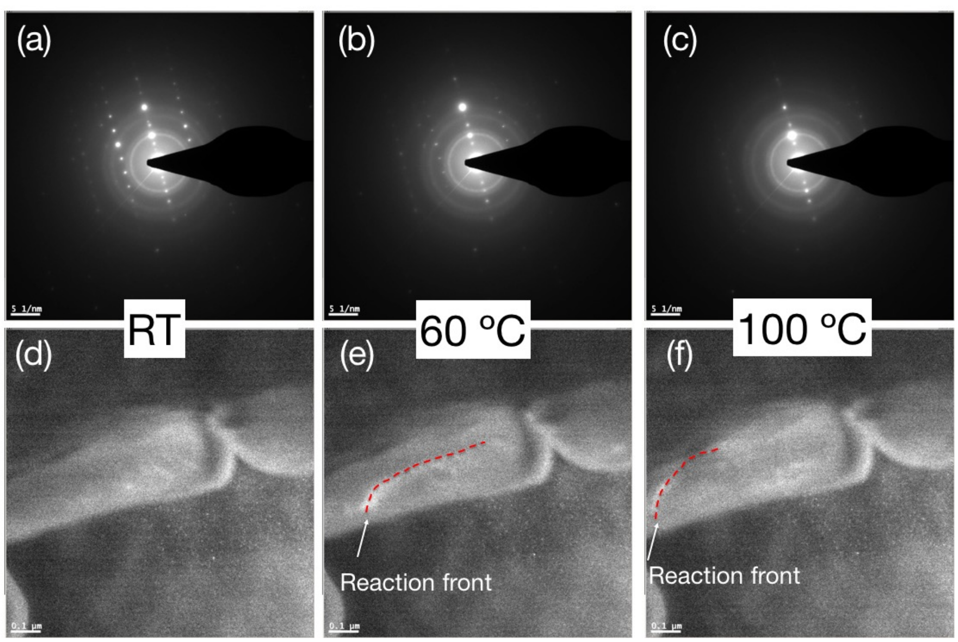

Figure 2. Snapshots of M→R transition. (a-c) Diffraction patterns at RT, 60 °C and 100 °C taken from the same region as shown in dark-field images (d-f). Dark-field images illustrate reaction front motion as indicated with the red dotted line.

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