Report on experiment #32-02/801 (06-07-2017 to 11-07-2017)

Current – induced metal-insulator transition in VO2 micro- and nanowires: an in-situ micro-diffraction study

Proposal abstract

We shall investigate the current-induced metal-insulator transition (MIT) and structural phase transition (SPT) in VO_2 micro- and nanowires (NWs). The use of perfect NWs will allow to get rid of extrinsic factor (strain, defects, domains) that commonly affect the MIT and the SPT in thin films and single-crystals. Attention will be paid to the determination of the mechanisms involved in the transition since it is still not settled whether the transition if of Peierls or Mott-Hubbard type. For this purpose we intend to characterize at the nanometer scale (using the sub-micrometer beam of the BM32 beamline) the structural evolution of VO_2 NWs placed between two electrodes while it experiences a MIT driven either by the temperature or by the injection of electrons. The different possible transition mechanisms are expected to give rise to different nanostructures during the transition, and in particular in the negative differential resistance regions associated with the onset of the MIT.

Materials

VO_2 wires were grown on SiO_2/Si substrates by VLS. Three different types of wires have been investigated:

- Strained VO_2 wires: these correspond to as-grown wires on the SiO_2/Si subtrates
- Free standing VO_2 wires: Mo electrodes have been deposited at both sides of the wires and the underlying substrate has been etched out using HF.
- W:VO2 wires: W was introduced during growth in order to shift the MIT towards room temperature.

Methods

The wires were analyzed using the 0.5x0.5µm² polychromatic X-ray beam of the BM32 beamline. Laue patterns were collected on a CCD camera. The patterns were analyzed with the LaueTools software suite. The wires have been scanned through the X-ray beam and Laue patterns have been collected for different positions along the wires. When possible, the resistivity was monitored simultaneously.

It was initially planned to activate the MIT/SPT using temperature and electric current. However, because of a problem in the design of the electrodes (too widely spaced) it was not possible to electrically initiate the MIT/SPT without melting the wires. We therefore focused on the thermally induced transitions.

The camera exposure time was refined in order to have a sufficient signal from the VO_2 wires while avoiding to oversaturate the diffraction peaks coming from the underlying Si substrates (which would complicate data analysis). Because of the weak signal of VO_2 it is necessary to ensure that no parasitic diffraction takes place. The first two days of the experiment were therefore devoted to optimize the sample heating stage (resistive furnace) in order to exclude any parasitic diffraction signal coming from the heating elements.

Results

Free-standing wires

In the free-standing wires the MIT and the SPT take place simultaneously upon heating at ~71°C and the whole wire is affected by the transition. Fig. 1 displays patterns at room temperature (RT) and 150°C. The indexation reveals the M1 phase at RT and the R phase at 150°C, as expected for perfect single crystals.

(a) (b)

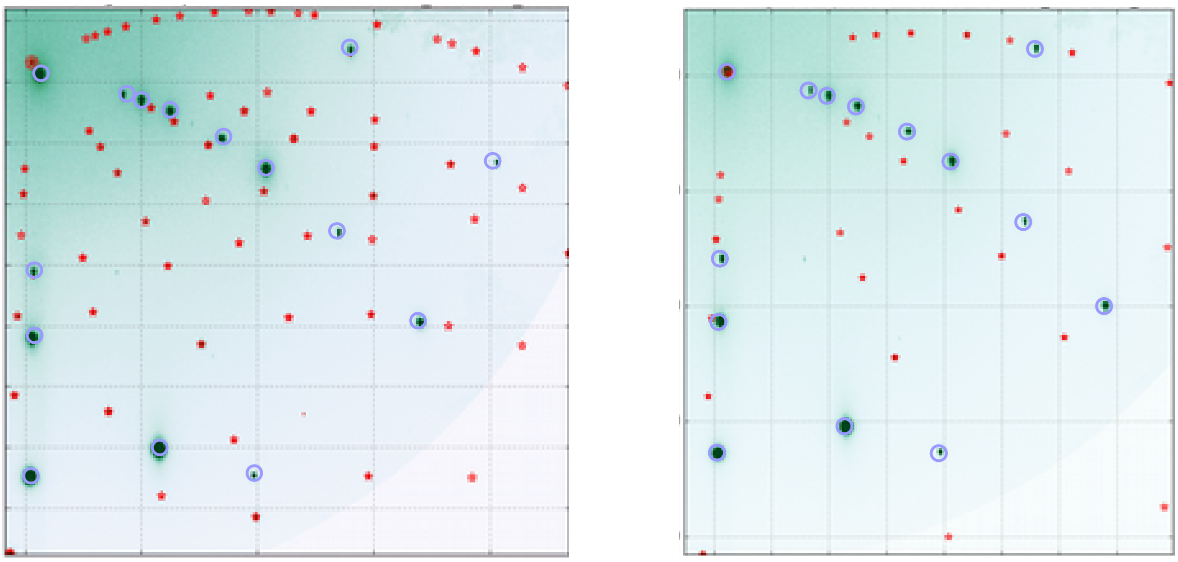

Fig. 1. Laue pattern at room temperature (a) and 150°C (b) of a free-standing wire. Blue circles: Si; red stars: VO_2 M1 phase (a) and R phase (b).

Strained wires

In strained wires the situation is much more complex. Since the wires are clamped on the substrate, transformation strain is released via the formation of twins and intermediate phases. In such cases, the transformation takes place heterogeneously along the wire (Fig. 2).

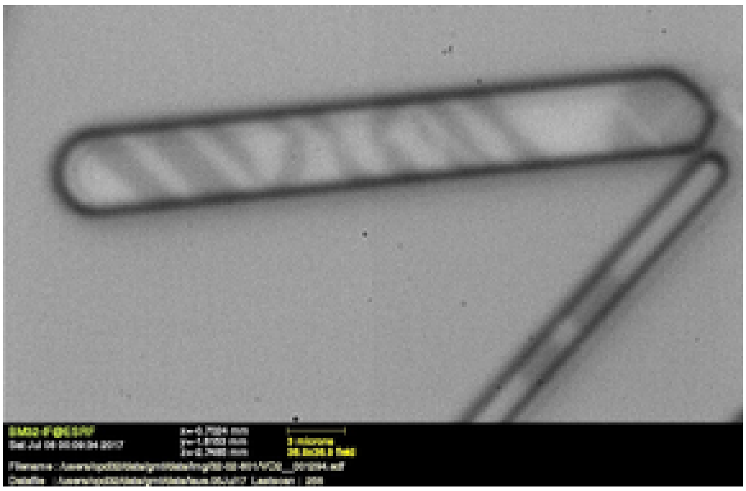

Fig. 2. Optical micrograph of a wire undergoing the MIT. Dark/white regions correspond to conducting/insulating domains.

Scanning the X-ray beam from a white to a dark region reveals a complex transformation sequence, as illustrated in Fig. 3.

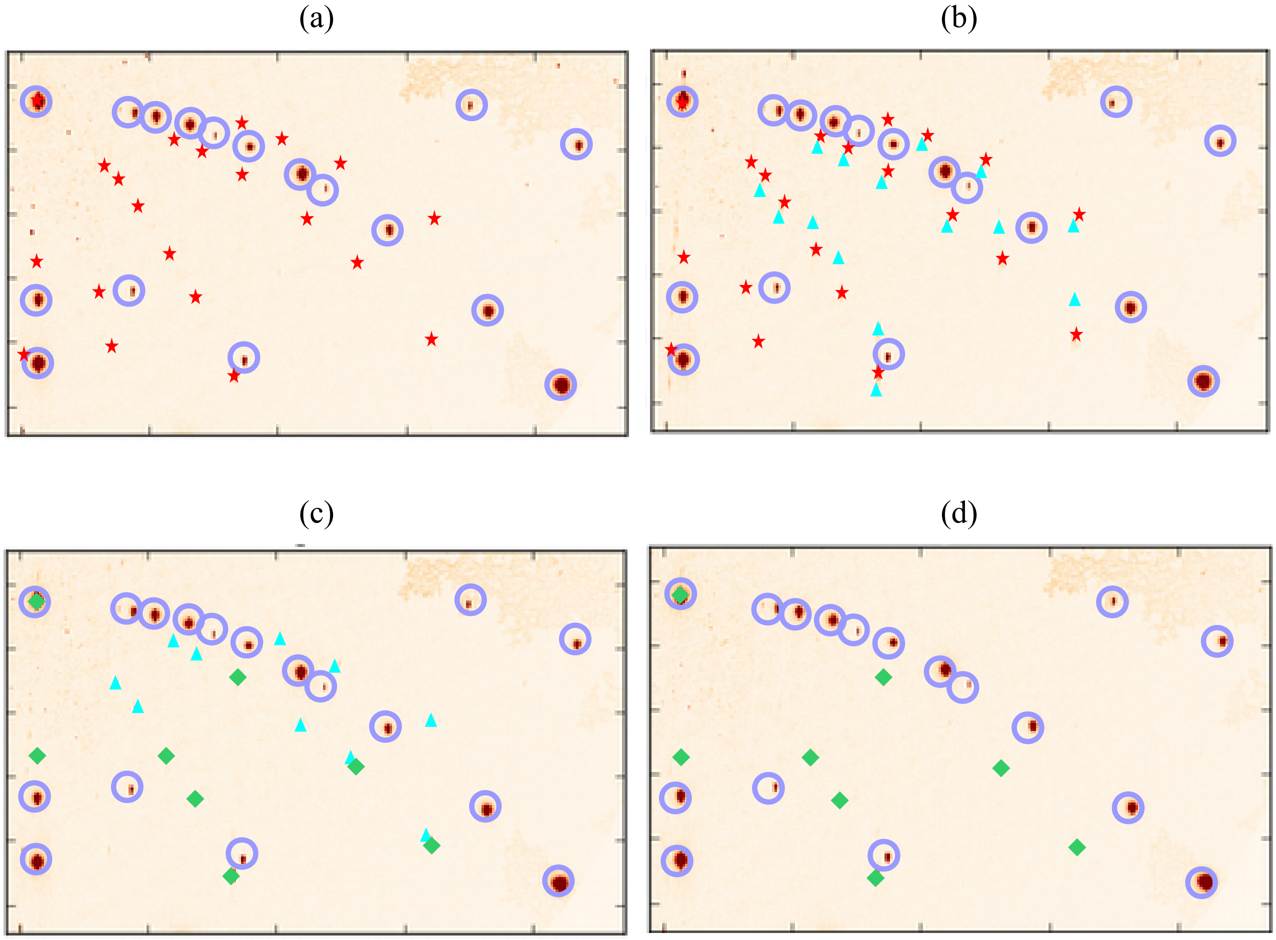

Fig. 3. Laue patterns recorede from a white region (a), up to dark region (d). (c) and (d) are intermediate positions. Blue circles: Si; red stars: VO_2 M1 (011) and (0-11); blue triangles: VO_2 M2 (-201); green diamonds: VO_2 R.

In Fig. 3(a), the domain can be indexed in the M1 phase. However, contrarily to free-standing wires, twins are formed during cooling down so that two M1 orientations are detected. The M1 domains progressively transform into the intermediate M2 phase (untwinned), Fig. 3(b), which progressively transforms into the R phase, Fig. 3(c). The transformation is complete in Fig. 3(d).

Doped wires

All W-doped wires were found to be in the R phase at room temperature. Initiating the SPT requires a cooling element which was not possible during the experiment.

Perspectives

Now that the experimental conditions have been optimized and the transformation sequences clearly identified, further experiments are required to refine and complete this first experiment on VO_2 wires:

- repeat measurement for smaller wires in order to enable electrical activation
- perform experiment with narrow heating steps in order to clearly detect the MIT and the SPT both during heating and cooling
- perform measurement for temperature lower than RT for doped wires.