ESRF	**Experiment title:** X-ray microscopy study of the VO_2 transition temperature modified by ion irradiation	**Experiment number**: MA-3576
Beamline: ID01	**Date of experiment**: from: 12. February 2018 to: 17. February 2018	**Date of report**: March 5, 2018
Shifts: 18	**Local contact(s)**: Dr. Tao Zhou	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Dr. Andreas Johannes*, Dr. Tao Zhou*

ESRF 71 avenue des Martyrs CS 40220 FR - 38043 GRENOBLE Cedex 9

Prof. Carsten Ronning*, Dr. Jura Rensberg*

Friedrich-Schiller-Universität Jena, Institut für Festkörperphysik,
Max-Wien-Platz 1, 07743 Jena, Germany

Report:

Upon heating vanadium dioxide (VO_2) undergoes a phase transition from a low-temperature, monoclinic (M1), insulating phase to a high-temperature, rutile (R), metallic phase. Ion beam irradiation changes the transition temperature, making it an interesting technological tool, however, the mechanisms behind the modification of the transition are under discussion. This experiment investigated the phase change and associated stress in a focused ion beam irradiated thin-film of VO_2 grown on mica.

On the investigated sample, markers made using focused ion beam (FIB) Pt-deposition at the University of Jena before the experiment facilitated easy spatial navigation on the sample. Using the optical microscope (rough) and small XRD maps (fine), the regions of interest patterned with the FIB were easily found. The epitaxial relation between the mica substrate and the thin film to be investigated was used to navigate in reciprocal space. After the first day of experiments using the classical scanning XRD setup at ID01 to find and map the out of plane VO_2 $(020)_{M1}$ Bragg peak, it was decided that the intensity in the Bragg-peaks to be investigated was too low to be feasible to use the full field X-ray diffraction microscopy (FFXRDM) technique originally envisaged in the proposal. The main factor limiting the signal is the small collimation in the FFXRDM with respect to the large width in reciprocal space that the Bragg peaks of interest exhibited. Also, using the diffractometer rotating out of the plane of the synchrotron ring would make it possible to image Bragg peaks at higher scattering angles close to 90 ° without suffering loss of scattered intensity from the polarization factor. Therefore, all the experiment was conducted in scanning XRD mode.

First an area of the sample in which fields irradiated with increasing ion fluence was mapped in 3D (2 real-space coordinates + sample rocking). First results in Fig.1 show that the expected decrease in transition temperature with increasing irradiated ion fluence was clearly reproduced. These results were used to identify those ion fluences at which the clearest XRD results could be obtained. From the available prefabricated structures those irradiated with an ion fluence of 0.05 and 0.1 pCµm^{-2} were deemed to be suitable.

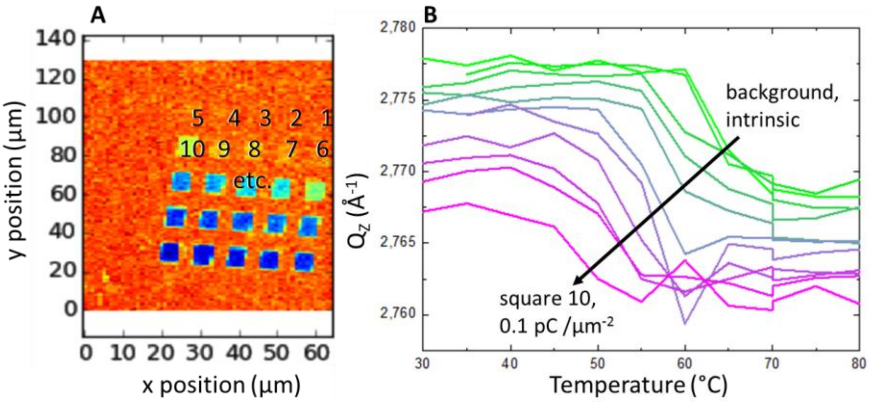

Figure 1: *Heatmap (A) of the unmodified (020)$_{M1}$ Bragg peak showing the area irradiated with squares of increasing iradiated ion fluence. The phase transition in the VO_2 thin-film is clearly visible in the plot of the value of Q_z vs temperature (B) for the lowest 10 ion fluences.*

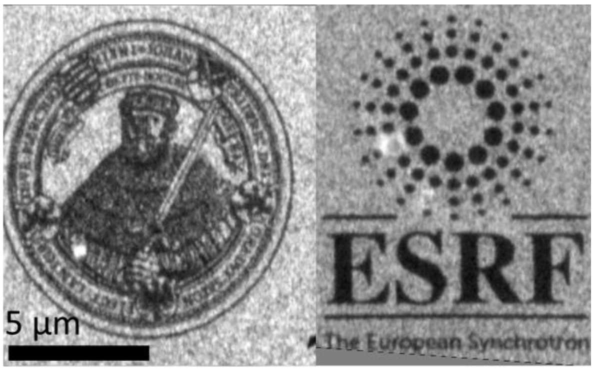

Figure 2: *Greyscale heatmap of the unmodified (020)$_{M1}$ Bragg peak for exemplary structured irradiation patterns.*

Figure 2 shows exemplary high resolution 2D maps showing the fine features obtainable with focused ion irradiation on a VO_2 film. Full 3D maps were performed to investigate the strain present along the edges of irradiated areas at 40 °C and 60 °C. At the higher temperature the irradiated area has transitioned to the metallic state, while the unirradiated areas are still insulating. A detailed analysis of these 3D maps is underway to investigate the impression that the granularity of the film defines the maximal resolution obtainable by the focused ion beam structuring.

Without the strict geometric constraints of the FFXRDM, it was further possible to image similarly patterned areas on the sample at a variety of Bragg-conditions summarized in table 1. The information from these scans will corroborate the conclusions about the effect of the granularity of the film on the edges of the irradiated region. Unfortunately, it was not possible to do full 3D maps at the asymmetric peaks within the allocated time.

Table 1: *Investigated Bragg-Peaks on VO_2 thin film during this experiment.*

Miller index	Dimensions mapped	comment
(020)$_{M1}$	3D	Various fluences 0-2.5 pCµm^{-2} and temperatures 30 – 80 °C
(040)$_{M1}$	3D	High resolution at 40 °C and 60 °C
3 x (310)$_R$	2D	3-fold symmetry caused by epitaxial growth Peak is degenerate in the monoclinic M1 phase
3 x (301)$_R$	2D	3-fold symmetry caused by epitaxial growth Peak is **not** degenerate in the monoclinic M1 phase

We already consider these results from this preliminary analysis highly interesting and are working on the detailed data analysis. To finalize the conclusions drawn from the XRD data, this sample will be also investigated by AFM (acknowledgements to the PSCM) and STEM (University of Jena).