

and k , which is clearly separated from its neighbouring reflections. The x-ray powder diffraction could be indexed with tetragonal unit cells with an approximate axis $a = 6.15$ Å and with c axis between 13 and 35 Å depending on the type of amines used.

We performed X-ray diffraction measurements on vanadium oxide nanotubes on the beamline BM16 by using X-ray of wavelength $\lambda = 0.40576(1)$ Å. We determined the diffraction intensities at room temperature and also at $T = 15$ K by using a helium flow cryostat. A comparison of the synchrotron data (upper, red curve in Fig. 1) with the data obtained with a conventional powder diffractometer (lower, black curve in Fig. 1) shows, that the limited resolution of the overlapping reflections is indeed caused by the sample itself. Cooling does also not lead to a better resolution of the reflections. There is no significant difference between the measurements at room temperature and 15 K (Fig. 2, red curve RT measurement, blue curve 15K measurement). Nevertheless the resulting powder pattern allowed the determination of the structure type: All vanadium oxide nanotubes observed so far are built with one of vanadium oxide sheets, first found in $\text{BaV}_7\text{O}_{10}\cdot n\text{H}_2\text{O}$ [3]. The sheets consist of bilayers formed of quadratic VO_5 -pyramids and VO_4 -tetrahedra. Rietveld refinement of the laboratory X-ray diffraction data was not possible because of strong overlapping of the reflections, the preferred orientations and the different peak profiles caused by the pronounced anisotropic crystallite size of the tubes. Several other vanadium oxide layers described in the literature could be ruled out by comparison of their calculated diffraction diagrams with the observed patterns.

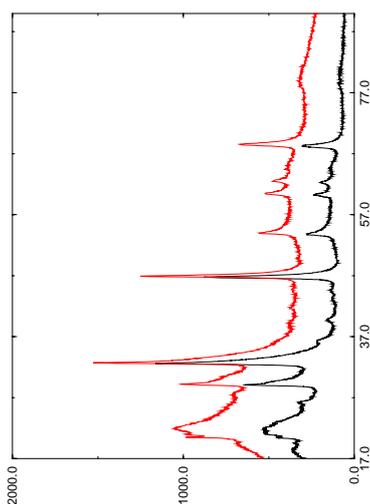


Fig. 1: Synchrotron data (upper, red curve) conventional diffractometer data (lower, black curve). The synchrotron data were scaled to $\text{Cu K}\alpha$ radiation.

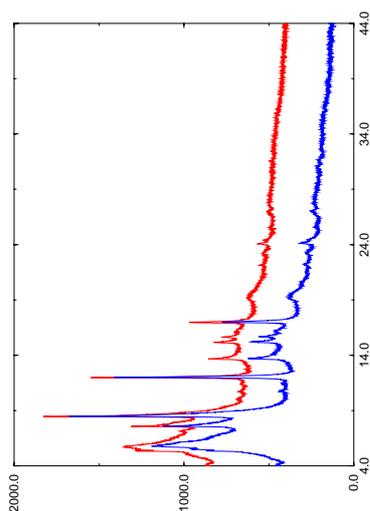


Fig. 2: 15K measurement (blue) RT measurement (red)

References:

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