SbVO$_4$ is a key element in the catalysis of propane to acrylonitrile. The system maintains the rutile structure during the whole existence range accommodating the non-stoichiometry in a "soft way", i.e., without extended defects, which are so common in other rutile systems. The great structural flexibility exhibited, very important for its catalytic performance, involves cation vacancies, changes of the oxidation state of vanadium (V$^{4+}$, V$^{3+}$, and Sb$^{5+}$), long range ordering, structural modulations, short range ordering, etc. [1,2] In this work we study the structure of one of the key points of the system, the composition limit Sb$^{1.0}$V$^{1.0}$O$_4$.

The samples have been prepared by heating Sb$_2$O$_3$ and V$_2$O$_5$ in an equimolar ratio (1:1) at 800°C under argon atmosphere in two runs of 12 hours with a careful grinding in the interval. The sample has been characterized by powder X-ray diffraction in a Powder Diffractometre Bruker D8 Advance with Cu Kα radiation with rapid detector (lynxeye). Electron diffraction experiments were carried out in a JEOL 2000FXII transmission electron microscope with a double-tilt holder. The sample has also been characterized by neutron diffraction experiments (Instruments D1B-ILL, Grenoble and E6, E9 at HZB, Berlin), magnetic susceptibility measurements, DSC calorimetry and Raman spectroscopy. The sample exhibits a basic rutile powder X-ray diffraction pattern without extra characteristics. TEM exhibits very crystalline crystals with a typical shape of tetragonal prisms capped at both ends. However, electron diffraction shows the presence of intense diffuse lines that, after careful tilting experiments, are demonstrated to be two-dimensional wavy diffuse sheets. Notice that the diffuse intensity lines are absent in the [001] zone axis due to the fact that the diffuse intensity sheets are perpendicular to the [001] direction. While these diffuse sheets seem to be very straight in zone axes such as [110] and [101], they reveal a wavy nature when tilting away from these and specially at the [100] zone axis. This diffuse intensity condense in a two fold superlattice when the sample is prepared under nitrogen atmosphere. Neutron diffraction and magnetic susceptibility measurements reveal magnetic ordering at T$_{N}$ < 50K from the ordering of vanadium magnetic moments [3].

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Fig. 1: SAED patterns of SbVO₄ crystals orientated along different zone axes. The sharp diffraction maxima belongs to the basic rutile lattice. The diffuse lines between the Bragg maxima are caused by SRO phenomena.

Fig. 2: a) HRTEM processed image of a crystal of SbVO₄ oriented along the [100] zone axis showing the presence of SRO. b) FFT of the original image