

Experimental report

30/09/2016

Proposal: 5-41-856

Council: 4/2016

Title: Magnetic structure determination of coherently strained (010) oriented o-TbMnO₃ thin films using neutron diffraction.

Research area: Materials

This proposal is a new proposal

Main proposer: Saumya MUKHERJEE

Experimental team: Laurent CHAPON
Saumya MUKHERJEE

Local contacts:

Samples: TbMnO₃

Instrument	Requested days	Allocated days	From	To
D10	10	10	05/09/2016	15/09/2016

Abstract:

We plan to determine the magnetic structure of the multiferroic orthorhombic (o-) TbMnO₃ thin film deposited on (010) oriented o-YAlO₃ substrate by measuring 20-30 magnetic reflections with four-circles diffractometer D10. By performing neutron diffraction experiments with the triple-axis spectrometer RITA-II at SINQ, we identified an incommensurate (IC) magnetic phase below $T_N \sim 41\text{K}$, which locks to a commensurate phase ($0 \leq q_k = 0.5$) below $T_{\text{lock}} \sim 30\text{K}$. The epitaxial strain in the film changed the bulk-like IC phase ($0 \leq q_k \sim 0.29$) to a commensurate structure. Macroscopic ferroelectric (FE) measurements on these films demonstrate large polarization ($\sim 0.4 \text{ } \mu\text{C cm}^{-2}$) along the crystallographic a-axis below $T_{\text{FE}} \sim 41\text{K}$. This response is in sharp contrast to the weak polarization along the c-axis in bulk o-TbMnO₃ below $T_{\text{FE}} = 27 \text{ K}$ [1]. Resolving the magnetic structure in these films will provide an intrinsic understanding of the change in microscopic mechanism induced by the epitaxial strain. The sample to be measured is a 44 nm thin (010) oriented o-TbMnO₃ film.

Magnetic structure determination of coherently strained (010) oriented o-TbMnO₃ thin films using neutron diffraction. (Report on experiment: 5-41-856)

In this experiment, a 44 nm TbMnO₃ thin film deposited on (010) oriented YAlO₃ substrate is used to study the symmetry of magnetic order using neutron scattering at D10, ILL (France). Set of magnetic reflections (q_h q_k q_l) was planned to be measured at different temperatures across the observed phase transitions. A transition from paramagnetic to an incommensurate antiferromagnetic-ferroelectric phase at 39 K was followed by a transition to commensurate- ferroelectric phase at 31 K. Also, since the Tb spins order below 15 K, we will measure reflections sensitive to this ordering. So we attempted measuring reflections at 10 K and 35 K.

We measured nuclear reflections of the substrate and refined the UB matrix to search for the film nuclear reflections. Fig. 1 shows a q-scan for (1-1 0) YAlO₃ to identify the TbMnO₃ film (1 -1 0) peak.

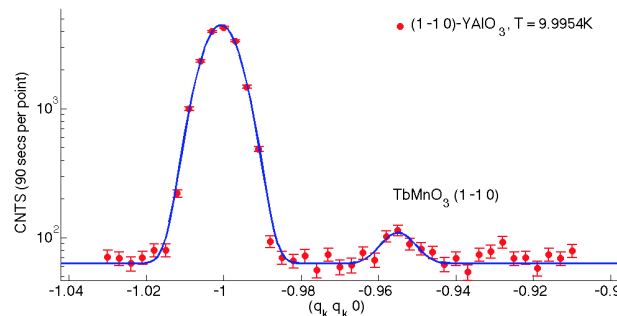


Fig.1

Nuclear reflections (023), (110), (221), (131), (122) and (120) for the film was measured at T = 10 K to refine its the low temperature lattice parameters: $a = 5.17$, $b = 5.919$ and $c = 7.353$ Å and the UB matrix to was estimated. Table.1 shows comparison of the measured and calculated intensity of magnetic reflections. These integrated intensities will be used to refine the magnetic structure at T = 10 K. Fig. 2 show few of the measured reflections. Tb ordering was absent above 6 K. But at 4 K we observed the (0 ½ 2) reflection sensitive to Tb ordering (Fig. 3).

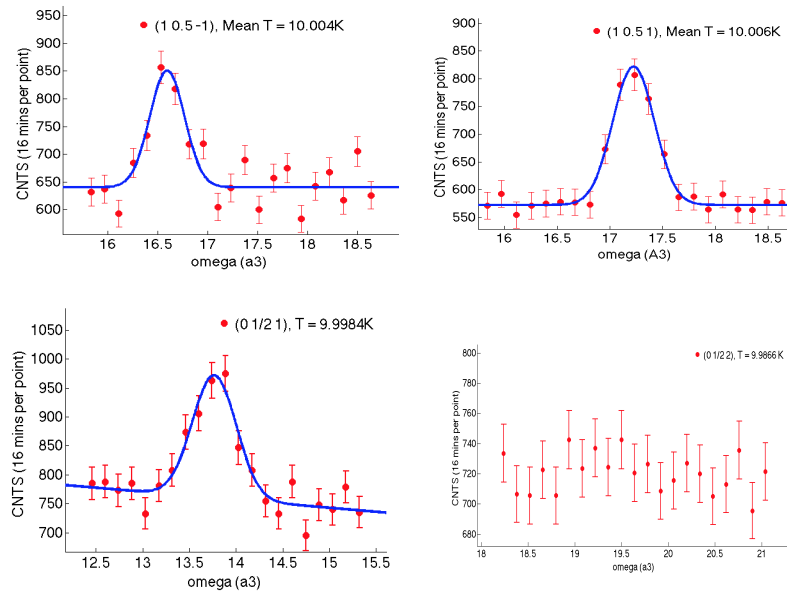


Fig.2

Table.1

H K L	Intensity (Cal)	Intensity(Not normalised) observed
1 1/2 1	3000	88.45
1 1/2 -1	3000	65.63
0 3/2 0	18	13.91
0 1/2 1	6000	92.92
0 1/2 -1	6000	103.65
0 1/2 2	1.3	0
0 1/2 -2	1.3	0
0 3/2 -1	550	15.4
0 3/2 1	550	18.3
0 1/2 0	180	0
1 -1/2 1	3000	62.5
1 -1/2 -1	3000	63.3
2 1/2 1	2530	30.9
0 1/2 3	2875	38.9

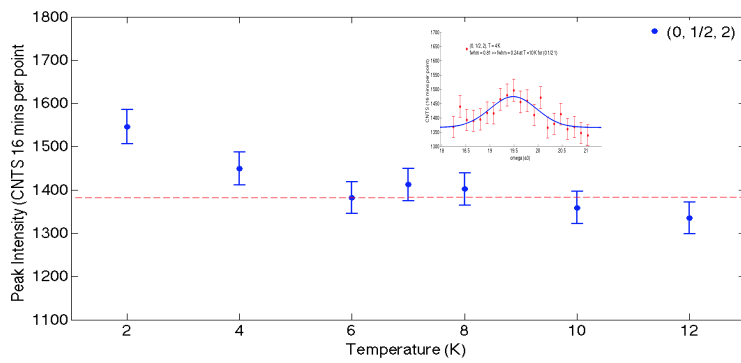


Fig. 3

A major challenge was to avoid movement of the sample holder, which has to be modified due to change of the cooling technique at D10. This caused fast cooling or warming of the sample inducing slipping of the sample out of the sample holder. On addition of glue the problem was fixed but that was not an efficient solution since based on cooling rate we had to face the same problem quite regularly. Also the glue gave additional background. All this led to increase in counting time and we did not manage to measure magnetic reflections at $T = 35$ K. Therefore, to complete the understanding of the magnetic structure in the high temperature incommensurate phase we decided to request beam time as a continuation work.