Experimental report

25/01/2022

Proposal: 5-54-341 **Council:** 4/2020

Title: Neutron diffraction in epitaxial antiferromagnetic oxide films for application in spintronics : effect of the thicknesses

Research area: Materials

This proposal is a new proposal

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Samples: (LaSrMnO3)4(BaTiO3)3
(LaSrMnO3)3 (BaTiO3)4

Instrument	Requested days	Allocated days	From	To
D10	20	10	28/01/2021	02/02/2021
			18/06/2021	27/06/2021

Abstract:

We have demonstrated (see report 5-54-290) that we are able to measure the antiferromagnetic structure in epitaxial films of LaSrMnO3 and BaTiO3 superlattices at low temperature. (LaSrMnO3)n (BaTiO3)m.
We have unpublished results on n=m=4 and n=m=3 films.
To be able to confirm the relevence of our model, it is necessary to study the evolution of the structure whan we vary the values of n and m independently.
We then propose to study here a n=3 and m=4 and n=4 and m=3 samples, where the coupling among layers is different, as well as the symmetry, but the structure of the layer it self is similar andthe opposite.
10 days was demonstrated to be needed in a previous measurement for each sample.

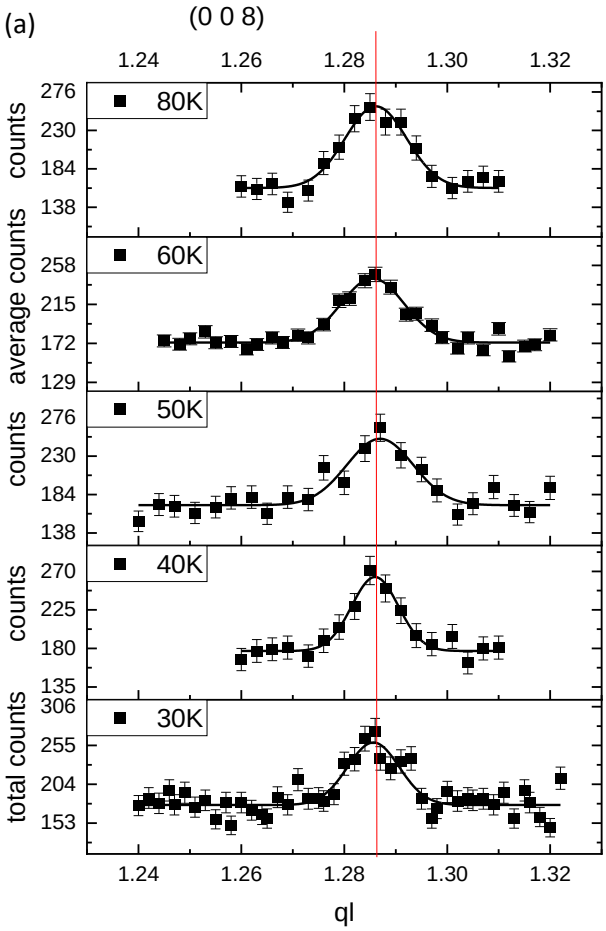
Experiment - 5-54-341 (D10)

During the 5 days of beamtime in January 2021, superlattice $(La_{2/3}Sr_{1/3}MnO_3)_3(BaTiO_3)_3$ repeated 24 times deposited on a lattice matched $SrTiO_3$ (001)-oriented substrate, was measured. The sample size is 5x5mm^2 with a total thickness of typically 60 nm.

Temperature dependence of few peaks of this sample was already studied during the experiment 5-54-290. The (0 0 8) peak seemed to have satellites in the temperature range 40 – 60 K, which could be related to incommensurate magnetic structure. But the statistics were not good enough to confirm. So, the aim of this experiment was to study the temperature dependence of (0 0 8) and other magnetic peaks, with good statistics.

We have used 12 million monitor, taking about ½ hour to measure one point. Each peak contains 23 points, taking almost 12 hours for one measurement. The film was held in a cryostat on the D10 diffractometer. Incident wavelength of 2.36 Å1 from a pyrolytic graphite monochromator was used. The energy analyzer was used to reduce the signal coming from the substrate. Orientation of the film was done at each temperature on two independent peaks of the substrate. The data plots corresponding to this are shown in fig. 1. Few preliminary conclusions:

- As evident from the graphs, there are no significant changes in the (0 0 8) peak with temperature.
- Based on this experiment, the (0 0 8) peak doesn't seem to have any satellites, contrary to the observations in the previous experiment on the same sample.



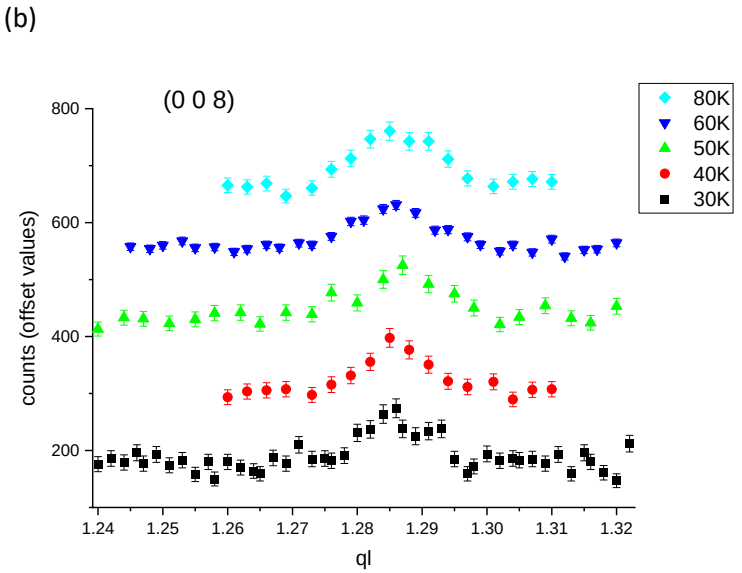


Fig. 1a,1b: (0 0 8) peak of 3-3 sample measured at various temperatures. The measurement time at each temperature is almost 12 hours on a 12 million monitor.

During the beamtime in June 2021, we measured the superlattice $(La_{2/3}Sr_{1/3}MnO_3)_2(BaTiO_3)_3$ repeated 29 times, deposited on a lattice matched $SrTiO_3$ (001)-oriented substrate. The sample size is 5x5mm^2 with a total thickness of typically 60 nm. From our previous experiments on the 3-3 sample, we were able to measure various peaks (both nuclear and magnetic) at 30 K. The aim of this experiment was to repeat the same measurements for the 2-3 sample, which has only 2 layers of LSMO in the bilayer. This type of study will help us understand the magnetic coupling in-between LSMO layers. Preliminary SQUID measurements on this sample suggest antiferromagnetic coupling in the structure.

In order to identify the most intense reflections, theoretical nuclear intensities were calculated for a DFT optimized structure. The selected reflections were then measured at 30 K with different statistics, depending on the peak strength.

Incident wavelength of 1.53 Å from a Cu (2 0 0) monochromator was used. The four circle setup was used with energy analyzer to reduce the signal coming from the substrate. The lattice parameters (in Å) obtained are as follows:

Substrate (at RT): 3.90 3.90 3.90, Space group symmetry: P m 3 m

Film - RT: 3.90 3.90 20.17, LT (30 K): 3.89 3.89 20.12

In total, about 14 different reflections were measured with good statistics. On average, it took about 25 minutes to measure a single point with 2 million monitor. Few of the measured peaks are shown in figure 2. It seems like there is intensity only for peaks with L multiple of 5.

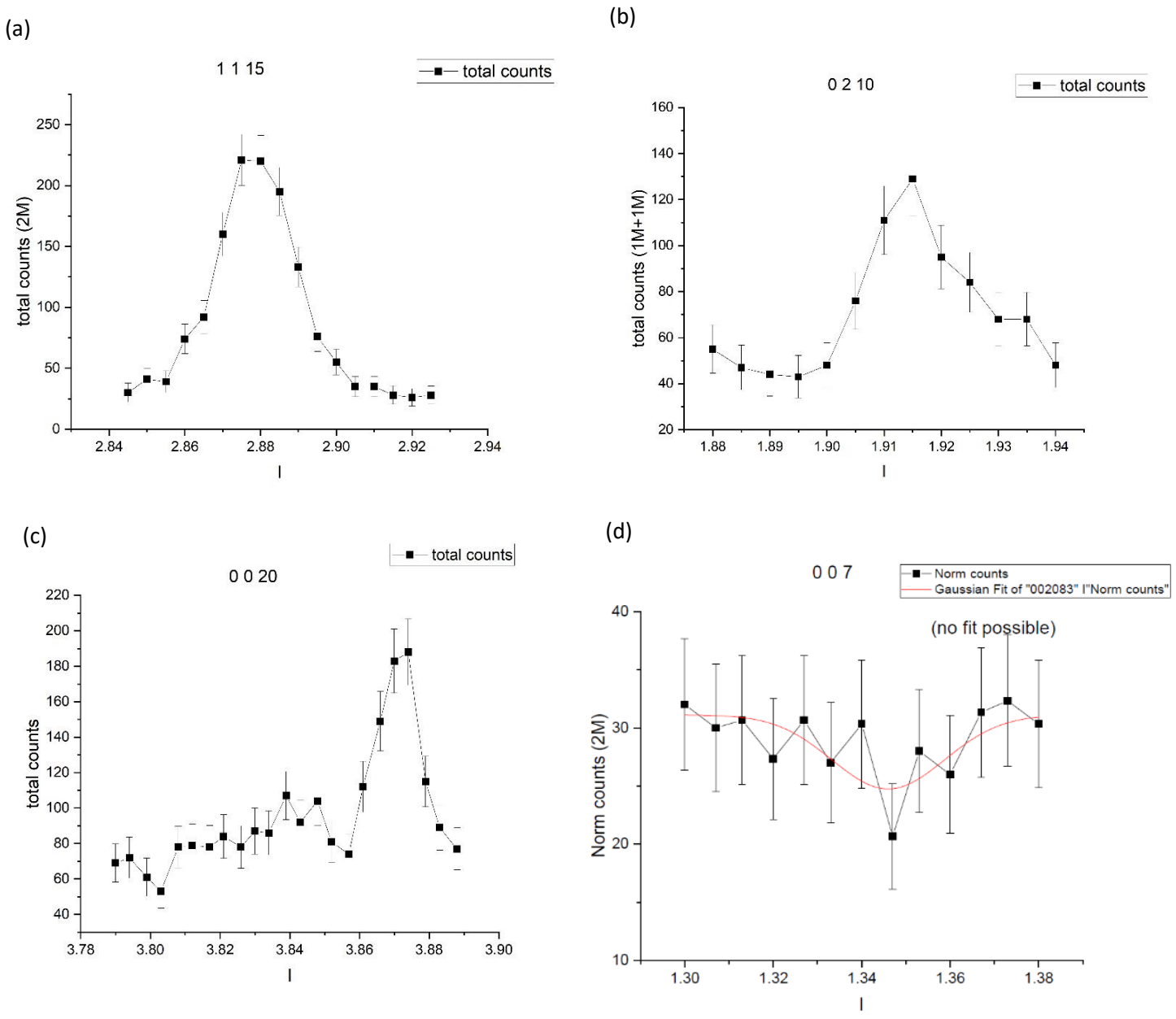


Fig. 2: Few of the measured peaks of the 2-3 sample at 30 K