ESRF

Experiment title:	**Experiment number:**
Magnetic X-ray Scattering from films of transition metal oxides	He-304

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Report:

During this first experiment on transition metal oxide thin films we were working with two different samples. The first one was a 650Å thin film of MnO grown by pulsed laser ablation on a sapphire substrate, its growth direction is (111). The second sample was a MnO bulk crystal with a (001) surface which was used in order to compare bulk and film behavior.
We mainly studied temperature dependence in the non-resonant regime, but resonant test experiments near the k-edge of Mn were also made for both , film and bulk crystal. From the rough data it can be concluded that there is strong resonant enhancement near the k-edge.
The main result obtained during the experiment is the fact that the antiferromagnetic phase transition becomes of second order in the film, whereas the transition in the bulk crystal is of first order, as already shown by D. Bloch et al. [l]. This crossover from first to second order can be explained either the lower dimensionality of the film or by magnetoelastic effects due to the presence of the substrate.
Experiments were performed at 6.52 keV which is far enough from the k-edge of Mn (6.538keV) in order to be in the non-resonant regime.
The film was mounted on the four-circle diffractometer of ID20 with the (111) axis in the scattering plane, this alignment allows specular scattering. At room temperature the quality of the thin film was tested. Its mosaicity was found to be of the order of 0.1°, which is fairly good for a thin film. Around the (111) reflection the Kiessig oscillations were measured in the reciprocal space (see figure 1). They give access to the film thickness d as well as the surface roughness σ. Best fits were obtained for d=650Å and σ=4Å.
In order to see the magnetic reflections, the sample was cooled down to 50K. At this temperature MnO is ordered in a type II antiferromagnetic structure, with the magnetic moments ferromagnetically aligned in the (111) planes, which are stacked antiferromagnetically.

We measured two different magnetic reflections the $(\frac{1}{2}\frac{1}{2}\frac{1}{2})$ and $(\frac{3}{2}\frac{3}{2}\frac{3}{2})$ and made polarization analysis on them. For both reflections the rotated intensity I,, evidenced the magnetic character of the signal. The intensities were from 3 to 20 counts a second in the various geometries. This shows that **650Å** is the lower limit of film thickness that can be measured in a reasonable time.
The temperature dependence was made on the most intense reflection, the $(\frac{1}{2}\frac{1}{2}\frac{1}{2})$ reflection, in the $\sigma\sigma$ channel. Data are shown in figure 2. There is a clear evidence that the transition becomes of second order and that the Neel temperature of 143K is much bigger than the bulk one (118K).The loss in intensity around 130K is not understood yet and has to be looked at in detail in a new experiment.

Due to the (001) surface of the bulk crystal, it was not possible to work in specular geometrie. The crystal was mounted with the (010) direction perpendicular to the scattering plane. In this configuration seven magnetic reflections were found in direct beam without polarization analysis. Polarization analysis was made on the most intense peaks (2000cts/s). The presence of a signal in the rotated channel I,, proved the magnetic character of the intensity. The best signal to background ratio was found for $(\frac{1}{2}\frac{\bar{1}}{2}\frac{5}{2})$ in the $\sigma\pi$ channel. Temperature dependence was made on this magnetic reflection. Figure 3 shows that with X-rays we observe the same first order transition at 118K as with the neutrons.

Future experiments will deal with resonance and more detailed temperature studies.

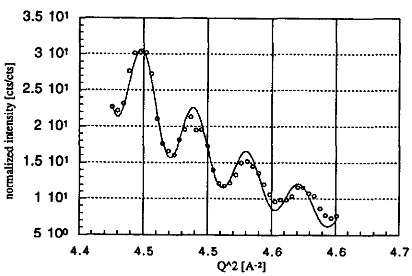

Fig.1: Kiessig oscillations around the (111) reflection. They give access to film thickness and roughness.

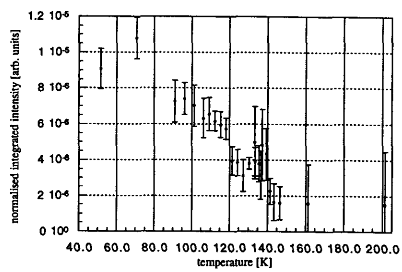

Fig.2: Temperature dependence of the integrated intensity of the $(\frac{1}{2}\frac{1}{2}\frac{1}{2})$ reflection in $\sigma\pi$. In the film case a second order phase transition with a Néel temperature of 143K is observed.

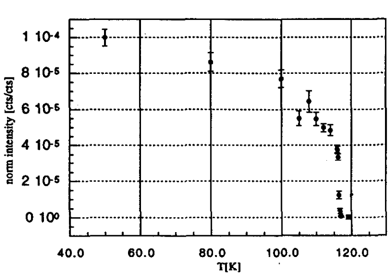

Fig.3: Temperature dependence of the integrated intensity of the $(\frac{1}{2}\frac{\bar{1}}{2}\frac{5}{2})$ reflection in $\sigma\pi$. In the bulk case a first order phase transition with a Neel temperature of 118K is observed.

Reference: [l] D. Bloch, R. Maury, C. Vettier, W.B. Yelon, Phys. letters (1974) 354-356.