ESRF	**Experiment title:** Superstructure of ultrathin magnetite (Fe3O4) layers	**Experiment number**: SI-728
Beamline:	**Date of experiment**: from: 03-oct-01 7:00 to: 10-oct-01 7:00	**Date of report**: 28-2-02
Shifts:	**Local contact(s)**: Dr. Bernd Struth	*Received at ESRF:*

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Report: The aim of the experiment was to investigate the in-plane and out-of-plane structure of the interface of Fe_3O_4 and MgO. Three samples were actually studied during the run: 1) a 12 nm thick Fe_3O_4 film on a MgO(001) substrate capped with a few nm of MgO, 2) an uncapped 12 nm Fe_3O_4 film, and 3) a thin burried Fe_3O_4 film. Sample 1) was most extensively studied. Both the substrate and film diffraction spots could be measured without much difficulty. The magnetite diffraction spots were very broad in directions parallel to the interface (of the order of 1 degree) in contrast to the extremely narrow MgO substrate spots. This is probably related to the presence of anti-phase domains in the magnetite layer. We could not find the superlattice spots corresponding to a primitive cubic lattice visible in electron diffraction patterns for similar but free-standing magnetite films. So, apparently the in-plane superstructure is not present in the burried layers of this study in contrast to the free films of the electron diffraction study. The conclusion is that neither the bulk of the magnetite films, nor the MgO/Fe_3O_4 interfaces give rise to a superstructure in X-ray diffraction.

The second part of the experiment put forward in the research proposal was to determine whether the occupation of the interface layers is consistent with the physical requirement of charge neutrality and non-polarity of the film. To answer that question we concentrated during the rest of the run on measuring the structure of a number of crystal truncation rods (CTR). Representative rodscans performed are shown in fig. [1-3]. The period of the fringes observed in the rodscans corresponds to the total thickness of magnetite film and caplayer. Unfortunately this period was not fully reproducible. In Fig. 1 the reflectivity at small angles measured during the first day and 2 days later is compared. It is seen that the small period increased after having exposed the sample to the X-ray beam for a few days and that the long period modulation of the fringes related to the MgO caplayer disappeared completely. This observation tellls us that during the experiment the MgO caplayer disappears physically. The same observation was made for sample 3). Even for sample 2) some small changes were noticed in the period of the thickness fringes after long exposures, although there is no caplayer in that case. A speculative explanation of this phenomenon is that the insulating sample is charged up to the 8kV of the X-ray beam due to photo-electron emission. This charged sample then is bombarded by charged particles from the surrounding atmosphere, sputtering off the outer layers of the sample. Consequently the intensity profile of the rodscans changes in time and a fit to a structural model is not possible. Nevertheless, from a qualitative comparison with simulations we may tentatively conclude that the magnetite layer is most probably bounded by only half a layer of iron atoms at the interface, making the interface non-polar as predicted. However, to deliver the final proof it will be necessary to collect reliable rodscan data after solving the film deterioration problem. Our next attempt will be to cover the sample with a grounded amorphous carbon layer to prevent charging. In march 2002 we hope to collect the new data and to finish the project.

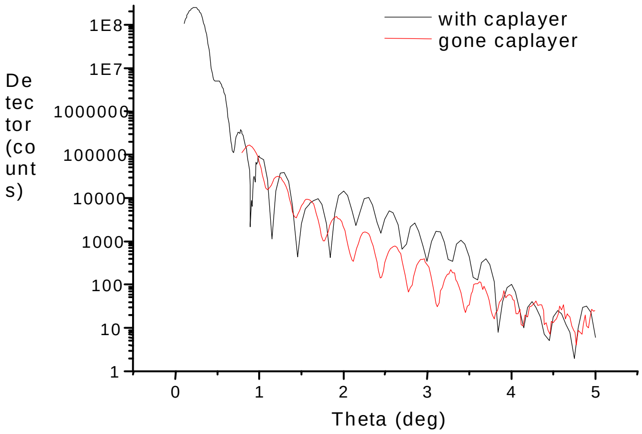

Fig1. X-ray reflectivity changes in time. Red: measurement after 2-3 days. The caplayer has disappeared, i.e. the large period is gone and the small period has increased somewhat.

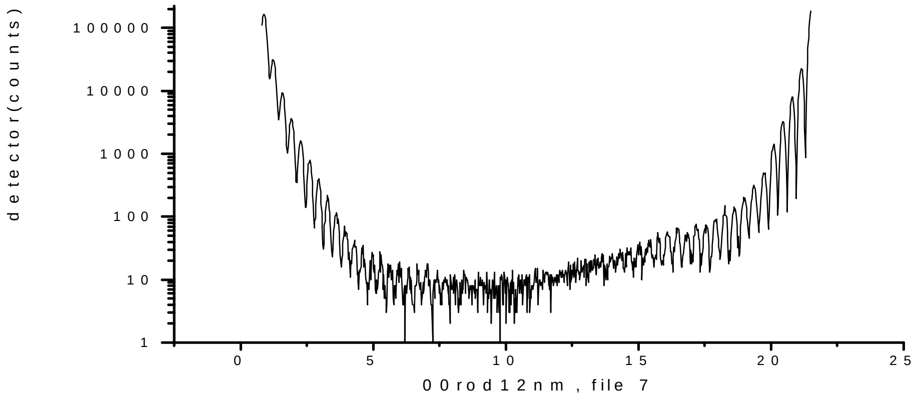

Fig. 2 (00)-rod scan of a 12 nm Fe_3O_4layer on MgO(001) (sample 1). The caplayer has disappeared almost completely.

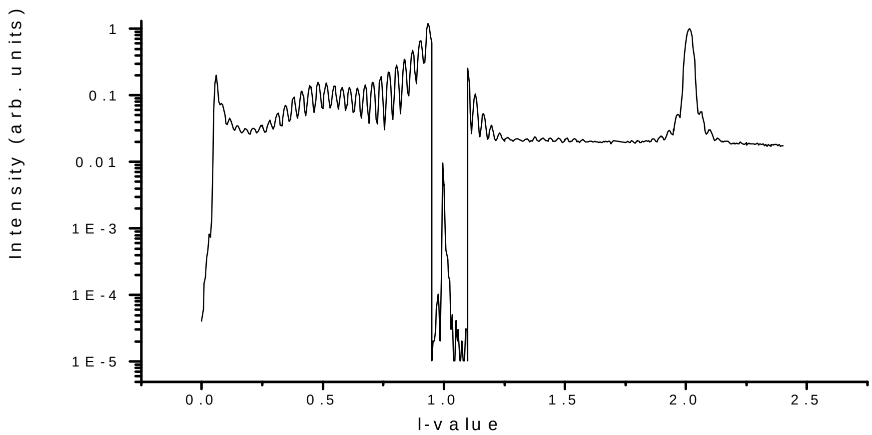

Fig. 3 (11)-rod scan of a 12nm Fe_3O_4 layer on MgO(001) (sample 2). No caplayer was applied in this case.