	Experiment title: Structures of the simple terminations of Fe3O4(111)	**Experiment number**: SI-1364
Beamline: ID32	**Date of experiment**: from: 03/05/06 to: 09/05/06	**Date of report**: 30/08/06
Shifts: 21	**Local contact(s)**: Dr Isabelle Joumard	*Received at ESRF:*

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Report:

In this experiment we attempted to carry out a quantitative structural determination of Fe_3O_4(111) using surface x-ray diffraction (SXRD) and scanning tunnelling microscopy (STM). *In situ* preparation was carried out in the UHV facilities located in the Surface Characterisation Laboratory (*SCL*) by repeated cycles of argon sputtering and annealing in up to 10^{-7} mbar O_2. Multiple domains were observed by LEED and STM. These were previously observed [1]. This is due to the anneal temperature being too high, which seemed to result from an inaccurate (underestimation) temperature reading. It is not possible to recover the sample to suppress the multi domain without polishing. Nevertheless we proceeded with the SXRD measurements to check the quality of the sample. The sample was transferred to the beamline under UHV, using a *Baby* chamber [2]. SXRD measurements were performed on beam line ID32, employing the diffractometer in EH1. X-ray experiments were performed at 17.7 keV. SXRD data were collected at room temperature using conventional rocking scans, in which the sample is rotated about its surface normal while scattered x-ray intensity is measured. For a given *(h,k)* these were performed at different *l*, enabling crystal truncation

rods (CTR's) to be compiled. 3 CTRs were measured, with a total of 136 reflections. In the CTRs, spurious peaks appear at some *l*=integer (arrows, see figure 1) that are due to the presence of multiple domains/crystallites. This probably arises from the high temperature anneal. As the possible terminations of Fe_3O_4(111) give rise to similar CTRs (see figure 1), a full complete analysis requires a more extensive set of data.

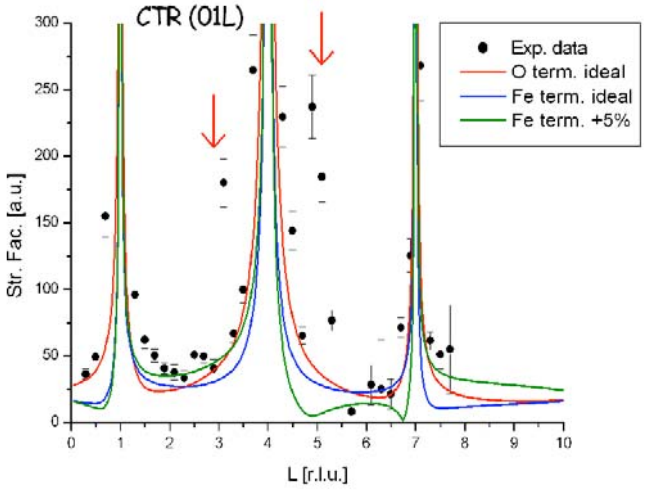

Figure 1. Crystal truncation rod (01L) for the Fe_3O_4(111) surface. The curves correspond to the ideally O terminated (red) and Fe terminated (blue) surfaces, and a Fe-terminated surface with 5 % relaxation of the interlayer distance (green).

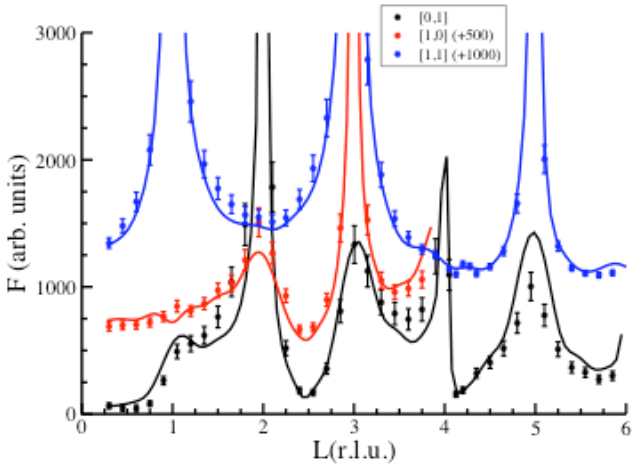

Figure 2. (01L), (10L) and (11L) crystal truncation rods for the TiO_2(110)-1x1 surface. Each CTR is represented in a given colour. Curves are offset for clarity. Continuous lines correspond to the obtained fit.

The TiO_2(110)-1x1 surface termination has also been measured to elucidate the discrepancies existing between recent experimental analysis on the same surface obtained with LEED [3], MEIS [4] and early X-ray diffraction measurements [5]. A rather large data set was measured involving 15 non-equivalent CTR's containing more than 700 reflections. This large data set was required to determine the modulus and directions of the atomic relaxations of the topmost surface atoms. The results obtained from the analysis of the XRD data coincide with those obtained by LEED and Ion scattering (MEIS). The controversy regarding the results obtained form different methods on this system is consequently resolved.

References:

[1] R.A. Fellows et al., Surf. Sci. 445, 11 (2000)
[2] www.esrf.fr/UsersAndScience/Experiments/SurfaceScience/ID32/SurfaceLab/
[3] R. Lindsay et al., Phys. Rev. Lett. 94, 1 (2005)
[4] G.S. Parkinson et al., Phys. Rev. B 73, 245409 (2006)
[5] G. Charlton et al., Phys. Rev. Lett. 78, 495 (1997)