ESRF	**Experiment title:** Determining the structure of TiO_2 (110) (1x1) at low temperature using surface X-ray diffraction	**Experiment number**: SI - 666
Beamline ID 3	**Date of experiment**: from: 22/03/01 to: 29/03/01	**Date of report**: 20/08/01
Shifts: 21	**Local contact(s)**: Dr Paul Steadman	*Received at ESRF:*

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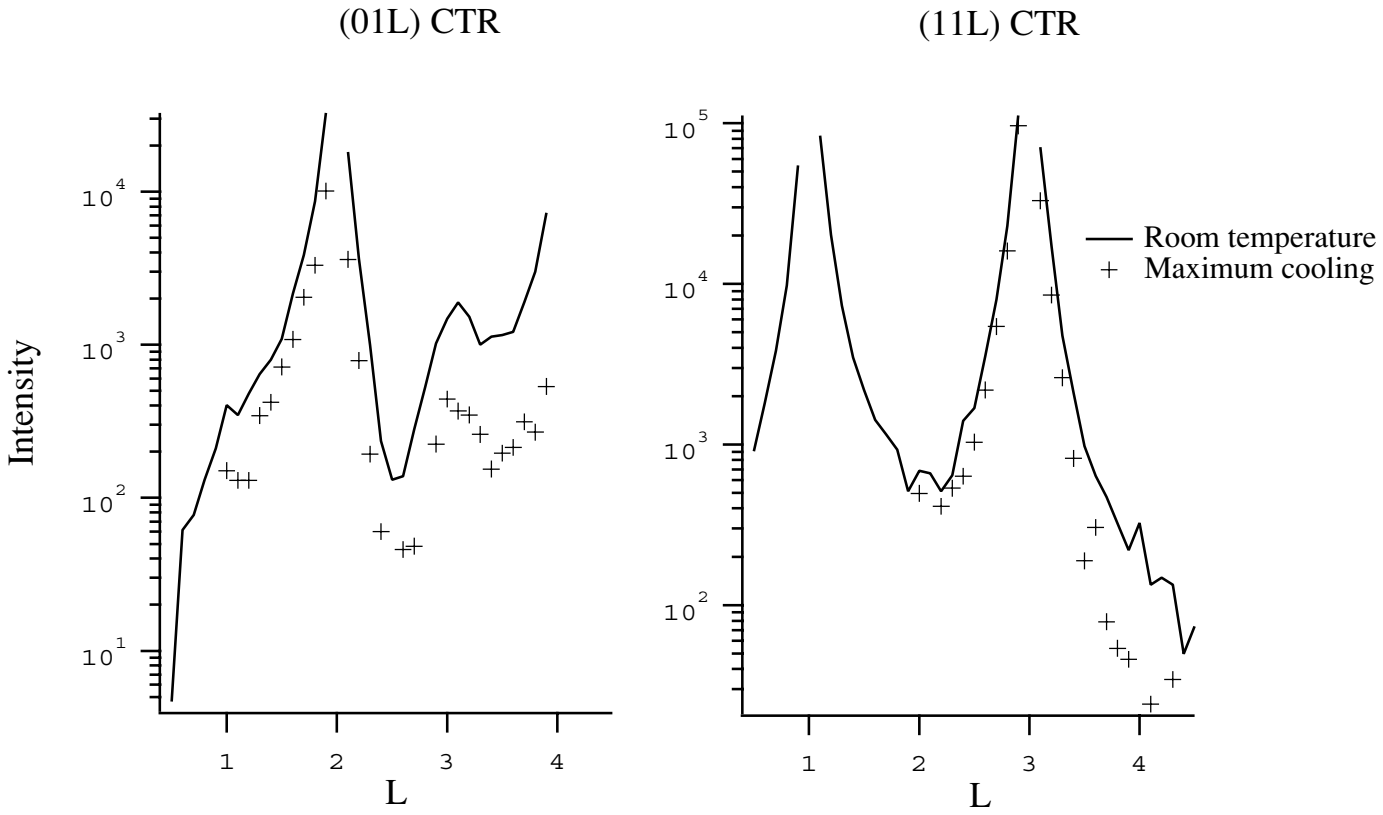
Report:

This experiment aimed to accurately locate atomic positions on the TiO_2 (100) 1x1 surface using Surface X-ray Diffraction at temperatures near 50K. This work was undertaken as theoretical models of this surface [1,2,3] agree poorly with our previous room temperature determination of this surface structure using SXRD [4]. Recent calculations [5] suggest that this difference may arise from soft anisotropic and anharmonic surface vibrations. By cooling the sample with liquid helium we expected to damp these vibrations, allowing us to test the validity of this hypothesis and obtain definitive values for the surface atomic positions.

Crystal truncation rods were measured at room temperature to compare with our previous work. The data closely matched our earlier work, ensuring the surface preparation had been successfully reproduced. Measurements were repeated with the sample cooled with liquid helium. We observed only a small difference in the data from the room temperature rods. Two rods are displayed overleaf. The divergence was always downwards and increased throughout the measurement. By comparison with our models we took this to suggest that the cold sample was acquiring significant surface contamination over the time scale of the measurement even at the chamber pressure of $2x10^{-10}$ mbar. This relatively high pressure was partly due to the chamber pressure falling slowly after the sample had been cleaned. This sample contamination meant we were unable to accurately investigate the effect of cooling on the crystal truncation rods. Improved chamber pressure would be required to successfully complete this work.

Of the remaining three shifts, two were employed to continue a previous experiment at ESRF (SI-313) where we attempted to investigate the structure of TiO_2 (100) - 2x1 formate, which has been previously observed by a variety of techniques [6]. We concluded that the intense X-ray beam causes the formate to dissociate. Here we repeated this experiment with liquid helium cooling in the hope that we could obviate this problem, and found we were still unable to form a stable overlayer, suggesting that the desorption process is not primarily the result of local heating. The remaining shift was used to collect data from the clean surface at room temperature. We were able to obtain a larger data set than in our previous work at the ESRF and the SRS [4] which will form a sound basis for a future attempt at investigating the

TiO_2 surface structure by modeling the vibrational modes, in collaboration with the Daresbury theory group.



In addition to contamination problems, we encountered a number of problems with beamline equipment. The first sample mount used was built in several pieces, so heat transmission to the sample was poor. This combined with a boroelectric heater, rather than an electron beam heater, meant it was difficult to attain a high enough temperature to order the surface. This was swiftly replaced with a custom built monolithic backplate, which allowed us to heat (and cool) the sample more efficiently. These difficulties were exacerbated by the absence of LEED or RHEED diagnostics. Both the heater and the platinum resistance thermometer used to determine sample temperature suffered repeated intermittent failures, and the thermometer did not measure reliably at low temperatures. In addition, cryocooling was very problematical, with frequent, unexplained rises in sample temperature during cooling. It took several attempts to successfully cool the sample.

We received high quality support from beamline personnel, particularly Dr Steadman who worked tirelessly as a member of the experimental team.

References:

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