DUBBLE	**Experiment title:** **Towards *in-situ* monitoring of the PLD process by synchrotron X-rays** **Step 3: Surface reconstruction of the $SrTiO_3$ (001) surface.**	**Experiment number:** **26-02-157**
Beamline: BM26	**Date(s) of experiment**: From: 21-06-2003 To: 27-06-2003	**Date of report**: 18-09-2003
Shifts: 18	**Local contact(s)**: I. Dolbnya	

Names and affiliations of applicants (* indicates experimentalists):

S. Harkema[1*], V. Vonk[2*], H. Graafsma[2], A.J.H.M. Rijnders[1], D.H.A. Blank[1] and H. Rogalla[1]

[1]Low Temperature Division and MESA+ Research Institute, University of Twente, POB 217, 7500AE, Enschede, The Netherlands
[2]European Synchrotron Radiation Facility, Grenoble, France

Report:

$SrTiO_3$ is a widely used substrate-material in thin film deposition processes like Pulsed Laser Deposition (PLD). High quality single crystals of e.g. high-T_c superconductors can be grown using these substrates.
The (001) surface has two different terminations, i.e. SrO and TiO_2. By chemically etching, single-terminated surfaces can be obtained, as confirmed by Atomic Force Microscopy. A second treatment, annealing in flowing oxygen at 950 ºC, assures that any oxygen vacancies formed in the etching process are filled again, in order to obtain a stable surface. Two annealed samples have been used in the measurements, one that was etched (TiO_2. terminated) and that was not etched (mixed termination). The samples used were low miscut substrates with a miscut angle of 0.07^0.

Since deposition takes place at elevated temperatures between 600 and 800 ºC and often in an oxygen environment, it is important to have knowledge about the surface-structure under these conditions.
A furnace that has been used in previous experiments on already deposited films was used to achieve the required temperature. In order to avoid overheating the aluminum cap of the furnace, the pressure was lowered to the 10^{-3} mbar range, which is also typical for deposition.

The main aims of the present experiment were to study the apparent C(4x4) surface reconstrucion observed in our last experiment (26-02-129) on DUBBLE and to improve the surface diffraction measurements of both samples using low-miscut samples.

The surface reconstruction observed in previous measurements could not be reproduced. The reason for this has been investigated in detail and it became clear that the observed non-integer reflections found last time were caused by higher harmonics (?/4) in the beam. The effect has been enhanced by the apparent beam hardening caused by the Al-cap used in the experiments and a non optimal setting of the detection chain.
The absences of the apparent C-centering can be explained by the intensity of the different reflections involved.

The higher harmonics problem was studied in more detail. The results are collected in a separate technical report on the DUBBLE run.

For both samples a number of Crystal Truncation Rods and in plane reflections have been measured. For the TiO_2 terminated sample measurements were made at room temperature, at 800 ºC and at room temperature again. For the mixed termination sample measurements were made at room temperature only.
The disagreement factor of symmetry related CTR's (see the figure for examples) is between 5 and 10%. CTR's and in-plane structure factors are presently being analyzed to obtain the surface structure. The results of the mixed termination sample will be used for a critical evaluation of the method described in the literature to obtain the ratio of SrO and TiO_2 termination of the surface (G.Charlton et al. *Surface Science* **457** (2000) L376-380).