ESRF	**Experiment title:** Towards *in-situ* monitoring of the PLD process by Synchrotron X-rays Step 1: Temperature-induced changes in the $SrTiO_3$(001)	**Experiment number**: 26 02 129
Beamline: BM26	**Date of experiment**: from:10-09-2002 to:16-09-2002	**Date of report**: 05-11-2002
Shifts: 18	**Local contact(s)**: I. Dolbnya	*Received at ESRF:*

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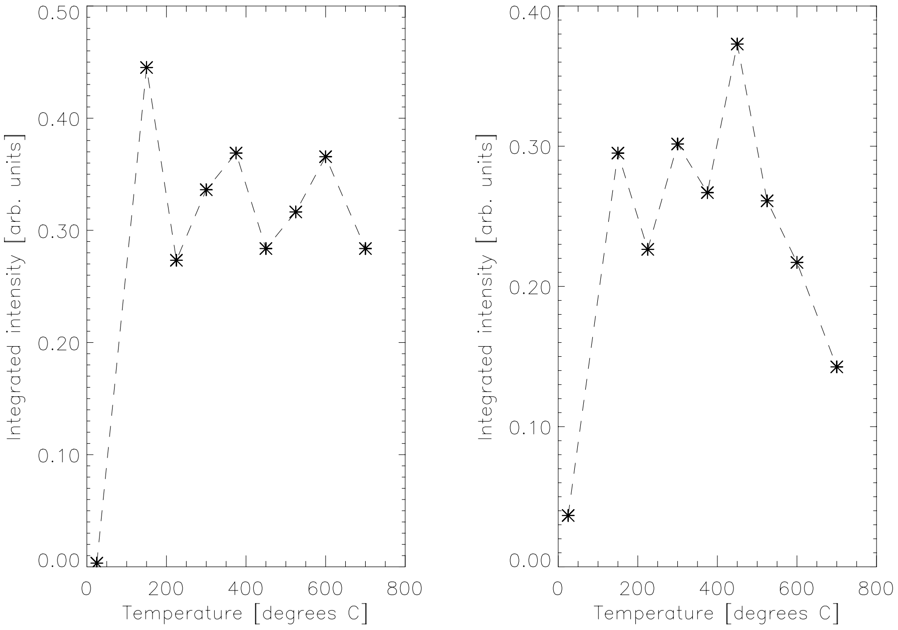
Report:

$SrTiO_3$ 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 samples have been used in the measurements, one that was etched and one that was annealed as well.

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 exact surface-structure in 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 aluminium cap of the furnace, the pressure was lowered to the 10^{-3} mbar range, which is also typical for deposition.

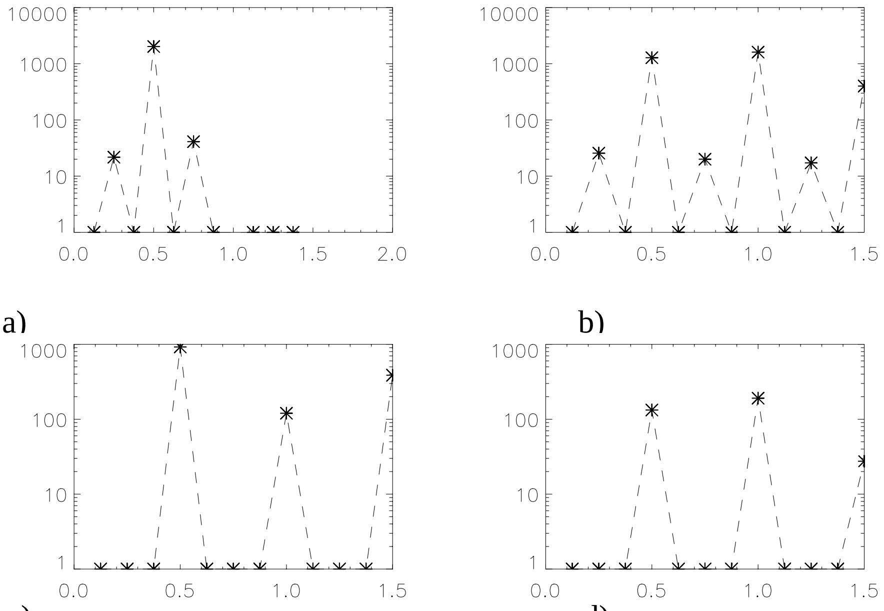
Surface X-ray Diffraction (SXRD) using the 2+3 circle diffractometer at BM26 was employed to study the aforementioned structure. A wavelength of λ=0.780 Å was selected with the Si(111) double-bounce monochromator. In addition a Pt-coated mirror was used to further reduce any higher harmonics. An ion chamber just in front of the diffractometer and after a pair of sample slits (0.5x1 mm^2 HxV) was used to monitor changes in intensity. The samples, which were mounted on a hexapod, were aligned by means of a laser in such a way that their optical surfaces were perpendicular to the ω-axis. The angle of incidence was fixed at 1° to enhance surface sensitivity. Several rods were measured up to l=4 for both samples at room-temperature. Although the chosen wavelength is absorbed considerably by the 1mm thick aluminium cap of the furnace, the signal-to-noise ratio was acceptable. The annealed sample was heated to 700 ºC and here reconstruction reflections were found indicating c(4x4) reconstruction, which are formed due to the ordering of oxygen vacancies which are created at high temperatures. In figure 1 the integrated intensities of the ½01 and ¼¼1 superstructure reflections are plotted as function of temperature.



a) b)

Fig. 1 Integrated intensities as function of temperature of the a) ½01 and b) ¼¼1 reflections.

Clearly the peaks vanish upon cooling to room-temperature, indicating that either the ordering disappears or oxygen diffuses back into the crystal. Measurement of the rods of the reconstruction reflections reveals the modulation as can be seen in figure 2.



a) b) c) d)

Fig. 2 CTR’s (horizontal axis shows ℓ, vertical the integrated intensity in arbitrary units) of a) ½0 b) ½½ c) 1½ and d) $^{3}/_{2}$½1 For clarity all the data points not showing a peak are set to 1.