	Experiment title: *In-situ* monitoring of the PLD process by synchrotron X-rays: STO on DyScO	Experiment number: 26-02-405
Beamline: BM26	**Date(s) of experiment:** From : 06-11-07 To : 12-11-07	**Date of report:** 06-01-08
Shifts: 18	**Local contact(s):** W. Bras, K. Kvashnina	

Names and affiliations of applicants (* indicates experimentalists):

S. Harkema[1*], D.H.A. Blank[1], H. Graafsma[2*], A.J.H.M. Rijnders[1], P. Tinnemans[3*], E. Vlieg[4*]

[1] Faculty of Science & Technology and MESA+ Research Institute, University of Twente, Enschede, The Netherlands
[2] Deutsches Elektronen Synchrotron, Hamburg, Germany
[3] European Synchrotron Radiation Facility, Grenoble, France
[4] IMM Dept. Solid State Chemistry, Radboud University, Nijmegen, The Netherlands

An important class of oxidic materials is formed by the perovskites: complex transition metal oxides. Depending on composition, this class of materials includes itinerant and local ferro-magnets, high Tc superconductors, ferroelectrics, insulators, semiconductors and half-metallic magnets. In view of the technological importance of these compounds and especially of thin layers of these materials, they are extensively studied in our group.
The preferred technique for the growth of these thin films is Pulsed Laser Deposition (PLD). The PLD process can be monitored by high pressure Reflection High Energy Diffraction (RHEED). The RHEED method, however, only probes the topmost layers. Furthermore, due to the strong interaction, the theoretical interpretation of the result is complicated. When using (synchrotron) X-rays, the periodicity is probed on a much larger scale, making the method less sensitive for contaminations. The theoretical interpretation (kinematical theory) is much simpler. Therefore, PLD and surface diffraction is combined by means of synchrotron X-rays to in-situ monitor intensity oscillations during PLD and to study the thin (few unit cell) layers produced this way.
Earlier experiments of this project were 26-02- 271, 292, 309, 332 and 373.

Previous experiments showed the possibility to study layer-by-layer growth of several complex oxides ($LaTiO_3$ on $SrTiO_3$, $PbTiO_3$ on $SrTiO_3$), using X-rays.
During this experiment the system $SrTiO_3$ (STO) on $DyScO_3$(110) was investigated. The novel substrate $DyScO_3$ has special properties (e.g. a larger in-plane lattice constant) as compared to other commonly used substrates, creating the possibility to obtain, of example tensilely strained ferroelectric phases of extremely thin films of the normally cubic STO.
The thin film needs to be grown at elevated temperature (650^{o}C). Due to the difference in thermal expansion coefficient, the crystal lattice of the substrate and film material at this temperature match up and a good epitaxial film is grown. When the substrate and film are cooled down, the difference in thermal expansion coefficient creates the strain into the film.

The new setup, used in this experiment, is described in the report of the previous experiment (26-02-373).
The first 6 shifts (2 days) in this experiment were used to align the diffractometer and to mount/align the *in-situ* PLD chamber.

The growth of the thin film was monitored by measuring scattered intensity at the "anti-Bragg" position (0 0 0.5). Growth oscillations can be seen in figure 1: each half oscillation corresponds to one monolayer of STO. The constant signal, after the laser is stopped, indicates a stable thin film. Subsequent measurements of the specular rod (fig. 2) shows an epitaxially strained film, the fringes in the rod conforming the number of deposited monolayers. From analyses of the measured dataset the crystal structure of the film can be reconstructed. A first fit of the data indicates an extremely tensilely strained STO3 film, about 9%, compared to the 2-3% mentioned in literature for thicker films.

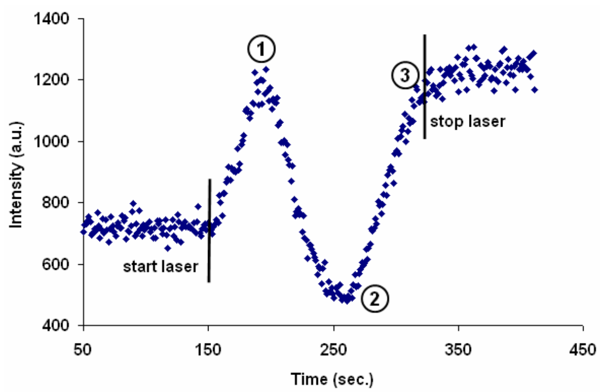

Fig 1: Intensity oscillations at the "anti-Bragg" position during growth of 3 monolayers STO on $DyScO_3$. The numbers indicate the number of monolayers completed..

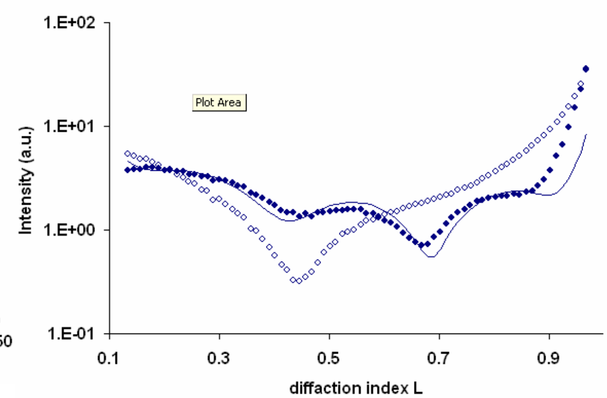

Fig 2: Specular rod of the bare $DyScO_3$ substrate (open circles) and the substrate with the deposited STO thin film (filled circles). The line is a model where the STO film is extremely strained.

Additional features of the bare $DyScO_3$ substrate have been observed during the measurements. Measuring the bare substrates at different stages revealed some unexpected results. In figure 3, the specular rod at room temperature and elevated temperature are clearly different. This can be expected if one considers, for example, a water or oxygen layer on top of the substrate at room temperature, which disappears at elevated temperature (650°C). Though, during the heating of the substrate, the scattered intensity does not decrease monotonically from one position to the other (dashed arrow, figure 3). In figure 4, rocking curves at different temperatures show an oscillating behaviour. The origin if his unexpected behaviour will be further investigated.

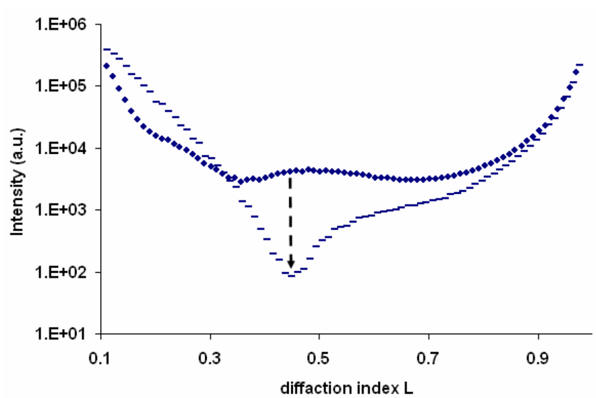

Fig 3: Specular rod of the bare $DyScO_3$ substrate at room temperature (filled circles) and deposition temperature (650°C) (dashes).

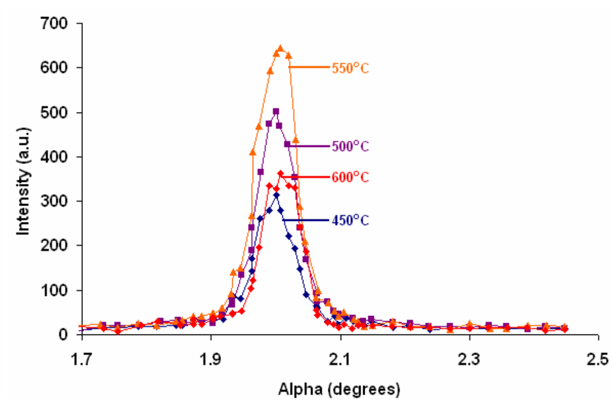

Fig 4: Rocking curves of a bare $DyScO_3$ substrate at different temperatures.