	Experiment title: *In-situ* monitoring of the PLD process by synchrotron X-rays	Experiment number: 26-02-373
Beamline: BM26	**Date(s) of experiment:** From : 05-03-07 To : 12-03-07	**Date of report:** 14-06-07
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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. SrTiO3 (001) substrates are widely used in thin film growth of related oxide materials by 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. Further-more, 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, we started a project to combine PLD and surface diffraction 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 26-02-129,157, 224, 248, 271, 292 and 309.

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.
This experiment the system $SrTiO_3$ on $DyScO_3$ was investigated. The novel substrate $DyScO_3$ has special properties as compared to other commonly used substrates, creating the possibility to obtain, of example ferroelectric phases of extremely thin films of the cubic $SrTiO_3$.

A new *in-situ* PLD chamber was used in this experiment (figure 1). The design of the new sample chamber now allows for:
1) Full rotation of the sample, so full crystallographic data can be taken without any blind spots.
2) A bigger x-ray exit window, with the possibility to explore a bigger range in reciprocal space.
3) The use of multiple targets to produce multilayer systems or capping layers
4) Fixation of chamber and laser on the same base to avoid realignment of the laser.

The first 9 shifts (3 days) were used to align the diffractometer and to mount the new sample chamber.

During the subsequent days of the experiment the new sample chamber performed well and only minor adjustments in the setup had to be made. The scattered intensity at an “anti-Bragg” position (0 0 .5) was monitored (figure 2) to determine the number of monolayers deposited.. A clear oscillation pattern is measured, but the overall intensity drops. This indicates a non perfect layer-by-layer growth as can also be concluded from the decrease in intensity after deposition.
Possible causes for the non prefect layer-by-layer growth may be:

a) The target could not be rotated.
b) The energy density of the laser was too high and this created pits in the target, reducing the quality and shape of the plasma.
c) A not optimal target-substrate distance.

Despite this non perfect layer-by-layer growth, the produced film are of rather good quality, as seen in subsequent measurements. Kiessig fringes are visible in the diffraction patterns (figure 3). Crystal truncation rods were measured to obtain information about the atomic structure of the film.

The new sample chamber has shown to function properly. Several small adjustment are being made for a better performance in the next run. The collected data (crystal truncation rods) are being analysed.

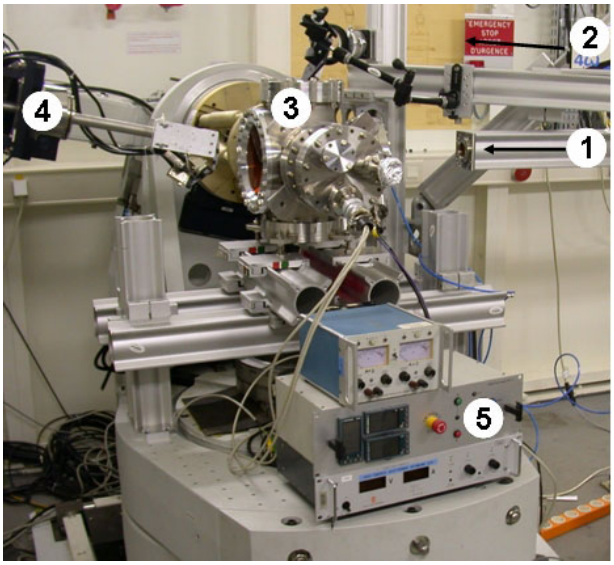

Figure 1: (top) new PLD setup on the DUBBLE surface diffractometer.
1). X-ray beam
2). laser
3). PLD-chamber
4). detector arm
5). electrical and temperature control.

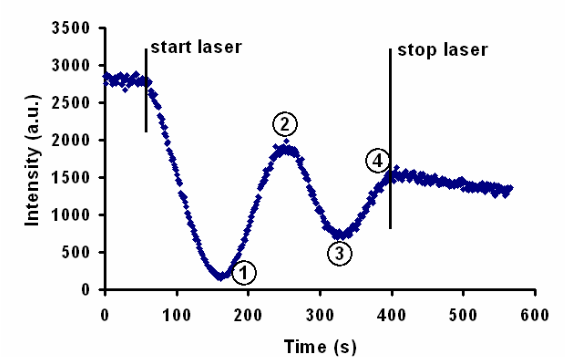

Fig 2 Intensity oscillations during growth of 4 monolayers STO on DyScO. The numbers indicate the number of monolayers at that moment.

Fig 3 Kiessig fringes around the 001 reflection. The distance of the fringes is related being to the number of monolayers.