	Experiment title: In-situ monitoring of the PLD process by synchrotron X-rays	**Experiment number:** 26-02-332
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Report:

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 ferromagnets, 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.

Recently new perovskite single crystals of scandates ($DyScO_3$, $GdScO_3$) were developed and they show good potential for strain engineering thin films. During this run thin films of $SrTiO_3$ (STO) were epitaxially grown on $DyScO_3$ (DyScO) 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. Furthermore, due to the strong interaction, the theoretical interpretation of the result is complicated. A structure determination of the grown film can therefore only be done using X-ray diffraction.

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.

The first two days (3 shifts and the MDT) had to be used to fix two mechanical problems of the diffractometer. The third day the diffractometer and X-ray optics were aligned and a stable 20 keV beam was obtained. The beam (1.00x0.35mm) remained positionally stable during the whole run with a nice flux of $1x10^{11}$ photons/sec. Although previous runs where troubled by contamination of higher harmonics, this run only a bit of $\lambda/2$ as detected and no other higher harmonics.

The last one and a half shift of the run were lost due to a cooling problem of the monochromator.

The bare DyScO substrate was characterized at room temperature and at deposition conditions (T=820°C, p=4*10^{-2} mbar O$_2$). The observed changes in the measured intensity with time indicate a change (possibly roughening) of the bare substrate, when exposing it to elevated temperature for a prolonged period of time.

Several depositions of STO on DyScO were carried out, varying the STO film thickness. All measurements where performed at temperatures of 650 or 820°C in a low pressure oxygen environment.

To check the system and deposition parameters a 20 monolayer film was grown. In the ridge scan (fig. 1) fringes can be seen, from which the film thickness can be determined. The thickness of the film corresponds to the number of oscillations seen at the “anti-Bragg” position.

A full dataset (CTR) of one monolayer of STO on DyScO has been measured. The current system seems to be stable at a film thickness of one monolayer. From this data the structure of a single monolayer STO on DyScO can determined.

A small dataset of 5 monolayers of STO on DyScO was taken at the end of the run (650°C). The lower temperature was chosen to prevent possible roughening of the film and substrate. The specular rod (fig. 2) nicely shows oscillations due to the finite thickness of the layer. In the measured out of plane rods (not shown) no fringes are observed, however. AFM seems to rule out roughening of the layer in time as a possible explanation for the absence of oscillations in the out of plane rods. It could be that the STO film is incommensurate.

A full dataset of 6 monolayers of STO on DyScO was taken at elevated temperature (820°C) and reduced pressure. Here again absence of the fringes in thenon-specular rods was found. In this case AFM indicates that the long time at this high temperature may have roughened the film.

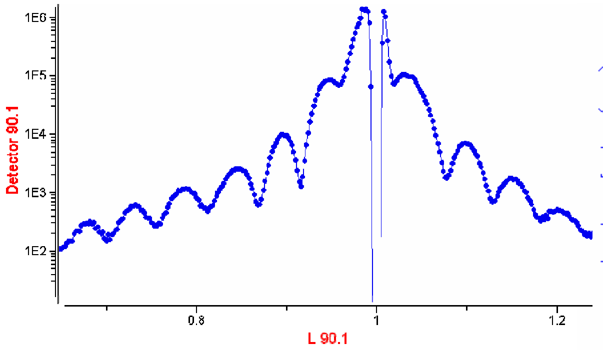

Fig. 1: Ridge scan around the (0,-1,1)-rod with *l* varying between 0.65 and 1.25. The signal goes to zero at the Bragg peak due to saturation of the detector. The pronounced Kiessig fringes on either side of the Bragg peak allow for thickness determination.

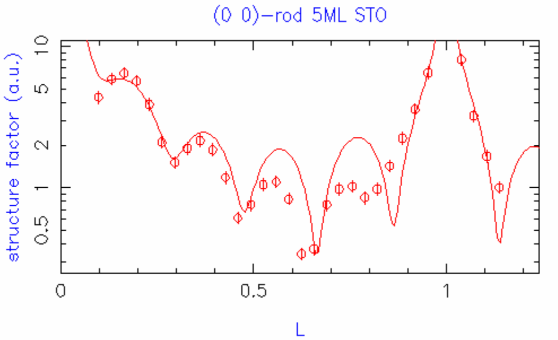

Fig. 2: The specular rod at 820°C of DyScO covered with about 5 monolayers of STO (circles). The solid line is a preliminary fit, indicating a closed and smooth outer layer.

In conclusion: surface crystallographic datasets of the STO/DyScO system were successfully obtained. The bare substrate has been investigated at high temperature. Data of different thin films were taken and can be used to solve the atomic structure.

Subsequent experiments of this system will be performed at slightly lower temperature (650°C), as compared to the films grown at the University of Twente(820°C). This to ensure stability of the film during the measurements at high temperature.