DUBBLE	**Experiment title:** *In-situ* monitoring of the PLD process by synchrotron X-rays: STO on DyScO	**Experiment number:** 26-02-429
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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.
The preferred technique for the growth of these thin films is Pulsed Laser Deposition (PLD). A high intensity laser ablates a target material and the plasma created condensates on the substrate (figure 1). With each laser pulse a fraction of a monolayer is deposited.

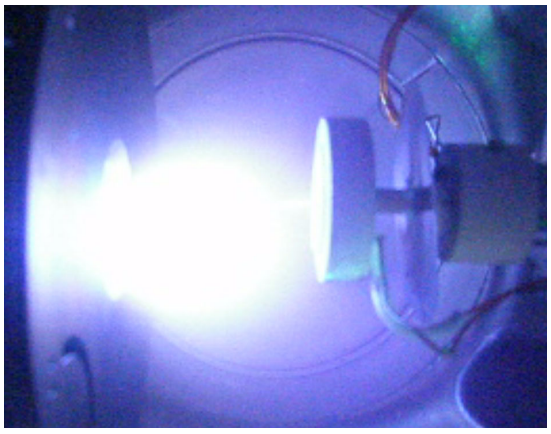

Figure 1: PLD process. A high power pulsed laser beam enters the vacuum chamber (hole bottom right) and ablates the target material (middle left. The plasma condensates on the substrate (centre).

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 are 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 and 405.

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 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.

Several technical failures were encountered during this run. During the whole week of beamtime the injection of the beam was troublesome and due to a problem with one of the RF cavities of the storage ring, the lifetime and stability of the beam was limited. Furthermore the diffractometer broke down. In total an estimated 5-6 shifts were lost due to these complications.

Four thin films of STO where deposited on DyScO substrates with different film thicknesses and different growth conditions.
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 2: each half oscillation corresponds to one monolayer of STO. The constant signal, after the laser is stopped, indicates a stable thin film.

Figure 2 and 3 show dat for two STO films, each two monolayers thick deposited with different energy densities of the laser. When the energy density is higher (pink), more material is deposited per pulse as compared to the lower energy density (blue). So the period of the growth oscillations with higher energy density is shorter.
The quality of the resulting films seems to be different. Thickness fringes (fig 3.) are less pronounced in the film grown at lower energy density, indicating more surface roughness of the film. Though not entirely visible, the same positions of the minima and maxima indicates the same number of layers grown.

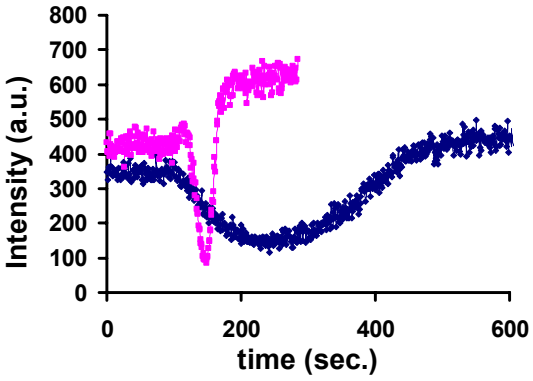

Fig 2: Intensity oscillations at the "anti-Bragg" position during growth of 2 monolayers STO on $DyScO_3$. Film grown with high energy density (pink) and lower energy density (blue).

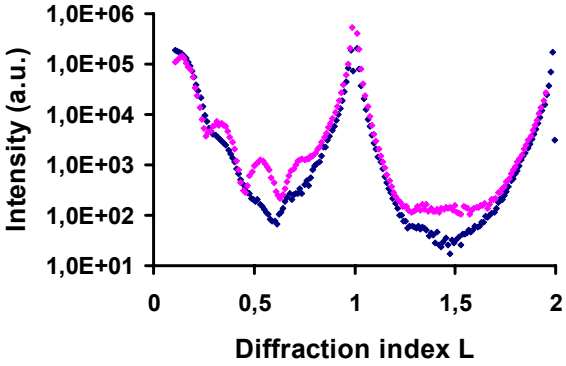

Fig 3: Specular rod of 2 monolayers STO on $DyScO_3$ at 650^{o}C. High energy density (pink) during growth and lower energy density (blue).

The two other remaining films, four monolayer STO and again two monolayer STO but at 850^{o}C where also characterized at high temperature.

Though it is relevant to measure a structural data set both at deposition temperature (650^{o}C) and at room temperature, the limited amount of remaining beamtime allowed for only one thin film to be measured at room temperature. Arrangements have been made to have the remaining films measured (at ANKA, Karlsruhe) and so to complete this run.