DUBBLE	**Experiment title:** **Towards *in-situ* monitoring of the PLD process by synchrotron X-rays** **Step 3: Testing and implementation of the chamber for monitoring the PLD process by synchrotron X-rays.**	**Experiment number**: **26-02-224**
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Names and affiliations of applicants (* indicates experimentalists):

S. Harkema[1*], V. Vonk[2*], H. Graafsma[2], A.J.H.M. Rijnders[1], D.H.A. Blank[1] and H. Rogalla[1]
K.Driessen[1,2#], M.Huijben[1#],

[1]Low Temperature Division and MESA+ Research Institute, University of Twente, POB 217, 7500AE, Enschede, The Netherlands
[2]European Synchrotron Radiation Facility, Grenoble, France
[#] experimentalists, non-applicants

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.

$SrTiO_3$ (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. 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, 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.

The first steps of this project were taken in experiments 26-02-129 and 157

The main aims of the present experiment were the testing and implementation of the chamber for monitoring the PLD process, recently constructed at the ESRF, and to perform the first *in situ* measurements.

During the first buffer day the optics were aligned to obtain a suitable X-ray beam at the sample position. After that we spent three days setting up the remainder of the experiment. Here, several problems were encountered, both in hardware and software of the existing ID equipment at Dubble. To summarize the difficulties : low intensity of the X-ray beam, higher harmonics in X-ray beam, the control of the hexapod and the diffractometer. The diffractometer and higher harmonic problems were for this particular experiment not a nuisance since we measured most of the data at a fixed point in reciprocal space.

Nevertheless, the experiments have shown that the PLD chamber, constructed for use on Dubble, is properly working and that the PLD process can be monitored.

Two deposition processes have been studied *in situ*: the deposition of $SrTiO_3$ on $SrTiO_3$ (001) and deposition of **c**-axis oriented YBCO on $SrTiO_3$ (001), both at 800^o C. The intensity oscillations measured on the specular rod, for both experiments, are given in the following figures. From the oscillations observed it can be concluded that the growth mode is layer by layer. Also shown are two scans of the 001 reflection of the YBCO layer, made during intermissions in the deposition process. From these scans, the thickness of the films can be calibrated.

The results of this, in our opinion quite successful, run are presently being worked out.