	Experiment title: **Towards *in-situ* monitoring of the PLD process by synchrotron X-rays** **Step 4:** ***Study of the formation of thin layers by interface diffraction methods.***	**Experiment number:** **26-02-248**
Beamline: BM26	**Date(s) of experiment:** From: 28-01-2005 To: 31-01-2005	**Date of report:** 07-02-2005
Shifts: 12	**Local contact(s):** W. Bras, F. Meneau	

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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.

$SrTiO_3$ (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 26-02-129,157 and 224

In our previous run, we have succesfully tested and implemented the sample chamber, which is designed especially for use on the Dubble interface diffractometer. Therefore, the main aims of the present experiment were a systematic study of several deposition runs, verifying the reproducibility of the process.

During the first buffer days the optics were aligned to obtain a suitable X-ray beam at the sample position. Although this took much more time than anticipated, we obtained a positionally very stable 1x1 mm^2 beam, with a flux of about 10^{11} photons/s. Due to the increase in beam intensity, compared with previous runs, this opens possibilities to perform data-collections of CTR's with our sample chamber mounted.

After the initial beamline start-up we spent three days setting up the remainder of the experiment. Here, the same problems as always with the ID equipment at Dubble were encountered: slits that do not move properly (this has been mentioned since 2000 in the ID logbook) and communication between SPEC and the motor controllers. Since we are not permanently working at the beamline and therefore not specialists, these problems cause a lot of delay, and interfere with setting up for the laser and its optical elements, i.e. the actual set-up.

With the final set-up two samples were measured. Since it takes about a day from mounting the sample to cooling it down again after deposition, time is very precious. Unfortunately, due to a problem with the laser during the deposition of the first sample, these measurements are of limited value. The second sample gave a very good run and excellent results (figures 1, 2 and 3). However, during cool-down of the second sample a collision of the sample chamber occured, thereby damaging the connection to the vacuum pump beyond the state of easy repair. Although this was a major set-back, we used the last day of beam-time for careful ex-situ measurements of the grown thin film. The results are presently being worked out.

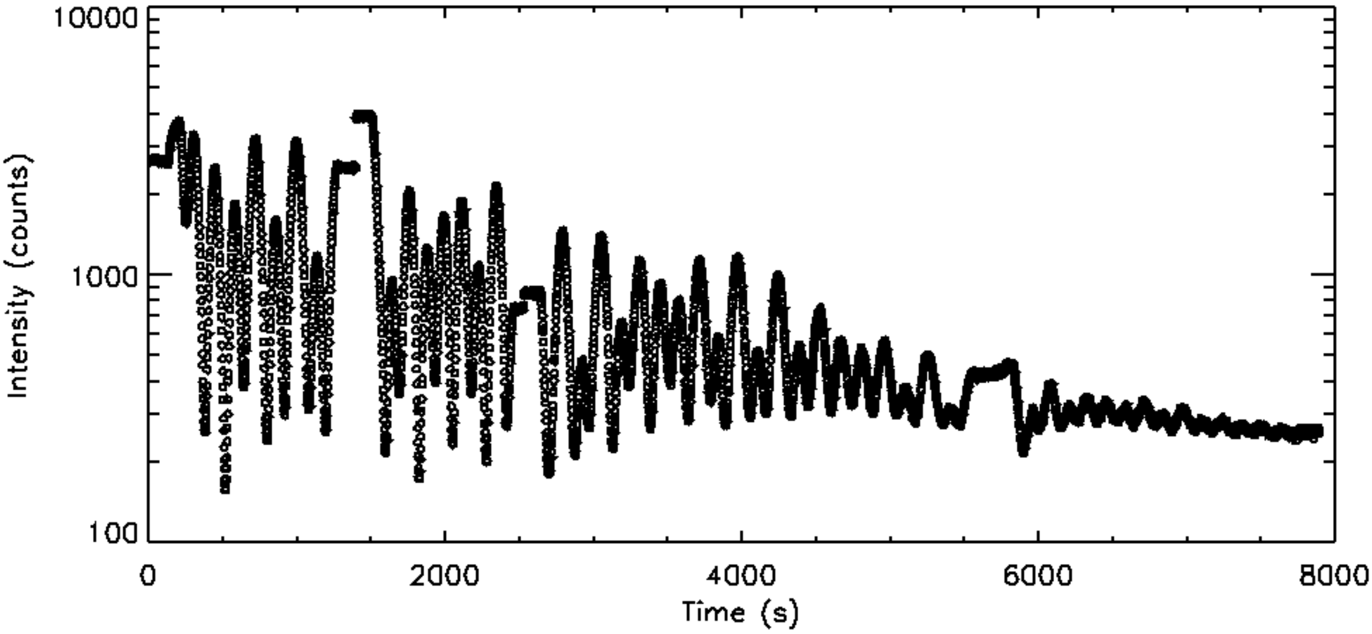

Fig.1 Intensity oscillations of the reciprocal point (0,0,0.2) in STO lattice units during deposition of YBCO. Each maximum corresponds to the growth of another monolayer.

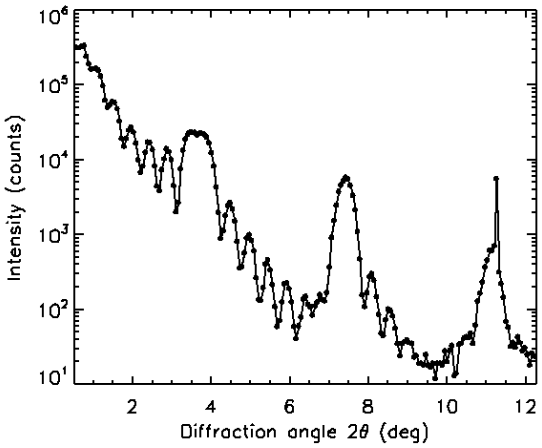

Fig. 2 Bragg reflections of the YBCO film after deposting about 10 nm.

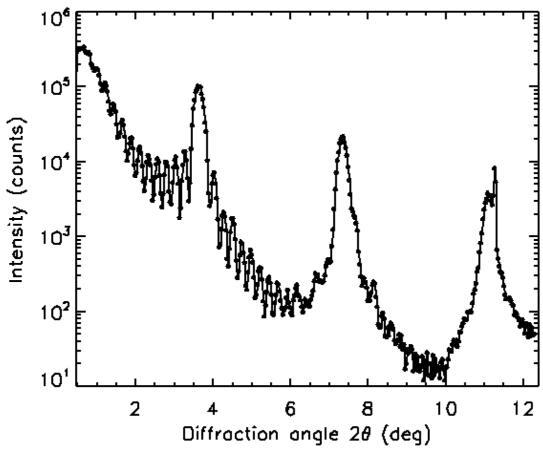

Fig. 3 Bragg reflections of the YBCO film after depositing about 20 nm.

In conclusion we can mention that although this run we have not succeeded in meeting our goals, it is clear that the set-up works in a reproducible way. The data obtained during deposition are of high quality, and allow for a detailed analysis of the growth. The ex-situ data allow for comparison between the high temperature structure and the room temperature one.