ESRF	**Experiment title:** Diffraction studies of ferroic materials under non-ambient conditions	**Experiment number**: 01-02-746
Beamline: BM01-A	**Date of experiment**: from: 31.08.06 08:00 to: 05.09.06 08:00	**Date of report**: 10.10.06
Shifts: 15	**Local contact(s)**: Dmitry Chernyshov	*Received at ESRF:*

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Report:

alpha-D-Glucose complex: The first part of the experiment period was devoted to a collection of data for the ferroelastic alpha-D-glucose · NaCl · H_2O (6:3:3) complex. Good data for one of the crystal forms of this compound is lacking and is needed in order to complete this project.

Crystals of suitable size were cut from a larger specimen and checked by diffraction. A crystal was mounted and centred in our thermostat sample cell which was conditioned at t = 23 ^oC and relative humidity (RH) = 65 %. Crystals of the complex are sensitive to radiation, and dehydrate quickly in a too dry environment under exposure to X-rays. Intensity data were collected with a CCD, wavelength λ = 0.7097 Å.

Two sets of data comprising ~ 54.860 reflections were collected to d_{min} ~ 0.59 Å. Merging excluding Friedel pairs gave a set of 24.160 unique reflections; R_{int} = 0.025 for all data. A preliminary refinement including H atoms converged at R(F^2) ~ 0.036. A closer analysis of the data and the results of the refinement remains to be done.

Epitaxial ferroelectric films: Ferroelectric films constitute the major target for study in our programme on 'Diffraction studies of ferroic materials under non-ambient conditions'. Our samples were epitaxial films of $PbTiO_3$ (PTO) deposited by RF magnetron sputtering onto substrates of $SrTiO_3$ (STO). They were mounted in our sample cell between two transparent capacitor plates made from Kapton with a thin layer of gold deposited by evaporation. The samples of size 5 x 5 mm or 7 x 7 mm were mounted horizontally on four 1.5 mm long glass pegs that were attached to one of the capacitor plates.

Ferroelectric $PbTiO_3$ is tetragonal, its (0 0 1) plane forms a nearly perfect match with the base plane of cubic $SrTiO_3$. Hence, the polar ***c*** axis of the $PbTiO_3$ film will be oriented along the growth direction, parallel to one of the main axes of the $SrTiO_3$ substrate.

Two samples with nominal film thicknesses ~ 21 nm (A), ~ 57 nm (B) were studied by 1- and 2-dim. *q*-scans to explore substrate and film reflections and satellite peaks in the vicinity of the latter reflections. The satellite peaks relay a short-range order that can be attributed to the average domain periodicity in the

ferroelectric film. The two samples and a third one with film thickness ~ 5 nm (C) were also studied by $\omega/2\theta$ scans to map out thickness fringes. A few selected reflection profiles (sample B) were studied without and with an applied DC field of ~ 2500 V/cm, and two data sets were collected w/wo field. All *q*-scans were made with an aperture ~ 150 x 300 μm. A major aim of these experiments was to find out what can be obtained by diffraction from epitaxial thin films on the BM01-A station with the beam quality and the existing instrumentation there and with our own equipment.

Fig. 1 shows the profile from a q_x-scan (transversal scan) of the PTO (0 0 1) reflection in sample A (~ 21 nm film). The Bragg peak and the satellite maxima have been fitted to a pseudo-Voigt function. The average distance from the Bragg position to the satellite maxima, $\Delta(h)$ = 0.017 x a^*, gives a domain periodicity of 23.0 nm. A similar scan on sample B (~ 57 nm film) gave a domain periodicity of 32.5 nm, *i.e.* an increase with film thickness. Fig. 2 shows an iso-intensity contour map from q_{xz}-scans (reciprocal plane mapping) of the substrate and film reflections (1 0 4) from sample B. The strong peak from the STO substrate in the upper part is located at h = 0.997, l = 4, the lower PTO (1 0 4) peak at l ~ 3.76, h is unchanged. The elongated shape of the two main peaks reflects the strain in the (h 0 l) plane, the satellite topography around the PTO peak shows the anisotropic and nearly orthogonal distribution of coherent domains in the film. Figures 3 and 4 are $\omega/2\theta$ scans of the STO and PTO (0 0 1) peaks for samples A and C, respectively. The black curve in Fig. 4 is the accumulation of 10 scans, for the red curve a correction for the sloping background has been applied.

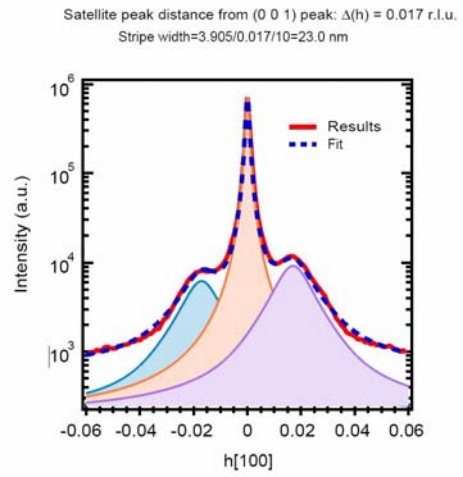

Fig.1. q_x-scan through PTO (0 0 1) peak, sample A.

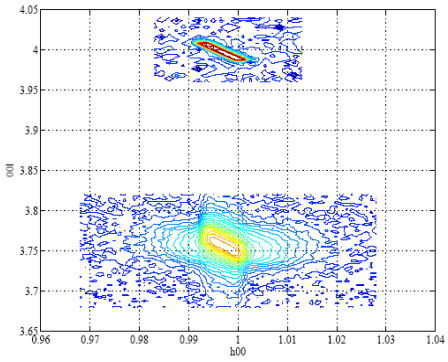

Fig. 2. Iso-intensity contour map around the STO and PTO (1 0 4) reflections, sample B.

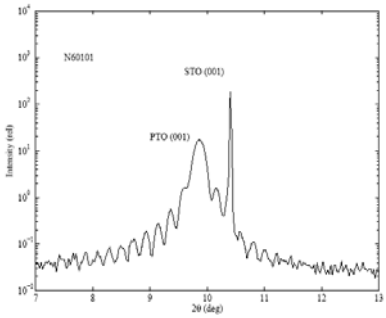

Fig. 3. $\omega/2\theta$ scan of the (0 0 1) peaks, sample A.

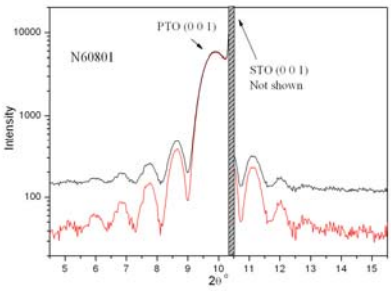

Fig. 4. $\omega/2\theta$ scan of the (0 0 1) peaks, sample C.

The work with an applied field was not conclusive and must be repeated.

One important purpose of this study was to identify limiting conditions and plan modifications in procedure and instrumentation that are required to improve the experiment. Some modifications:

- A sample mount with very precise translations/rotations and with remote control of at least two translations for the fine positioning of the film while scattering radiation.
- An additional 'manual' centring of reflections is probably necessary prior to the scans. The orientation matrix obtained from automatic centring is not good due to the large, plate shaped and strongly absorbing substrate sample.
- The 2-dim. *q*-scans are very time consuming. An alternative method is needed. Access to a 2-dim. detector with angular resolution ≤ 0.01° would be the preferred alternative.
- The sample must be mounted completely independently of the capacitor plates in order to eliminate possible movements of the sample caused by the capacitor.