ESRF

Experiment Report Form

The double page inside this form is to be filled in by all users or groups of users who have had access to beam time for measurements at the ESRF.

Once completed, the report should be submitted electronically to the User Office via the User Portal:

https://wwws.esrf.fr/misapps/SMISWebClient/protected/welcome.do

Reports supporting requests for additional beam time

Reports can be submitted independently of new proposals – it is necessary simply to indicate the number of the report(s) supporting a new proposal on the proposal form.

The Review Committees reserve the right to reject new proposals from groups who have not reported on the use of beam time allocated previously.

Reports on experiments relating to long term projects

Proposers awarded beam time for a long term project are required to submit an interim report at the end of each year, irrespective of the number of shifts of beam time they have used.

Published papers

All users must give proper credit to ESRF staff members and proper mention to ESRF facilities which were essential for the results described in any ensuing publication. Further, they are obliged to send to the Joint ESRF/ ILL library the complete reference and the abstract of all papers appearing in print, and resulting from the use of the ESRF.

Should you wish to make more general comments on the experiment, please note them on the User Evaluation Form, and send both the Report and the Evaluation Form to the User Office.

Deadlines for submission of Experimental Reports

- 1st March for experiments carried out up until June of the previous year;
- 1st September for experiments carried out up until January of the same year.

Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report, in English.
- include the reference number of the proposal to which the report refers.
- make sure that the text, tables and figures fit into the space available.
- if your work is published or is in press, you may prefer to paste in the abstract, and add full reference details. If the abstract is in a language other than English, please include an English translation.

ESRF	**Experiment title:** Impact of Strain Anisotropy on Piezoelectric Domains in MOCVD Grown $NaNbO_3$ Thin Films Studied by Grazing Incidence X-Ray Diffraction	**Experiment number**: **MA-2233**
Beamline:	**Date of experiment**: from: 16 July 2014 to: 21 July 2014	**Date of report**: 29 August 2014
Shifts: 15	**Local contact(s)**: Hubert Renevier	*Received at ESRF:*

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Report:

Note in advance:

In the framework of the experiment MA-2233 we have investigated the piezoelectric domain structure of $NaNbO_3$ and $(K,Na)NbO_3$ thin films grown on different substrates. The experimental data has not been evaluated in detail yet since the end of the experiment is just five weeks ago and we have accumulated a lot of data. We instead give a very preliminary report here and would like to emphasize that

(i) the proposed experiment was successfully carried out

(ii) the experimental station BM02 (D2AM) is suitable for carrying out such an experiment (although the rather low intensity of the primary beam at a bending magnet is critical)

Preliminary results

We have investigated piezoelectric domains in $NaNbO_3$ and $(K,Na)NbO_3$ epitaxial thin films that have been grown on complex oxide substrates by MOCVD. We have applied in-plane grazing incidence x-ray diffraction (GIXD) in order to investigate the impact of strain (induced by various substrates with different lattice mismatch to the films) on structural properties of the domains. These properties determine the ferroelectric domain formation. Three samples could be investigated during the beam time.

We have worked at an x-ray energy of 10 keV and we were using a linear position sensitive detector (Vantec) for analysing the intensity of the diffracted x-ray beam as a function of the exit angle to the sample surface. In order to tune the x-ray penetration depth into the sample we measured at two different angles of incidence slightly below ($\alpha_i = 0.20°$) and slightly above ($\alpha_i = 0.40°$) the critical angle of total external reflection.

We have performed (i) angular scans in order to measure the monoclinic distortion angle of the in-plane unit cell and (ii) radial scans to measure the corresponding in-plane strains. Also in-plane reciprocal space maps combining both, angular and radial scans, have been carried out. As an example, Fig.1a shows the lateral PFM image of a $K_{0.45}Na_{0.55}NbO_3$ film grown on (110) $DyScO_3$ substrate. Here, multi-domains of type a_1a_2/c are observed. They form a complicated but well ordered pattern with stripe domains which are aligned

± 45° with respect to the $[001]_{DSO}$ substrate direction. The corresponding x-ray diffraction results are presented in Fig.1(b) (radial out-of-plane section) and Fig. 1(c) (in-plane intensity distribution).

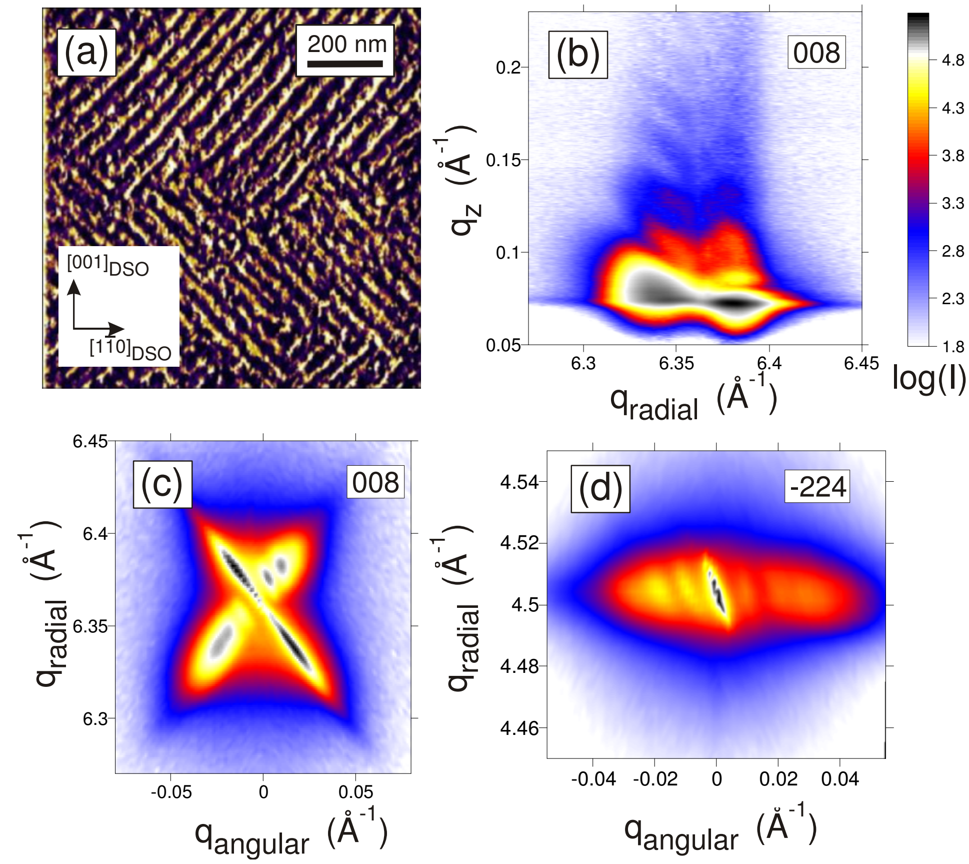

Fig.1: (a) lateral PFM image (phase) of a 25 nm $(K,Na)NbO_3$ film grown on (110) $DyScO_3$ substrate
(b) radial out-of-plane section close to the 008 reciprocal lattice point of the $DyScO_3$ substrate
(c) in-plane section measured close to the 008 reciprocal lattice point of the $DyScO_3$ substrate
(d) in-plane section measured close to the -224 reciprocal lattice point of the $DyScO_3$ substrate

In the in-plane intensity distribution the two types of domains show up as a cross with about 90° opening angle (Fig.1(c)). The observed intensity distribution is caused by a superposition of (i) monoclinic distortion of the in-plane unit cell and (ii) positional correlation of the periodic domain pattern. The disentanglement of both effects is not trivial and requires the investigation of many Bragg reflections. For example, we can have a look at the -224 reflection. Here, the scattering vector is collinear with the alignment of the +45° domain type, while it is perpendicular to the alignment of the -45° domain type. Consequently it is sensitive to the monoclinic distortion of only one type of domains. For that reason, the corresponding in-plane intensity distribution displayed in Fig.1(d) shows only one branch of the cross observed in Fig.1(c). This means that the monoclinic angles and lateral period of both domain types can be measured independently. From all of these measurements a mean value for the lateral period of about 60 nm can be estimated, which is in good agreement with the PFM image (Fig.1(a)), while the in-plane monoclinic angle amounts to about $\beta = 0.30°$.

However, the disturbed symmetry of the intensity pattern in Fig.1(c) indicates that the two types (± 45°) of domains are not equivalent. The reason for this asymmetry is the slightly different monoclinic distortions β of the in-plane unit cells for the +45° and -45° variants.

Also the radial out-of-plane section (Fig.1(b)) shows interesting features: the vertical rods exhibit intensity fringes which are inclined indicating that the 'vertical' domain walls of the stripe domains are also tilted from the surface normal.

Conclusion

The measurements delivered a rich variety of information. The data evaluation is expensive and still in progress.