EUROPEAN SYNCHROTRON RADIATION FACILITY

INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

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:** Grazing Incidence X-Ray Diffraction from Ferroelectric Domains in $KNbO_3$ Films Grown on $NdScO_3$	**Experiment number**: **MA-2604**
Beamline: BM 20	**Date of experiment**: from: 11 March 2015 to: 16 March 2015	**Date of report**: 27 August 2015
Shifts: 15	**Local contact(s)**: Valentina Cantelli	*Received at ESRF:*

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Report:

In the framework of the experiment MA-2604 we have investigated the piezoelectric domain structure of $K_{0.90}Na_{0.10}NbO_3$ thin films grown on (110) $NdScO_3$ substrate by grazing incidence x-ray diffraction. The experiment has been performed at BM 020 station at an x-ray energy of 11.5 keV which corresponds to a wavelength of $\lambda = 1.0781$ Å. A one dimensional linear detector has been used and oriented parallel to the sample surface. A small pinhole was placed just after the sample, allowing a fast two-dimensional in-plane reciprocal space mapping by a single rocking scan of the sample. This multi-detection technique ensures fast data acquisition with good counting statistics within a reasonable time and sufficient angular resolution. A more detailed description of the technique can be found in [1]. The angle of incidence was chosen just above the critical angle of total reflection ensuring maximum sensitivity to the film.

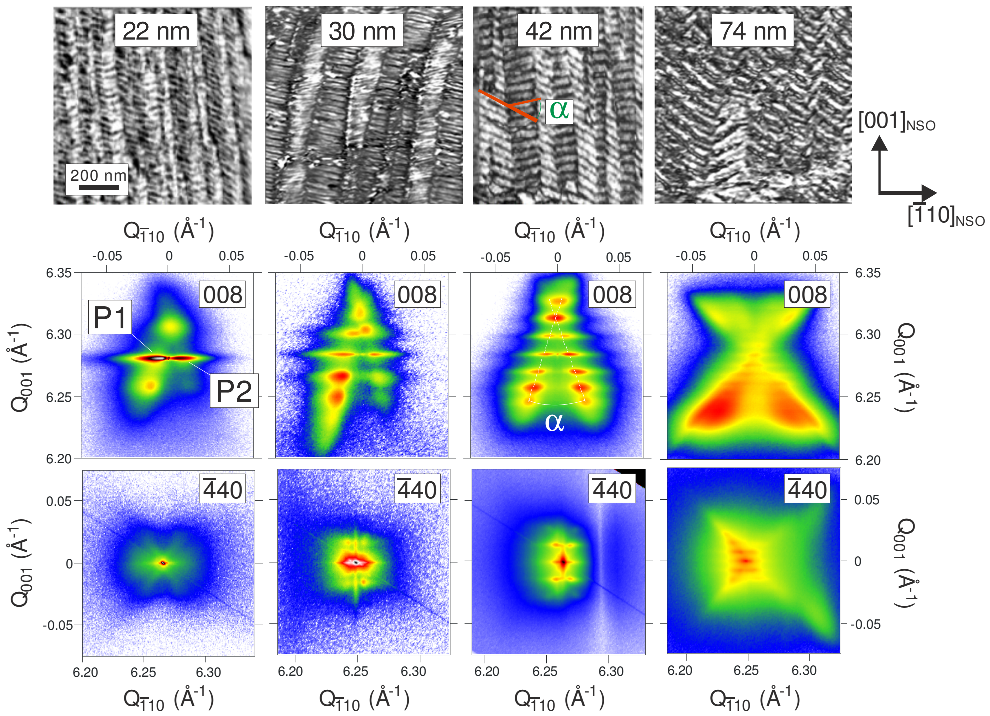

Fig.1: **Top**: Lateral PFM images (amplitude) of $K_{0.90}Na_{0.10}NbO_3$ thin films grown on (110) $NdScO_3$ substrate for four different film thicknesses (22 nm, 30 nm, 42 nm, 74 nm). **Bottom:** Corresponding in-plane intensity distribution in the vicinity of the 008 and -440 $NdScO_3$ reciprocal lattice points.

Our experiment was quite successful, and we were able to investigate six samples with different film thicknesses ranging from 22 nm up to 74 nm. For each sample in-plane reciprocal space maps were measured in the vicinity of five different reciprocal lattice points of the $NdScO_3$ substrate (-220, -440, 004, 008, 2-2-4). From these measurements the in-plane monoclinic distortion angle β, the domain wall angle α, the domain periodicity, and the strain state could be investigated as a function of film thickness.

In Fig.1 a selected choice of the measurements is shown along with corresponding lateral piezoresponse micrographs displaying the domain pattern of the $K_{0.90}Na_{0.10}NbO_3$ thin films. The domain pattern – symmetry, domain sizes, periodicity, and degree of ordering – changes as a function of the film thickness. The results can be summarized as follows:

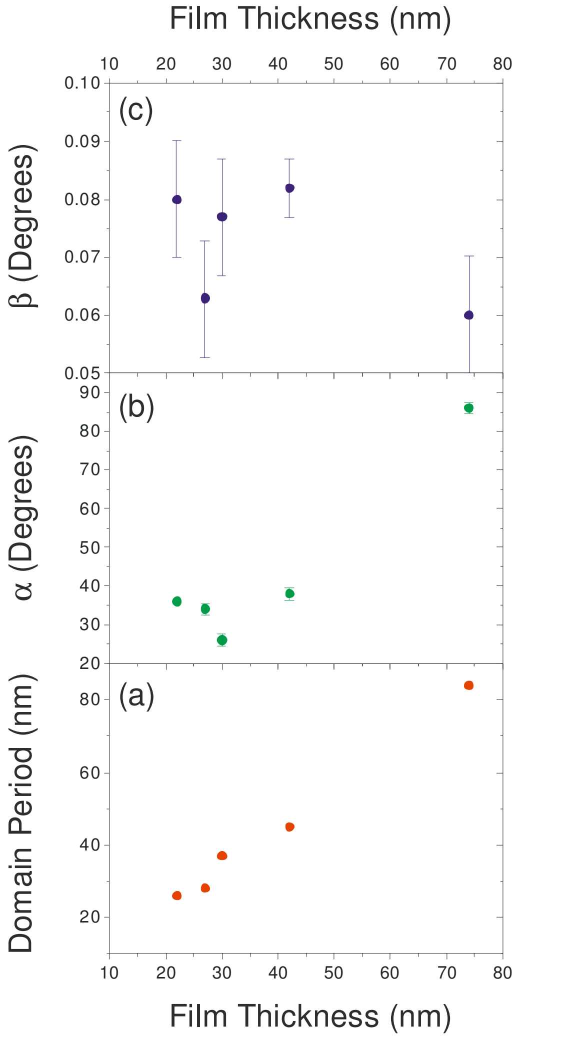

Fig.2: (a) Lateral domain period along $[001]_{NSO}$, (b) domain wall angle α, and (c) in-plane monoclinic distortion angle β as a function of the film thickness.

(i) For small thicknesses the domain period along the $[001]_{NSO}$ substrate direction is small and continuously increases as a function of film thickness (Fig.2a). Here PFM data and x-ray data are in full agreement. Moreover, the degree of ordering of the domain pattern is also enhanced at larger thicknesses. This is reflected in the corresponding x-ray data, where the number of observed satellite peaks is largely increased when going from 22 nm to 74 nm film thickness (See Fig.1 bottom)

(ii) The domain wall angle α (please see Fig.1 for the definition of α) between adjacent super-domains exhibits a more complicated behavior. It shows a minimum at about 30 nm film thickness (see Fig.2b).

(iii) While the morphological parameters (domain wall angle α, domain period, and domain ordering) vary with film thickness, the monoclinic angle β seems to remain constant in the observed thickness range (Fig.2c). Obviously, the monoclinic angle β in $K_xNa_{1-x}NbO_3$ thin films depends solely on the Potassium content x. This is not fully understood and seems to be in contradiction with the model given by Bokov and Ye [2] where the monoclinic angle should vary as a function of strain and domain wall angle α. This question will be further investigated in the future (a proposal for ESRF has been prepared)

(iv) Although satellite peaks – reflecting the lateral domain periodicity – are present in the -440 reflections these do not show a horizontal peak splitting (into P1 and P2) caused by an in-plane monoclinic distortion of the unit cell. This proves that the lattice remains coherent at the domain walls running along $[001]_{NSO}$.

References:

[1] M. Schmidbauer, P. Schäfer, S. Besedin, D. Grigoriev , R. Köhler, and M. Hanke, Journal of Synchrotron Radiation **15**, 549 (2008).

[2] A.A. Bokov and Z.-G. Ye, J. Appl. Phys. **95**, 6347 (2004).