EUROPEAN SYNCHROTRON RADIATION FACILITY

INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

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*

Deadlines for submission of Experimental Reports

Experimental reports must be submitted within the period of 3 months after the end of the experiment.

Experiment Report supporting a new proposal ("relevant report")

If you are submitting a proposal for a new project, or to continue a project for which you have previously been allocated beam time, you must submit a report on each of your previous measurement(s):

- even on those carried out close to the proposal submission deadline (it can be a *"preliminary report")*,
- even for experiments whose scientific area is different form the scientific area of the new proposal,
- carried out on CRG beamlines.

You must then register the report(s) as "relevant report(s)" in the new application form for beam time.

Deadlines for submitting a report supporting a new proposal

- 1st March Proposal Round - **5th March**
- 10th September Proposal Round - **13th September**

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.

Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report in English.
- include the experiment number 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:** Spatially resolved nanodiffraction of quantum dot based solar cells: The role of Sb in the capping layer	**Experiment number**: MA-5580
Beamline: ID01	**Date of experiment**: from: 25/04/2023 to: 29/04/2023	**Date of report**: 03/07/23
Shifts: 12	**Local contact(s)**: Edoardo Zatterin	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Jaime Dolado[1]*, Fernánda Malato[2]*, Malte Schwarz[3] and Gema Martínez-Criado[1,2]

ESRF, European Synchrotron Radiation Facility, 38043 Grenoble, France; [2] ICMM Instituto de Ciencia de Materiales de Madrid Cantoblanco; [3] ISOM and Departamento Ingeniería Electrónica, ETSI Telecomunicación, Universidad Politécnica de Madrid, 28040 Madrid, Spain.

Report:

Here, we report the nanodiffraction study of PbTe films. The main topic of the proposal was to study the effect of Sb in InAs/GaAsSb quantum dot systems. The short preparation time since the acceptance of the beamtime (one month) meant that we only had one sample for the experiment. The contacts of the prepared sample loosened during the trip, and we were unable to contact them properly again (see Figure 1). Since the proposed measurements could not be carried out, it was decided to study the PbTe films.

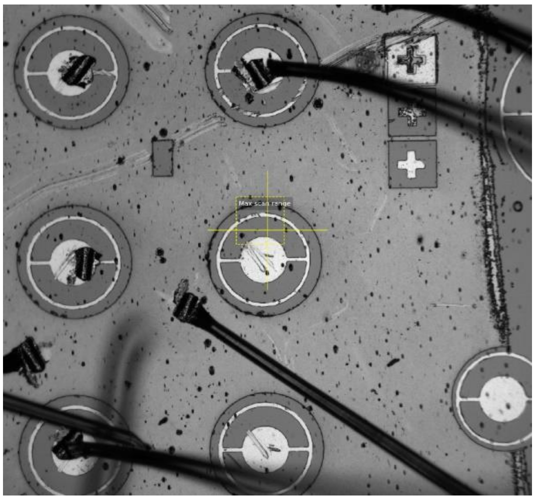

Figure 1: Optical microscope image of the InAs/GaAsSb sample, where it can be seen that the contacts are detached.

Heteroepitaxy enables the engineering of novel properties, which do not exist in a single material. Two principal growth modes are identified for material combinations with a large lattice mismatch, Volmer-Weber, and Stranski-Krastanov. Both lead to the formation of three-dimensional islands, hampering the growth of flat defect-free thin films. This limits the number of viable material combinations. Here, we study PbTe films obtained from a different growth method, based on molecular beam epitaxy of PbTe on InP initiated by pregrowth surface treatments[1]. Early nucleation forms islands analogous to the Volmer-Weber growth mode, but film closure exhibits a flat surface with atomic terracing. Remarkably, despite multiple distinct crystal orientations found in the initial islands, the final film is single crystalline. This is possible due to a reorientation process occurring during island coalescence, facilitating high quality heteroepitaxy despite the large lattice mismatch, difference in crystal structures, and diverging thermal expansion coefficients of PbTe and InP. This growth mode offers a new strategy for the heteroepitaxy of dissimilar materials and expands the realm of possible material combinations.

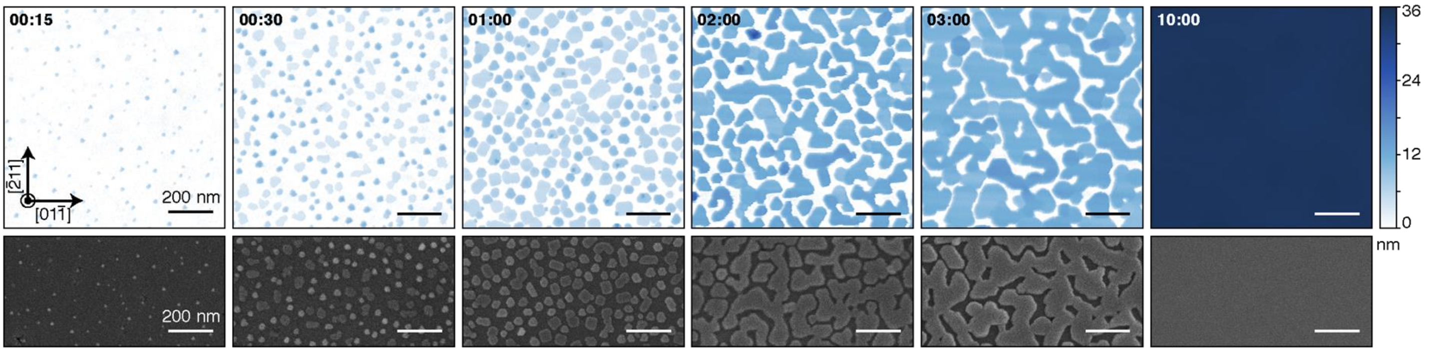

Figure 1: PbTe layer formation. (a) AFM scans depict the growth stages from initial island formation, to their coalescence, and the development of a closed layer. The in-plane crystal directions of the InP substrate are indicated in the first panel and kept consistent throughout. (b) SEM micrographs of the same samples.[1]

PbTe films were studied at the nanodiffraction beamline ID01 at the ESRF. By scanning X-ray diffraction microscopy (SXDM) technique, the position and shape of different Bragg reflections are recorded along the PbTe film, allowing to obtain laterally resolved spatial maps of the three components of the scattering vector Q. The displacements in the scattering vector |Q| provide local changes in the interplanar distance (see Figure 2a-c) and lattice rotation (see Figures 2d-f) measured in each sample position.

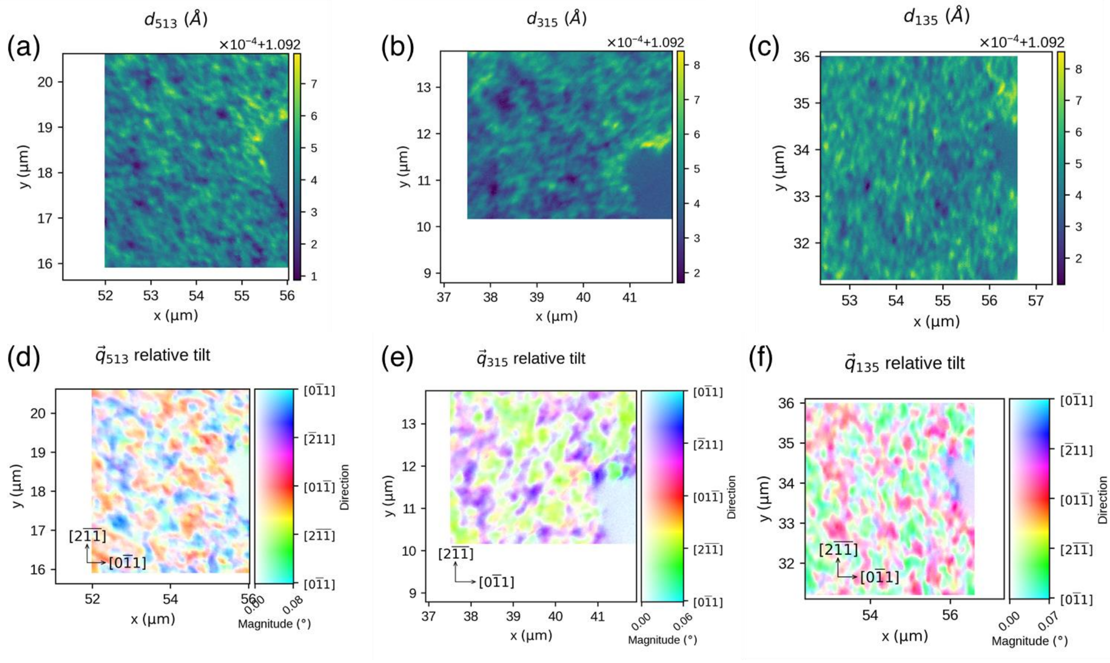

Figure 2: SXDM maps for 513, 315 and 135 Bragg peaks on the PbTe film: (a)-(c) Variation of the d-spacing. (d)-(f) Magnitude and direction of the relative tilt of Q vector with respect to different crystallographic orientations.

Once three diffraction maps have been acquired for three different asymmetric Bragg reflections, it is possible to calculate the six lattice parameters (a, b, c, α, β, γ). Figure 3 shows the local values of the lattice parameters along the InSb network, providing the lattice distortions relative to the PbTe lattice:

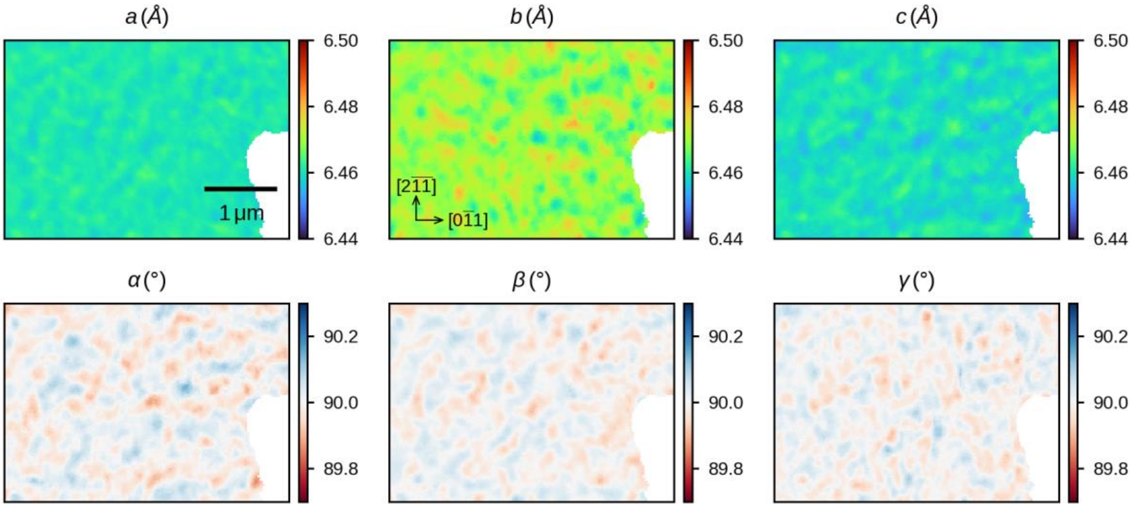

Figure 3: Local values of the six lattice parameters (*a, b, c, α, β, γ*) along the PbTe layer.

To calculate the strain tensor, we define an orthogonal reference system setting $\hat{x}$ and $\hat{y}$ along the $[0\bar{1}1]$ and $[2\bar{1}\bar{1}]$ crystallographic axes (in-plane directions) respectively. Thus, $\hat{z}$ is parallel to the [111] axis (out-of-plane direction). Figure 4 shows the maps of all six strain tensor components in the PbTe film, derived from SXDM data:

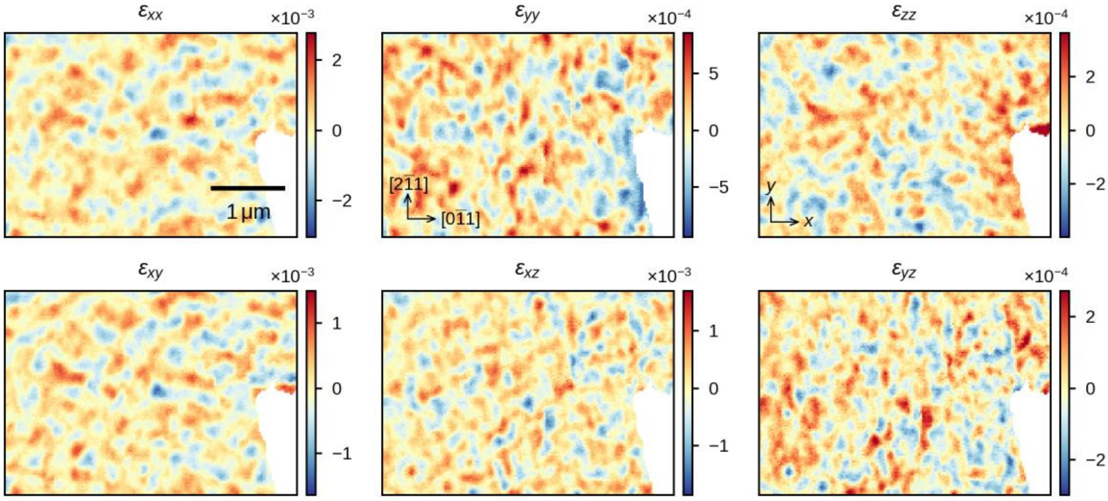

Figure 4: Maps of all six strain tensor components along the PbTe layer.

In addition, Figure 5 shows the maps of the associated small lattice rotations, where small-angle misalignment are detected:

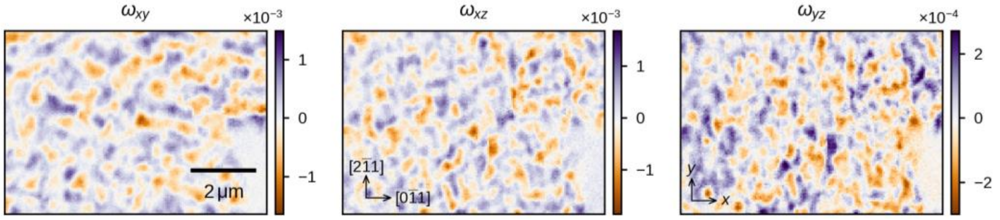

Figure 5: Maps of the three lattice rotation tensor components along the PbTe layer.

Finally, similar measurements were attempted on PbTe nanoestructures grown in the same way as the previous PbTe films. However, after several hours of exposure to the X-ray nanobeam, the sample began to degrade, until it stopped at a certain point (see Figure 6). This behaviour may be due to an oxidation process of the sample induced by the X-ray beam. The experiment should be repeated with an N_2 atmosphere to remove the O_2 on the surface of the sample.

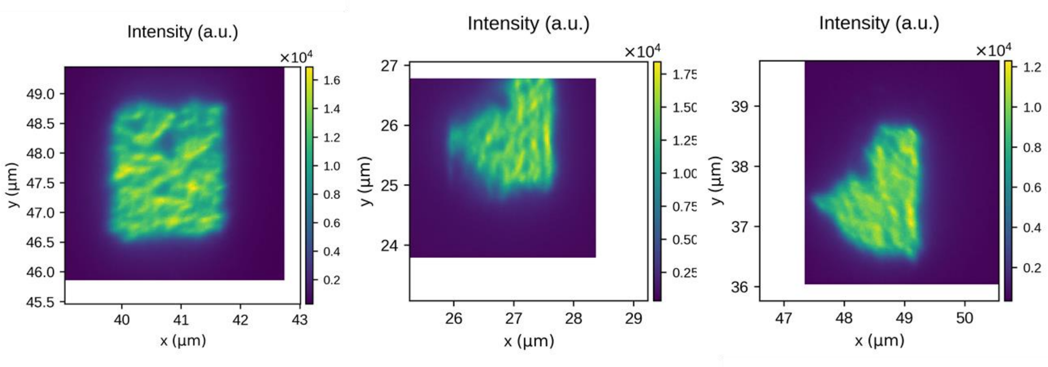

Figure 5: SXDM scattered intensity map, where the deterioration of the sample after exposure to the X-ray beam can be seen.

Therefore, SXDM offers a non-destructive means to study the strain landscape in epitaxial material systems, in particular ultrathin strained layers. By superimposing the strain and rotation fields over the PbTe films, it is possible to gain knowledge about local distortions. These results highlight the reorientation process that occurs in these samples during the growth stage, facilitating the formation of a predominantly single-crystalline film. Future work will explore the heteroepitaxial growth of topological crystalline insulators, namely, SnTe and PbSnTe that are expected to exhibit comparable growth behavior.

References

[1] Jung, Jason, et al. "Single-crystalline PbTe film growth through reorientation." *Physical Review Materials* 7.2 (2023): 023401.