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:** 2D chalcogenides for valleytronics grown E-ALD: an operando SXRD investigation	**Experiment number**: MA-3340
Beamline: ID03	**Date of experiment**: from: *12/07/2017* to: *18/07/2017*	**Date of report**:
Shifts: 18	**Local contact(s)**: Raja Znaiguia	*Received at ESRF:*

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Report:

Aims

The aim of the present proposal was the in-operando characterization of the growth process and structure of $MoSe_2$ 2D layers and thin films obtained by means of Electrochemical Atomic Layer Deposition (E-ALD). The present experiment represents the first attempt to characterise such systems grown by E-ALD. The structural analysis of the films has been carried in-situ by Surface X-ray Diffraction and X-ray reflectivity.

Methods (1): experimental set up

The experiment was performed in the hutch EH1 of the ID03 beamline, using the six circle diffractometer equipped with the ID03 electrochemical flow cell setup (represented in figure 1), already used in our previous experiments MA-2082, MA-2251, MA-2636 and MA-3071.

The experimental set up included the Maxipix detector mounted on the diffractometer arm and a Pilatus 300k-w detector used for fast acquisition of in-plane powder diffraction pattern (covering a 2θ range between 10 and 20 with one single images at the energy of 24 KeV) and the XIA detector (for XRF) from the instrument pool.

Methods (2): samples

Different kind of samples were considered in this operando experiment . Samples were realised according to the E-ALD procedure, i.e. alternating the underpotential depositions of the considered elements, assembling the 2D structure and the thin films. At the end of the experiment an attempt of codeposition of Se and Mo from a MoO_4^- and SeO_3^- both 10 mM at selected potential on Ag(111).

Methods (3): in situ measurements

In situ measurements were also realised in the electrochemical flow cell setup, located in the hutch, with the flow cell mounted on the diffractometer.

In particular, we performed:
- l and (h, k) scans on two different Bragg reflections of the CdS phase (every 10 E-ALD cycles of the n layer)
- reciprocal space maps after the growth
- X-ray diffraction maps at several relevant l quotes of the obtained material
- XRF spectrum every cycle of the growth

Preliminary Results
The XRF, acquired during the growth, clarified that while the amount of Mo grows during the deposition cycles, the amount of Se grows only during the first cycle. The reciprocal space maps report only powder pattern probably due to a polycrystalline molybdenum oxyde.

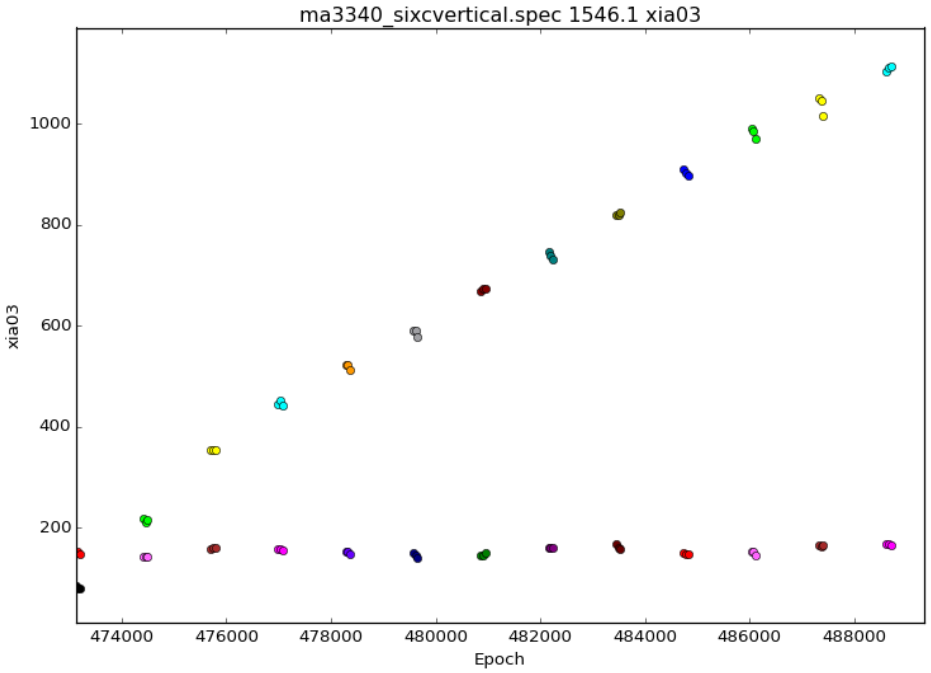

Figure 1: XRF signal integrated at selected ROI acquired every cycle.

During the last shift of the experiment we performed a potentiostatic treatment of a solution of MoO_4^- and SeO_3^- both 10 mM at selected potential on Ag(111) electrode. The reciprocal space maps present a Bragg's peaks pattern compatible with the metric of MoSe. Such epitaxial growth, obtained with a one step potentiostatic electrodeposition process, is an important result but require more study to understand the structure of the film and the growth process.

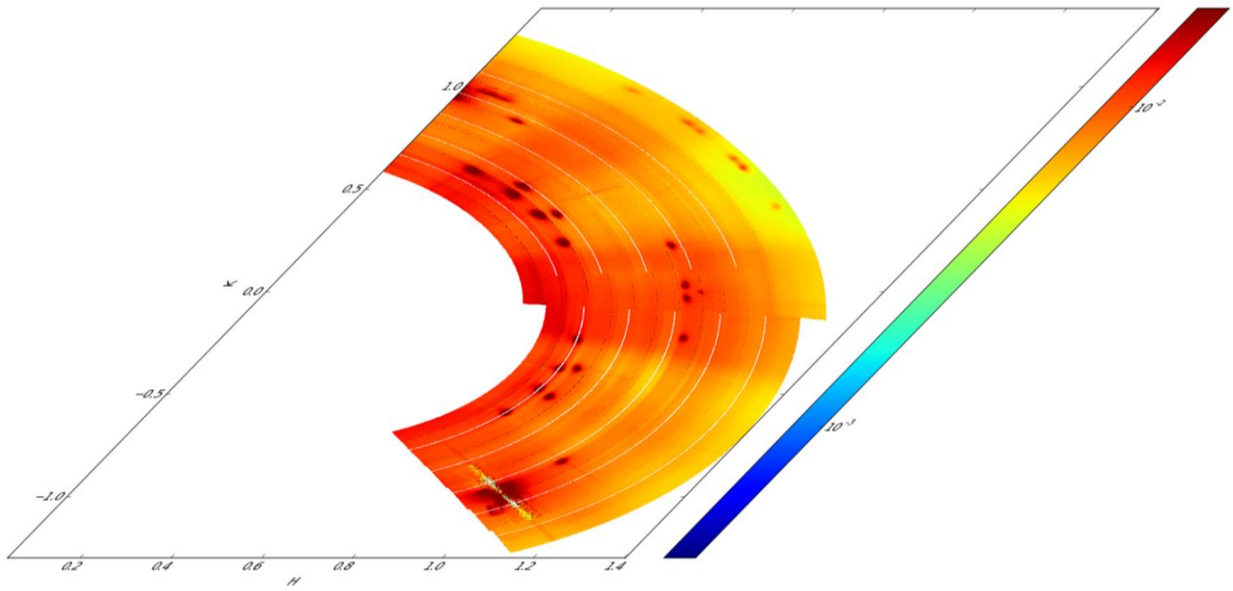

Figure 2: reciprocal space acquired at l=1.8 during the last shift after potentiostatic deposition of $MoSe_x$.