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:** *In-situ SXRD characterization of CdS/Cu₂S and Cu₂S/CdS thin films for photovoltaics application*	**Experiment number**: MA 3071
Beamline: ID03	**Date of experiment**: from: *22/02/2017* to: *28/02/2017*	**Date of report**:
Shifts: 18	**Local contact(s)**: Francesco Carlà	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

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Report:

Aims

The aim of the present proposal was the in-operando characterization of the growth process and structure of p-n junctions of thin films semiconductors obtained by means of Electrochemical Atomic Layer Deposition (E-ALD). The present experiment represents the first attempt to characterise a p-n junction grown by E-ALD. The structural analysis of the films has been carried out both ex situ and 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 and MA-2636.

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).

Methods (2): samples

Two different kind of samples were considered in this operando experiment: CdS over Cu_2S (hereafter labelled as CdS/CuS) and Cu_2S over CdS (hereafter labelled as CuS/CdS). Samples were realised according to the E-ALD procedure, i.e. alternating the underpotential depositions of the considered elements, assembling layer-by-layer the junction. Equal number of deposition cycles (60) were used for both the p and n layers.

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. As in our previous experiments we got an exhaustive characterisation of the p layer (Cu_2S), we deposited this layer without intermediate

characterisation. After the 60 cycles of deposition were realised, we performed a fast characterisation to verify the quality of the layer, and we moved to the deposition of the n layer, alternating synthesis and characterisation. In particular, we performed:

- XRR before starting the E-ALD of the p layer
- XRR after ending the E-ALD of the p layer
- l and (h, k) scans on two different Bragg reflections of the CuS phase (every 10 E-ALD cycles of the n layer)
- l and (h, k) scans on two different Bragg reflections of the CdS phase (every 10 E-ALD cycles of the n layer)
- XRR afte ending the E-ALD of the n layer
- X-ray diffraction maps at several relevant l quotes of the obtained junction.

Preliminary Results

Several problems occurs during the experiment: at lest 5h of beam time has been lost due to beam failure and some instability of the detector limited the exploration to the structure of the CuS/CdS. However, important data have been gather on the sample up to 60 cycles of CdS and 30 of Cu_2S (due to lack of solution in one of the reservoir). The data are very important for the study of this p-n system, showing that Cu_2S grows steadily without changing the structure of the CdS/Ag(111) substrate. On the basis of this data, it will be possible to characterize the growth of this layered system, since there is no interference between the growth of CdS and Cu_2S from a structural standpoint the system has the features of a p-n junction. This would be the first p-n junction grown by means of E-ALD.