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 using the **Electronic Report Submission Application:**

http://193.49.43.2:8080/smis/servlet/UserUtils?start

Reports supporting requests for additional beam time

Reports can now 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:** Crystallographic study of ferromagnetic semiconductor multilayers based on tin oxide	**Experiment number**: HS-3455
Beamline: BM25B	**Date of experiment**: from: 2 September 2007 to: 11 September 2007	**Date of report**: *Received at ESRF:*
Shifts: 12	**Local contact(s)**: Dr. Juan RUBIO-ZUAZO	

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Report:

Diluted magnetic semiconducting oxides (DMS) have been intensively studied in last years because of their potential use in spintronics devices. Wide band gap oxides (as ZnO, TiO_2 and SnO_2) doped with 3d ions are the most promising candidates since recent experimental results revealed ferromagnetism (FM) above room temperature. However, the origin of the FM is still under debate and the formation of metastable phases at grain boundaries or interfaces have also been demonstrated to occur experimentally. In this frame, where defects and interfaces play a fundamental role in the magnetic interactions, we have investigated two systems based on Mn and SnO_2: i)Mn/SnO_2 multilayers and ii)(Mn,In)-doped SnO_2.

The multilayers under study were Mn/SnO_2 multilayers (2 nm SnO_2/ x nm Mn)$_N$ deposited on Si(100) and R-cut sapphire by magnetron sputtering. The nominal Mn layer thickness ranged from 0.4 up to 6 nm. Some of them were annealed in air. As-grown films presented a FM phase with Tc>300K while annealed samples presented no significant FM at T>50 K. [1]

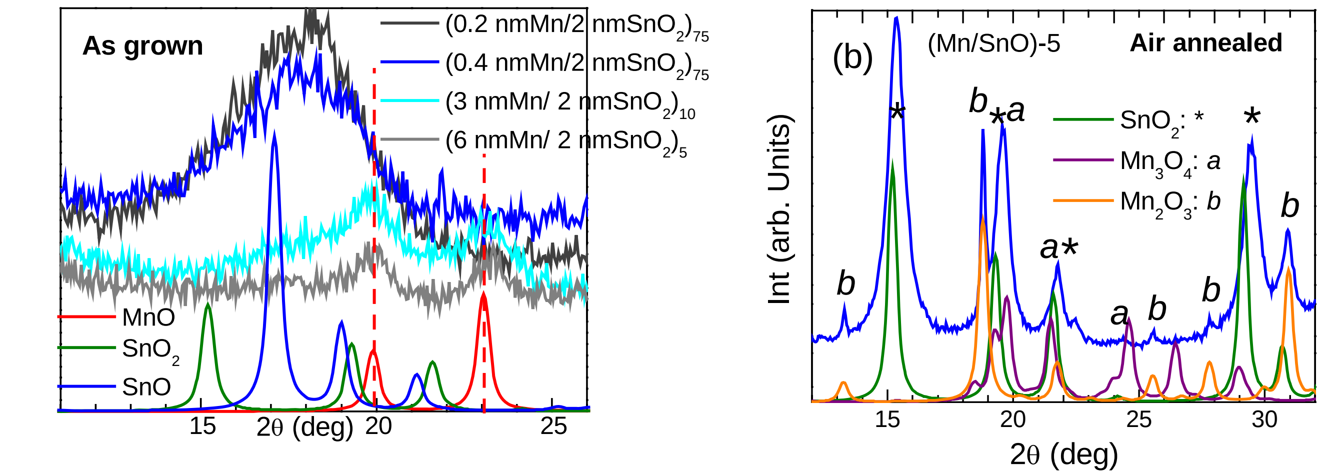
5%(Mn,In)-doped SnO_2 films were deposited also on Si(100) and Al_2O_3 (R-cut) by RF magnetron sputtering at different deposition power, sputtering gas mixture and substrate temperature (room temperature and 550ºC). [2]

According to the proposal project, we performed high-angle X-ray diffraction measurements with a six-circle diffractometer available at the beamline. Grazing incidence geometry (0.75 deg) was used in order to enhance the thin film contribution and avoid the very strong peaks from the substrates. The energy selected for a accurate angle resolution was 14 KeV (λ = 0.8857 Å). We also performed reflectivity measurements in order to obtain the total and bilayer thicknesses values as well as the quality of the interfaces (rugosity and interdiffusion). All measurements will be carried out at ambient pressure and at room temperature.

i) <u>Mn/SnO_2 multilayers</u>

The first obtained information is that as-grown multilayers contain basically SnO and MnO nanocrystals and slightest suspicion of amorphous SnO_2. So, the nominal SnO_2 layers are formed by nanocrystals of SnO_{1+x}. The diffraction pattern of an as-grown thick (200nm) SnO_2 film grown in the same conditions as the multilayers shows the same features except the Mn contributions. The presence of MnO, i.e Mn with valence +2 is quite consistent with X-ray absorption spectroscopy (XAS) measurements at Mn K-edge energy performed in a previous ESRF experiment.

On the other hand, annealed films presented polycrystalline SnO_2 and MnO transforms to Mn_2O_3 and Mn_3O_4 oxides, which is self consistent with its magnetic behaviour and XAS measurements.

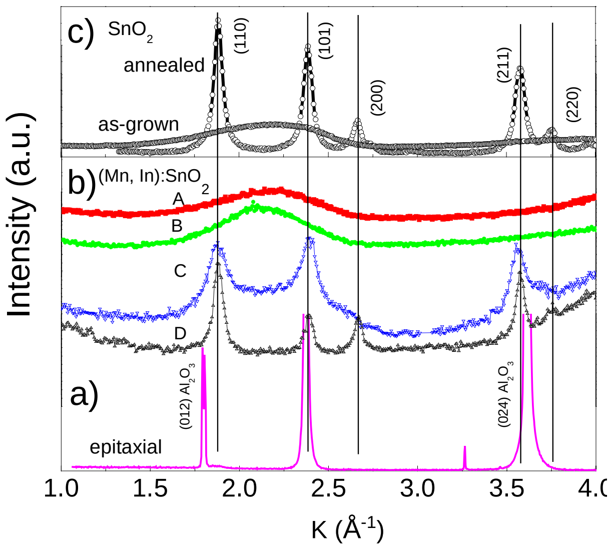


[1] A. Espinosa, E. Céspedes, C. Prieto, M. García-Hernández, J. Rubio-Zuazo, and A. de Andrés. J. Appl. Phys. 103, 07D129 (2008).

ii) <u>(Mn,In)-doped SnO_2 films</u>

In case of doped SnO_2 samples, as-grown films obtained at low power and temperature were amorphous while annealed films revealed crystalline SnO_2. As the deposition power, substrate temperature or O_2 proportion are increased, SnO_2 nanocrystals are formed. Epitaxial SnO2 films were obtained on Al_2O_3 at 550ºC. No secondary phases or segregation of dopants were detected

Due to the amorphous nature of most of the samples which are correlated with a FM phase, this structural characterization has been possible thanks to a grazing incidence configuration and a tuned selected energy that provides an accurate angle resolution for this experiment.



[2] A. Espinosa, N. Menéndez, J. Rubio-Zuazo, C. Prieto and A. de Andrés. J. Non-Cryst. Solids (accepted)