ESRF	**Experiment title: Characterization by X-ray radiography and topography of the solidification of multi-crystalline silicon for photovoltaic applications**	**Experiment number**: MA-1274
Beamline: BM05	**Date of experiment**: from: 22/06/2011 to: 28/06/2011	**Date of report**: 09/03/2012
Shifts: 18	**Local contact(s)**: Tamzin LAFFORD	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

TANDJAOUI Amina*, MANGELINCK-NOEL Nathalie*, REINHART Guillaume*, NGUYEN-THI Henri *, FURTER Jean-Jacques*, *IM2NP, UMR CNRS 7334, Campus Saint-Jérôme, Case 142, 13397 Marseille Cedex 20, France*
LAFFORD Tamzin*, GUICHARD Xavier*, *ESRF*

Scientific Objectives:

The general scientific objectives of our experiments are to provide benchmark data concerning the solidification of multi-crystalline Si (mc-Si) for photovoltaic (PV) applications. These original experiments consist in synchrotron X-ray imaging characterisation of the mc-Si during its solidification.

These experiments and proposal are part of a larger project entitled Si-X (Characterisation and understanding of the crystallisation of photovoltaic **Si**licon: **X**-ray synchrotron imaging) which is funded by the HABISOL (Habitat Intelligent et Solaire Photovoltaïque) program of the French National funding agency (ANR). ESRF is a full partner of this project.

During MA-1138 campaign, the device for X-ray radiography was validated and the experimental parameters set for the multicrystalline silicon material. During MA-1274 campaign, we improved our invsetigation means by using alternately X-ray radiography and topography during the same solidification experiment. Additionnaly, one of the main objectives of MA-1274 was to acheive the solidification of three different silicon grades showing differing levels of impurity: Czochralski (Cz), Metallurgical Grade (MG) and Solar Grade (SoG).

Experimental method:

The experiments were performed on the beamline BM05 achieving X-ray radiography and topography alternately. We aimed to perfom the solidification experiments by two ways: decreasing the temperature of the two heaters or by pulling the sample from the hot zone to the cold one. The crucibles used in this campaign are made of pyrolitic boron nitride and their housing for the sample have a surface of 40 x 6 mm^2 and a depth of 300µm. The samples dimensions have been modified in comparison to those from MA-1138 and have a final surface of 38 x 5.6 mm^2 and a thickness of 300µm to avoid capillary effect at the housing edges.

From the X-ray imaging point of view, contrarily to experiments performed during MA-1138 using monochromatic beam, in these experiments we used a white beam to be able to perform topography. In radiography mode, the transmitted beam is monochromatised (17.5 keV) by the post-specimen monochromator made of two Si crystals (111) and situated after the topography device (see Figure 1). To perform topography during one experiment, we cut the transmitted direct beam using a beam-stop and collect the diffracted beams on the topographs. Topographs are obtained by collecting the diffracted beams on AGFA structurix D3-SC X-ray sensitive films.

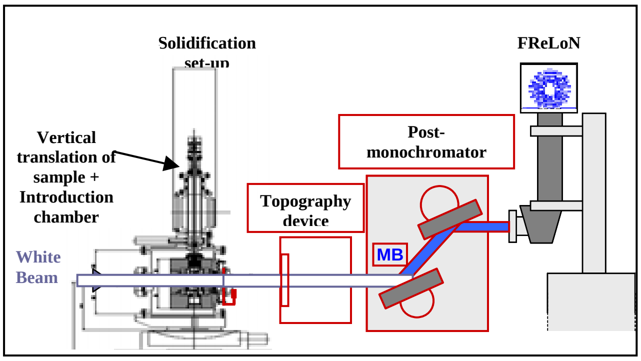

Fig. 1: Schematic drawing of the installation of the experimental device for both X-ray radiography and topography in radiography mode.

IM2NP developed an additional device on top of the specific solidification furnace to achieve X-ray topography characterization. This device allows to expose 30 topographs during one single solidification experiment without stopping the X-ray beam with a very good reliability in terms of positioning of the topographs. Using X-ray topography, we obtain information on the growth of isolated grains, strains, defects and grain orientations.

Results:

Figure 2 shows a topographic film taken during the solidification of MG-Si under the conditions mentionned in the caption. The yellow rectangles indicate the reflexion of the same grain at different positions on the film for its different crystallographic planes. When zooming on one of the reflexions we could observe the equalthickness fringes, called “Pendellosüng” fringes which indicate that this zone of the grain is almost perfect and contain less than 10 Cm/Cm^3 of dislocations.

Thanks to the device allowing to record 30 topographic films during one experiment, we can follow the growth of the grains during all the solidification experiment (See Fig. 3.c) and even build topograph moovies.

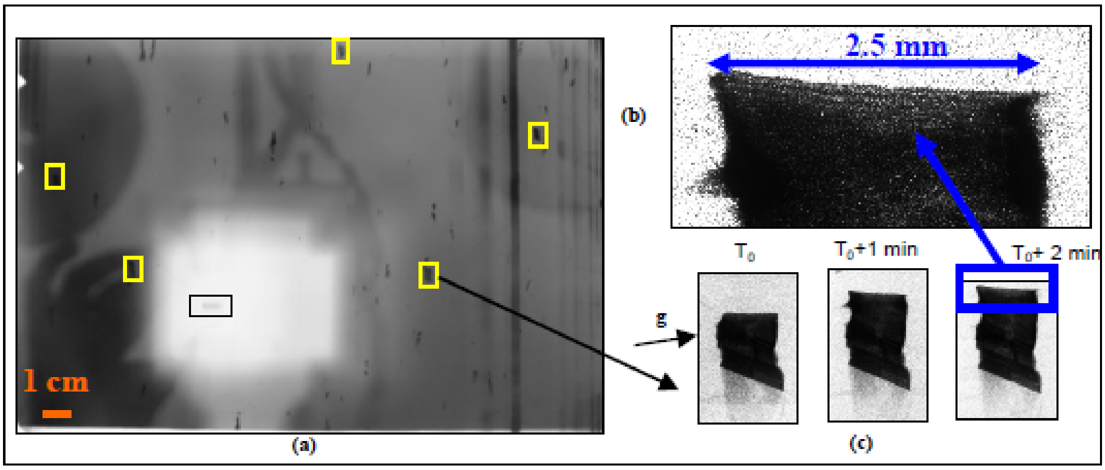

Fig. 3: X-ray topograph of MG-Si under G = 12.5 K/cm, R = 0.5 K/min a) Topographic film with reflections of the same grain indicated by yellow rectangles, b) Pendellosüng fringes in the protruding zone and, (c) Time sequence of one reflection of the grain growing during solidification. “g” is the diffraction vector.

Future work

The MA-1274 campaign allowed to provide benchmark data concerning the mc-Si solidification. We were able to study silicon dendrites, facetted and rough growth, grain competition, groove formation, twin dynamics. For example, the occurrence of twins, their growth and arrangments was observed and characterised using both complementary X-ray imaging methods (radiography and topography). These original results are under further analysis and we are in the process of writing publications on them. They also showed the need for further investigations by varying the processing parameters or by exploring new silicon grades. Indeed, in our experiments, there is a demonstrated effect of the impurities shown by the differences observed during the solidification of the silicon grades we used. However, to better understand the origin of the phenomena observed, we need to perform some experiments with controlled impurities.