| ESRF | **Experiment title:** *In situ* **studies of dendritic growth and fragmentation in Ga - In alloys** | **Experiment number**: MA 4075 |
|---|---|---|
| **Beamline**: ID 19 | **Date of experiment**: from: 31.03.2018 to: 03.04.2018 | **Date of report**: 01.03.2019 |
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**Report:**

Radiography as a 2D imaging method provides dynamical data of high time and spatial resolution at a low noise level. However, 2D radiography data are not enough for verification of the existing 3D microstructural models. Tomography, unfortunately still with a much lower time resolution, allows us to obtain 3D spatial information about the morphology and to get very accurate 3D images about the evolution of the dendritic sidearm structure itself.

In this set of experiments, in situ synchrotron tomography and diffraction methods have been combined to study the evolution of dendritic microstructures during the solidification of Ga - In alloys. The study of indium dendrite growth has been of fundamental interest because binary Ga – In alloy is a model system for different solidification phenomena [1-3]. The paper is focused on the correlation of 3D dendritic morphology with crystallographic orientation of indium crystals.

A tomography/ radiography experiment during the alloy solidification process was carried out using a solidification setup tested at HZDR [2, 3]. The nominal composition of the Ga–35 wt. % In alloy was prepared from 99.99% Ga and 99.99% In. The alloy was melted and filled into capillary samples (diameter ~ 1 mm). The capillary sample was cooled down from 60° to 20 °C at a cooling rate of 0.01 K/s. During this stage, the sample was controlled by real-time radiography to control growth of dendrites. Then, cooling was stopped, leading into an isothermal stage that was maintained over a period of 8 hours at a constant temperature of 20 °C. While maintained in a constant temperature, the dendrite structure continues to evolve through isothermal coarsening.

The capillary sample was exposed to a monochromatic X-ray beam with an energy of 70 keV. Absorption-based X-ray tomography was performed using a PCO.edge camera, which was

coupled with ID19’s camera optics offering a field of view (FOV) of 2048 x 2048 pixels and a pixel size of 0.65 µm. One tomogram consisted of 720 projections over 180° with an exposure time of 0.95 s per projection, the total time per tomogram was approx. 18 minutes including data readout, backward movement, dark field and flat field images. The distance between the detector and sample was ~10 cm. For the diffraction experiment a Laue like diffraction geometry was chosen using a monochromatic X-ray beam with an energy of 70 keV that ensures us the heat load by an X-ray beam during the solidification experiments should be much as possible reduced. Diffractograms were recorded using the ESRF FreLoN camera (charge-coupled device CCD-based camera) located 20 cm after the capillary sample. The Laue patterns were recorded at 181 angular positions with step 1° for the rotating capillary. Initial positions of the capillary were the same as during tomography measurements. Each diffractogram was recorded for 5 s. Quantitative analysis of the diffractograms is still in progress.
During an isothermal holding phase, the side-branch structure coarsens continually, resulting in an increase of the secondary dendrite arm spacing and diameter of the primary trunks. The three-dimensional morphology of isolated dendrites after coarsening is shown in Fig. 1 (a). Exemplary of a cross-sectioned slice extracted from tomographic volumes (Fig. 1(b)) indicates four-fold symmetry of sidearms. The angle between the primary trunk and sidearm directions is ~90°, indicating that the primary and side arms are growing in the <100> directions of FCC lattice.

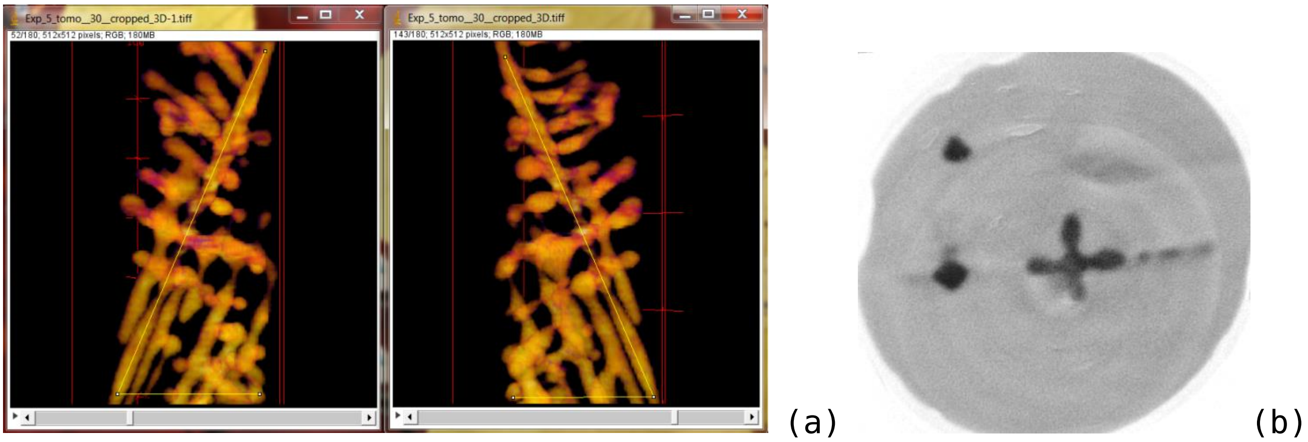


Fig. 1: (a) Three-dimensional observation of indium dendrites in Ga-35 % In alloy after an isothermal holding at a temperature of 20 °C (b) exemplary of a cross-sectioned slice.

The current experiment demonstrates that synchrotron tomography of a thin sample is a highly suitable experimental technique for studying the interface dynamics [4] of dendritic structures in metallic alloys.
This experiment is the first step towards the validation of a more realistic 3D pinching model providing statistically relevant 3D experimental data of the interface dynamics of dendritic structures and of the evolution of individual sidearms.

**References**

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[3] N. Shevchenko, et al. J. Crystal Growth, 417 (2015) 1-8
[4] H. Neumann-Heyme, et al. PHYS. REV. E, 92 060401 (2015)