ESRF	**Experiment title:** *In situ* **studies of dendritic growth and fragmentation in Ga - In alloys**	**Experiment number**: MA 3243
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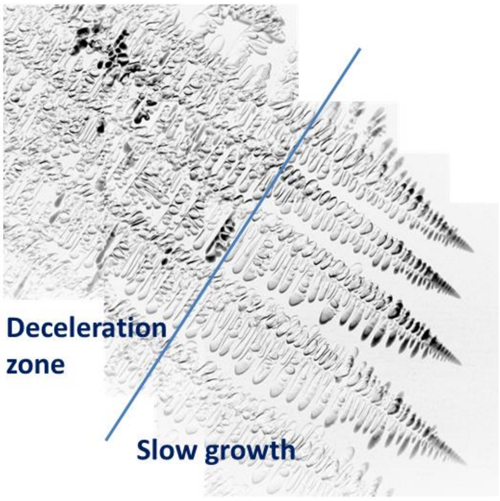
Report:

The detachment of dendrite side arms is an important fragmentation mechanism and is considered as one of the major unresolved questions in the field of solidification. In particular, two questions are discussed intensively in the literature: i) which mechanism governs the detachment of fragments from the dendritic skeleton and ii) how do the fragments reach the region ahead of the solidification front. The first issue has been investigated within our resent experiment. Our previous research was limited to isothermal coarsening [1]. In the present study, the pinching dynamics are investigated at slow cooling conditions.

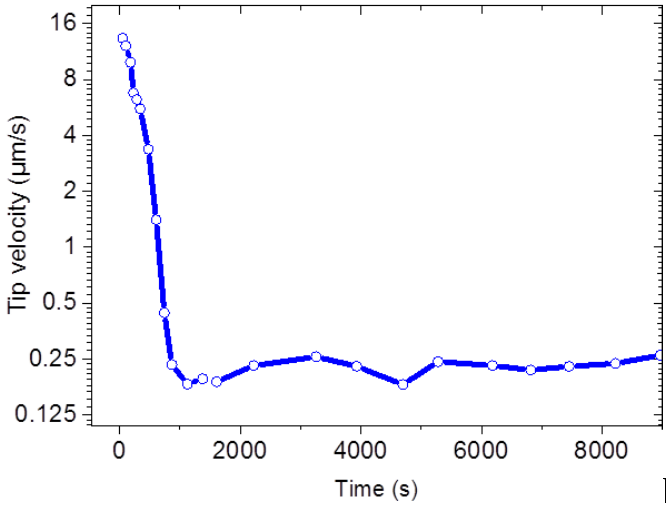
A radiography experiment during the alloy solidification process was carried out using a solidification setup developed at HZDR [2]. The nominal composition of the Ga - X wt%In (where X = 25, 35, 50 wt%) alloys were prepared from 99.99% Ga and 99.99% In. The alloy was melted and filled into a windowed Hele-Shaw cell with a liquid metal volume of 23 × 23 × 0.15 mm^3. During the experiment, a monochromatic beam (40 keV) was used to penetrate the sample, and a high speed CCD camera (PCO Edge) was used to record the images at a spatial resolution of ~1 µm.

The Ga–In alloy was solidified in vertical direction starting from the top of the solidification cell at a controlled cooling rate of 0.002 K/s and at a temperature gradient of ~2 K/mm. Figure 1(a) contains exemplary images illustrating the growth process of dendrites reconstructed from several images along a growth direction. The dendrite tip velocity was calculated by tracking the individual dendrite tips using ImageJ, and then averaging the individual dendrite velocities (Figure 1(b)). The average tip velocity is decreased from 14 µm/s to 0.2 µm/s over a transient period of 1000 second. After that the dendrite tip velocities show small fluctuations near a value of ~0.2 µm/s. In general, all fragmentation events are located in the deceleration zone that is formed during an initial phase of the solidification.

Under slow growth conditions, which are probably close to steady state conditions, it is observed that coarsening in the mushy zone, behind an advancing growth front, does not involve a significant detachment of side arms. The experimental data are promising to quantify morphological transitions such as retraction, coalescence and pinch-off of the side arms in the deceleration zone. Quantitative analysis and image processing are still in progress.



a)



b)

Fig. 1: (a) X-ray radiographs of top-down solidification of the Ga-35 wt% In alloy. (b) Average tip velocity of four chosen dendrites shown on Fig 1(a).

High resolution measurements are very important to study the evolution of the geometry of the growing dendrite tip that can provide the selection constant σ*, which is essential for the characterizing of the dendritic growth stage. The growth process of dendrites is characterized by the dendrite tip radii, the secondary arm spacing, the primary dendrite spacing and the amount of solid fraction (not shown here).

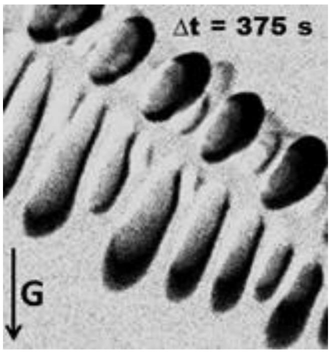


Fig. 2: Zoom of the secondary arms: black areas – new solid; light grey areas – melted zone of arms.

Under the given experimental conditions the high temporal and spatial resolution of the experiment allowed us to identify an additional migration process that is influenced by the existing temperature gradient called a Temperature Gradient Zone Melting (TGZM) process (see Fig. 2). A detailed study of some side arms with respect to its edges revealed a migration rate of 0.01 µm/s. However, the results of our analysis suggest that this process does not play a significant role for the side arm detachment process itself.

In a next step, we will develop a quantitative comparison between the synchrotron data and a numerical model [3] describing the evolution of a single dendrite arm in collaboration with two theoretical groups (TU Dresden & University of Iowa). The expected results have important implication for understanding the curvature-driven coarsening. Using numerical and experimental data we are able to derive fundamental characteristics and limit the pinching instability at a sidearm neck.

References

[1] Neumann-Heyme, Shevchenko et al., Acta Mater. 146 (2018) 176
[2] N. Shevchenko, et al. J. Crystal Growth, 417 (2015) 1-8
[3] H. Neumann-Heyme, et al. PHYS. REV. E, 92 060401 (2015)