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Report:

X-ray absorption contrast techniques are an important diagnostic tool for investigating solidification processes in metallic alloys. This work is devoted to the in situ visualization of the dendritic growth during the bottom-up solidification of a Ga-25wt% In alloy under natural convection. Many effects of melt flow on the mushy zone structure were observed by standard X-ray radiography with a spatial resolution of 5-10 microns [1, 2]. The visualization experiments uncover a complex interaction between dendritic growth and melt convection. Many effects of melt flow on the solidification process can be observed, but, the actual manifestation of all phenomena depends sensitively on the local conditions like the dendrite arm spacing and orientation, a detachment of side branches, the local direction and intensity of the flow [1, 2]. A more detailed analysis of the particular processes requires a better spatial resolution.

Specific phenomena such as the dendrite side-arm development, fragmentation or flowinduced segregation are studied in collaboration with other, theoretical groups and experimental groups. Experimental high-resolution data are very important for the verification of existing microstructural models [3, 4].

We improved our experimental setup (a solidification cell made of Plexiglass and temperature control) for synchrotron experiments. The nominal composition of the Ga–25wt% In alloy is prepared from 99.99% Ga and 99.99% In. The alloy was melted and filled into the Plexiglas Hele-Shaw cell with an area of ~230 x 230 mm² and an inner cell thickness of 150 μ m. The radiography/ diffraction experiments were performed at the beamline BM20 at an energy of 28.5 keV.

The temporal dynamics of morphological transitions such as retraction, coalescence and pinch-off of the side-arms were observed in-situ. Figure 1 shows an example of the evolution of the side-arm shape during a pinch-off process. The narrow neck is formed above the junction between the sidearm and the parent stem. Later, the sidearm pinches off at the neck and the resulting fragment coarsens into a spheroid. The evolution of morphological parameters like the neck radius was quantified by image processing and compared with numerical simulations.



Fig. 1: (a) X-ray radiograph of dendrites showing pinch-off of the side-arms during bottom-up solidification. The red box in (a) indicates the area that is magnified and shown in (b)-(c). (c) The arrow shows the narrow neck above the junction between the side arm and the parent stem. The time between the two images is about 80 minutes.



Fig. 2: Fitting the diffraction pattern.

The coupling of X-ray imaging with X-ray diffraction techniques could provide a reconstruction of a crystallographic orientation of growing dendrites. The image (Fig. 2) shows the results of the fitting procedure using CaRIne crystallography for the diffraction image. On the bottom of the images one can see the orientation of the unit cell (bottom right). The red arrow which is also highlighted on the corresponding first dendritic image indicates the (110) orientation. Identifying the (110) direction is related to the point that according to literature dendrites with body-centered tetragonal unit cell grow exactly along this direction. One can see that the (110) orientation nicely fits to the orientation of the dendrite trunks. The corresponding fitting shows that the reflections on the left-hand side are belonging to one particular orientation of the unit cell shown in the inset of Fig. 2.

Outlook: This experiment demonstrates that in-situ synchrotron X-ray experiments are able to provide benchmark data for the validation and improvement of the numerical models.

References

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