Time-resolved X-ray Imaging of Solidification Phenomena of Metallic Alloys

Observation is the first step for understanding various phenomena. In the field of crystal growth, solidification and casting processes of metallic alloys, it is an important issue to avoid some defects, which are introduced during the processes, to reduce segregation, which is the inhomogeneous distribution of constituent elements, and consequently to improve the quality of products. There are still ambiguities in understanding the mechanisms, since we cannot see what really happens inside the products using conventional microscope systems.

The third generation synchrotron radiation facilities such as SPring-8 enable us to use monochromatized hard X-ray that is attractive for observing the microstructure of various materials. Recently, time-resolved X-ray imaging was successfully performed to observe the crystal growth and solidification of Sn-based and Al-based alloys [1-3]. The observations improve our understanding of the solidification mechanism and help us develop numerical simulation techniques with high reliability.

In our research projects at SPring-8, we focus on the fragmentation of dendrite arms during the solidification of metallic alloys. One of the major defects is the stray crystals that have undesirable crystal orientations. There are two possible mechanisms for the formation of the stray crystals.

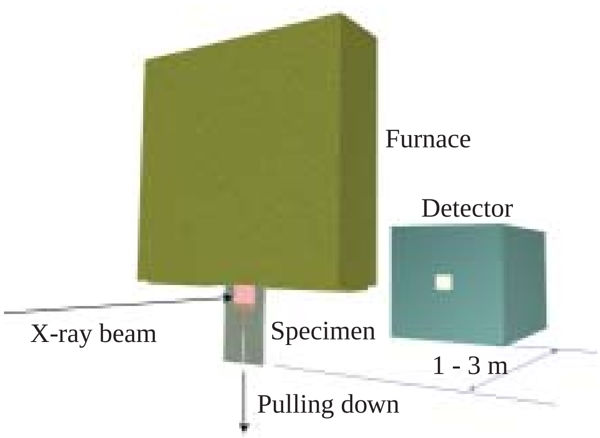

Fig. 1. Configuration of specimen, furnace and X-ray detector for observing solidification of Sn-Bi alloys and Al-Cu alloys.

One is nucleation ahead of the advancing dendrites, and the other is the detachment of fragmented dendrites from the mushy zone. Until now, it is uncertain whether the fragmentation is an origin of the stray crystals or not.

The experiments were performed at beamline **BL20B2**. A large beam size at the experimental hutch (approximately 300 mm in the horizontal direction and 20 mm in the vertical direction) has an advantage for observing rather macroscopic phenomena at a high spatial resolution. Figure 1 shows a schematic illustration of the observation systems. An image detector consisting of an X-ray direct-sensing pickup tube SATICON was used (10 or 4 μm/pixel, 1024 pixels × 1024 pixels and 10-bit resolution). The sample-to-detector distance was 2.5-3.0 m, which gave rise to the phase contrast along with the absorption contrast. The specimen was melted in the furnace and unidirectional solidification was performed by pulling the specimen at a given rate.

It is of interest to investigate the detachment of dendrite arms when the dendrites grow in the enlarged region after passing the re-entrant corner, since the stray crystals were often observed in the enlarged region for turbine blades. Figure 2 shows a sequence of the obtained images (Sn-21mass%Bi alloy, 100 μm in thickness) after the dendrite passed the re-entrant corner. The dendrites immediately covered the enlarged region after passing around the corner (t = 16 s). The detachment of a fragmented dendrite arm was seen at the position indicated by an arrow (33.07 s). The detached crystals grew ahead of the advancing front and consequently the stray crystal was formed (138.67 s). The observations showed that the deceleration of the growth rate significantly enhanced the fragmentation and detachment. The observation proved that the detachment of the segmented crystals provided seeds of the stray crystals.

The direct observation was also performed for Al-Cu alloys. Figure 3 shows a sequence of the solidification of the Al-15mass%Cu alloy at a pulling rate of 10 μm/s. The Al dendrites are rapidly developed in the undercooled region and then grew in the vertical direction. The fragmentation frequently occurred and the detached crystals became stray crystals ahead of the advancing dendrites from the

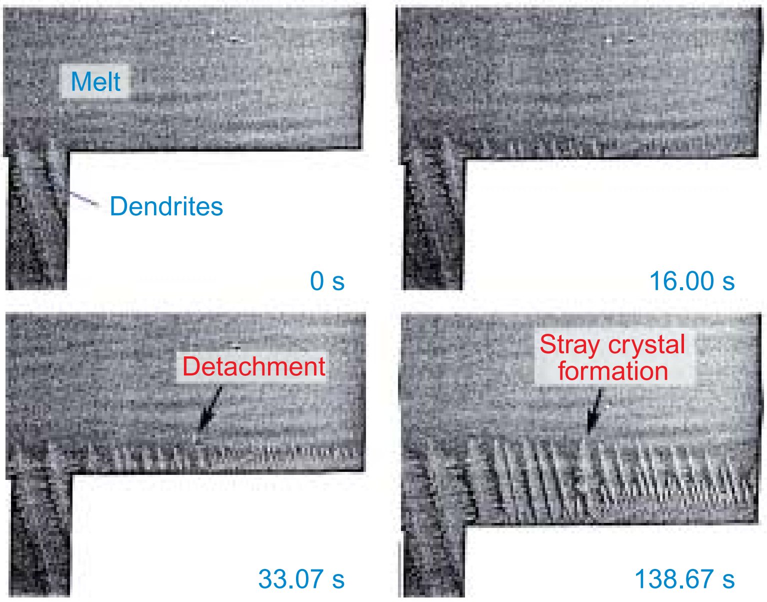

Fig. 2. Sequential photographs of microstructure after dendrites pass re-entrant corner during unidirectional solidification of Sn-21mass%Bi alloy at growth rate of 10 μm/s. The X-ray energy was 29 keV, which is just below the Sn edge.

bottom. In this case, the equiaxed grain structure, which consisted of fine grains with isotropic shape, was produced due to the frequent fragmentation. The observations show that the fragmentation dominantly contributes to the formation of the stray crystals and equiaxed grains, comparing the nucleation in the melt.

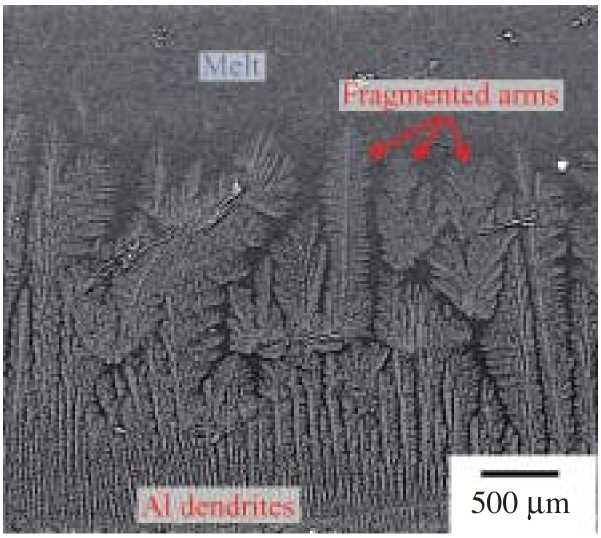

Fig. 3. Sequence of solidification for Al-15mass%Cu alloy at pulling rate of 10 μm/s. The X-ray energy was 29 keV.

The time-resolved imaging using synchrotron radiation is the best technique for observing solidification phenomena of metallic alloys. The development of X-ray imaging techniques is expected to improve understanding of various phenomena.

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References

[1] R.H. Mathiesen *et al.*: Phys. Rev. Lett. **83** (1999) 5062.
[2] R.H. Mathiesen *et al.*: Metall. Mater. Trans. B **33** (2002) 613.
[3] *H. Yasuda, I. Ohnaka, K. Kawasaki, A. Sugiyama, T. Ohmichi, J. Iwane and K. Umetani: J. Crystal Growth* ***262*** *(2004) 645.*
[4] B. Li *et al.*: Phys. Rev. E **70** (2004) 062602.