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Abnormal macrosegregation induced by formed porosity during solidification of an Al–Sn alloy

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This paper reports in situ investigation of porosity formation and its influence upon the subsequent microstructure development during solidification of an Al–6 wt.% Sn alloy using X-ray imagining and directional solidification and X-ray computed tomography techniques. The growing porosity induces abnormal macrosegregation of Sn above the pores, which is attributed to mass transportation caused by local variations in thermal and solution concentration. The motion of pores due to the abnormal macrosegregation during solidification is also described.

Keywords: Porosity; Macrosegregation; Solidification; Aluminum alloy; Casting

Macrosegregation and porosity are two major defects in Al alloy castings, and result in not only non-uniformity of structure and mechanical properties, but also reduced corrosion resistance [1–3]. The macrosegregation is generally thought to be associated with the motion of liquid in the mush zone driven by gravity and solidification shrinkage, while porosity is considered to form due to both solidification shrinkage and evolution of dissolved gas (like hydrogen). As a result, both defects are treated separately and no direct correlation is assumed between them in most numerical and experimental studies [4–12]. Recently, however, Rousset et al. [13] and Voller et al. [14] coupled inverse segregation and microporosity in their one-dimensional simulation of binary alloys. It was shown that inverse segregation decreased with the volume fraction of microporosity since the shrinkage of metal could be compensated by the development of microporosity. Using Al–0.25 wt.% Au alloy, Catalina et al. [15] observed a solution segregation band left in the solidified microstructure with a thickness of 70–80 μm around a pore after the pore was engulfed by a planar solid/liquid interface.

To better understand macrosegregation formation and its interaction with porosity during solidification, the solidification process of an Al–6 wt.% Sn alloy was observed in situ with a real-time X-ray imagining and directional solidification (XIDS) apparatus. Quantitative analysis of porosity and macrosegregation was also conducted using X-ray computed tomography (XCT), scanning electron microscopy (SEM) and energy-dispersive spectrometry (EDS). A detailed description of the XIDS has been given elsewhere [16].

The Al–6 wt.% Sn alloy melt was prepared in an electric resistance furnace using commercially pure Al (99.9%) and pure Sn (99.99%). The high hydrogen level in the Al–Sn melt (named HH alloy) was obtained by bubbling a mixture of Ar and H_2 ($Ar:H_2 = 4:1$) into the melt for 15 min. For comparison, an Al melt of the same Sn content with low hydrogen level (LH alloy) was prepared by degassing the melt with argon for 15 min. LECO™ combustion measurements of the as-cast samples showed an H_2 content of 0.36 ppm in the HH alloy sample and of 0.08 ppm in the LH alloy sample. After processing, the melt was quickly poured into a sample boat (with typical cavity of $300 \times 10 \times 3$ mm^3) in the XIDS apparatus. Simultaneously, the XIDS cooling system was activated to start unidirectional solidification from the bottom to the top and X-ray radiographs and videos were recorded automatically. The temperature gradient during solidification (measured by two K-type thermocouples in the sample boat) was about 2.5 °C mm^{-1}. The solidification velocity was characterized by measuring the vertical moving velocity

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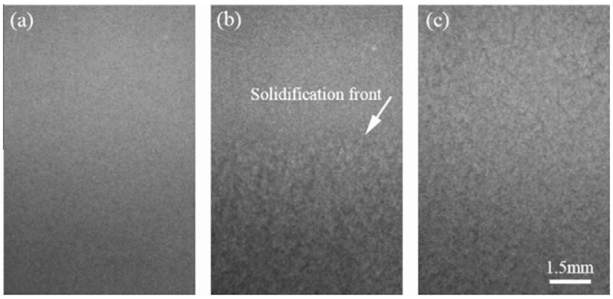

Figure 1. Solidification process of the Al–6% Sn alloy with low hydrogen content (LH alloy; the solidification velocity is about 180 μm s^{-1}): (a) 0 s; (b) 30 s; (c) 75 s. All images in the figure are on the same scale.

of the solidification front observed in the real-time radiographs. The XCT and SEM specimens were sectioned from the area that was actually observed in real time.

Figure 1 shows radiographs captured in real time during the solidification process of the LH alloy. With Al–Sn alloy, the solidification front can be seen clearly by XIDS, and is indicated by the arrow in Figure 1b. Figure 1c is the radiograph taken immediately after the solidification front had passed over the upper borderline of the X-ray view window. It can be seen that in the LH alloy no porosity forms and no notable macrosegregation occurs.

Real-time radiographs of the solidification process of the HH alloy are shown in Figure 2. The solidification velocity in this experiment was about 200 μm s^{-1}. Figure 2a shows the state when the solidification front just came into the bottom of the XIDS view window (this moment is defined as 0 s). After 25 s, the solidification front had moved up to the middle of the XIDS view window and many pores had precipitated in the mushy zone below the solidification front (Fig. 2b) due to the high hydrogen supersaturation. After about 45 s (Fig. 2c), the solidification front had passed through the upper borderline of the view window and the solidifying liquid in the entire view window became semi-solid. With the progress of solidification in the mush zone, many new pores were observed to form and the old pores grew continuously (Fig. 2d). After about 80 s, however, very few new pores formed and the old pores grew more slowly (Fig. 2e–h).

During the early stage of solidification, it was found that the growing pores could move around and even quickly escape from the view window. For example, pores #1, #2, #3 and #4 (indicated in Fig. 2b and c) moved upward by distances of 0.5, 0.8, 2 and 2.5 mm, respectively, within 20 s. In comparison with pores #1 and #2, pores #3 and #4 moved further as they were closer to the solidification front, where the temperature and liquid fraction was higher. Similar to the situation reported by Han using transparent organic alloys [12], the escaped pores were observed and recorded on video, but could not be captured in the radiographs in this study due to the limited shutter speed of the camera used.

Another interesting phenomenon observed in the real-time X-ray experiments is the abnormal macrosegregation of Sn solute induced by growing pores during solidification. As shown in Figure 2, particularly in Figure 2d–h after the solidification front had passed across the view window, the dark areas or shadows associated with all individual pores are Sn-rich segregations. At the beginning of solidification, as in Figure 2c, the gray level around the pores in the bottom half of the image is slightly darker than the pore-free matrix. Most of the dark shadows are above the pores, but a few are below them. As solidification progresses, the gray level of those dark areas around the pores becomes darker, indicating increased Sn segregation (Fig. 2d–f). Finally, almost all the pores look like having “black hats” when the solidification in the view window is completed (Fig. 2h). As described in the literature [13], the pores growing during solidification of Al alloys reduce segregation by compensating for the volume shrinkage. However, in this study, the abnormal macrosegregation phenomenon is induced by the presence of pores, which is different from inverse segregation and cannot be explained simply by Stock’s convention as a result of the shrinkage force.

It was also found that the induced macrosegregation of Sn at the top of pores could move the pores around. For example, the #1 pore moved gradually downward during the entire solidification (Fig. 2e–h), while the #2 pore first moved slowly downward from 45 to 120 s (Fig. 2c–f), while the segregation increased in size, then jumped back to the top of the black zone (Fig. 2g). Afterwards, the pore moved downward again (Fig. 2h). A similar phenomenon was observed with other pores, such as pores #3, #7 and #8 (#7 pore was merged from #5 and #6 pores, another motion pattern of pores). Although jumping of pores in the mushy zone towards the regions of higher temperature along the dendrites was also observed by Han in an organic alloy [12] and by Arnberg and Mathiesen in an Al–Cu alloy [17], no such finding of pores moving to lower temperature regions is reported in the literature. It should be pointed out that interaction between pores and the dark zones around the pores continues through the entire solidification process. Some dark channels are also seen in the radiograph, as indicated by the arrow in Figure 2g, and they become wider as the pore radius becomes larger (Fig. 2h). The formation of dark channels is believed to be related to the motion of pores during solidification.

Figure 3 presents XCT tomographs showing the actual distribution of pores and associated Sn segregation in three dimensions for the region that was observed in situ by XIDS. The colored balls dispersed in the sample (Fig. 3a) are pores observed in radiographs, while the red color zones with irregular shapes in Figure 3b correspond to the black (Sn macrosegregation) areas indicated in Figure 2h. The #1 and #2 pores observed in Figure 2 are also identified in Figure 3b. It can be seen that all the irregularly shaped red colored areas in Figure 3b are connected to the spherical pores.

Figure 4a shows a SEM image taken from the cross-section of a pore observed by both XIDS and XCT. The alloy formed a cellular–dendritic microstructure. It can be clearly seen that there is a large interconnected bright phase located immediately above the pore. According to EDS analysis (Fig. 4b and c), the large network-like