In-situ observation of porosity formation during directional solidification of Al-Si casting alloys

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Abstract: In-situ observation of porosity formation during directional solidification of two Al-Si alloys (7%Si and 13%Si) was made by using of micro-focus X-ray imaging. In both alloys, small spherical pores initially form in the melt far away from the eutectic solid-liquid (S/L) interface and then grow and coagulate during solidification. Some pores can float and escape from the solidifying melt front at a relatively high velocity. At the end of solidification, the remaining pores maintain spherical morphology in the near eutectic alloy but become irregular in the hypoeutectic alloy. This is attributed to different solidification modes and aluminum dendrite interactions between the two alloys. The mechanism of the porosity formation is briefly discussed in this paper.

Key words: in-situ observation; porosity formation; Al-Si casting alloy; micro-focus X-ray; directional solidification

CLC number: TG146.2 + 2

Document code: A

Article ID: 1672-6421(2011)01-014-05

Porosity in cast aluminum alloys has long been recognized as one of the most detrimental factors affecting the mechanical properties, and in particular fatigue resistance of the material[1]. While numerous attempts have been made to minimize the porosity through optimal gating/riser design and process optimization, one would like to accurately simulate porosity formation, and predict porosity population and pore sizes in the cast components, and eventually to design and manufacture the cast components with high reliability. To achieve this, one needs to fully understand the physics governing porosity formation in aluminum castings. In the last couple of decades, a number of investigations have been conducted to study the influence of processing variables and melt quality, such as hydrogen concentration, melt purity and melting processing, on the size and volume fraction of porosity in the solidified microstructures of the aluminum castings[2–6]. However, little progress has been made in understanding how the porosity forms during solidification because of the lack of capability in real time observation. R. Fuoco et al. used a quenching technique to study the mechanism of porosity formation during solidification of an A356 alloy[7]. Although this method is more advanced compared with traditional microstructure observation of the fully solidified castings, it cannot really track bubble movement, interaction, and morphology change. In 1960, A. E. Carte studied air bubble formation in freezing ice using an optical method; and found that the size, morphology and density of porosity are strongly affected by solidification rate and gas saturation[8]. Q. Han[9] used a stereomicroscope attached to a video camera to observe the behavior of porosity formed during the solidification of cyclohexane, a kind of transparent organic material. It was found that many pores move quickly at an inconceivable velocity towards regions of higher temperature in the mush zone. Using XTGS (X-ray Temperature Gradient Stage) technique, P. D. Lee et al. were able to observe in real time the formation of porosity during solidification in a hypoeutectic Al cast alloy for the first time[10-11]. The valuable data of nucleation temperature and size variation of porosity were obtained for the alloy from the XTGS experiments. To accurately simulate porosity, more information is still needed on nucleation and forming process of porosity, as well as its interaction with solidifying microstructure. In this paper, a micro-focus X-ray imaging system and directional solidification technique were utilized to observe the evolution of porosity formation during directional solidification of both near eutectic and hypoeutectic Al-Si alloys with added strontium.

1 Experimental setup and procedure

In-situ observation of porosity formation during solidification of aluminum castings was conducted in the X-ray temperature gradient system (XTGS), schematically shown in Fig. 1. The XTGS consists of a micro-focus X-ray imaging source (14 µm beam size), a modified directional solidification unit, and an
image intensifier connected to a CCD camera.

In the unidirectional solidification unit, there is a stainless steel crucible with internal dimensions of 320 mm × 10 mm × 3 mm. The crucible and the aluminum sample can be moved up or down at a controlled velocity with a special traction unit. Forced cooling water is applied to the bottom of the sample crucible. A Bridgman furnace, constructed with three zones: namely hot, heat insulation and cold zones, is employed to obtain controllable temperature gradient conditions in the sample crucible.

The near eutectic alloy (Al-13%Si-0.35%Mg) was prepared in an induction furnace using commercial Al-12Si alloy, pure silicon (99.98%) and pure magnesium (99.7%). The hypoeutectic alloy (Al-7%Si-0.35%Mg) was an A356 alloy. Both alloys were modified using Al-10%Sr master alloy to obtain a Sr addition of about 0.02wt.%. No degassing was made to either alloy (H2: ~0.20 mL/100g Al). To make the specimens for XTGS experiments, the aluminum melt was first cast in a metal mold with a cavity of 310 mm × 60 mm × 3 mm and the solidified slice was then cut into the typical dimensions of 310 mm × 10 mm × 3 mm that can be placed into the sample crucible. In the XTGS experiment, the sample crucible was first held in the hot zone for about 30 min to re-melt the sample completely; and the sample was then moved down and held at the heat insulation zone to start unidirectional solidification. In the XTGS experiments, the temperature gradient was controlled at about 2.2 °C/mm and the average solidification rate was about 64 μm/s. Radiographic images were captured at a sampling rate of 25 images per second. The microstructures of the solidified samples were taken using a stereomicroscope and the samples were prepared according to standard metallographic technique.

2 Results and discussion

Figure 2 shows the radiographs taken from the Sr-modified near-eutectic Al-13%Si alloy during solidification. At the beginning of solidification, the sample was completely liquid and the background of the X-ray image (Fig. 2a) is uniform. When the cooling water was turned on, the unidirectional solidification started from the bottom of the specimen and the time was counted as 0 s. After about 55 s, three spherical pores (~50 μm) first appeared in the view window of the XTGS (as indicated by the arrows in Fig. 2b). With continued cooling, more new pores appeared in the view window and the previously formed pores continued to grow (Figs. 2c-d). After 195 s, however, only a few new pores formed. At the same time (195 s), the eutectic solid-liquid (S/L) interface moved up into the view window (as indicated by an arrow in Fig. 2d). At about 245 s, the S/L interface moved up to the middle of the view window. After 265 s, the metal in the view window was completely solidified and most of the pores were entrapped by solid (Fig. 2f). As the contrast between the liquid and solid of Al-Si alloy is low, it is hard to see the eutectic solid-liquid interface.

Fig. 1: A schematic illustration of the XTGS setup

Fig. 2: X-ray macrographs showing porosity formation during solidification in the near-eutectic Al-13%Si alloy
interface (S/L) directly from the unprocessed radiographic images, as in Fig. 2. But, the eutectic solid-liquid interface can be discerned by enhancing the contrast in the radiographic images, as shown in Fig. 3.

Fig. 3: The enhanced radiographic images showing porosity formation and the solid/liquid interface (S/L as indicated by an arrow) during directional solidification of the near eutectic Al-13%Si alloy

It was also observed that several pores (denoted as I–VII in Fig. 2d-f) moved out of the view window so quickly that they could not be captured by the X-ray imaging system with a sampling frequency of 25 s⁻¹. Based on the experimental setup and X-ray imaging system sampling rate, it is anticipated that the pores move at a speed of about 150 mm/s, which is similar to the velocity of bubble jumping (140 mm/s) during solidification of a cyclohexane observed by Q. Han[9].

Figure 4 shows the evolution of porosity formation during solidification of the hypoeutectic Al-7%Si alloy. There are two stages that can be readily identified in the process of porosity formation. In the early stage (from 0 to 90 s as shown in Figs. 4a-c), small spherical pores continuously nucleate from the liquid and the previously formed pores continue to grow without changing too much in their morphology. This is similar to the observations made in the near-eutectic alloy. The second stage (shown in Figs. 4d-f) of the porosity formation in the hypoeutectic Al7%Si alloy is characterized by a remarkable change of porosity morphology and sizes. As the solidification proceeds, many pores become elongated in the solidification direction and some of them exhibit cone-shaped morphology (as denoted by I and II in Figs. 4d-e). With the continuous cooling and solidification, most of the pores become spindle-shaped along the solidification direction (Figs. 4e-f). At the end of solidification (after 235 s as shown in Fig. 4f) all the remaining pores exhibit irregular shape; which is different from the corresponding observation made for the near-eutectic alloy. This is attributed to the formation of a mushy zone ahead of the eutectic front and the interaction of the porosity with dendrite network prior to the eutectic reaction. It was also observed that some pores (as denoted by III, IV and V in Fig. 4e) disappeared and/or moved out of the view window. This indicates that pores can still move around in the mushy zone during the solidification of the hypoeutectic Al-Si alloy.

From the radiographic images shown in Figs. 2 and 3, it is clear that the morphology of all the pores formed in the near-eutectic alloy is spherical during the entire evolution process of the porosity formation. When the eutectic solid/liquid interface passes, the pores are immediately entrapped, the so-called “in-situ submergence”, with almost no change of their morphology and position. Metallographs (Fig. 5a) also show that the final morphology of the solidified pores is spherical. Most of the
pores are entrapped into the Al-Si eutectic grains directly by the eutectic S/L interface. This is slightly different from the observation made by Catalina et al.\textsuperscript{[12]}. They investigated the interaction process between porosity and liquid-solid interface during solidification of pure aluminum and Al-Au alloy. A considerable change of porosity morphology from sphere into ellipsoidal was observed while porosity was encountering the interface. It should be noted that the solidification rate used in Catalina et al.’s study was 4 \textmu m/s, which is about one magnitude slower than that we used in this work. The slower solidification rate tends to leave longer time for porosity to grow even though part of the pore is fixed by the interface. Figure 5b shows the solidified macrostructure and morphology of porosity in the hypoeutectic Al-7%Si alloy. It is seen that almost all pores are located in the inter-dendritic regions. The irregular shape of the pores is the result of geometric constraints from the growing dendritic network. In the hypoeutectic alloy, dendrites nucleate and grow first and leave the eutectic liquid in the inter-dendritic regions. As observed in XTGS, the pores form initially in spherical shape and continue to grow with the spherical morphology until they encounter dendrites. As the solidification proceeds, the growth of pores is confined in the inter-dendritic regions leading to their morphology being distorted. Because of the combined effect of dendrite impingement and shrinkage of eutectic solidification, many pores first change their morphology downward and then upward, as denoted by I and II in Figs. 4d and 4e. When entrained in the inter-dendritic regions, the pores will block the channel of feeding and cause their morphology to change downward first to compensate the eutectic shrinkage. This is different from the model proposed by P. D. Lee et al. that the pores change their morphology upward after impinging upon the dendrites during directional solidification\textsuperscript{[13]}. It should also be noted that the irregularly shaped pores observed in the hypoeutectic alloys cannot be simply considered as shrinkage-based porosity since they are gas driven at the early stage of the forming process.

Quantitative analysis of the number density and area percentage of porosity observed in the view window of XTGS is shown in Fig. 6 for both alloys. The change in number density of porosity during solidification is comparable for both alloys. The number density reaches a maximum when the time is between 110 and 140 s, and then decreases slowly because of the disappearance of some pores. Similarly, the area percentage of porosity increases quickly at an approximately constant rate in the beginning; and then gradually levels out or even decreases slightly in the case of the hypoeutectic Al-7%Si alloy. The maximum area percentage of porosity is at about 265 s for

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**Fig. 5:** Macrostructures of the directionally solidified Al-Si alloys

(a) Near eutectic Al-13%Si alloy  
(b) Hypoeutectic Al-7%Si alloy

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**Fig. 6:** Quantitative analysis of the number density and area percentage of porosity observed in the view window of XTGS during the solidification process of near eutectic Al-13%Si alloy (a); and hypoeutectic Al-7%Si alloy (b)
the near eutectic alloy and 140 s for the hypo-eutectic alloy. The constant increase of the area percentage of porosity at the early stage of solidification is attributed to the continuous nucleation of new pores and growth of the previously formed pores.

By further examining the XTGS radiographs in Figs. 2 through 4, it can be seen that although a majority of new observable pores form right before the eutectic S/L interface approaches and passes through the view window; the first group of pores nucleate at 55 to 75 s in locations far away (more than fifteen millimeters) from the S/L interface. That is to say, porosity could nucleate and grow in the liquid far away from the eutectic S/L interface. Certainly, this does not exclude the possibility that very small pores (< 50 μm) may form in the liquid very close to the eutectic S/L interface because of the limit of resolution of the XTGS system employed in this work. There is a significant difference in the solubility of hydrogen between the liquid and solid of aluminum (equilibrium partition ratio \( k \)) is 0.057 at 660°C in pure aluminum system according to Ransley and Neufeld \([14]\). As a result, hydrogen will be rejected into the melt and accumulated in front of the liquid-solid interface during solidification due to limited diffusion of hydrogen in the liquid \([15]\). In addition, the solubility of hydrogen in liquid aluminum increases with temperature \([16]\). Therefore, it is generally accepted that the hydrogen is more saturated in front of the S/L interface than that in the liquid far away from the S/L interface. That is why in the numerical simulation of gas porosity in aluminum castings nucleation of porosity is usually based on stochastic nucleation theory with a threshold value and the growth of porosity is controlled by hydrogen diffusion \([13]\). However, the diffusion coefficient of hydrogen in liquid aluminum is much greater than other solutes usually present in aluminum alloys, such as Si, Mg and Cu \([18]\). The differences in hydrogen supersaturation between the liquid in front of the S/L interface and that far away from the interface may be not as considerable as expected. In addition, it is known that there exist an enormous number of heterogeneous sites in the liquid metal for porosity nucleation, of which the difference of their abilities may be remarkable. In this study, porosity was observed to form in the liquid far away from the eutectic S/L interface, where the hydrogen supersaturation is usually believed to be very low. This further confirms that the nucleation of gas porosity depends not only on the hydrogen supersaturation but also on the existence of those powerful heterogeneous sites, such as gas bubbles that have survived in the re-melting or oxide inclusions with large gas gap in the liquid. This should be taken into account when simulating porosity in aluminum castings.

### 3 Conclusions

(1) In the near-eutectic (Al-13%Si) alloy, spherical pores nucleate and grow in the liquid melt ahead of the eutectic S/L interface. The pores remain spherical in shape when they encounter the S/L interface.

(2) In the hypo-eutectic (Al-7%Si) alloy, the spherical pores nucleate from the melt and grow into irregular morphology in the confined inter-dendritic regions. When they encounter the eutectic S/L interface, the elongated pores grow further downward and then upward due to lack of feeding during directional solidification.

(3) The different porosity morphology observed in the two alloys is attributed to the different solidification modes between them.

(4) Porosity can nucleate in the liquid far away from the eutectic S/L interface although the hydrogen supersaturation may be low, because of the existence of other heterogeneous nucleation sites.

### References


This work is funded by the Natural Science Foundation of China under grant No: 50771031 and GM Research Funding under contract No: GM-RP-07-211.