Crystallographic investigation of grain selection during initial solidification

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Abstract. Normally, macroscopic solidified structure consists of chill, columnar and equiaxed zones. In a chill zone, many fine grains nucleate on the mold surface and grow their own preferred growth direction. Only a few of them continue to grow because of grain selection. In order to understand the grain selection process, crystallographic investigation has been carried out in the zone of initial solidification in this study. 10 g of Al-6 wt%Si alloy was melted at 850 °C and poured on the thick copper plate. Longitudinal cross section of the solidified shell was observed by a SEM and analyzed by EBSD. The result of EBSD mapping reveals that crystallographic orientation was random in the range of initial solidification. Further, some grains are elongated along their <100> direction. Columnar grains, whose growth directions are almost parallel to the heat flow direction, develop via grain selection. Here, a dendrite whose growth direction is close to the heat flow direction overgrows the other dendrite whose growth direction is far from the heat flow direction. However, sometimes we observed that dendrite, whose zenith angle is large, overgrew the other dendrite. It can be deduced that the time of nucleation on the mold surface is not constant.

1. Introduction

When molten alloy is poured into a mold cavity, many small and random grains nucleate on the mold surface [1, 2]. These small grains are called chill grains. Some of them continue to grow inward but some stop growing, because of the grain selection [2]. A grain is composed by some columnar dendrite arms, which have the same crystallographic orientation and may be grown from one nucleus. If the growth direction of these dendrites is not favoured, their growth is interfered with by the growth of dendrites which form another grain. Finally this former grain disappears during growth. In the same way, favoured grains continue growing and well-developed columnar zones would form. Understanding of the solidified structure of the as-cast product is very important to evaluate the mechanical properties of products. Since the process of chill to columnar zone transition is continuous, understanding of formation of chill zone and chill to columnar transition is very important. Therefore, this study has been performed to understand the process of grain selection, which takes place in the initial solidification region using EBSD.

2. Experimental procedure

The substrate used in this study was thick copper plate and its surface was polished unidirectionally by
Emery paper of #80. Al-6wt%Si alloy was prepared from pure aluminum and silicon. The purities of raw materials were 4N and 6N, respectively. 10g of Al-6 wt%Si alloy was melted at 850ºC and cast on the substrate. Solidified shell was approximately 25mm in diameter and 7 to 8 mm in height. Longitudinal cross section of the solidified shell, which is perpendicular to the direction of polishing, was observed metallographically. The surface of the specimen was carefully prepared using colloidal silica and treated with Ar-milling. Then crystallographic orientation was analyzed using EBSD. In order to visualize the growth of dendrites in this region, the model dendrites are constructed by 3D-CAD.

3. Experimental results and discussions

SEM image of analyzed region is shown in figure 1. The growth direction is from bottom to top. Eutectic grains can be observed in the interdendritic region and were aligned in a certain direction. This indicates that dendrites, which were in a solidified grain, grew in the same direction.

![SEM image of longitudinal section of Al-6 wt%Si alloy.](image1.png)

Figure 1. SEM image of longitudinal section of Al-6 wt%Si alloy.

![EBSD mapping result in y-direction of longitudinal cross section.](image2.png)

Figure 2. EBSD mapping result in y-direction of longitudinal cross section.

Figure 2 shows the EBSD mapping result in y-direction with rainbow colour, which is the same area as shown in figure 1. Crystallographic misorientations exceeding 10° are indicated by thick black lines. At the bottom of this mapping, the grains were small and the crystallographic orientation was random. Therefore, it is safe to judge that the surface area of the solidified shell was chill zone. On the contrary, approximately 1 mm from the mold surface, which is the top of this mapping, the columnar zone developed and the grains were elongated vertically. Furthermore, the width of these grains was large. These grains were reddish and this means that the crystallographic orientations in y-direction
were close to <001>. Therefore, chill-to-columnar transition took place in this area indicated in this mapping. Here, typical grains are named 1, 2, 3 and 4 as shown in figure 2. EBSD mapping results in x-direction and in z-direction are also shown in figures 3 and 4, respectively.

3.1 Normal grain selection
The growth of grain #1 is analyzed in detail. The grain #1 was a rather long columnar grain. All <001> directions of this grain were parallel to the direction of x, y and z. Because all colours indicated in figures 2-4 were almost red. Therefore, the grain #1 was perfectly aligned. The Euler angles of the grain were obtained from EBSD analysis. Using these angles, the zenith angle, which is the angle between dendrite trunk and heat flow direction, was calculated. The zenith angle of the grain #1 was 7.2 degrees.

A model dendrite in grain #1 was constructed with 3D-CAD and is shown in figure 5. Here, the array of dendrite is thought to be square and secondary dendrite arms sintered together and form a plate with uniform thickness [3-5]. The secondary dendrite arm plates were almost parallel to the x- and z-direction. This description agrees well with the EBSD mapping result described above. Furthermore, the dendrites in grain #1 were almost perpendicular to the mold surface. The grain #1 overgrew other small grains, a, b, c, d and e as shown in figure 2. The zenith angles of these grains are 44.4°, 21.7°, 21.9°, 49.4° and 43.7°, respectively. In this case, the dendrites with low zenith angle (#1) were selected. After that it became a well-developed columnar grain. Unfortunately, since the grain #1 did not touch the bottom surface, the nucleation point of the grain #1 was not observed on this cross

Figure 3. EBSD mapping result in x-direction of Al-6wt%Si alloy.

Figure 4. EBSD mapping result in z-direction of Al-6wt%Si alloy.

Figure 5. Model dendrites of grain #1 constructed by 3D-CAD.
section. It must be a certain distance far from this cross section in z-direction.

As shown in figure 2, the grain #2 was small but elongated. It inclined approximately 45°. The colours of this grain in x- and y-directions were almost the same and green. Thus, the orientation in x-direction and y-direction were approximately <011>. Therefore, the growth direction of grain #2 was <001>, or primary dendrite trunk inclined in this direction. Thus, a dendrite can grow along its preferred growth direction, regardless of heat flow direction as pointed out by Kurz and Fisher [2].

![Figure 6. Model dendrites of grain #2 constructed by 3D-CAD.](image)

A model dendrite of this grain is shown in figure 6. The direction of primary dendrite can be again confirmed. The zenith angle of this dendrite is 45.1°. This angle is quite large and this growth direction is not favourable for the growth of columnar grain. Therefore, it indeed overgrew some small grains (f-i as shown in figure 2) but was stopped growing by other favorable grains, j and k as shown in figure 2.

Almost all grain selections analyzed are the same as indicated above, namely favourable grains are selected and unfavourable grains disappear.

### 3.2 Abnormal grain selection

Occasionally, unfavourable grain overgrew favourable grain. Competitive growth between grains #3 and #4 was analyzed. Here, the growth of grain #4 was stopped by the grain #3 as shown in figure 2. The zenith angles of the grain #3 and #4 were calculated from the Euler angles and to be 38.4° and 25.4°, respectively. A bird’s eye view of model dendrites of grain #3 and #4 is shown in figure 7. It is easy to understand that the grain #3 overgrew the grain #4 and both dendrites inclined in the same direction. If the normal grain selection takes place, the grain #4 would overgrow the grain #3, because the zenith angle of grain #4 is smaller than that of grain #3. However, the experimental result was different: the grain #3 overgrew grain #4, which did not agree with the description in the textbook [2].

A probable hypothesis is that the nucleation of grain #3 should be earlier. The grain #3 might nucleate and grow earlier, it could overgrow the grain #4. However, at approximately 1 mm from the mold surface, the local non-uniformity of initial solidification might disappear. Therefore, the grain #3 was stopped growing by other favourable grains (l and m as shown in figure 2). Because the grain #3 is not favourable for columnar dendrites.
The condition of initial solidification may be complex. For example, flow of molten alloy may not be uniform. Furthermore, the contact between molten alloy and mold surface may not be uniform. Therefore, some unsteady growth may locally take place in this region. One example is that the nucleation does not take place simultaneously. Surface defects of cast products, such as surface cracking, formation of stray crystals, may be originated in the non-uniformity of initial solidification.

4. Conclusions
Crystallographic investigation of solidified grains in initial solidification region has been carried out using Al-6wt%Si alloy by EBSD. The abnormal grain selection as well as the normal grain selection has been observed. In abnormal grain selection, the unfavourable grain overgrew the other favourable grains. This may be due to the fact that the nucleation does not take place simultaneously.

References
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