

SURFACE DEFECTS ON DIRECT CHILL AS-CAST 6XXX ALUMINUM BILLETS

Mikael Erdegren, Torbjörn Carlberg

Mid Sweden University, Department of Natural Sciences, Engineering and Mathematics, 851 70 Sundsvall, Sweden

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Abstract

Surface defects found during air-slip direct chill casting are today a major quality factor for extruded aluminum, as they can cause increased segregation, pores and unwanted precipitations. The surface zone in billets of the aluminium alloys 6063, 6005 and 6082 have been analysed by metallographic methods and by chemical analysis. Surface defects, of the type vertical drags, were investigated and compared to defect free surfaces. Inverse segregation to the surface was quantitatively analysed. The concentration profiles were coupled to the appearance of the defects and to microstructures from corresponding areas. It was shown, for vertical drags on 6005 billet surfaces, that either the segregation depth or the precipitated particles were different from surface areas without defects. For the 6063 alloys on the other hand the vertical drag zones contained different particles than surfaces without defects and the segregation had noticeably increased.

Introduction

In the production of aluminum billets a major problem has been identified, which is addressed in this project, namely surface defects. This problem is of large economic importance, and is today the main cause to scrap during vertical direct chill (DC) casting. Billet surface quality is for example evaluated by measuring surface appearance, segregate zone thickness, large Mg_2Si -particle near the surface and area fraction of pores. Surface defects is said to increase the segregation zone, but also the size and number of the Mg_2Si -particles. The presence at the surface of many Mg_2Si - and β -particles can lead to pick-up defects on the aluminum profiles during extrusion [1]. Vertical drags (VD) are one of the most common defects found at DC casting. A possible cause for formation of VD at air-slip hot top casting could be that, due to uneven and/or low air-pressure in the graphite ring, liquid aluminum sticks to the ring in the DC casting mould [2]. The purpose of this article is to analyze various surface defect structures, the segregation zones and to identify the intermetallic phase particles in the surface region. Most of the previous works connected to this research were done on a macroscopic level [3], but this paper involves studies of both microstructure and macro-segregation.

Experimental Procedures

The 6xxx series aluminum alloys are primary made for extrusion. Three of the most commonly extruded alloys were therefore chosen for this study. These three were 6005, 6063 and 6082. From the aluminum industry 30 mm thick discs were cut from the billets, as-cast by air-slip technology (Wagstaff). From the discs different samples were extracted and table 1 shows all the samples presented in this article. Samples A-G were chosen to show different types of defects on homogenized billets. They consisted of three VD defects from 6005 billets and three from 6063 billets. Sample F was taken from a considerably older billet compared to

the rest, and the studied defect was very large and less usual than the rest. According to statistics 6082 billets do not usually have VD defects going along the whole billet. They rather have a characteristic rough surface with small random ridges a few millimeters long, which for this type of alloy, is considered normal. That type of surface is represented in sample G.

Samples C, E and G were selected for a segregation analysis, and comparisons were made between areas with and without defects on the surface of the same billets. Those billets were homogenized, but also two not homogenized ingots H and I were analyzed in the same way. All samples mentioned were also used to identify intermetallic phase particles.

Table 1. The samples designated letters for respective billet type. VD = Vertical drags and UH = Unhomogenized.

Sample name	Al alloy	Type	Billet diameter [mm]
A	6005	VD	178
B	6005	VD	178
C	6005	VD	228
D	6063	VD	178
E	6063	VD	203
F	6063	VD	178
G	6082		203
H	6005	UH+VD	178
I	6063	UH+VD	178

The surface defects were first studied at lower magnifications in a stereo light microscope (SLM). Before further microscope studies all samples were mounted in a polymer with hardness similar to that of the alloys to avoid edge effects during grinding. They were then ground with wet silicon paper and polished with a polishing-system. No water was used in the last step of polishing, as water etches the water sensitive Mg_2Si particles. After polishing the samples were studied with a light optic microscope (LOM) axially and radially.

Later a scanning electron microscope (SEM), with an energy dispersive x-ray spectrometer (EDX), was used to identify intermetallic phase particles. This was done by calculating the Fe:Mn:Si:Mg ratio for particles with different shape and color [3]. After the phase identification the samples with surface defects were etched in a solution of 0.5% hydrofluoric acid (HF) for 10 seconds, restudied in a LOM and the defects and segregation zone depth were measured with the software Kappa Imagebase Metero 2.7.

The grain sizes at different depths were also measured, at both defects and at defect free areas. To do this the samples were anodized to improve the grain measurements. They were placed as

anode in a solution, consisting of 24 ml ethanol, 1 ml HF (50%), 1 ml fluoroboric acid (HBF₄)(35%) and 74 ml tap water. The cathode was a platinum basket. The voltage was 50 V and the anodization time was 90 seconds. When inspecting the anodized samples in a LOM polarized light was applied. Grain size variations on the macro scale for these three alloys have also been presented in previous work [4].

To properly study the segregation in a sample the composition at different depths from the surface had to be measured. This was accomplished with a method that was used in [5]. The billet discs were put in a milling-machine so the surfaces of the billets could be cut out like ring segments, 3-4 mm thick. These samples were then straightened with a 20-ton press and placed in an optical emission spectrometer (OES) for a composition measurement. After each run in the OES the samples were ground and again put in the OES, until the depth from the original surface was 1000 μm. First five measurement points were done with 10 μm steps as the segregation shifts fast near the surface.

Results

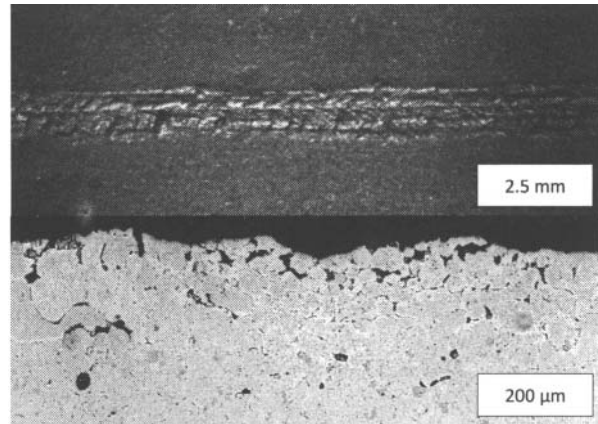
Looking at the billets surface, the VD defects could have varying length, location and “track” appearance. Sometimes they spanned across the whole length of the billet and other times just short sections. Common VD appearances were named: tire tracks (figure 1a), wave tracks (figure 1b), zipper tracks (figure 1d) and fish bone tracks (figure 1f). The width and depth of the VD’s also varied along the billets.

The defect profiles for most of the VD defects tended to go both in and out in relation to the cast surface. When following the billets axis saw-tooth or wave shaped patterns were observed. These types of patterns created corresponding variations also in the structure at the defects, i.e. the amount of precipitations, the types of precipitations and the influenced depth. For 6005 and 6063 billets, both beside and at defects, the structure variations were coupled to the meniscus lines. At positions where the meniscus lines went deep into the billets, or just before the start of the lines, the zone depths were large but just after the lines started at the surface the segregation zones were thin.

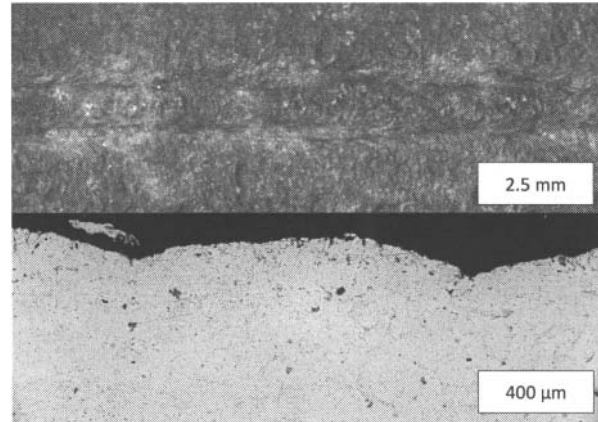
For two of the three 6005 samples, A and B, the grains seemed to have started coming apart and move away from the surface (figure 1a and 1b), thus creating an increase of the porosity. In none of the 6005 VD cases did the radial segregation zone depth, seen as a zone with increased amount of precipitations, change at the defects compared to at defect free surfaces. In figure 3a the surface zones of a homogenized 6005 alloy is shown. The segregation zone depth had an average of 90 μm and the segregation consisted of thick plate- or rod-shaped β-Al₅FeSi particles.

For the 6063 samples the analyzed particles were found to be of different types in the defect zones compared to defect free surfaces. The defect free homogenized 6063 surfaces contained mostly thin needle-shaped β-particles, figure 3b, but at the defect zones Chinese script-shaped α-Al₈Fe₂Si particles dominated, figure 3c. At the surface they can have the appearance of small cracks as in figure 1d. Following the axial direction two observations could be made. Firstly, the segregation zone depths varied a lot more at the defects compared to at a normal surface. In the 6063 samples studied, the average normal segregation zone depth was 70 μm, but for defects the variations were strong, and a maximum depth

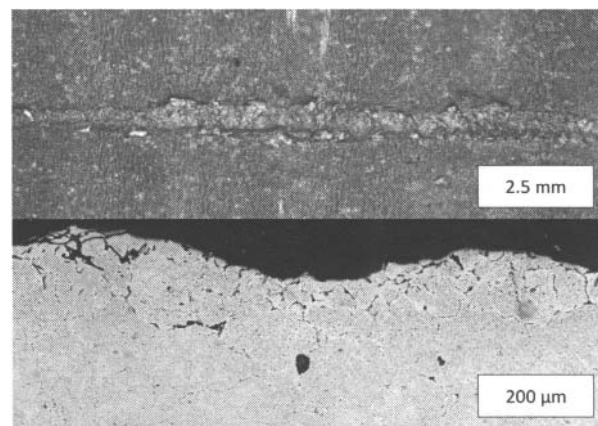
of 400 μm was recorded. The second observation was that the α-Al₈Fe₂Si particles in the defects followed the meniscus lines. In samples E α-particles could sometimes be seen 1 mm along these lines.



(a) 6005 VD, sample A.

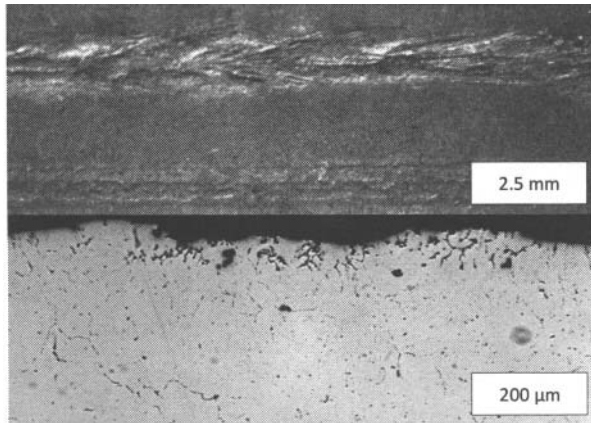


(b) 6005 VD, sample B.

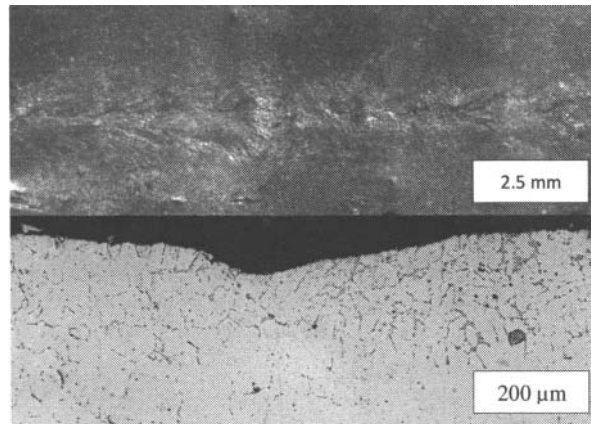


(c) 6005 VD, sample C.

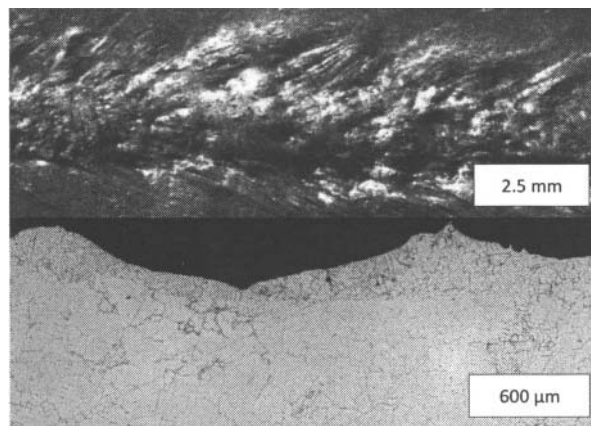
Figure 1. LOM surface pictures of samples A-G with their respective radial structure.



(d) 6063 VD, sample D.

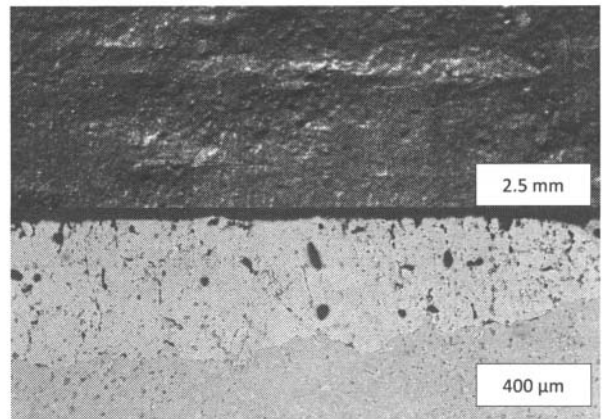


(e) 6063 VD, sample E.



(f) 6063 VD, sample F.

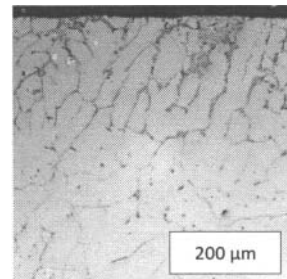
6005 and 6063 unhomogenized billets have rather similar structures, but the fraction eutectic is larger in 6005 billets, figure 2. Comparison to the homogenized billet in figure 3a shows that the particles thicken while the amount of eutectic areas decreased during homogenization. Both alloys contain some needle-shaped particles, which remain to a larger extent in the 6063 alloy after



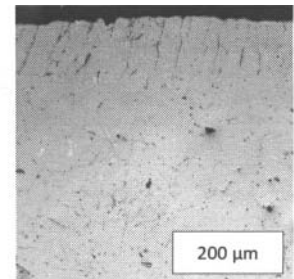
(g) 6082, sample G.

Figure 1. (Continued) LOM surface pictures of samples A-G with their respective radial structure.

homogenization. In 6005 all the particles become thicker during homogenization.



(a) 6005 UH, sample H.



(b) 6063 UH, sample I.

Figure 2. Surface segregation zone for the UH samples.

The 6082 sample, G in figure 1g, had a segregation zone that continuously throughout the billet surface, randomly changed in depth. When viewed radial and axially, the segregation zone looked the same and the depth of the zone varied between 50 μm and 500 μm. The zone contained many large pores and a high concentration of them often occur at the bottom of the segregation zone creating a porous line parallel to the surface. This area was clearly visible by a color difference between the segregation area and the bulk of the sample, caused by the etching. The particles found were mainly α -Al₁₅(FeMn)₃Si₂ (figure 3d), probably due to the higher Mn concentration in this alloys.

Mg₂Si particles appear as black areas when studied with a LOM or SEM. Black areas could sometimes be pores, but pores are generally larger than Mg₂Si particles. When the samples are observed in a LOM instantly after polishing with no water, the particles remain visible and appear as slightly darker gray areas than α - and β -particles. It was hard to compare the amount of Mg₂Si between the samples, however, they tend to lie near the α - and β -particles. Therefore an increase of these particles normally implied an increase of the number of Mg₂Si particles.

The phase particles mentioned in this section were confirmed by EDX measurements and by comparison with previous work in the same field [6-8]. Composition ratios and shape of a particle revealed the phase type. All the spot-analyses gave higher than expected Si concentration, which made some of the ratios deviate from expected values, but not enough to make identification impossible. The thick and thin $\beta\text{-Al}_3\text{FeSi}$ particles shown had a Fe:Si ratio of 1:1.1 and 1:1.3 respectively, the Chinese script-shaped $\alpha\text{-Al}_8\text{Fe}_2\text{Si}$ particle had a ratio of 2:1, and the $\alpha\text{-Al}_{15}(\text{FeMn})_3\text{Si}_2$ particle had the Fe:Mn:Si ratio of 3.3:3:3.6.

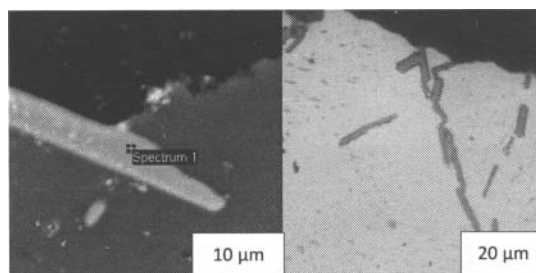
The grain size measurements in the surface zones were rather inconclusive. The average grain size was random. Neither at defects nor at regular surfaces could a clear pattern be obtained. Grain sizes ranged between 80-130 μm for all samples. A rough pattern was that for the VD samples, regardless of alloying type, smaller grains appeared near the surface, but at 200-300 μm from the surface some larger grains were found. Even further away from the surface, the grain size started to decrease again. The grains near the surface had grown slightly more columnar towards the surface hence making them appear smaller when measuring along lines parallel to the surface, which was the method used. The measurements were performed to the maximum defect depth, which was at 500 μm .

Results from the composition measurements are presented in figure 4. First measurement points are at 10 μm from the surfaces as the spark patterns obtained on the not grinded surfaces were not always reproducible. The concentration profiles compared well with most of the segregation zones as they were observed in LOM, i.e. the thicknesses of the zones with increased particle precipitation coincided with the starting points of strongly increased concentrations.

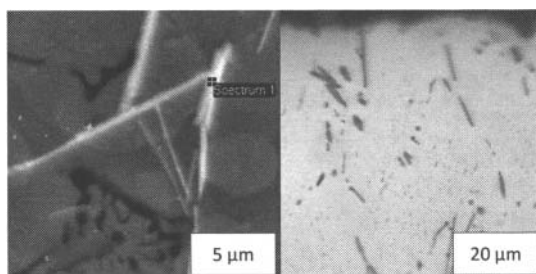
For the 6005 samples (C and H), when profiles beside a defect and at a defect are compared, no significant differences could be distinguished. Comparing the different billet slices C and H, the homogenized and not homogenized, respectively, clear differences can be seen. The former goes deeper into the billet, to about 400 μm , while the latter has an increased concentration starting about 200 μm from the surface. For the 6063 samples (E and I) the VD defects have caused segregation to go further into the billets causing deeper segregation zones, especially in the homogenized case. Near the surfaces of the VD defects the Fe and Si contents have been lowered compared to the defect free surface areas. For the 6063 and 6082 samples the defects had varying segregation depths, which meant different concentration profiles for selected measurement locations, but the total area for the OES measurement points at each depth was considered large enough to take this into account and give an average value. All surfaces except for billet (I) seemed to have a Fe content of about 1.5 wt% close to the surface. When looking near the surface the differences between a 6005 VD and a 6063 VD were the content of Mg and Si. 6005 VD had ~1.5 wt% Mg and ~2.5 wt% Si, while 6063 VD had ~0.8 wt% Mg and ~1.5 wt% Si.

Discussion

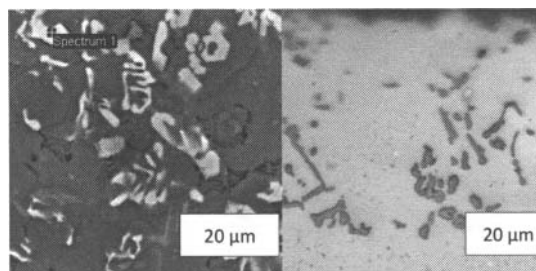
The VD defects can have very different appearances as can be seen in figure 1, which implies that it is not possible to give a general explanation to the formation of the defects or to the structures and segregations related to them. The inverse segregation to



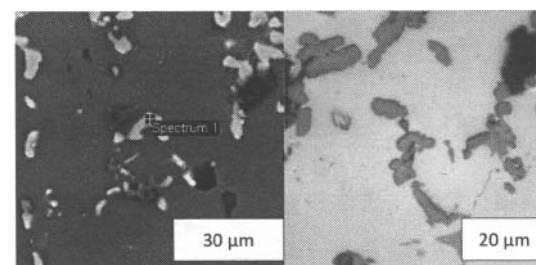
(a) Plate- or rod-shaped $\beta\text{-Al}_3\text{FeSi}$ particles found in 6005 billets.



(b) Needle-shaped $\beta\text{-Al}_3\text{FeSi}$ particles found in 6063 billets.



(c) Rod- and Chinese script-shaped $\alpha\text{-Al}_8\text{Fe}_2\text{Si}$ particles found at VD defect in 6063 billets.



(d) $\alpha\text{-Al}_{15}(\text{FeMn})_3\text{Si}_2$ particles found in 6082 billets.

Figure 3. SEM and LOM pictures of different intermetallic particles from surfaces of homogenized billets. The darker pictures to the left are taken from SEM.

the surface defects is dependent on defect type, but the differences between defects and smooth surfaces sometimes are smaller than differences between different billets, indicating that the casting parameters are important for controlling the surface segregation.

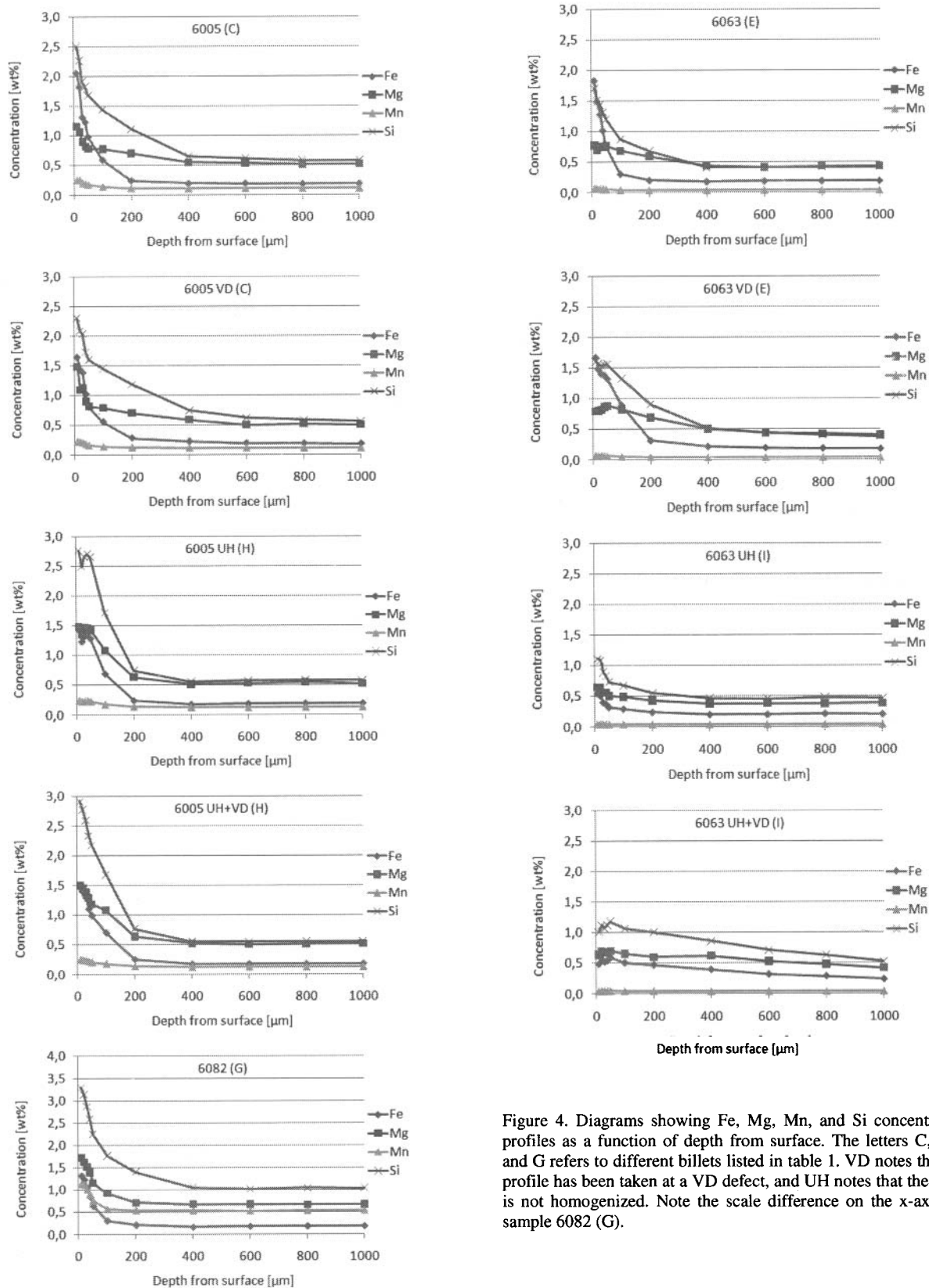


Figure 4. Diagrams showing Fe, Mg, Mn, and Si concentration profiles as a function of depth from surface. The letters C,E,H,I and G refers to different billets listed in table 1. VD notes that the profile has been taken at a VD defect, and UH notes that the ingot is not homogenized. Note the scale difference on the x-axis for sample 6082 (G).

One of the reasons for formation of VD defects, mentioned in the introduction, is that aluminum sticks to the graphite ring due to drop in air pressure, and that metal is coming out of the oxide skin at these point where the skin is strained. The sticking to the ring can explain the grain cracking for the larger of the 6005 VD defects shown in figure 1, A and B. The strong segregation, figure 4 C and H, and thus large fraction eutectic formed at the surface of 6005, figure 2a, decrease the coherency between the dendrites which, together with sticking, can open up the structure at the surface with oxides and pores as a result. This can be the reason why 6005 alloys have a high sensitivity to formation of VD defects. The inverse segregation to the surface in this alloy creates a local composition with relatively large areas with low melting point, and thus a loose structure, which becomes sensitive to sticking at the graphite ring. This can be the reason why 6005 alloys have a high sensitivity to formation of VD defects. The inverse segregation to the surface in this alloy creates a local composition with relatively large areas with low melting point, and thus a loose structure, which becomes sensitive to sticking at the graphite ring.

A slightly different defect type can be seen in figure 1c. Here the defect profiles mostly go inwards and the structure is not loose or porous as in figure 1a and 1b. A possible cause for this type of defect can be that oxides or refractory particles are stuck between the graphite ring and the billet surface [9]. An old graphite ring can sometimes have very clear indentions, with a scratch below probably caused by the release of the particle.

The different phases precipitated in the segregation zones are dependent on the local alloy composition, and the concentration profiles shown in figure 4 shows that there are completely different alloy contents at the surface than in the bulk of the billets. The ratio between the components controls the type of precipitation, and for the AlFeSi phases the Fe:Si ratio is important. For the 6005 alloys, C and H in figure 4, the Si content is higher in the whole surface zone, and the dominating phase is as expected β -phase in the shape of relatively thick plates, figure 3a. For the 6063 sample E in figure 4, the Fe:Si is about 1 at the very surface, and in this alloy, and especially at defects, α -particles were found, figure 3c. Different cooling conditions can also influence the type of precipitates, and for some of the defects in the 6063 alloy a slower cooling rate, combined with a smaller Fe:Si ratio, can explain the appearance of α -particles.

The surface segregation, both at smooth surfaces and at VD defects, is basically a result of shrinkage in the solidified shell, which causes interdendritic liquid of high alloy content to be sucked to the surface. This results in the typical increased concentrations at the surface and sometimes in exudation of enriched liquid out of the surface. The shape of the composition profiles shown in figure 4 shows a large variety, which could be confirmed also by analysis of more samples not included in this presentation. Both defects and smooth surfaces can have different types of surface segregation. In an analysis of inverse segregation [10] the influence of heat transfer at the surface was discussed, and it was found that high heat transfer gave steeper profiles at the surface and less penetration. High concentration of a certain element, in that case Cu, could also increase the steepness of the profiles. The present investigation indicates that, in cases where a difference can be seen, the defects rather give a deeper penetration of the segregation, i.e. higher contents in a thicker layer inside of the surface. Here the contributions from cooling rate differences

probably are of less importance than the movements of the shell. If the shell, dependent of defect type, is stretched out or pressed together the enriched liquid will be sucked in or pressed out, respectively, giving different types of macro-segregations in the surface zones.

Conclusions

The surface segregation in the studied 6005 billets was not changed by VD defects, while the segregation in a 6063 billet showed an increased depth and a high addition of α -particles. The segregation zone for 6082 varies randomly in depth and contains large pores. No defect in this research caused the segregation zone to go deeper than 500 μm . All analyzed as-cast billets have different concentration profiles at the surface, implying that the dimension of the billets as well as the casting process are as important as the defects for the surface structure and composition.

Acknowledgments

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