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EFFECT OF GRAIN REFINING ON DEFECT FORMATION IN DC CAST Al-Zn-Mg-Cu ALLOY BILLET

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Abstract

In direct chill (DC) casting, the effect of grain refining on the prominent defects such as hot cracking and macrosegregation remains poorly understood, especially for multi-component commercial aluminum alloys. In this work, DC casting experiments were conducted on a 7075 alloy with and without grain refining at two casting speeds. The grain refiner was introduced either in the launder or in the furnace. The concentration profiles of Zn, Cu and Mg, measured along the billet diameter, showed that the increasing casting speed raises the segregation levels but grain refining does not seem to have a noticeable effect. However, hot cracking tendency is significantly reduced with grain refining and it is observed that crack is terminated with the introduction of grain refiner at a lower casting speed. These experimental results are correlated with microstructural observations such as grain size and morphology, and the occurrence of floating grains.

Introduction

Direct Chill (DC) casting remains a major processing route to produce large aluminum alloy ingots, which are used for downstream processing. Macrosegregation, which characterizes the inhomogeneous distribution of alloying elements on the length scale of the casting, has to be minimized since it remains virtually unaffected by subsequent thermal treatments. Hot cracking that initiate above the non-equilibrium solidus temperature of the alloy, has to be prevented due to its direct influence on quality and productivity.

During DC casting, the cooling conditions and shape of the transition (semisolid) region determine to a large extent the structure and associated defect formation [1]. This region can be conventionally divided into slurry zone and mushy zone, based on the definition of coherency temperature at which solid grains start interact with each other [2]. Further below this temperature within the mushy zone, a continuous solid network forms (rigidity isotherm). In general, it is known that macrosegregation is more influenced by convective and shrinkage-induced flows in the slurry region [3,4]. Hot cracking, however, initiates in the mushy zone below the rigidity temperature between 0.85 and 0.95 solid fractions (the ‘vulnerable region’) due to thermal contraction and shrinkage stresses when metal feeding is no longer possible [2].

Out of DC casting process parameters, casting speed is the important variable as it greatly affects the transition region (the sump depth and its dimensions across the billet cross section) and hence exercises significant influence on defect formation. It is well known that the severity of macrosegregation and hot cracking tendency strongly increase with casting speed [5-7]. However, the effect of grain refining on the defect formation remains unclear although experimentally the beneficial role of grain refining in reducing the hot cracking tendency has been reported [8, 9].

But for macrosegregation, the issue remains open as whether grain refinement is beneficial or not. Gariepy and Caron [10] showed that grain refiner practices in commercial Al alloys influence the level of centerline segregation, which was, for example, more than doubled by the addition of grain refiners in a 5182 alloy. Glenn et al [11] also observed greater centerline depletion of Mg in grain refined 5182 alloy, which was explained by the formation and transport of isothermal dendrites (or “floating grains”). Grain refining caused more severe centerline segregation in a sheet ingot of Al-Mg alloy [12]. However, opposite trends are observed by Finn et al. [4] who showed that grain refining produced positive centerline segregation due to improved permeability of the mushy zone.

It is an experimentally observed fact that the introduction of grain refiners reduced the susceptibility to hot cracking by facilitating columnar to equiaxed transition. But, often in the literature, these effects are reported for permanent mould castings during a specific hot cracking test [8], or in a test designed to simulate the conditions existing during DC casting [9]. Hence data on the commercial scale DC casting experiments is lacking. Alloys having the widest solidification interval with small eutectic fractions are more problematic with respect to hot cracking [2]. In this respect, high- strength, precipitation hardenable 7000 series Al alloys are recognized as being difficult to cast and are prone to cracking during DC casting. Hot cracking tendency of different Al-Zn-Mg alloys have been investigated [9, 13, 14]. Matsuda et al [14] observed that lower casting speeds are helpful in reducing the hot cracking in a DC cast 7075. Hot cracking diagrams of various Al-Zn-Mg alloys indicate [13] that addition of Cu impairs hot tearing resistance, the maximum of hot tearing shifting to low Zn concentrations on increasing Cu content. This is reflected [9] in the cracking tendency of a 7050 alloy (2.2 wt. % Cu), which is marginally more susceptible than a 7010 alloy (1.6 wt.% Cu). The difference is attributed to a larger volume fraction of iron-rich intermetallic phases, which formed in the wider mushy region of the 7050 alloy. With grain refiner additions to these alloys, the hot cracking tendency is significantly reduced. Further, it has been observed that increased amounts of grain refiners are detrimental and there is a threshold level above which the addition is not beneficial [9]. This minimum level depends on the chemical composition (0.005 wt.% Ti and 0.3 wt.% Ti for 7010 and 7050 respectively). The reason is ascribed to the change in grain morphology with fine equiaxed dendritic grains reducing the hot cracking susceptibility while a cellular grain structure acts in the opposite direction [9, 15]. This paper reports the work on a DC cast Al-Zn-Mg-Cu alloy with and without grain refining. This work is a part of the ongoing experimental program to study the role of grain refining on defect formation of various DC cast commercial Al alloys.

Materials and Experimental Methods

Experiments were conducted in a pilot DC casting installation at the Delft University of Technology using a 200-mm hot top mould. A detailed description of this unit can be found in ref. [6]. Experiments were carried out with and without grain refining using a 7075 type alloy (Fe: Si >1), with the composition (in wt. %): 5.63 Zn, 1.51 Cu, 1.39 Mg, 0.29 Cr, 0.10 Fe, 0.07 Si. In our casting trials, two casting speeds (8 and 12 cm/min) were used while maintaining constant water flow rate (170 l/min) and melt temperature in the furnace (715°C). Grain refiner is added in the form of commercial Al-5Ti-1B master alloy rods either in the launder or in the furnace.

After casting, round billets of 192-mm ϕ were immediately loaded in a furnace maintained at 400°C for a stress-relief anneal. For macrosegregation, the billets are longitudinally sectioned in the center and samples of approximately 20 mm wide and 20 mm high were cut at suitable locations in the horizontal cross section of the billet. Care was exercised to ensure that the sampling represents the steady state conditions during DC casting. These rectangular bars are analyzed by spark spectrum analyzer across the billet diameter on all 4 sides at regular intervals of approx. 10 mm, and the average values are reported. In addition, the chemical composition at the billet surface was analyzed. The absolute error in these measurements is 0.05 wt. % for Zn, 0.01 wt. % for Cu and 0.01 wt. % for Mg. For microstructural observations, smaller samples were cut at different locations along the billet radius. Grain structure was studied under cross-polarized light after anodizing the samples in a 3% HBF_4 water solution. Other structural features such as dendrite arm spacing (DAS) and 'floating grains' are observed after etching the samples with 2% NaOH solution. Grain size is measured on photographs using the random line intercept technique.

In the following sections, grain refined (GR) samples are differentiated according to the method of addition, i.e., whether it is made in the launder (GR-launder) or in the furnace (GR-f/c). These are compared to the non-grain refined (NGR) samples.

Results

Macrosegregation

Composition profiles of the major alloying elements (Zn, Cu and Mg) plotted across the whole diameter (192 mm) indicate negative segregation in the center and minor positive segregation at the mid-radius. Further, strong segregation zones are noticed close to the surface.

The effect of grain refinement and casting speed on the macrosegregation is shown in Figure 1. At a casting speed of 8 cm/min, with lower segregation levels, it can be seen that the composition profiles are somewhat similar for both GR and NGR billets (Figure 1a & b). Also these patterns did not change for GR billet with respect to the method of addition (launder or furnace).

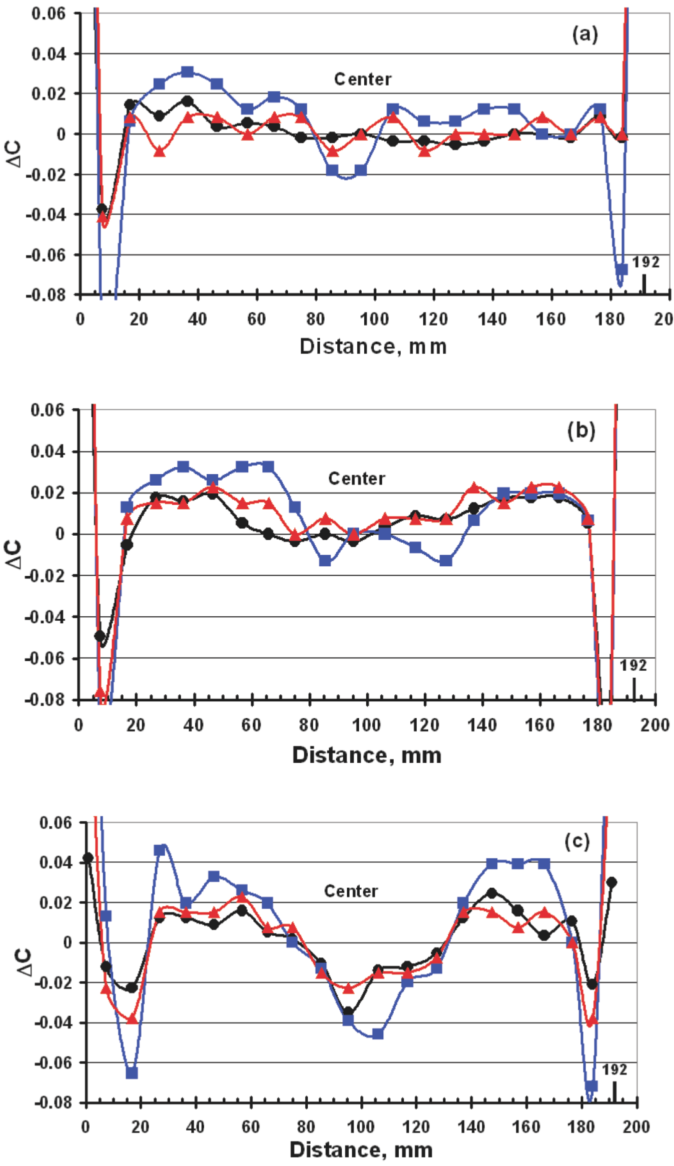


Figure 1: Composition profiles for Zn (●), Cu (■) and Mg (▲), (a) NGR – 8 cm/min, (b) GR-furnace – 8 cm/min, and (c) GR-furnace – 12 cm/min. ΔC = Relative deviation of concentration from the average, $(C_i\text{-}C_{ave})/C_{ave}$ where C_i is the concentration at the radial position and C_{ave} is the average concentration.

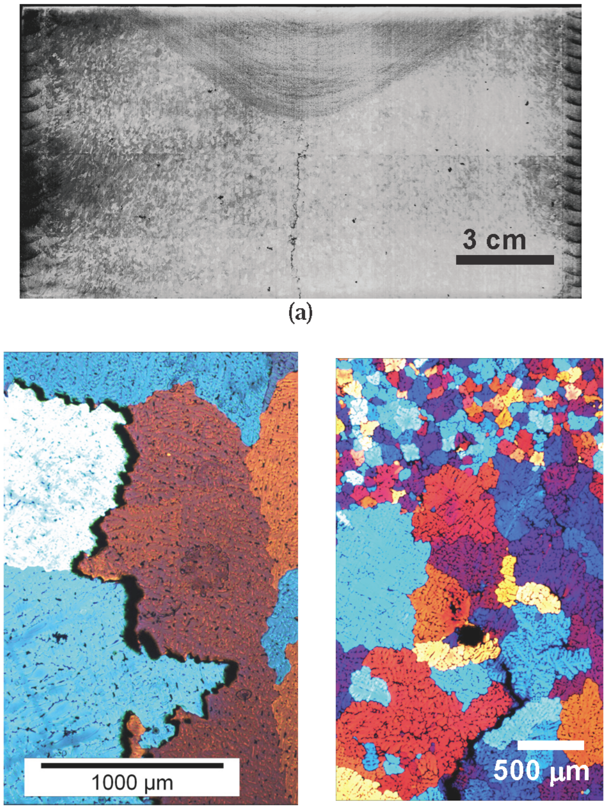
On the other hand, at a higher casting speed of 12 cm/min, deeper segregation profiles are observed, particularly in the central portion of the billet (see Figures 1b & c for GR-f/c condition).

Further in all the cases the extent of segregation differed depending on the element under consideration with Cu exhibiting larger variation compared to Mg and Zn. Another notable feature is the severe depletion of alloying elements in the subsurface followed by a strong positive segregation very close to the surface (1 mm).

Hot cracking

Macrostructures of the selected longitudinal sections were examined for the details of the cracking. As expected, centerline hot cracking had initiated in this alloy in the NGR billet (thin macro crack visible to the naked eye on a well-prepared section) at the casting speed of 8 cm/min (Figure 2a). Without exception, the crack follows the path along grain boundaries (Figure 2b). At the same casting speed, when the grain refining is initiated in the launder, it is observed that the cracking is completely eliminated. The macrostructure of the transition zone (Figure 2a) shows that the crack stops at the onset of grain refining. The solidification profile is clearly outlined due to different structure of the grain-refined area. A magnified picture (Figure 2c) of the anodized sample of the transition (from NGR to GR) further proves that the crack does not exist in the grain-refined portion.

The measured Ti concentration in the GR billets for both input conditions (furnace and launder) is 0.004 wt.%. Another experiment with the billet obtained directly from the grain-refined melt in the furnace (GR-f/c) also did not exhibit any cracking at 8 cm/min. Further no cracks (macro or micro) are observed close to the billet surface in any condition.



(a)

(b)

(c)

Figure 2: Termination of hot crack in AA 7075 with the introduction of grain refiner (a) macrostructure exhibiting the crack arrest, (b) initial intergranular crack in NGR billet (b) and (c) anodized structure at the NGR to GR transition zone.

At the casting speed of 12 cm/min, hot cracking is observed in the billet that is grain refined in the launder whereas the billet obtained with grain refiner in the furnace did not exhibit any cracking.

Microcracks and porosity

Observations are made on unetched metallographically prepared samples for microcracks and porosity with attention confined to the central portion of the billet (Figure 3). For the NGR billet at 8 cm/min, micro cracks are observed along the grain boundary (Figure 3a) in close proximity (5 mm) around the hot crack. Often these microcracks are connected by a eutectic-rich path. Observations on GR billet showed no microstructural evidence of microcracks for the conditions where either the cracking is prevented or the hot cracking did not initiate. With respect to porosity, irregular shaped pores are noticed (Figure 3b) at the inter-dendritic locations (shrinkage porosity) in all the conditions (i.e, irrespective of hot cracking).

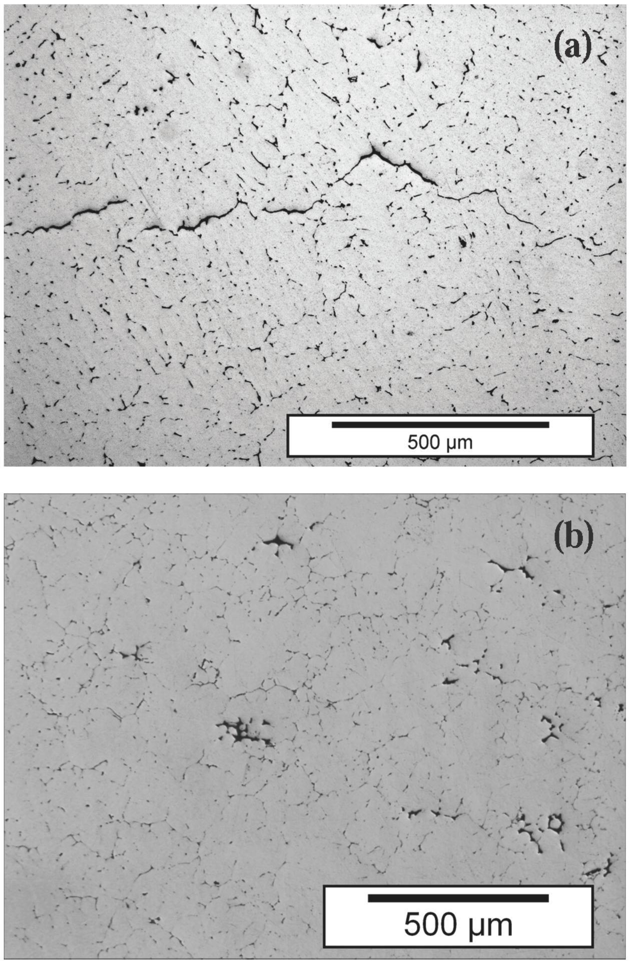


Figure 3: Microstructure in the centre of the billet (a) Microcracks close to the hot crack in NGR billet (b) Absence of microcracks with shrinkage porosity in the GR billet

Microstructure

Significant structural refinement is witnessed with the grain refiner additions as low as 0.004 wt.% Ti (Figure 4). The decrease in grain size in the center is approximately from 1100 µm to around 170 μm. With similar amount of grain refiner, GR-launder sample showed slightly finer grain size than the GR-f/c sample in the center. The radial distribution of grain size across the billet is shown in Figure 5 for both NGR and GR portions at a casting speed of 8 cm/min. In general, the grain size is increased from the periphery to the center of the billet. It is interesting to note that this variation is more pronounced for the NGR billet compared to the GR billet. With respect to grain morphology, again non-grain refined billet showed much variation across the billet diameter. After a thin layer of equiaxed grains, columnar grains have grown up to approximately 30 mm deep beneath the surface, which gave way to gradually increasing equiaxed grains towards the center (Figure 4a). Grain refining resulted in the expected disappearance of these morphological variations across the billet diameter.

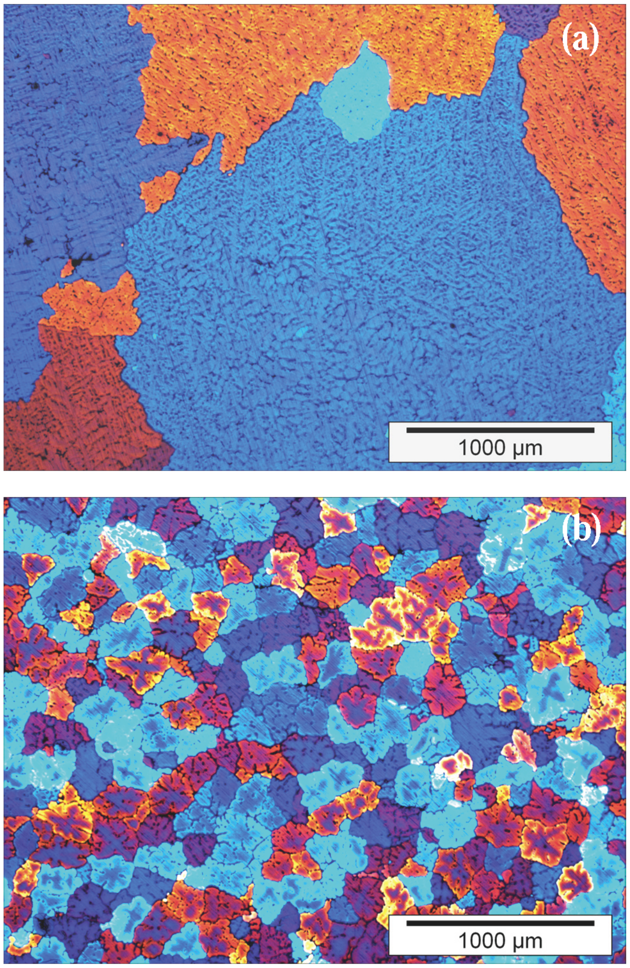


Figure 4: Equiaxed grain structures in the DC cast billet center (a) non-grain refined, and (b) grain refined.

At a finer level, dendritic microstructures are observed in both GR and NGR conditions. Examination in the billet central portion (Figure 6) showed no specific duplex structures in the non-grain refined ingot (Figure 6a). In GR billet, however, the presence of duplex microstructure is evident with clusters of coarse grains (Figure 6b) with a larger DAS compared to the rest of the matrix.

Discussion

The transition region between the liquidus (in the present case 635°C) and the non-equilibrium solidus (477°C) in the billet determines the structure and defect formation during DC casting. With respect to macrosegregation, the large-scale movement of solute-rich liquid and the solute-lean solid in the slurry zone accounts for the segregation patterns observed in DC casting. In addition to the natural convection of solute-rich liquid, the transport of solute-lean solid phase from the periphery of the billet to the center may also add to the negative centerline segregation. It is generally accepted that these isothermal dendrites with coarse cells observed in the center of the billet increase the severity of negative segregation [3, 5, 16]. Unlike our earlier observations [6, 17] of isothermal dendrites in Al-Cu alloys (both binary and 2024), we did not observe any obvious patterns of coarse dendrites in the central portion of the DC cast 7075 alloy in the non-grain refined billet (Figure 6a). But duplex structures are observed in the grain-refined billet (Figure 6b). Similar observations were made by Finn et al [4] where coarser structures are observed in the grain refined Al-Cu alloy but not in the parent material. However, duplex microstructures are reported in an Al-Zn-Mg-Cu alloy in the non-grain refined condition [3] although it is stated that fine dendrites, being those detached early in the solidification, are responsible for the negative centerline segregation. Thus it remains an open question as whether coarse dendrites (or 'floating grains') contribute to the negative segregation in this system. At a given casting speed, the sump depth do not change much for the GR billet with respect to NGR billet as had been verified by our previous measurements [18] on a 6061 alloy under identical experimental conditions. And with almost the same macrosegregation patterns in both NGR and GR billets, it seems probable that the increase in mushy zone permeability in GR billet is high enough for the solute-rich fluid to flow towards the centerline and compensate the depletion of alloying elements caused by 'floating grains'. But it remains a speculation and further work is necessary to establish the significance of the coarse grains as shown in Figure 6b in terms of their composition.

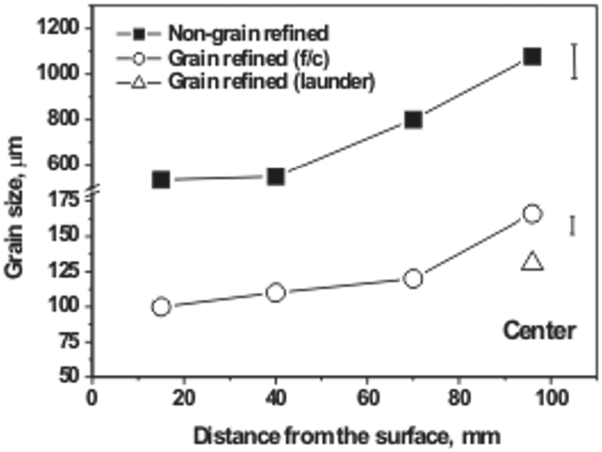


Figure 5: Radial variation of grain size in DC cast billet of 7075 alloy at a casting speed of 8 cm/min

On the other hand, increased casting speed (from 8 to 12 cm/min) had increased the sump depth significantly (e.g. from 25 mm to 68 mm for a 6061 alloy [18]). Casting speed is known to increase the severity of the macrosegregation in DC cast Al alloy billets due to widening of the liquid-solid transition region, especially in the central portion of the billet [6,16] and to the increased slope of the solidification front [19]. This means that the increase in the severity of segregation at a higher casting speed (Figures 1b & c) can be directly related to expanding distance between the liquidus and the coherency isotherm (the slurry portion of the transition region [6]) and the increasing chance of transport of the solute-depleted solid phase towards the center. At the same time, the increased steepness of the solidification front will promote the shrinkage-driven flow of the solute-rich liquid towards the periphery of the billet [19]. The deeper transition region (as shown by sump depth measurements) and associated flow patterns [6, 19] at higher casting speed will lead to increased macrosegregation levels (Figure 1c).

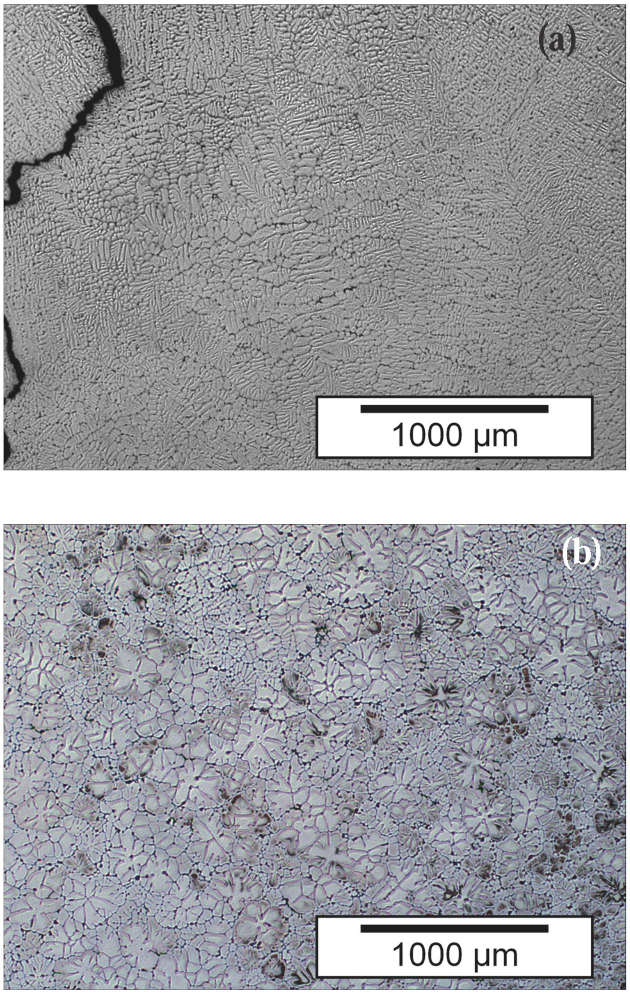


Figure 6: Etched microstructures in the center of the DC cast billet (a) NGR, and (b) GR

As was observed for commercial Al alloys, the degree of segregation is inversely proportional to the magnitude of the partition coefficient of alloying element [10, 17]. In the present work, the increased propensity for segregation for Cu could be related to its lower partition coefficient (0.17) compared to Mg (0.43) and Zn (0.45). This is consistent with the data reported [3] on DC cast Al-Zn-Mg-Cu alloy. The surface region of the DC cast material is often characterized by a strong segregation zones as observed in the present study (Figure 1) which is due to the shrinkage driven flow of solute-rich liquid [4,16].

With reference to cracking, microstructural evidence of crack following the grain boundaries (Figure 2b) points out that it is a hot crack propagated through the interdendritic liquid film. Also an annealing treatment was given immediately after the casting so as to reduce the risk of cold cracks. The present results confirm the general beneficial role of grain refining in reducing hot cracking even with low contents of grain refiners. In the alloy, which has 1.6 wt.% Cu (similar to the present alloy), a significant decrease in crack length is noted with only 0.002 wt.% Ti [9]. In the present case with similar amounts (0.004 wt.% Ti), there is complete annihilation of crack. These results demonstrate that fine equiaxed-dendritic grains minimize hot cracking. Although the exact mechanisms are not clear yet the role of grain refining can be explained in terms of mushy zone permeability, length of the 'vulnerable' solidification region and role of eutectics in healing of potential hot spots in the presence of critical stresses and strains caused by solidification shrinkage and thermal contraction.

Further to above it is noted [8] that decreasing the grain size will reduce the deformation to be accommodated, by liquid feeding, at each grain boundary within a hot spot. As cracking occurs intergranularly, any increase in GB area per unit volume will reduce the strain to be accommodated at each grain boundary. Grain refining delays the onset of strength development and load transfer in the mushy zone, reduces the liquid film thickness thereby reducing the hot tearing susceptibility [15, 20].

In order to avoid hot cracking, the localized hot spot should be initially accommodated by liquid flow through grain boundary and interdendritic channels. Hence the amount of liquid available during the critical stage of solidification is very crucial. It is indeed demonstrated [7] that the amount of non-equilibrium eutectics (the last liquid to solidify) is an important structural parameter in evaluating the hot cracking tendency. The availability of this eutectic at the grain boundary (see Figure 3a) versus inside the grain (at the inter-dendritic locations) is another factor that can control the initiation of hot cracks. Mushy zone permeability thus becomes important. From the present results on macrosegregation (Figure 1a & b) and from the literature data [4], it is apparent that grain refined alloys exhibit high permeability, which thus helps in inhibiting the crack. Also, the absence of any fine cracks in GR billet (Figure 3c) is another indication that the mushy zone is strong enough to withstand the thermal gradients. Further studies are, however, necessary to explain the hot cracking behavior observed in this alloy at the higher casting speed of 12 cm/min.

Conclusions

Direct chill casting experiments with and without grain refining at different casting speeds were conducted on Al-Zn-Mg-Cu alloy. Significant structural refinement is observed with the addition of grain refiner. At 8 cm/min, grain refining does not seem to have any considerable effect with respect to macrosegregation. Hot cracking is prevented with grain refining at a casting speed of 8 cm/min. At this drop rate, prior crack existed in the non-grain refined billet is completely arrested with the introduction of grain refiner.

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