Characterization of the Micro-Segregation in an AlMgMn Alloy Using an Innovative In-Situ Homogenization Process During Direct Chill Casting

Sootae Lee¹, Mark Gallerneault² and Robert Wagstaff³

¹Novelis Ulsan Innovation Centre, #1 Yeochun-dong, Nam-gu, Ulsan 680-090 Korea
²Novelis Global Technology Centre, 945 Princess Street, Kingston, Ontario, Canada, K7L 5L9
³Novelis - Solatens, 18001 E. Euclid, Suite A, Spokane Valley Washington, USA, 99216

It is well-known in aluminum alloys that during direct chill (DC) casting the imposed cooling rate, and difference in solid and liquid phase solubility, result in elemental micro-segregation. The level of micro-segregation may be sufficiently large that the DC ingot can only be subsequently processed with the addition of a formal homogenization step; wherein the micro-segregation is reduced to an acceptable level. In this work we outline an innovative change in the DC process that results in a significant reduction in the level of micro-segregation; to such an extent that homogenization may be eliminated; known as In-Situ Homogenization. This process involves the control of the DC secondary cooling effect with the specific aim of utilizing a portion of the latent heat of fusion, during the solidification process, to favorably control the level of micro-segregation.

Keywords: DC Casting, micro-segregation, In-Situ Homogenization, solidification

1. Introduction

Direct chill (DC) casting has been used for more than 60 years in the aluminum industry to produce large ingots (typically 0.5m thick x 2m wide x 5m long) that are subsequently thermo-mechanical processed to thinner gauge plate or sheet products. Indeed, except for the manufacture of foil and container alloys, DC casting remains the primary source of rolling stock and is used in products ranging from beverage containers to aerospace applications. From the microstructural standpoint it is convenient to think of the solidification of DC casting as comprised of two distinct parts, see Fig. 1(a). Firstly, the primary cooling zone, wherein the liquid aluminum contacts a water-cooled mold and begins to freeze, producing a solid alloy shell. Next, the secondary cooling zone, which begins at the point where the now-solidifying ingot exits the mold and is met by a series of aligned & impinging water jets; supplied via the primary mold cooling water. The water is supplied in sufficient quantity to provide a cooling film of water over the remaining length of the solidifying ingot.



Fig. 1 Direct Chill (DC) casting schematic with primary and secondary cooling zones (a) without and (b) with the in-situ technology.

As a consequence of this nonequilibrium cooling sequence, the microstructure of as-cast ingots must be homogenized in order to reduce the level of micro-segregation and dissolve eutectic phases at the grain boundary so that the ingot does not fracture on subsequent rolling. In the case of AlMg alloys, the rolling surfaces must cool sufficiently for the rolling surfaces to be scalped before rolling. Scalping is typically done prior to the homogenization stage. Hence, up to the point of hot rolling, a typical DC ingot will undergo cycling of temperatures: cooling from the melt during solidification, a reduced rate of cooling before scalping, and finally re-heating during the homogenization process followed sometimes by a short heating or cooling excursion prior to hot rolling. In the In-Situ Homogenization - ISH [1] process, see Figure 1(b), however, we aim to improve the overall thermal efficiency of the process by utilizing some of the available latent heat of fusion during solidification to re-heat the ingot prior to solidification and cooling to room temperature, necessary steps prior to scalping and rolling. These two situations are shown schematically in Fig. 2.



FIGURE 2: Schematic temperature cycle for (a) conventional and (b) in-situ homogenization processing.



Varying the rate of heat extraction during the DC casting process has been used for many years in the casting of certain high-strength aluminum alloys that exhibit particular challenges with stress build-up during (and post) solidification [2]. In a typical embodiment, wipers (which are used to direct the secondary cooling water film off the ingot) are positioned around the periphery of the ingot with the aim of reducing the overall cooling rate. The reduction in cooling rate will affect the build-up of thermal stresses and the ingot may be successfully cast and processed. The ISH process departs from this approach by introducing the wipers at a far earlier stage of the DC casting process, though still within the secondary cooling regime, but prior to the completion of solidification through the ingot cross-section. Thus, ISH is able to utilize both the sensible heat of the ingot and available latent heat of fusion from the semi-solid region to re-heat the surface of ingot. Importantly, since solidification is not complete, the sudden change in cooling will also affect the solid phase growth morphology (e.g. dendrites and cells will become blunted) and there will be increased time for the un-solidified inter-dendritic and inter-cellular liquid to solidify. Consequently this will permit more time for solute diffusion – which is the prime objective for the homogenization process.

Prima facie, this approach, wherein there must be a balance between the normal casting process parameters such as melt temperature, ingot drop rate, mold type, etc. and the position of the wiper system, would appear to be significantly more difficult to control than the traditional DC homogenization approach. While there is no doubt that there are significant differences between this approach and the conventional DC technique, the levels of thermal and process control required are well within the limits of modern DC processing technology. In this paper we will outline some of our

findings from work relating to the development of the ISH process with specific reference to a commercial alloy in Russia, AlMgMn alloy – an alloy for which satisfactory homogenization is critical to the successful subsequent processing to sheet products.

2. Procedure

DC ingots approximately 500mm thick were processed using the Russian designated alloy 1560 (Al-6wt%Mg-0.6wt%Mn) aluminum alloy. Typical casting conditions were used (720 °C trough temperature 50-70 mm/min, 0.5 - 1.5 l/min/cm). During casting, 3mm diameter, stainless steel sheathed, K-type (chromel-alumel) thermocouples were positioned:

1) at the ingot centerline (mid-width),

- 2) at the mid-point between the cast surface and ingot centerline, and,
- 3) in the as-cast surface.

Thermocouples were solidified in place which permitted the direct measurement of local ingot temperature across the slab width. For the tests outlined in this work, wipers, comprised of a compliant, heavy gauge, thermally-resistant polymer were positioned along the quench surface at different times prior to the point that the centerline was completely solidified. This gives rise to a sudden reduction in the heat extraction rate and the ingot establishes a new temperature distribution, with the effect being most strongly observed at the ingot surface. Metallographic samples were extracted from the ingots in close proximity to the thermocouples. These samples were subjected to optical and electron microscope examinations. Quantitative energy dispersive x-ray (EDS) techniques were employed to establish the variations in solute level.

3. Results and Discussion

For comparison purposes, thermocouple measurements were taken from a conventional DC casting process using the same ingot size and casting parameters (i.e. drop rate, melt temperature, water flow rates, etc.). For the given casting conditions we observed near-classic thermal behavior, see Fig. 3.



These included: 1) a solidification plateau at the centerline for about 350 seconds and a total of approximately 500 seconds to pass the non-equilibrium solvus (i.e. the time for the ingot to cool below the AlMg eutectic temperature of 450°C), 2) a near monotonic decrease in surface temperature with distance, 3) at the quarter point the thermocouple exhibited an intermediate

cooling profile, with solidification complete after about 500 seconds and, 4) a total cast plus cooling time of approximately 1,200 seconds. For the ISH casts we used criteria detailed above with the exception of the introduction of the wipers at a point (approximately 8 cm) below the ingot exit from the primary cooling mold. Regardless of the position of the wipers, we observed dramatic changes in the ingot solidification/cooling process and the resulting redistribution of heat/temperature, see Fig. 4. This was monitored via the ingot surface thermocouple - which exhibited the highest temperature increase. The change (increase) in surface temperature and the point it approaches the temperature of the solidifying ingot at the quarter and center positions, we termed the "rebound" temperature.



Features of the ISH cooling curves were similar to those of the conventional DC results until the introduction of the wiper. In the presence of a wiper 1) a solidification plateau at the centerline for about 450 seconds, 2) after rebound occurs there is a small temperature gradient between the surface and centerline temperatures, but 3) it takes about 3600 seconds for the ingot to cool to the AlMg eutectic temperature, and 4) that the reduced rate of heat removal increases the overall total cooling time to approximately 6000 seconds, a time which extends well beyond the ingot's removal from the casting pit. One would expect that given this extra time above the eutectic, in the presence of both solid and liquid phases, a visible change in the decrease in the level of micro-segregation and population density of the Al₈Mg₅ phase.



Fig. 5 Conventionally processed 1560 ingot optical microstructures at (a) surface, (b) ¹/₄ thickness and (c) centerline. (NH₄BF₄ electrochemically etched.)

Optical microscopy of the conventionally cast ingot revealed a typical cored AlMg microstructure with a population of eutectic material at the grain, cell/dendrite boundaries with the extent of it being greater towards the surface of the ingot, diminishing progressively towards the cast centerline, see Fig. 5. Clearly visible are the overall grain structures comprised of cored cellular/dendritic AlMg primaries with occasional patches of the Al₈Mg₅. The ISH processed ingot, see Fig. 6, showed a far decreased level of coring particularly at the surface and ¹/₄ thickness locations. Indeed, even close to the cast surface, the ISH processed ingot displayed a microstructure devoid of Al₈Mg₅ eutectic.



Fig. 6 ISH processed 1560 optical microstructures at (a) surface, (b) ¼ thickness and (c) centerline. (NH₄BF₄ Electrochemically etched)

In order to investigate this more quantitatively we used SEM/EDS compositional techniques similar to that proposed by Lacaze et al [3]. The procedure involves taking a large number of point analyses (we used 400 points for each sample) on a regularly-spaced grid that comprises a statistically significant (1500 μ m x 1500 μ m) representative area of the cast structure. By ordering the collected values of composition from the most solute rich to the lowest solute level, we obtain an overall elemental composition map for the particular alloy, see Fig. 7.



Fig. 7 Ordered EDS composition for Conventional and ISH processed ingot at the centerline, for elemental Mg contrasted to fraction liquid at the time of solidification.

The range of effective partition coefficient (k_{eff}) values (0 being Gulliver-Scheil and 1 being equilibrium) observed fall within those that would be expected in a conventionally homogenized ingot. From the data it is clear that the ISH processed material exhibits far lower levels of compositional variation that does the conventionally processed ingot. Since the EDS sampling volume is small compared to the size of the solidification structure, we can use a simple binary solidification model to fit the compositional profile for nonequilibrium solidification (viz Gulliver-Scheil) and back-out from that a value for, k_{eff} .

As per Lacaze's analysis, such values are an indication of the real partitioning during the particular solidification process. We found that the conventionally cast material yielded a k_{eff} of 0.43, while the ISH material exhibited a value of 0.78. That is, the ISH material showed a systematically lower level of solute segregation. The explanation for this effect, we believe, lies primarily in the extra time that the semi-solid alloy spends above the eutectic temperature. Under such conditions we would expect both solid state diffusion (which is what occurs in conventional homogenization practices) as well as a protracted period for solid-liquid diffusion as the material spends a significantly longer time than conventionally cast DC ingot in the two phase region. We believe that this is the origin of the improved ingot homogeneity.

While, from a practical standpoint, the effects of improved ingot homogeneity offered by the In-Situ Homogenization process have to be considered in terms of the overall sheet making process (and the alloys under consideration), the process offers advantages to improve the overall energy requirements for ingot production by reducing, if not eliminating, the need for a formal homogenization processing step. The results of this investigation, though for an AlMgMn alloy system, indicate that similar opportunities for the ISH process should exist in other aluminum (and nonaluminum) alloys systems that use DC casting for ingot production

4. Conclusions

- 1. It is possible to configure a conventional DC casting apparatus to take advantage of the heat available while still in the semi-solid to decrease the level of micro-segregation in an ingot.
- 2. Optical microcopy and EDS analyses confirm that the extent of solute micro-segregation of Mg in an AlMgMn alloy is significantly reduced by the use of the ISH process as compared to the conventional DC casting process.

Acknowledgement

The authors wish to thank Novelis Inc. for permission to publish this paper. We also wish to recognize the efforts of the many engineers and technicians who assisted in producing the samples and preparing the analysis for the discussion in this paper.

References

- [1] U.S. Patent No. 7,516,775. R.B. Wagstaff, (April 14 2009)
- [2] U.S. Patent No. 4,237,961. T.C. Zinninger, (December 9 1980)
- [3] A. Hazotte, J.S. Lecomte and J. Lacaze, Mat. Sci & Eng'g, A413-414 (2005): p223-228.