Microstructure Evolution in Melt Conditioned Direct Chill (MC-DC) Casting of Fe–Rich Al-alloy

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Abstract

In order to fabricate high quality aluminium products, it is first essential to produce high quality billets/slabs. One of the key objectives associated with casting processes is to be able to control the as-cast structure. A novel direct chill (DC) casting process, the melt conditioned direct chill (MC-DC) casting process, has been developed for production of high quality aluminium billets. In the MC-DC casting process, a high shear device is submerged in the sump of the DC mould to provide intensive melt shearing, which in turn, disperses potential nucleation particles, creates a macroscopic melt flow to uniformly distribute the dispersed particles, and maintains a uniform temperature and chemical composition throughout the melt in the sump. The effect of intensive shearing on the complex microstructure evolution observed after MC-DC is explained on the basis of nucleation and growth behavior. Complete suppression of typical columnar grain growth and significant equiaxed grain refinement is observed. The solidification mechanisms responsible for the significant grain refinement by intensive shearing and the morphological evolution of Mg_2Si and Fe–containing intermetallic phases are discussed.

Introduction

Microstructure controls the mechanical performance and processability of metallic materials. The mechanical properties of any alloy component are greatly enhanced by fine grain size and intermetallic phases. A fine and equiaxed grain structure imparts uniform distribution of second phase, higher yield strength, higher toughness, good extrudability, improved machinability, good surface finish, resistance to hot tearing and various other desirable properties [1-4]. Therefore, understanding the evolution of solidification microstructure and ways to control it, are of broad scientific interest and technological importance.

Al alloys have widespread industrial usage in the areas of automotive, aerospace and military due to their excellent castability, high specific strength, superior corrosion resistance and good wear properties [1,4]. It is standard practice to modify the solidification microstructure of Al alloys in order to control the morphology and distribution of different phases and improve both castability and mechanical property [2-6]. Explicitly, the production of wrought Al alloys starts with direct chill (DC) casting to produce billets or slabs of convenient shape and size for thermo-mechanical processing. Some of the problems encountered in conventional DC casting are formation of coarse and non-uniform microstructure, severe chemical segregation, undesirable morphologies of Fe–containing intermetallics, gas and shrinkage porosity together with hot tearing, which all have a detrimental effect on rollability and extrudability and in turn result in poor mechanical properties of both semi-fabricated and finished products [5,7]. Achieving a fine and equiaxed grain structure, whilst preventing cracks and hot tearing, is typically achieved by chemical grain refinement. In the Al metal casting industry, for instance, it is common practice to introduce nucleating agents to a liquid metal, referred to as inoculation, for the formation of fine, uniform and equiaxed non-dendritic grains. Al alloys are commonly inoculated by Al–Ti–B or Al–Ti–C type grain refiners [8]. However, addition of grain refiner is not widely known to modify the Fe–containing intermetallics and eutectic morphology and additionally the grain refiner particles and the unmodified

intermetallics all make it difficult to recycle the alloy [4]. Therefore, as an alternative to chemical grain refining, physical means have been exposed for microstrucutre modification of Al and Mg alloys such as rapid quenching, ultrasonic vibration, electromagnetic vibration and intensive shearing.

In this paper, we present the effect of intensive shearing on the complex microstructure evolution on the basis of nucleation and growth behavior of primary α–Al phase and the morphological evolution of Mg_2Si and Fe–containing intermetallics. The discussion is mainly focused on the solidification process under intensive shearing and outlines the advantages of the MC-DC casting process.

Experimental procedures

Aluminium alloy of the chemical composition, Al-3Si-2Mg-0.5Mn-1Fe was prepared in a clay graphite crucible in an electric resistance furnace by dissolving the corresponding hardeners at 760°C and holding the melt for 2 hours for homogenisation. All compositions in this article are given in weight percent unless otherwise stated. The alloy composition was checked by optical emission spectroscopy and degassed by using Nitral C19 tablet (2 g/Kg), prior to casting.

The casting experiments were carried out on a DC caster fitted with an 80 mm diameter mould. The melt was poured at 740 ± 3°C into the DC casting mould via a launder system to fill the hot-top and then held for ~15 s to ensure adequate shell formation before starting to cast at a steady speed of 220 mm/min with a water flow rate of ~25 liters/min. The MC-DC process is shown schematically in Fig. 1, where the high shear rotor-stator unit is submerged in the sump of a conventional DC mould to provide intensive melt shearing during casting [9]. The shearing device was pre-heated to 750°C before being submerged into the melt. The high shear device was operated at rotational speeds of 2000 and 5000 rpm. During shearing, liquid metal is drawn into the high shear device and is subjected to intensive shearing between the rotor and stator and then forced through the holes on the stator as fine jet at very high velocity. This escaping liquid forms a unique macroscopic flow pattern in the sump, as shown in Fig. 1.

The cast billets measured about 500 mm in length and were sectioned longitudinally for extracting samples for microscopy which were then prepared by grinding on SiC papers and polished using silica suspension. The un-etched samples were analysed for the microstructure and intermetallic phases using a ZEISS Axioscop2 MAT optical microscope equipped with AxioVision image analyzing software. In order to properly observe the grain structure, samples were also electro-etched by anodizing in Barker's reagent (7 ml HBF_4 48%, 93 ml H_2O) at 20 V for 70 s using a stainless steel cathode.

Results

Computational thermodynamics: The alloy composition in this work was specifically chosen to explore the formation of various Fe–containing intermetallics. By using the CALPHAD software package, a vertical phase diagram section of the Al-3Si-2Mg-0.5Mn-Fe alloy system was calculated, as shown in Fig. 2. The sequence of phase transformations (solidification) of the chosen alloy according to non-equilibrium thermodynamic calculations is listed in Table 1. The solidified microstructure of this alloy after conventional DC casting is shown in Fig. 3 (a–c). The individual phases are labelled on the given micrograph. At first, the solidification commences at 648.96°C with the formation of solid $Al_{15}(Fe,Mn)_3Si_2$ intermetallic phase, followed by the α–Al phase when the temperature drops to 629.86°C. The volume fraction of α–Al phase increases until all the liquid is consumed till the temperature reaches to 610.86°C. Subsequently, formation of α–AlFeSi, β–AlFeSi, Mg_2Si, and $Al_8(Fe,Mg)_3Si_6$ intermetallic phases starts sequentially as the temperature drops to 610°C, 598.14°C, 568.36°C and 564°C, respectively. The calculated volume fractions of α–AlFeSi, β–AlFeSi and $Al_8(Fe,Mg)_3Si_6$ phases are 0.5, 0.7, and 0.4, respectively. In this multi-element alloy system, the phase transformation is quite complex which makes it difficult to identify these individual phases by optical microscopy. It should be noted, however, that the exact

temperature of phase transformations may differ from the computational thermodynamics calculation. The primary objective in this work is to investigate the effect of intensive melt shearing on the morphological formation and distribution of the α–Al primary phase, the Fe–containing intermetallics and the Mg_2Si phase.
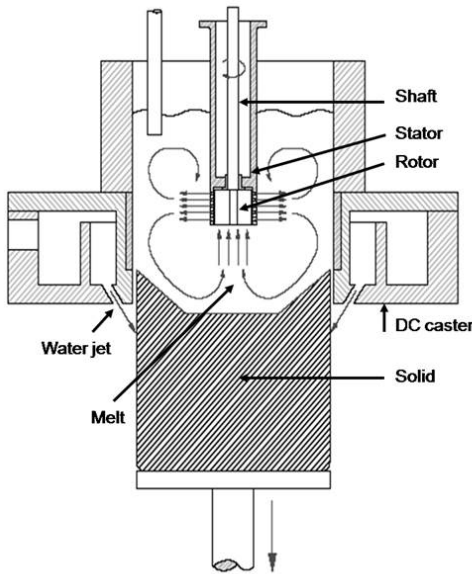

Fig. 1. Schematic illustration of the melt conditioned direct chill casting (MC-DC) process, with the high shear device submerged in the sump of a conventional DC casting mould, also showing the macroscopic melt flow pattern generated by intensive shearing.
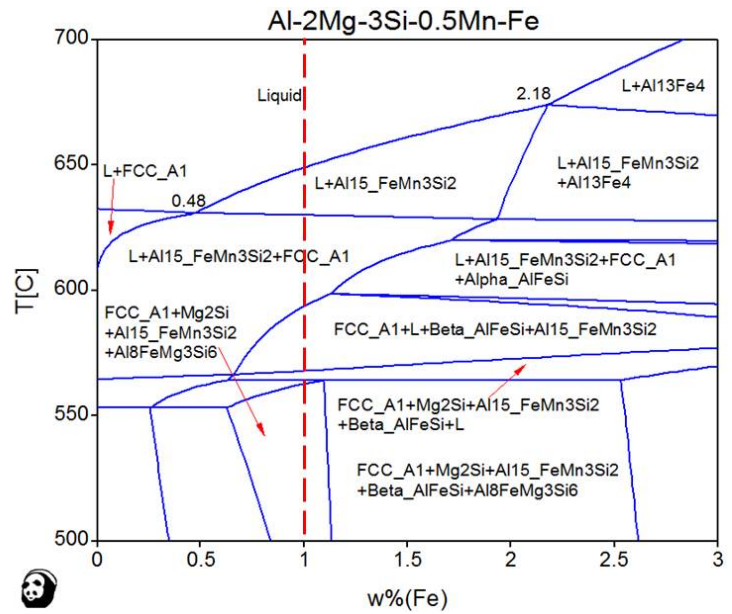

Fig. 2. CALPHAD calculated vertical phase diagram section of the Al-3Si-2Mg-0.5Mn-Fe alloy system.

Table 1. Calculated sequence of non-equilibrium (Scheil model) phase transformation states of the Al-3Si-2Mg-0.5Mn-1Fe alloy system.

Phase transformation temperature (°C)	Phase reactions
> 648.96	Liquid
648.96 - 629.86	Liquid – $Al_{15}(Fe,Mn)_3Si_2$
629.86 - 610.86	Liquid – $Al_{15}(Fe,Mn)_3Si_2$ + α–Al
610.86 - 598.14	Liquid – $Al_{15}(Fe,Mn)_3Si_2$ + α–Al + α–AlFeSi
598.14 - 568.36	Liquid – $Al_{15}(Fe,Mn)_3Si_2$ + α–Al + β–AlFeSi
568.36 - 564.06	Liquid – $Al_{15}(Fe,Mn)_3Si_2$ + α–Al + β–AlFeSi + Mg_2Si
564.04 - 553.19	Liquid – α–Al + Mg_2Si + $Al_8(Fe,Mg)_3Si_6$

Effect of intensive shearing on the solidification microstructure: In order to establish the effect of intensive shearing on the selected Fe–rich Al alloy, three main experiments were performed on the DC caster; (i) conventional DC casting with no shearing (0 rpm), (ii) MC-DC casting at a low shear speed of 2000 rpm and (iii) MC-DC casting at a high shear speed of 5000 rpm. Optical micrographs obtained from the billet DC cast without shearing are presented in Fig. 3 (a–c), illustrating the overall microstructure showing the α–Al phase, Fe–containing intermetallics and Mg_2Si morphologies. The microstructure of the non-sheared billet consists of primary α–Al dendrites with fully grown primary and secondary arms, a few millimeters in size, mainly separated by the Fe–containing intermetallic and Mg_2Si phases. The microstructures of the MC-DC billets cast at low and high shearing speeds are given in Fig. 3 (d–i). It is clearly observed, that with intensive melt shearing, the microstructure becomes more uniform and the grain structure is significantly reduced in size at both shearing speeds. The results from the microstructure analysis are given in Table 2. The grain size of the DC cast billet without shearing was measured to an average size of 3 mm compared to an average size of 300 μm obtained by MC-DC casting at shearing speed of 2000 rpm and 120 μm at shearing speed of 5000 rpm. The morphology of the α–

Al grain was also observed to have changed from coarse dendritic structure to a fine dendritic structure upon shearing at low intensity followed by a fine and equiaxed rosette type structure upon shearing at high intensity.
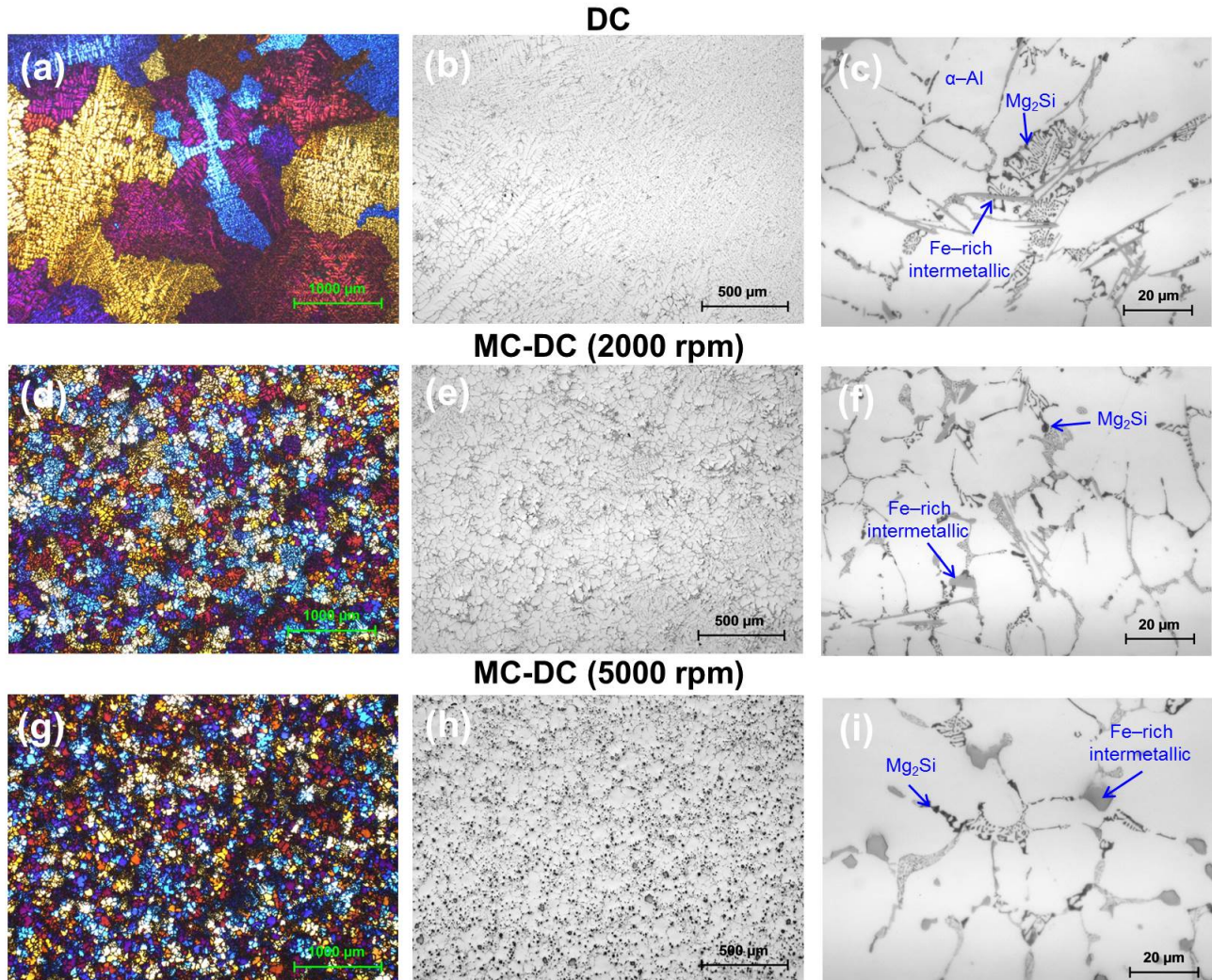

Fig. 3. Optical micrographs of the Al-3Si-2Mg-0.5Mn-1Fe alloy billets solidified with and without shearing during DC casting: (a), (d), (g) illustrating the overall change in grain structure (anodized samples), (b), (e), (h) overall un-etched microstructure, and (c), (f), (i) showing the morphological change of the Fe–containing intermetallics and distribution of the Mg_2Si phase.

Table 2. Comparison of the results obtained from the microstructure analysis of the DC and MC-DC billets, showing the size reduction of the α–Al grain and Fe containing intermetallic particles.

Casting	Shearing speed (rpm)	Avg. primary α–Al grain size (μm)	Avg. Fe–containing intermetallic particles size (μm)
DC	0	3000	20.6 (Plate-like)
MC-DC	2000	300	11.5 (Polygonal)
MC-DC	5000	120	4.3 (Globular)

The high magnification un-etched microstructure (Fig. 3c) of the DC cast billet shows the Fe–containing intermetallic phase formed with a long plate–like morphology and the Mg_2Si phase formed with the typical Chinese script–like morphology. By application of intensive shearing, a most prominent morphological change is observed for both the Fe–containing intermetallics and the Mg_2Si phase. The Fe–containing intermetallic is observed to form two distinct morphologies; large number of polygonal and compact plates after shearing at low intensity (Fig. 3f), and a more compact and globular morphology after shearing at high intensity (Fig. 3i). The plate-like morphology was measured to be an average size of 20.6 μm, compared to shearing at low and high intensity where the polygonal morphology measured 11.5 μm and the compact and globular

morphology measured 4.3 μm, respectively (Table 2). Similar morphological change was also observed with the Mg$_2$Si phase becoming more compact and finely distributed especially with shearing at high intensity.

Discussion

It is clear from the DC and MC-DC results, that the application of intensive melt shearing has a significant grain refining effect and can improve the uniformity of the microstructure in the as-cast billets. It is also believed that, the intensive shearing not only has an important contribution to the microstructural grain refinement but also reduced or eliminated chemical segregation. In order to understand primary α–Al grain refining mechanism by intensive shearing, temperature measurements inside the hot top and casting mould have revealed that, in conventional casting the temperature in the sump is quite non-uniform and the further the distance from the solidification front, the higher the melt temperature [5]. However, with application of intensive melt shearing in the sump, the temperature is more or less uniform throughout the hot-top and the mould, mainly due to the unique macroscopic melt flow pattern created during shearing. The temperature curves also show that, after application of the intensive shearing, the melt temperature becomes very close to the liquidus temperature of the alloy being cast, eventually reaching a plateau which is slightly lower that the liquidus temperature [5]. Additionally, liquid melts of Al and Mg alloys inevitably contain oxide in forms of clusters or films with a non-uniform distribution. Such oxides may act as substrates for nucleation but are not effective for grain refinement due to their poor wettability and low number density. However, such oxide clusters and films can be dispersed effectively by intensive melt shearing, which is known to enhance heterogeneous nucleation and promote grain refinement [2,10]. Similar sets of grain refinement results have also been achieved in various Al wrought alloys of the 2xxx, 6xxx and 7xxx series [5], including some experimental cast alloys as well and various Mg alloys such as AZ91 and AZ31 [3].

A lot of work has been reported on the refinement of the primary α–Al phase by application of shearing during DC casting with limited focus on its effects on the morphology of Fe–containing intermetallics and other solidification phases. In the present work, significant refinement and morphological transformation from long plate-like to fine globular Fe–containing intermetallic particles have been observed by application under intensive shearing. Similar coarsening and globularisation effect is also observed for the Mg$_2$Si phase. The relative domination of the overall enhanced heterogeneous nucleation may have an important contribution to the morphological evolution of the Fe–containing intermetallics. It has also been reported, that some of the intermetallic phases such as primary α–Fe(AlFeSi), β–Fe(Al$_5$FeSi), and π–FeAl$_8$(Fe,Mg)$_3$Si$_6$ have been known to nucleate on double oxide films [7]. In the case of processing with intensive shearing, the already existing oxide clusters and films are well dispersed and distributed throughout the volume of the melt hence this can also promote nucleation of the intermetallic phases and in turn suppress their subsequent and irregular growth. Therefore, the significant refinement and morphological change of Fe–containing intermetallics and the Mg$_2$Si phase can be attributed to the following factors;

- Dispersion and distribution of fine oxide particles generated by intensive shearing increases the number of potent sites for nucleation of the subsequent intermetallic phases,
- The morphology of α–Al phase, governs the subsequent intermetallic phase formation,
- Homogenisation of the solute composition at the intermetallic growth front due to the forced convection created during shearing, suppresses the typical directional growth and promotes spherodisation resulting in a more compact and polygonal intermetallic morphologies.

In summary, the main advantages of the MC-DC casting process can be outlined as to; promote physical grain refinement, achieve uniform microstructure with homogeneous chemical composition, refinement on secondary intermetallic phases into less detrimental morphologies for enhanced mechanical properties and most importantly, the MC-DC process can be applied to benefit recycling of scrap Al-alloys which mainly contain Fe as the major contaminant.

Conclusions

1. MC-DC casting of a Fe-rich Al-Si-Mg alloy system has been investigated in comparison with conventional DC casting and the resulting solidification microstructures cast under identical DC casting parameters have been reported and discussed.
2. Formation of coarse dendritic α–Al grains is completely suppressed and significantly refined grains are promoted under intensive melt shearing.
3. The strong fluid flow generated by the intensive shearing and the dispersion of oxide particles with increased potency for nucleation is believed to suppress the formation of large intermetallic particles and results in significant size reduction and morphological change to facilitate the enhancement of mechanical properties.
4. The results obtained in this work demonstrate that, the MC-DC process can benefit recycling of scrap metals and also presents an opportunity for producing *in-situ* composite billets.

Acknowledgements

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