Magnesium Technology 2012
Edited by: Suveen N. Mathaudhu, Wim H. Sillekens, Neale R. Neelameggham, and Norbert Hort
TMS (The Minerals, Metals & Materials Society), 2012

MELT CONDITIONED DC (MC-DC) CASTING OF MAGNESIUM ALLOYS

Y B Zuo, B Jiang, Y Zhang, Z Fan
BCAST, Brunel University, Uxbridge, Middlesex, UB8 3PH, UK

Keywords: DC casting, Melt conditioning, Grain refinement, Microstructure, Magnesium alloy.

Abstract

A new melt conditioned direct chill (MC-DC) casting process has been developed for producing high quality magnesium alloy billets and slabs. In the MC-DC casting process, intensive melt shearing provided by a high shear device is applied directly to the alloy melt in the sump during DC casting. The high shear device provides intensive melt shearing to disperse potential nucleating particles, creates a macroscopic melt flow to distribute uniformly the dispersed particles, and maintains a uniform temperature and chemical composition throughout the melt in the sump. Experimental results have demonstrated that the MC-DC casting process can produce magnesium alloy billets with significantly refined microstructure and reduced cast defects. In this paper, we introduce the new MC-DC casting process, report the grain refining effect of intensive melt shearing during the MC-DC casting process and discuss the grain refining mechanism.

Introduction

Magnesium alloys are used in a wide variety of applications including automotive, aerospace, defense, electronics, healthcare and sports equipment because of their low density, high specific strength, high damping capacity, good castability and excellent electromagnetic shielding properties [1, 2]. However, due to their specific crystallographic structure, Mg-alloys have only a limited number of slip systems available, and therefore are difficult to deform at low temperatures. This poor deformability means low productivity and high processing cost, and consequently limited engineering applications. It is well accepted that a fine grain size can result in both improved formability and enhanced mechanical properties [3, 4]. In addition, fine and equiaxed solidification can reduce chemical segregation, hot tearing and formation of other casting defects [5], especially when producing large billets.

DC casting is a major technology for processing wrought magnesium alloys. However, the billets or slabs produced by the conventional DC casting process often have coarse and non-uniform microstructures, severe chemical segregation, porosity and hot tearing, which not only burden the downstream thermo-mechanical processing but also have a negative influence on the mechanical properties of the final products. Grain refinement by inoculation with chemical grain refiners is an effective approach for mitigating such problems [6, 7]. Although grain refinement of magnesium alloys has attracted intensive research [2, 8-10] the search for new and effective grain refiners and methods for grain refinement still continues. Up to date, the research on grain refinement of magnesium alloys is mainly focused on searching for new chemical grain refiners and the application of external physical fields during solidification processing, such as electromagnetic field, electrical pulsing and ultrasonic fields [11]. Recently, we found that intensive melt shearing provided by a twin-screw mechanism has a significant grain refining effect on both aluminium and magnesium alloys [12-15]. Based on a similar principle, a new high shear device has been developed for conditioning liquid metals prior to solidification processing [16], and the high shear device has been integrated into the conventional DC casting process to form a melt conditioned DC casting process for production of high quality Mg-alloy billets or slabs.

This paper introduces the newly developed MC-DC casting process with intensive melt shearing, reports our experimental results on microstructural refinement of MC-DC cast Mg-alloys and discusses the grain refining mechanisms.

Experimental Procedure

Commercial AZ91D Mg-alloy (Mg-8.67%Al-0.62%Zn-0.21%Mn, all compositions are in wt%), and AZ31 Mg-alloy (Mg-2.92%Al-0.85%Zn-0.36%Mn) were used in the present work. The measured liquidus is 600°C for AZ91D and 630°C for AZ31. The alloys were melted at 680°C in a steel crucible under a protective atmosphere containing a gas mixture of N_2 and 0.5 vol% SF_6. Mg-alloy billets were DC cast with and without intensive melt shearing. Figure 1 is a schematic illustration of the MC-DC casting process, which is effectively a combination of a conventional DC caster with the high shear device submerged in the sump of the caster [16]. The alloy melt was poured with 50K superheat, and the high shear device was operated at 5000-15000RPM. The same protective gas was supplied to the hot top during the DC casting process. 80mm diameter Mg-alloy billets were produced under the same conditions but with or without the high shear device being switched on.

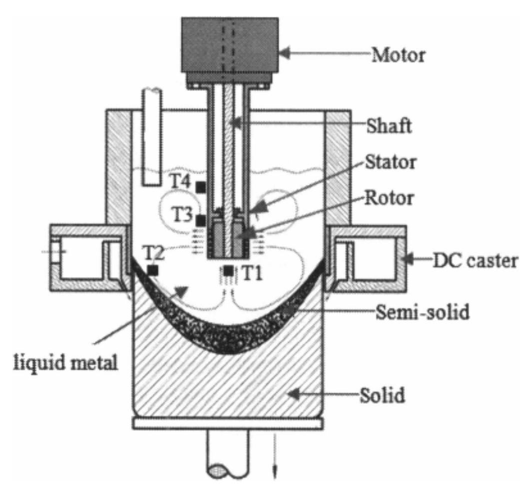

Figure 1. Schematic illustration of the melt conditioned direct chill (MC-DC) casting process, which is effectively a conventional DC caster with a high shear device submerged in the melt sump. The high shear device provides intensive melt shearing to disperse potential nucleating particles and a macroscopic flow pattern to homogenize the melt temperature and the composition in the sump. T1-T4 indicates the positions of thermocouples for temperature measurement.

Samples for both macroscopic and microscopic examinations were cut, prepared and then examined according to standard metallographic procedures. To further understand the effect of melt shearing during DC casting on the microstructure, the temperature field was measured by using thermocouples attached to the high shear device and a data logger during the DC casting process. T1-T4 in Figure 1 schematically indicate the positions of the thermocouples for measuring the melt temperature.

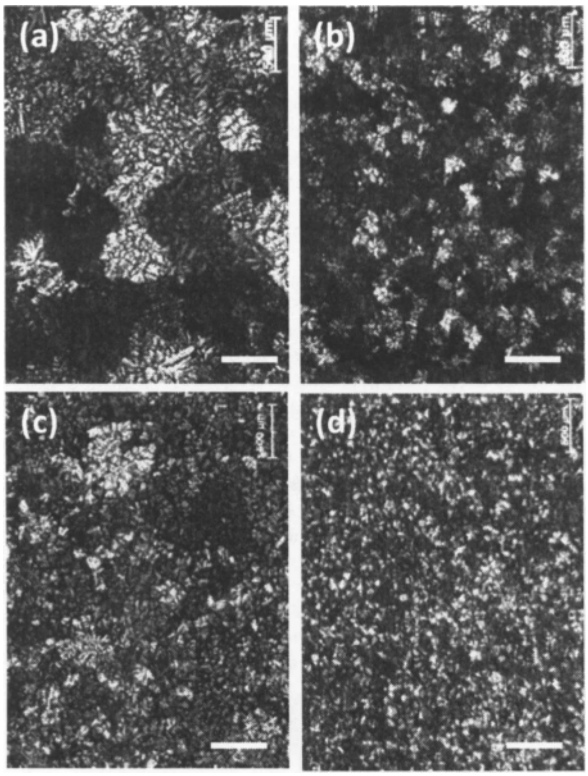

Figure 2. Microstructure of AZ91D magnesium alloy cast in a TP1. (a) 650℃, without shearing; (b) 650℃, with shearing; (c) 620℃, without shearing (d) 620℃, with shearing. The size bars in all the micrographs are 500μm.

Results

Microstructure of AZ91D Alloy with Intensive Melt Shearing

To demonstrate the effect of intensive melt shearing on the grain refinement of Mg-alloys, AZ91D alloy melts were sheared at different temperature and cast in TP1 mould to assess the grain size. Figure 2 shows the microstructures of AZ91D alloy cast at 650°C and 620°C in the TP1 mould with and without intensive melt shearing. The TP1 mould was operated under the standard conditions for grain size assessment with a constant cooling rate of around 3.5 K/s for aluminium alloys [17]. With intensive melt shearing, the microstructure becomes more uniform and the grain size is significantly reduced at both casting temperatures. The as-cast grain size was reduced from 550μm to 170μm when cast at 650°C (Figures 2a and 2b), and from 180μm to 90μm when cast at 620°C (Figures 2c and 2d). It is interesting to note from Figure 2 that the grain size of Mg-alloys has a strong dependence on melt superheat, and that such superheat effect is suppressed significantly by intensive melt shearing.

Microstructure of MC-DC Cast AZ91D Alloy

Figure 3 shows the macroscopic structures of the 80mm diameter AZ91D alloy billets produced by DC casting at 650°C with and without intensive melt shearing. Although both billets have an equiaxed structure, the grain structure is much finer and more uniform in the billet with intensive melt shearing. In addition, chemical analysis using optical emission spectroscopy indicates that there is no macro-segregation along either the longitudinal or the radial directions in both types of billets.

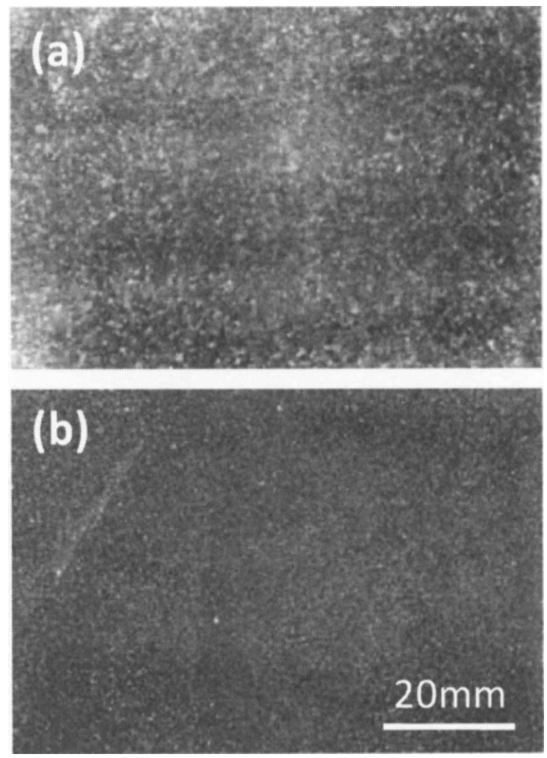

Figure 3. Macrographs of sectioned DC cast AZ91D alloy ingots cast at 650°C with and without shearing.

The microstructures of the AZ91D billets DC cast with and without intensive melt shearing are shown in Figure 4. The conventional DC cast billet (without intensive melt shearing) shows a coarse equiaxed dendritic microstructure with an average grain size of around 500μm near to the ingot surface (Figure 4a) and a coarser equiaxed grain structure with an average grain size of 700μm at the centre of the billet (Figure 4b). However, the AZ91D billet produced by the MC-DC process (with intensive melt shearing) have a consistently fine and equiaxed grain structure throughout the entire billet with an average grain size of 190μm (Figures 4c and 4d). Figure 5 compares the grain size variation between the AZ91D alloy billets produced by DC casting with and without intensive melt shearing. For the conventional DC cast billet, there is a thin layer with finer microstructure with an average grain size of 500μm, and apart form that layer, the microstructhre is dominated by coarse grains with an average grain size of 700μm. However the billet DC cast with intensive melt shearing shows a uniform grain size distribution through the entire cross section, with an average grain size of 190μm.

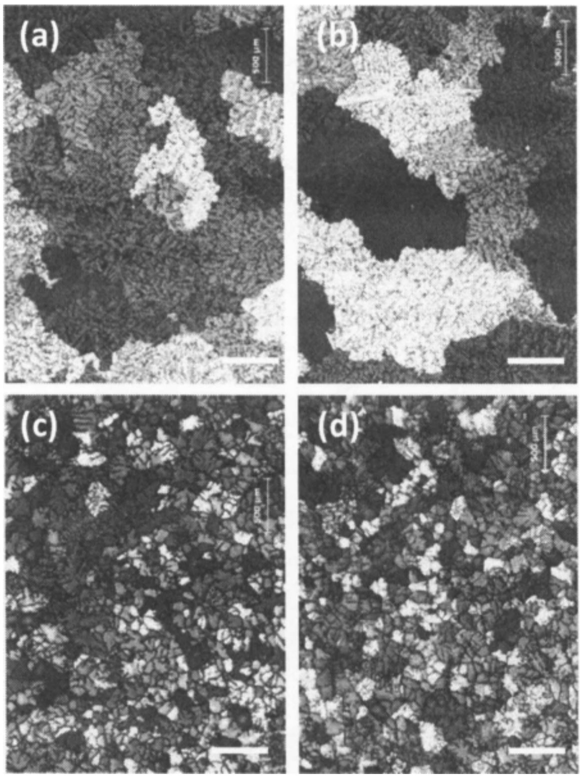

Figure 4. Comparison of the microstructures of DC cast AZ91D alloy billets at 650°C with and without intensive melt shearing. (a) edge without shearing; (b) centre without shearing; (c) edge with shearing; (d) centre with shearing. The size bars in all the micrographs are 500μm.

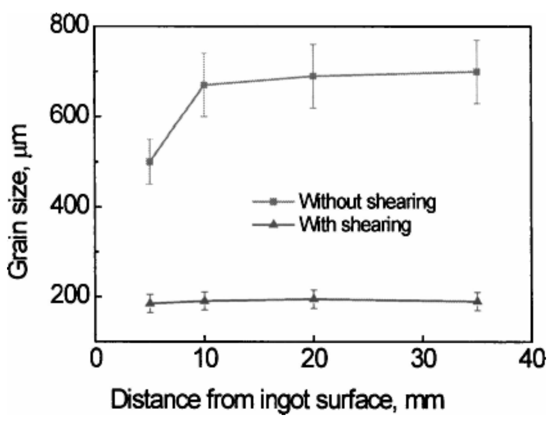

Figure 5. Variation of grain size as a function of the radial distance from the billet surface in AZ91D alloy billets produced by DC casting with and without intensive melt shearing.

In order to further demonstrate the effect of *in situ* intensive melt shearing on the microstructure of DC cast Mg-alloy billets, AZ91D alloy was cast by the newly developed MC-DC casting process by switching on and off the high shear device. The high shear device was submerged in the sump of the DC caster, and was switched off during the first half of the billet casting and switched on during the second half of the billet casting. Figure 6 shows the microstructure of DC cast AZ91D alloy with the transition from the section cast with high shear device off (bottom) to the section with the high shear device on (top). Without intensive melt shearing, the DC cast AZ91D alloy shows a coarse dendritic microstructure, while with intensive melt shearing it turns sharply into a fine and equiaxed microstructure. From the transition up wards, the AZ91D alloy billet maintains the same fine and uniform microstructure.

It is clear from the above results that during the DC casting process, the application of intensive melt shearing has a significant grain refining effect on AZ91D magnesium alloy and can improve the uniformity of the microstructure in the as-cast billets.

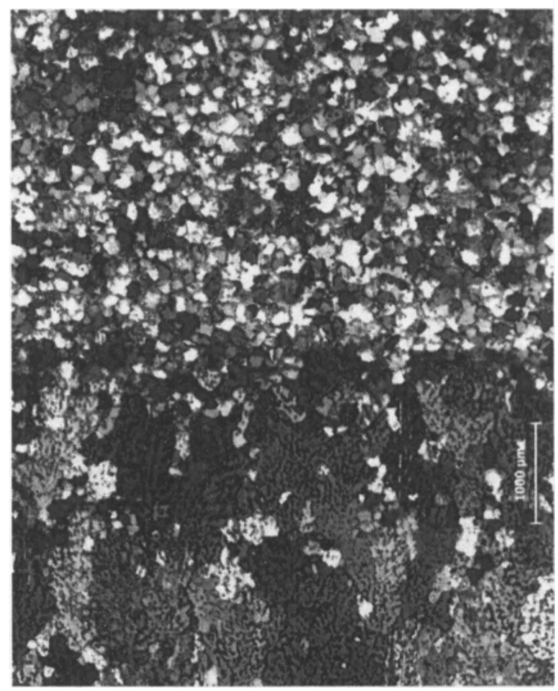

Figure 6. Microstructure of DC cast AZ91D alloy billet at 650°C with (top) and without (bottom) intensive melt shearing, showing the transition from coarse dendritic grain structure without intensive melt shearing (high shear device off) to a fine and equiaxed grain structure with intensive melt shearing (high shear device on).

Microstructure of DC Cast AZ31 Alloy

To further confirm the grain refining effect of intensive melt shearing, the AZ31 magnesium alloy ingots were cast at 680°C with *in situ* intensive melt shearing. Figure 7 shows the microstructure of AZ31 Mg-alloy ingot without (Figure 7a) and with (Figure 7b) intensive melt shearing. The microstructure of conventional DC cast AZ31 alloy ingot shows coarse columnar dendrites with a dendrite cell size of a few mm (Figure 7a);

however, with the application of intensive melt shearing the microstructure becomes finer (less than 200μm) and the grain morphology is also changed to equiaxed. This clearly demonstrates the power of intensive melt shear for grain refinement of Al-containing Mg-alloys.

It is well know that grain size is strongly dependent on the concentration of solute elements; the higher the solute concentration, the smaller the grain size due to the increased growth restriction factor with the increase of solute concentration. However, a comparison between Figures 4 and 7 suggests that intensive melt shearing during DC casting may significantly reduce the grain size dependence on solute contents.

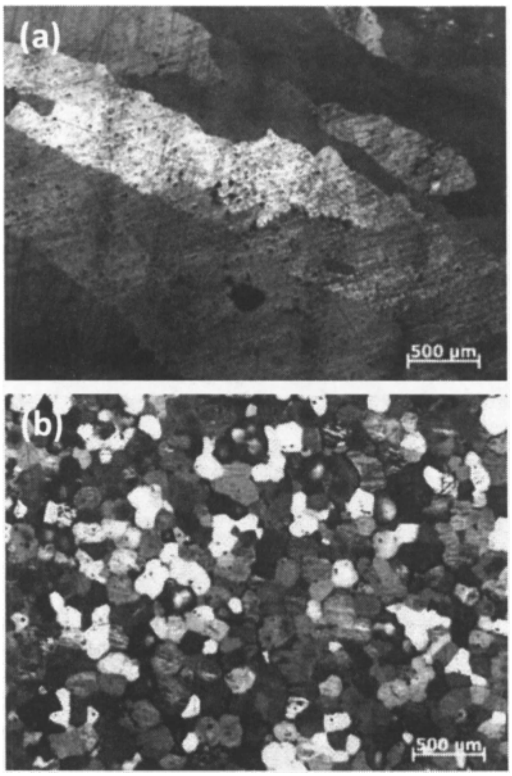

Figure 7. Microstructure of AZ31 alloy DC cast at 680°C (a) without and (b) with *in situ* intensive melt shearing, showing an order of magnitude grain size reduction by application of intensive melt shearing.

Temperature Uniformity during DC Casting

To facilitate the understanding of grain refining mechanisms by intensive melt shearing, temperature measurement during DC casting was carried out. The recorded temperatures during DC casting of AZ91D magnesium alloy are shown in Figure 8, which was obtained through the thermocouples attached to the head of the high shear device. The positions of thermocouples (T1-4) are schematically shown in Figure 1. Based on the direct measurement of the sump depth with stainless steel rods, during the casting process, the bottom of the high shear device is about 40 mm above the liquid/solid interface. Without melt shearing, the melt temperature in the sump is expected to be non-uniform; the further the distance from the solidification front, the higher the melt temperature. However, with the start of the high shear device, there is a sharp change of melt temperature distribution in the sump. Intensive melt shearing in the sump made the temperature curves become closer together, resulting in a more or less uniform melt temperature in the sump. The temperature curves also show that after the application of intensive melt shearing, the melt temperature becomes very close to the liquidus and eventually reaches a plateau which is around 7°C lower than the alloy liquidus. This could make an important contribution to the grain refinement of microstructure.

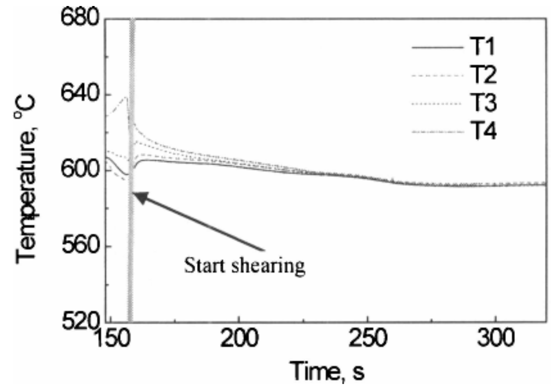

Figure 8. Temperature curves measured during DC casting of AZ91D alloy. T1, centre, 10 mm below the bottom of the high shear unit; T2, edge, 10 mm below the bottom of the high shear unit; T3, near to the centre, 30 mm above the bottom of the high shear unit; T4, near to the centre, 90 mm above the bottom of the high shear unit.

Discussion

The experimental results presented in the previous section clearly demonstrate that the MC-DC casting process is a simple and effective technology for production of high quality magnesium alloy billets (or slabs). Compared with the conventional DC casting process, the newly developed MC-DC process has the following anticipated advantages:

- Significant grain refinement
- Uniform and equiaxed microstructure
- Reduced/eliminated macro-segregation
- No large inclusions and oxide films
- Reduced/eliminated hot tearing
- Reduced sensitivity to alloying contents
- Easy to implement within existing DC casting facilities

Further research and development of the MC-DC process is under the way before industrial implementation.

The high shear device is the key element in the MC-DC process to deliver the above mentioned advantages. The high shear device provides *in situ* conditioning of the liquid alloy during the DC casting process. The major function of the high shear device can be summarized as follows:

A high shear dispersive mixing action: The centrifugal force due to the rotation of the rotor generates a negative pressure inside the stator, which in turn sucks the melt below the high shear device into the stator and forces it to escape at high speed through the openings in the wall of the stator. This process subjects the melt to a high shear action in the gap between the rotor and the stator and the in the openings in the stator wall. The shear rate could be as high as 10^5/s. As a consequence of this intensive melt shearing the clusters of inclusions and oxide films naturally occurring in the melt will be dispersed into individual particles [12], as shown in Figure 9. It is well documented that naturally occurring oxide films are hazardous to casting processes, and more importantly they deteriorate mechanical properties of the final cast products [18, 19]. In the MC-DC process, the intensive melt shearing makes the usually harmful oxide films and other inclusions not only harmless, but also useful for grain refinement.

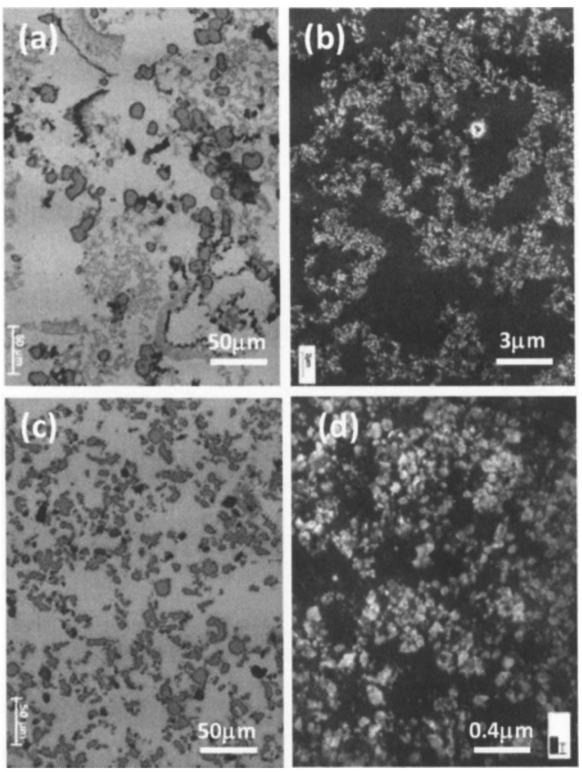

Figure 9. Optical micrographs (a, c) and SEM micrographs (b, d) showing the physical status of oxide in AZ91D alloy. Without intensive melt shearing oxide exists in the melt in the form of oxide films (a, b) consisting of discrete nano-sized MgO particles (b); after intensive melt shearing, oxide films become invisible under optical microscope (c) and are dispersed into more individual particles (d). All the samples here are produced by pressurized melt filtration. The large grey particles in (a) and (c) are Al-Mn intermetallic particles.

<u>A macroscopic flow pattern</u>: The rapid escaping liquid from the openings on the stator will create a macroscopic flow pattern in the sump, as schematically illustrated in Figure 1. This macroscopic flow provides a distributive mixing action in the sump, which homogenizes the temperature and chemical composition of the melt. As a consequence, the melt temperature and chemical composition in the sump will be uniform, at least around the tip of the high shear device (see Figure 8). In addition, this macroscopic melt flow will be strong near the tip of the high shear device, and weak near the surface of the melt. This will prevent the occurrence of gas entrapment from the melt surface.

<u>A reduced solute build-up at the solidification front</u>: Due to the partitioning solute elements in the solid and liquid phases during solidification, there is usually a built-up of solute elements at the advancing solidification front. The forced convection generated by the high shear devise will suck the solute elements at the solidification front and distribute them uniformly in the sump. Such uniform fields of both temperature (slightly below liquidus) and chemical composition will create a condition which promotes heterogeneous nucleation and equiaxed growth.

Enhanced heterogeneous nucleation in the MC-DC casting process is originated from the intensive melt shearing. Firstly, intensive melt shearing generates more potential nucleating particles by dispersing the oxide films (Figure 9). Previous study has confirmed that the potential number of nucleating particles can be increased by three orders of magnitude [20], and that MgO particles in a Mg-alloy melt do nucleate α-Mg [12]. Increased nucleation events lead to grain refinement. Secondly, the uniform temperature field in the sump can increase the survival rate of the existing nuclei by avoiding their dissolution in the hotter melt (temperature greater than the liquidus). This also contributes to finer grains. Thirdly, there is a possibility that intensive melt shearing promotes fragmentation of dendrite arms since the melt temperature in the sump is below the liquidus (Figure 8). This may also contribute partially to the grain refined microstructure in the MC-DC cast billets.

Summary

A new DC casting process, the melt conditioned direct chill casting (MC-DC) process, has been developed by submerging a high shear device in the sump of a conventional DC caster. The high shear device provides both intensive melt shearing to disperse the naturally occurring oxide films into individual oxide particles as potential nucleation sites and a macroscopic flow pattern to distribute the dispersed particles and to homogenize the melt temperature and chemical composition in the sump. Experimental results have demonstrated that the MC-DC process is capable of producing high quality magnesium billets/slabs with significantly grain-refined microstructure, homogenized chemical composition, reduced/eliminated cast defects. The grain refinement in the MC-DC casting process may be attributed to the increased number of potential nucleating particles by dispersing naturally occurring oxide films, increased nuclei survival rate in the homogenized temperature composition fields and dendrite arm fragmentation due to intensive melt shearing.

Acknowledgement

The financial support from the EPSRC and the Technology Strategy Board of UK is gratefully acknowledged.

References

[1] Mordike B L, Ebert T. Magnesium: Properties -- applications -- potential. Mater. Sci. Eng. A 2001;302:37.

[2] Emley E F. Principles of magnesium technology. Oxford: Pergamon Press, 1966.

[3] Zhang S M, Fan Z, Zhen Z. Direct chill rheocasting (DCRC) of AZ31 Mg alloy. Mater. Sci. Technol. 2006;22:1489.

[4] Qiu D, Zhang M X, Taylor J A, Fu H M, Kelly P M. A novel approach to the mechanism for the grain refining effect of

melt superheating of Mg-Al alloys. Acta Mater. 2007;55:1863.

[5] Nadella R, Eskin D G, Katgerman L. Role of grain refining in hot cracking and macrosegregation in direct chill cast AA7075 billetsRole of grain refining in hot cracking and macrosegregation in direct chill cast AA7075 billets. Mater. Sci. Technol. 2007;23:1327.

[6] Eskin D G. Physical metallurgy of direct chill casting of aluminum alloys. Boca Raton CRC Press, 2008.

[7] Murty B S, Kori S A, Chakraborty M. Graun refinement of aluminium and its alloys by heterogeneous nucleation and alloying. Inter. Mater. Rev. 2002;47:3.

[8] David H SJ, Ma Q, Easton M A, Cao P, Hildebrand Z. Grain refinement of magnesium alloys. Metall. Mater. Trans. A 2005;36:1669.

[9] Zhang M X, Kelly P M, Qian M, Taylor JA. Crystallography of grain refinement in Mg-Al based alloys. Acta Mater. 2005;53:3261.

[10] Easton M A, Schiffl A, Yao J-Y, Kaufmann H. Grain refinement of Mg-Al(-Mn) alloys by SiC additions. Scripta Mater. 2006;55:379.

[11] Cui J Z, Zhang Z Q, Le Q C. Direct-chilling casting of Mg alloy under electromagnetic and ultrasonic combined field. Trans. Nonferrous Met. Soc. China 2010;20:s297.

[12] Fan Z, Wang Y, Xia M, Arumuganathar S. Enhanced heterogeneous nucleation in AZ91D alloy by intensive melt shearing. Acta Mater. 2009;57:4891.

[13] Zuo Y B, Li H T, Xia M X, Jiang B, Scamans G M, Fan Z. Refining grain structure and porosity of an aluminium alloy with intensive melt shearing. Scripta Mater. 2011;64:209.

[14] Fan Z, Liu G. Solidification behaviour of AZ91D alloy under intensive forced convection in the RDC process. Acta Mater. 2005;53:4345.

[15] Zuo Y B, Xia M, Liang S, Wang Y, Scamans G, Fan Z. Grain refinement of DC cast AZ91D Mg alloy by intensive melt shearing. Mater. Sci. Technol. 2011;27:101.

[16] Fan Z, Zuo Y B, Jiang B. Apparatus and method for liquid metals treatment. UK patent, Application No. 1015498.7, 2010.

[17] The Aluminium Association. Standard test procedure for aluminium alloy grain refiners (TP-1). Washington DC, USA, 1990.

[18] Mirak A R, Divandari M, Boutorabi S M A, Campbell J. Oxide Film Characteristics of AZ91 Magnesium Alloy in Casting Conditions. Int. J. Cast Met. Res. 2007;20:215.

[19] Campbell J. Entrainment defects. Mater. Sci. Technol. 2006;22:127.

[20] Men H, Jiang B, and Fan Z. Mechanisms of grain refinement by intensive shearing of AZ91 alloy melt. Acta Mater. 2010;58:6526.