Grain refinement of DC cast magnesium alloys with intensive melt shearing

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Abstract. A new direct chill (DC) casting process, melt conditioned DC (MC-DC) process, has been developed for production of high quality ingots of light alloys by application of intensive melt shearing through a rotor-stator high shear device during the conventional DC casting process. The rotor-stator high shear unit can provide not only macro flow of liquid metal in the DC casting mould but also intensive melt shearing within the high shear device. In this paper, we report the grain refining effect of intensive melt shearing. Experimental results of DC casting of Mg-alloys with and without intensive melt shearing have demonstrated that the MC-DC casting process can produce magnesium alloy billets with significantly refined microstructure. Such grain refinement in the MC-DC casting process can be attributed to enhanced heterogeneous nucleation by dispersed naturally occurring oxide particles, uniform temperature and compositional fields at the solidification front, which promotes equiaxed growth.

1. Introduction
Magnesium alloys have low density, high specific strength, high damping capacity, good castability and excellent electromagnetic shielding properties, which make them suitable for applications in many industrial sectors including automotive, aerospace, defence, electronics, healthcare and sports equipment [1, 2]. DC casting is still the major technology for providing billets or slabs of magnesium alloys. However, the billets or slabs made by the conventional DC casting process often have coarse and non-uniform microstructures, severe chemical segregation, porosity and hot tearing, which not only limit 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 important and effective approach for mitigating such problems [3, 4]. Although grain refinement of magnesium alloys has attracted intensive research [2, 5-7] the search for new and effective grain refiners and methods for grain refinement still continues. Contemporary research on grain refinement of magnesium alloys has mainly focused on finding a new chemical grain refiner and on the application of external electromagnetic and ultrasonic fields [8]. Recently, Fan and his co-workers [9-12] have found that the intensive melt shearing provided by a twin-screw mechanism has a significant grain refining effect on both aluminium and magnesium alloys. Based on this principle, a new technology with simpler, more robust, equipment (a rotor-stator high shear device) for intensive melt shearing has been developed [13]. In the present work melt conditioned DC (MC-DC) casting, by combining the conventional DC casting with the rotor-stator high shear device was used for the production of high quality ingots of magnesium alloys.
Experimental procedure
Commercial magnesium alloys AZ91D, Mg-8.67%Al-0.62%Zn-0.21%Mn (all compositions are in wt %), and AZ31 Mg-2.92%Al-0.85%Zn-0.36%Mn were used in the present work. The measured liquidus is 600°C for AZ91 and 630°C for AZ31. The alloys were melted at 680°C in a steel crucible under a protective atmosphere containing a mixture of N\textsubscript{2} and 0.5 vol% SF\textsubscript{6}. Then ingots were DC cast with and without intensive melt shearing. AZ91D and AZ31 alloys were cast at 650°C and 680°C, respectively. Intensive melt shearing was achieved using a simple rotor-stator device comprised of a rotor, a stator and a housing for holding the stator and the rotor. A motor was used to drive the rotor at high speed (5000-15000RPM) to provide in situ intensive melt shearing in the sump of the DC mould. As shown in Figure 1, the high shear unit was fixed inside the DC mould and the melt was conditioned by intensive shearing during casting. Samples were sectioned, mechanically polished and then examined by optical microscopy (OM). 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. In Figure 1 T1-T4 shows the positions of the thermocouples.

2. Results
The high shear device was switched on during DC casting and the microstructure of the transitional zone was observed. Figure 2 shows the microstructural transition of DC cast AZ91D magnesium alloy ingot from the section without shearing to the sheared section. The bottom half of the sample shows coarse dendritic microstructure; but after switching on the high shear device, the microstructure is immediately transformed to a fine and uniform microstructure with a spherical morphology.

Figure 3 is a macrographs of the sectioned DC cast AZ91D alloy ingots with and without shearing cast at 650°C. The conventional DC cast sample without shearing has a coarse equiaxed grain structure but with intensive melt shearing the structure becomes much finer, and more uniform. The microstructure of the ingots is shown in Figure 4. The conventional DC cast ingot shows a coarse equiaxed dendritic microstructure with an average grain size around 550 µm (Figure 4a) near to the ingot surface and a coarser grain structure with an average grain size around 750 µm (Figure 4b) at the centre. The microstructure of the ingot with intensive melt shearing is much finer and more uniform.

Figure 1, Schematic illustration of the DC casting process with the application of a rotor-stator high shearing unit. T1-T4 indicates the positions of thermocouples for temperature measurement.

Figure 2, The transition zone from non-shearing to shearing (switching on the high shear unit) of AZ91D alloy ingot cast at 650°C.
Both near the surface, Figure 4c, and at the centre of the ingot show, Figure 4d, the microstructure is fine and uniform with an average grain size of 190 µm.

Figure 4, Comparison of the microstructure of AZ91 alloy ingots with and without intensive melt shearing. (a) edge without shearing; (b) centre without shearing; (c) edge with shearing; (d) centre with shearing.

The grain size distribution from the edge to the centre of the sheared and un-sheared ingots is shown in Figure 5. For the conventional DC cast ingot, there is a thin layer with a finer structure, apart from that layer, the structure is dominated by coarse grains. However, the ingot cast with intensive melt shearing shows a uniform grain size distribution across the entire

Figure 5, Variation of average grain size as a function of the distance from the ingot surface.
cross section. The application of intensive melt shearing has had a significant grain refining effect on the AZ91D magnesium alloy and can improve the uniformity of the microstructure.

Figure 6 shows the microstructure of the AZ31 Mg-alloy ingot without (Figure 6a) and with (Figure 6b) intensive melt shearing. The microstructure of the conventionally DC cast AZ31 alloy ingot shows a coarse dendritic structure; however, with the application of intensive melt shearing the microstructure becomes finer and the morphology is also transformed to an equiaxed structure.

Figure 6, Microstructure of DC cast AZ31 Mg-alloy, (a) without and (b) with intensive melt shearing.

The temperature curves during DC casting of the AZ91D magnesium alloy are shown in Figure 7, which were obtained through the thermocouples attached to the head of the high shear device. The positions of thermocouples (T1-4) are shown schematically in Figure 1. Based on the direct measurement of the sump depth with stainless steel rods during the casting process, the bottom of the head of high shear device was about 40 mm above the liquid/solid interface. With the start of the high shear device, there was a sharp change of the temperature curves. The temperature curves became closer, which means the temperature field around the high shear unit was more uniform. The temperature become very close to the liquidus and then reach a plateau which was around 7°C lower than the liquidus.

3. Discussions

The results show that intensive melt shearing has a significant grain refining effect on magnesium alloys.

Figure 7, The measured temperature curves during MC-DC casting 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.

The rotor-stator high shear device can treat the liquid alloy in situ during DC casting. The device
generates intensive melt shearing in the gap between the rotor and the stator and forced convection
that sucks in melt and spraying it out through the holes in the stator. The high shear disperses oxide
agglomerates and films, homogenises composition and temperature fields, and promotes the forced
wetting of oxide particles. The devise generates the intensive shearing and agitation mainly around
and just below the high shear unit and the flow near the liquid surface is very weak. Therefore the
liquid surface is quite stable, preventing the turbulence of surface and entrainment of oxide from the
surface.

Magnesium alloys inevitably contain oxides in forms of particle clusters or films with a non-
uniform distribution [14, 15]. Such oxides may act as substrates for nucleation but are not effective for
grain refinement due to their poor wettability and low number density. Such oxide clusters and films
can be effectively dispersed by intensive melt shearing and the well dispersed oxide particles can act
as effective nucleation sites for α-Mg [9]. The rotor-stator high shear device can not only provide
intensive melt shearing but also forced convection in the melt. The intensive melt shearing can
effectively disperse the naturally formed oxide films or clusters into individual particles and also
provide forced wetting of the oxide particles by the alloy melt. The forced convection flow pattern is
schematically shown in Figure 1. This flow pattern near the solid/liquid interface can not only increase
the formation and the separation of the nuclei near the mould wall but also distribute the nuclei
uniformly in the melt. The high shear device in the DC casting process therefore can enhance
nucleation and the formation of a fine and uniform microstructure, as shown in Figure 2, 3 and 4.

The temperature field also plays an important role in grain refinement. During DC casting, a
uniform temperature field and a low temperature (4-6°C below the liquidus) in the mould can lead to
the formation of a fine and uniform microstructure [16]. The temperature curves of DC casting AZ91D
show that the temperature field around the head of the high shear device and close to the solid-liquid
interface becomes more uniform and about 7°C below the liquidus shortly after switching on the high
shear unit. The low and uniform temperature field is caused by the intensive melt shearing and the
forced convection. The uniform and low temperature can activate more oxide particles and increase
the possibility of them acting as effective nucleation sites. In addition, the uniform temperature field
and low temperature also increase the survival rate of the formed nuclei.

The growth of the formed nuclei is under a uniform temperature and compositional field and in a
which is favorable for the spherical and/or equiaxed growth of α phases. Also, the grains can not grow
too large because there are sufficient nuclei provided by intensive melt shearing and they will touch
their neighbors.

Grain refinement in the MC-DC process can be mainly attributed both to: (1) the enhanced
heterogeneous nucleation by the dispersed oxide particles through intensive melt shearing and (2) the
increased survival rate of the formed nuclei in the homogenized temperature and composition fields.

4. Summary
A new melt conditioned DC (MC-DC) process has been developed for the production of high quality
ingots of magnesium alloys. In the MC-DC casting process, intensive melt shearing provided by a
rotor-stator device is used to control the solidification process. Experimental results of DC casting of
Mg-alloys with and without intensive melt shearing have demonstrated that the MC-DC casting
process can produce alloy billets with a significantly refined microstructure. The intensive melt
shearing can refine the grain size of AZ91D from about 750 µm to 190 µm. The mechanism of grain
refinement is that under intensive melt shearing well dispersed and uniformly distributed oxide
particles can act as nucleation sites for enhancing heterogeneous nucleation. In addition, the
homogenized temperature and composition fields developed by intensive melt shearing improve the
survival rate of the nuclei.
References