Extremely fine and uniform microstructure of magnesium AZ91D alloy sheets produced by melt conditioned twin roll casting

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Magnesium AZ91D alloy strips consisting of equiaxed grains with a mean size less than 100 μm were fabricated successfully by using the melt conditioned twin roll casting (MC-TRC) process. A melt conditioning by advanced shear technology (MCAST) process for conditioning liquid metals at temperatures either above or below the alloy liquidus using a high shear twin screw mechanism was combined with the twin roll casting (TRC) process to form an innovative technology, namely, the MC-TRC process for casting Al alloy and Mg alloy strips. During the MC-TRC process, liquid melt with a specified temperature is continuously fed into the MCAST machine. By intensive shearing under the high shear rate and high intensity of turbulence, the liquid is transformed into conditioned melt with uniform temperature and composition throughout the whole volume. The conditioned melt is then fed continuously into the twin roll caster for strip production. The experimental results show that the AZ91D MC-TRC strips with different thicknesses have a fine and uniform microstructure. It consists of equiaxed grains with a mean size of 60–70 μm, and also displays extremely uniform grain size and composition throughout the entire cross-section. Investigation also shows that the MC-TRC processes are extremely effective to reduce the formation of defects, particularly the formation of the central line segregations.

Keywords: Melt conditioned twin roll casting, Magnesium alloy, Microstructure, Grain size, Segregation

Introduction

It is well known that Mg metal, with a hexagonal close packed structure, has only three slip systems. This makes it difficult to undergo plastic deformation in the solid state Mg alloy to fabricate Mg alloy strips at room temperature. In the conventional process, Mg based alloy strips are fabricated by hot rolling slabs which are prepared by direct chill (DC) casting. As DC cast Mg alloys are coarse grained materials, a sophisticated rolling process has to be carried out carefully to fabricate much thinner strips. These include controlling high rolling temperatures exactly, reheating strips at each step of the rolling process, and utilising precise control systems for rolling and applying low rolling speed.1–5 These extra steps cause low production efficiency and high cost. To overcome the difficulties associated with the solid state deformation process of Mg alloys, improve production efficiency and reduce cost, a twin roll casting (TRC) technology has been developed to produce Mg alloy strips directly from liquid Mg alloys.1–8

In the recent years, the TRC technique has been developed extensively for producing both Al and Mg alloy strips with high quality and good mechanical properties, and research has also been made to improve the understanding of the solidification mechanism of the TRC process.5–7 The conventional TRC process integrates casting and rolling into a single process8–11 and especially stresses the large deformation in the hot rolling process. The ultimate objective of TRC is to produce a strip with a fine and uniform microstructure and little defects such as segregation, and a good surface finish. However, lots of problems have to be solved before such an ideal TRC strip is achieved. The major problems with the conventional TRC process include coarse columnar grains in the size of nearly 1 mm, many types of severe segregations and defects, only few alloys suitable for strip production, poor surface finish and low casting speed. Most of such problems are associated with the deformation process.11–13 The conventional TRC process involves a substantial amount of force to weld the two presolidified shells in the bite of the two rolls. As a consequence of this large deformation, the solute rich liquid is squeezed out from the growing dendrite, resulting in severe central line segregation and other casting defects such as bleedings. It is worth considering that TRC is a
casting process, and that high quality strips can be produced by relying on the control of the solidification behaviour rather than large deformation. Based on this consideration, a new MC-TRC (the melt conditioned twin roll casting) process was developed (Fig. 1), in which the function of the solidification process was enhanced significantly and high quality strips with little segregation were achieved.

In BCAST, the key technique in the MC-TRC process is a MCAST (melt conditioning by advanced shear technology) processor which is attached to a twin roll caster for producing Mg and Al based alloy strips. The MC-TRC process combines the advantages and functions of both the MCAST process and the TRC process, and can control effectively the solidification behaviour of liquid metals in the TRC process. This control process can be realised by the MCAST device (Fig. 1). As shown in Fig. 1, the MCAST device has a pair of co-rotating, fully intermeshing and self-wiping screws rotating inside a barrel with accurate temperature control. The screws have specially designed profiles to achieve high shear rates, high intensity of turbulence and desirable residence time, which ensures the uniform temperature, the uniform composition and the homogeneous heat transfer in the whole volume of liquid melt. Normally, in the MC-TRC process, the liquid alloy is continuously fed into the MCAST machine, where the melt is intensively sheared for a specified period of time and a fixed speed. The sheared melt is then fed continuously into the twin roll caster for strip casting.

In the present paper, successful preparation of magnesium AZ91D alloy strips by both TRC and MC-TRC processes is reported. The microstructure, grain size, compositional variation of both TRC and MC-TRC strips were investigated in detail. The understanding in the nucleation and growth of grains in both TRC and MC-TRC processes and the controlled solidification behaviour of sheared liquid melt in the MC-TRC process is beneficial to develop advanced materials and new production techniques, and to fabricate high quality Mg and Al based alloys in the near future.

Experimental procedure
Fabrication of AZ91D TRC strips
The twin roll caster used in this research was a small lab scale version of the TRC machine with a pair of opposed steel rolls of 318 mm in diameter and 350 mm in width, and it is capable of producing Al and Mg based alloy strips with different thicknesses (2–8 mm). The steel rolls with an inner water cooled system have a maximum speed of 40 rpm. Some core parameters including roll speed, setback, roller gap and strip thickness can be varied randomly, even while a cast is in progress. Under the protection of a N\textsubscript{2}+0.5 vol.-%SF\textsubscript{6} gas mixture, magnesium AZ91D alloy ingots were melted at 700–750°C and the melts were stirred to make the homogeneous composition. Then the liquid melts were transferred into the preheated header box which is also under the protection of a N\textsubscript{2}+0.5 vol.-%SF\textsubscript{6} gas mixture for twin roll casting.

Fabrication of AZ91D MC-TRC strips
The MCAST machine was attached to the twin roll caster. The preparation, melting and protection of AZ91D alloy has been described in the TRC process. The liquid melt was cooled down to specific temperature (700°C) and fed continuously into the MCAST machine under the protection of a N\textsubscript{2}+0.5 vol.-%SF\textsubscript{6} gas mixture. The MCAST machine was run with the specified temperatures (590–650°C) of barrel heat in advance for AZ91D alloy and the melt was sheared at the rotation rate of 500–800 rpm and the time period of 30–60 s. The sheared liquid was fed steadily into the twin roll caster to produce strips with different thicknesses dependent on the roller gap.

Characteristics of both TRC and MC-TRC samples
Samples cut from both TRC and MC-TRC AZ91D alloy strips were ground, polished carefully and etched in a 0.5% nitride solution. The microstructure was examined using an optical microscope equipped with an image analysis system. Further details were investigated by scanning electron microscopy (SEM) with an attached X-ray energy dispersive spectroscopy (EDS). Throughout the whole samples fabricated by both TRC and MC-TRC processes, the variations of the composition, grain size and microstructure were also studied. Specimens of both TRC and MC-TRC strips were etched specially 3–5 min by the solution of 75 mL ethanol, 25mL distilled water and 0.5mL acetic acid, and the grain sizes were measured precisely.

Results
Microstructure, grain size and composition of magnesium AZ91D TRC strip
Figure 2a shows the microstructure on the entire cross-section of the AZ91D TRC strip in the longitudinal direction produced at a pouring temperature of 650°C. Large amounts of columnar dendrites can be seen throughout the strips. It can also be noticed that serious central line segregations can also been observed in the central portion of the TRC strip. These segregations have irregular morphology, black colour and go through the strips in the longitudinal direction. Figure 2b–f presents more details of the microstructure along a
random line from the top to the bottom. From the top to the bottom in the strip, there are three layers (Fig. 2a), namely, the top layer, the central layer and the bottom layer. On the surface of the strip, there is a thin layer which consists of fine grains (Fig. 2b) and it is called the outer chill zone. Following the chill zone, a columnar zone which consists of columnar dendrites is developed. In this zone, the microstructure varies significantly. Some dendrites are able to grow up to 1300–1500 μm. Next to the columnar zones, a central zone that consists of large amounts of equiaxed grains and central line segregations is presented (Fig. 2d). The central region of the strip always has a markedly different structure. It can be noticed that the central portion has brighter contrast than other zones (Fig. 2a), suggesting that the composition in this zone is completely different from that of other zones. Figure 3 shows the SEM morphology of the segregations of the central region in the TRC strip. The segregations were formed by the squeezed liquid melt because of large amounts of deformation during the casting. The EDX compositional analysis shows that the composition of the segregation (location A in Fig. 3b) is 63.95Mg–31.75Al–4.30Zn. It is clear that the segregation is (Al, Zn) rich phases. The interface (location B in Fig. 3b) between the segregation and the matrix was also investigated. Figure 3c displays the microstructure of the interface which consists of some compounds and fine structures. The composition of the interface analysed by of EDX is 77.76Mg–19.68Al–2.21Zn–0.35Mn. It is noticed that Mn element was detected, which suggests that Al–Mn compounds formed in the interface area. The matrix structure was shown in Fig. 3d. It is completely different from the microstructure described above.

To check the variation of grain sizes in the different areas, the measurement of grain sizes through the thickness direction was investigated. The specimens were etched by the solution of 75mL ethanol, 25mL distilled water and 0.5mL acetic acid carefully. The colour photos of the microstructure were obtained and the grain sizes were measured precisely. The result was shown in Fig. 4. The grain size is smaller (about 220 μm) in the chill zone. However, in the column zone, with the increase in the distance along the thickness direction, the dendrite sizes rise sharply and have a mean size of about 600–750 μm dependent on the distance along the thickness direction. In the central zone (equiaxed grain zone), the dendrite size reduces significantly to about 300 μm. These results mean that the grain sizes of the TRC strips vary greatly with changing locations or zones.

The composition variation throughout the entire TRC strips was also investigated. Along the thickness direction from the top surface to the bottom surface, the content variations (wt-%) of Mg, Al and Zn elements with increase in the distance were shown in Fig. 5. At the beginning, with the increase in the distance, Al and Zn contents have no obvious variation. However, at the central region, both Al and Zn contents change and increase sharply. Al content reaches nearly 20% and Zn content up to 3%. This result suggests chemical segregation in the as-cast strip, which originates from the growth of columnar dendrites and rich liquid melt squeezed out from dendrite arm space into central areas during the deformation process. With further increase in the thickness from the centre to the bottom surface, both Al and Zn contents reduce, which has the same tendency with that from the centre to the top.
Microstructure, grain size and composition of magnesium AZ91D MC-TRC strip

The entire cross-section microstructure of the MC-TRC strip in the longitudinal direction was shown in Fig. 6a. No macrosegregations were observed throughout the entire samples. The outer morphology of the AZ91D strip fabricated by the MC-TRC process is also checked. The strip with a thickness of 4 mm displays a good shape, good edge, flat and good surface finish as well as high quality without macrodefects. To investigate the size, morphology and distribution of solid phases, different locations throughout the thickness direction (Fig. 6a) in the strip were chosen and their microstructures were shown in Figs. 6b–f. It is important to point out that no columnar dendrites were seen in all locations. Throughout the entire cross-section, only equiaxed grains with uniform size were observed. The entire cross-section microstructure of the MC-TRC strip in the transverse direction was shown in Fig. 7a. There are still no macrosegregations to be observed throughout the entire samples. Much more details of the microstructure were shown in Fig. 7b–f. It can be seen that the strip still consists of an extremely uniform structure and equiaxed grain size throughout the entire strips in the transverse direction. From the results described above, it suggests that the MC-TRC process is effective to improve the solidification behaviour, control the nucleation and growth, and reduce significantly the formation of defects such as segregations to achieve high quality strips. The SEM morphology of the central area of the MC-TRC strip was shown in Fig. 8. It can be noticed that the microstructure is extremely homogeneous. This further confirms the uniformity of the MC-TRC strips.

To measure the mean sizes of equiaxed grains, the samples were etched specially and the colour photos of the strip microstructure were also observed. The mean size of grains throughout the thickness in the MC-TRC
A microstructure of entire cross-section of MC-TRC AZ91D alloy strips at longitudinal direction and a-f microstructural variation throughout whole thickness direction from top to bottom showing extremely uniform structure.

The size of equiaxed grains is about 60–70 μm and is extremely uniform. Compared with the grain sizes of the TRC strip, the mean size of the equiaxed grains in the MC-TRC strip is only one tenth that in the TRC samples. On the other hand, in the MC-TRC strip, the morphology and size of grains are really uniform throughout the whole strips, but in the TRC strip, the morphology and size of grains vary significantly and have different morphologies and sizes in different zones. The significant reduction of the grain sizes and the extreme uniformity of the morphology, which are caused by the advanced MC-TRC process, are possible to improve significantly both strength and plasticity of the strips. The composition variation throughout the thickness direction of the MC-TRC strip was also investigated. From the top to the bottom, with increase in the distance, the content variations (wt-%) of Mg, Al and Zn...
elements were shown in Fig. 10. It is noticed that Al, Mg and Zn contents have no obvious change with the increase in the thickness. This result suggests that there is no obvious chemical segregation in the MC-TRC strip. It also means that the MC-TRC process is effective to prevent the formation of defects such as segregations and bleedings.

Discussion

The microstructure of TRC strips depends significantly on the solidification behaviour of the liquid melts in the TRC process. The basic solidification behaviour of the liquid melt has been described in detail. As the nucleation initiates from the roller surface, the temperature gradient between the roller surface and the liquid melt causes the formation of columnar dendrites easily. Some grains with the most favourably crystallographic orientation are able to grow to huge columnar dendrites. Under the above solidification conditions, the dendritic growth always results in enriched liquid in the growth front of solidification. As the casting proceeds and both the solidified shells approach the roll bite, their dendritic networks and residual liquid melts interact and combine to form the final solidified strips. As shown in Fig. 2, large amounts of columnar dendrites can be seen throughout the strips. For the MC-TRC process, the nucleation and growth of grains is completely different from that of the TRC process. The solidification behaviour of sheared liquid melt has been discussed by Fan et al. According to the classical theory of nucleation, when overheated liquid melt is poured into the relatively cold mould, heterogeneous nucleation takes place immediately in the undercooled liquid close to the mould wall. The majority of the nuclei are transferred to the overheated liquid region and dissolved, so only a small proportion as low as 0.3% of nuclei survive and contribute to the final microstructure, resulting in a coarse and non-uniform microstructure. This has been observed in the TRC strips that consist of columnar dendrites shown in Fig. 2. In the MCAST process, the fluid flow inside the barrel of the MCAST machine is characterised by high shear and high intensity of turbulence. A direct consequence of such melt flow characteristics is that the liquid melt inside the barrel of the MCAST machine has a uniform temperature, uniform chemical composition and fast heat removal rate. Such conditions will ensure a 100% nuclei survival rate. Perhaps more importantly, intensive melt shearing will convert the oxide particle clusters and oxide films usually present in the liquid metal into fine, almost mono-sized and well dispersed particles, which are completely wetted by the liquid metal under the shear force. According to the free growth theory, once a critical undercooling corresponding to the oxide particle size is reached, heterogeneous nucleation will occur throughout the entire volume of the conditioned liquid metal. Due to the uniform temperature and composition of the liquid metal, all the nuclei created will survive and grow without preferential orientation. Moreover, higher cooling rates provided by the rotating roller with an inner water cooled system can also increase the thermal undercooling for nucleation and hence increasing the nucleation rate. All these factors will promote a fine and fully equiaxed microstructure in the entire cross-section of the strip. Hence, the MCAST machine has in the first time eliminated successfully both the columnar grains and the macrosegregation in the as-cast strip.

To confirm how the shearing in the MCAST process affects the nucleation and growth of the grains, a simple trial was designed for it. The basic idea is to let the liquid melt just go through the running MCAST machine and check the structural evolution of the strips under the conditions of low shearing speed and short shearing...
time. First, the MCAST machine was switched on in advance, the barrel was heated to the specified temperature, the shear speed was turned up to 150 rpm and the valve was opened. Then, the liquid melt with a specific temperature (615°C) was poured into the MCAST machine. As the valve is open and the melt just goes through the running MCAST machine and flows into a twin roll caster. The sheared time is the time that the melt goes through the running MCAST machine and flows into a twin roll caster. The sheared time is the time that the melt goes through the running MCAST machine. It is measured to be about 5–10 s. The strip microstructure is investigated and shown in Fig. 11. It can be noticed that no columnar dendrites were seen. The strip has a microstructure that consists of equiaxed grains with a mean size of 120 μm. It is also important to note that both the morphology and the size of grains have no difference in different zones (Fig. 8b–c). These results show that the MCAST process is extremely effective to control the nucleation and growth of equiaxed grains.

Extensive experimental investigation in BCAST has identified the following advantages of the MC-TRC process in comparison with the conventional TRC process:

(i) much reduced or even eliminated central line segregation;
(ii) much reduced grain size (up to one order of magnitude reduction)
(iii) extended range of alloys
(iv) more tolerant to inclusions and impurity elements
(v) high grade scrap alloys can be directly cast into high quality strips
(vi) potential increase of casting speed
(vii) improved surface finish in the as-cast condition
(viii) reduced requirement for subsequent rolling
(ix) reduced capital investment
(x) reduced production cost.

Summary

Magnesium AZ91D strips with high quality and little segregation were fabricated successfully by the MC-TRC process. The microstructure, chemical composition and grain size of the strips were investigated. Throughout the entire MC-TRC strip, extremely uniform composition and grains were achieved. The mean grain size of the MC-TRC strip is about 60–70 μm, which is one order of magnitude smaller compared with that of the TRC strip. This originates from the uniform composition and temperature field throughout the whole volume of liquid melt as well as the well dispersed heterogeneous nucleation sites provided by the intensive shearing in the MCAST machine. Experiments confirmed that the MCAST process is extremely effective to control the nucleation and growth of equiaxed grains. Investigation also shows that the MC-TRC strips have few defects, suggesting that macrosegregations such as central line segregation can be prevented or reduced significantly by the MC-TRC process.

References


References
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