MICROSTRUCTURAL ANALYSIS OF SEGREGATED AREA IN TWIN ROLL CAST MAGNESIUM ALLOY SHEET

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Abstract

Twin roll casting process of magnesium alloy has been studied by various institutes since POSTECH started at the world first time. RIST has done the research and development program of twin roll casting and reverse warm milling of magnesium alloy sheet with 600 mm width. Presently, RIST is working on twin roll casting program of magnesium alloy sheet with 2,000 mm width. Twin roll cast magnesium alloy sheet includes segregated area which is important due to controllable quality of final product. Here we present the microstructural analysis of segregated area in twin roll cast magnesium alloy sheet to understand segregation phenomena. Especially, we studied on phase identification in segregated area, comparing the theoretically calculated simulation. Scanning electron microscopy, transmission electron microscopy and energy dispersive spectrometry were used for the phase identification. Our result shows that two kinds of segregation morphologies are observed in the segregated area, not depending upon the location in the sheet. Phase identification shows that segregated area includes densely continuous or dispersed beta and phi phase, and dispersed Al₈Mn₅ and Mg₂Si.

Introduction

Die-cast magnesium parts have been used widely for weight reduction of automobile so far, and then wrought magnesium part would be applied in the near future [1]. Especially, magnesium alloy sheet is approaching the market with competitive cost and new advanced technologies, such as strip casting process. Twin roll strip casting has been developed competitively by Universities, Research Institutes, and Industries [2-6], respectively, according to two issues. Firstly, twin roll strip casting is costcompetitive process for magnesium alloy sheet due to process consolidation. Secondly, demands of lightweight increase due to moving efficiency.

Market needs to improve the surface quality of magnesium alloy sheet and increase its width up to 2,000 mm to extend the application. Dent defect should be removed on the surface of asstrip cast magnesium alloy sheet. However, segregation could be included in the as-strip cast magnesium alloy sheet during solidification. The segregation is classified into center segregation and inverse segregation. It is well known that the center segregation could be reasonably included during solidification in twin roll casting. The inverse segregation might be possibly formed as well by roll pressure during solidification. Controlling the inverse segregation is an important issue to improve the surface quality. Therefore, it is necessary to analyze the microstructure of segregation area in as-cast sheet to understand segregation phenomena. Additionally, phase identification in segregation area is also an important issue to accomplish the post process [7-9]. This study is focused on understanding segregation phenomena in as-twin roll cast magnesium alloy sheet. Here we present the microstructural analysis of segregation morphology in

as-cast magnesium alloy sheet and phase identification of second particles in the segregation area.

Experimental

The magnesium alloy sheet with 600 mm width was manufactured by twin roll casting in RIST. The alloy was AZ31 (Mg-3wt%Allwt%Zn). Figure 1 shows the schematic drawing of the twin roll casting process and the manufactured alloy sheet with directions (rolling direction, transverse direction and normal direction). The transverse direction (TD) of the alloy sheet was observed mainly to analyze the segregation morphology. The microstructural analysis and phase identification were conducted by optical microscopy (OM), scanning electron microscopy (SEM), transmission electron microscopy (TEM) and energy dispersive spectrometry (EDS). TEM specimen was prepared by focused ion beam (FIB) to conduct the phase identification of the local area which was segregated in the sheet. EDS analysis of the TEM specimen was performed in scanning transmission electron microscopy (STEM) mode to increase the accuracy.







Figure 2. TEM specimen prepared by FIB.

Results and Discussion

Segregation microstructure

As shown in Figure 1, center segregation can be easily observed in the central area in twin roll cast AZ31 alloy sheet. Figure 3 (a) shows the center segregation observed at TD direction. Figure 3 (b) shows center segregation and inverse segregation which is also observed in an area, having different contrast on the alloy sheet surface. Minimizing inverse segregation is the key issue in twin roll casting process to improve the surface quality of magnesium alloy sheet because the inverse segregation is located near the sheet surface as shown in Figure 4. The inverse segregation is originated from center segregation and formed by roll pressure because it is vertically connected with center segregation and surface.



Figure 3. Optical images of (a) center segregation and (b) center and inverse segregation (TD direction).



Figure 4. SEM image of inverse segregation near sheet surface.

The segregation area includes second particles with α -Mg matrix. Dispersed second phases and continuous phases are observed in the segregation area. Figure 5 shows magnified microstructures of segregation area. Figure 5 (a) shows the center segregation and the inverse segregation. Two kinds of segregation morphologies are observed in the segregated area, not depending upon the location in the sheet (dispersed phases and continuous phases). Figure 5 (b) shows that the dispersed phases consist of Mg₁₇Al₁₂, Al-Mn and Mg₂Si particles according to EDS analysis (as shown in Figure 6 (a) and (b)). Dispersed phases are mixed with continuous phases as shown in Figure 5 (c). Figure 5 (d) shows the continuous phases is brighter phase in Figure 5 (d). The phi phase is

known as the last solidified phase according to calculated simulation result based on Scheil cooling of AZ31 alloy [9].



Figure 5. SEM images of segregation area showing dispersed phases and continuous phases.



Figure 6. EDS qualitative analysis of (a) and (b) dispersed, and (c) and (d) continuous second phases in Figure 5 (b) and (d), respectively.

Phase identification

TEM works were conducted to identify the second phases observed in dispersed and continuous segregation area. Figure 7 shows TEM image of dispersed phase segregation area. According to EDS quantitative analysis, dispersed segregation area consists of beta phase, Al_8Mn_5 , Mg_2Si and phi phase (as shown in Table I). The particle shape of phi phase is similar to Al_8Mn_5 phase, bright particle. Most of the second phases in the

dispersed segregation area are beta phases identified as $Mg_{17}Al_{12}$, including Zn element.





Figure 7. TEM image of dispersed phase segregation area.

Table I. EDS quantitative analysis of second phases in Figure 7.

(Wt. %)						
Spectrum	0	Mg	Al	Si	Mn	Zn
1	2.75	48.29	33.88			15.08
2	3.26	46.27	36.51			13.95
3	3.55	90.87	5.57			
4	4.54	91.34	4.12			
5	3.01	48.06	34.69			14.24
6	4.09	92.41	3.49			
7	3.50	37.75	15.68			43.07
8	2.46	7.37	52.94	1.09	36.14	
9			40.84	1.90	57.26	
(At. %)						
Spectrum	0	Mg	Al	Si	Mn	Zn
1	4.72	54.50	34.45			6.33
2	5.54	51.81	36.84			5.81
3	5.33	89.71	4.96			
4	6.76	89.59	3.64			
5	5.13	53.89	35.05			5.94
6	6.11	90.79	3.09			
7	7.27	51.56	19.29			21.88
8	4.94	9.73	62.97	1.25	21.12	
9			57.69	2.58	39.73	

Figure 8 shows TEM image of continuous phase segregation area. According to EDS quantitative analysis, continuous segregation area consists of beta phase and phi phase (as shown in Table II). The phi phase solidifies in the eutectic area after solidification of beta phase. The phi phase is brighter than beta phase in the micrograph, and identified as $Mg_{11}Al_5Zn_4$ according to diffraction pattern and EDS quantitative analysis.



Figure 8. TEM image of continuous phase segregation area.

Table	II.	EDS	quantitative	analysis	of second	phases	in	Figure	8.
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Spectrum	0	Mg	Al	Zn	(Wt. %)
1	3.61	90.22	6.17		
2	2.14	32.81	21.10	43.95	
3	2.46	34.72	19.98	42.83	
4	2.97	47.89	28.62	20.52	
5	6.39	87.48	6.13		
6	4.42	45.97	31.39	18.22	
7	5.32	91.88	2.80		
8	3.17	34.61	20.23	42.00	
9	2.65	51.06	31.63	14.66	
Spectrum	0	Mg	Al	Zn	(At. %)
1	5.41	89.10	5.49		
2	4.55	45.94	26.62	22.89	
3	5.17	47.96	24.87	22.00	
4	5.26	55.80	30.05	8.89	
5	9.45	85.17	5.37		
6	7.65	52.39	32.23	7.72	
7	7.88	89.65	2.46		
	1 1 10	47.24	24 88	21.32	
8	0.57	77.27	21.00		

Summary

As-twin roll cast AZ31 alloy sheet includes center segregation, or could include center segregation and inverse segregation under unsuitable casting condition. The segregation area can be classified into disperse phase segregation and continuous phase segregation. According to EDS quantitative analysis, dispersed segregation area consists of beta phase, Al_8Mn_5 , Mg_2Si and phi phase, and continuous segregation area consists of beta phase and phi phase. The beta phases is identified as $Mg_{11}Al_{12}$, including Zn element and the phi phase is identified as $Mg_{11}Al_5Zn_4$ according to this study. The continuous phase inverse segregation should be removed during twin roll casting process to improve the surface quality of magnesium alloy sheet.

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