Mechanical Properties and Microstructure of Twin-Roll Cast Mg-Zn-Y Alloy

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Mg97Zn3Y0.9 alloy has been subjected to twin-roll casting (TRC) process. As-cast microstructure consists of α-Mg dendrite and icosahedral interdendritic phase. The icosahedral phase present in TRC alloy is found to be thermally unstable and transforms to H-phase and W-phase during subsequent thermo-mechanical treatment (TMT) at 400°C. It shows that the pre-heating condition has a significant effect on the microstructural evolution during TMT. The specimen pre-heated at 400°C for 30 min shows elongated microstructure after TMT, while the specimen pre-heated at 400°C for 12 h shows equiaxed recrystallized structure after TMT. Such homogeneous microstructure of the specimen pre-heated at 400°C for 12 h results in better combinations of strength and ductility than the specimen pre-heated at 400°C for 30 min.

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1. Introduction

There has been a growing interest in Mg alloys due to their excellent specific strength and stiffness.1–3) Such properties of Mg alloys are quite attractive for applications in transportation systems that require light-weight components.4) Most of the Mg alloys currently in use in transportation systems are cast products. However, Mg sheet products are much needed since they have a larger potential for applications than their cast counterparts.5) It has been recently shown that the application of twin-roll casting (TRC) can effectively produce low cost Mg alloy sheets with the equivalent tensile properties to those of conventionally processed alloy sheets.6–9) Due to its relatively fast solidification rate, TRC can provide beneficial effects on microstructure such as refinement of microstructural features, extension of solid solubilities, and formation of metastable or non-equilibrium phases.3,10) Among various Mg alloy systems, Mg-Zn-Y alloys are quite interesting since they can incorporate novel second phase particles such as icosahedral phase11) or long period ordered (LPO) structure12) depending on Zn/Y ratio. It has been shown that these alloys, particularly icosahedral phase containing alloys, can have good combinations of strength and ductility due to the coherent interface between the icosahedral phase and Mg matrix.13,14) In the present study, TRC has been applied to the Mg-Zn-Y alloy containing icosahedral phase to fabricate its sheet product. The TRC sheets have subsequently been subjected to thermo-mechanical treatment (TMT) and their microstructure and mechanical properties have been investigated.

2. Experimental Procedure

Mg-Zn-Y alloy used in this study has the composition of Mg97.3Zn2.3Y0.4, similar to the one reported to form second phase of icosahedral phase by gravity casting.11) The alloy was melted under an inert atmosphere of CO2 and SF6 mixture. After melting, the alloy was subjected to TRC with the roll gap of 2 mm and roll speed of about 4 m/min. The as-cast alloy was subjected to TMT consisting of pre-heating at 400°C for 30 min and 12 h and rolling at 400°C with the total reduction of 75%. The rolled sheets were subsequently annealed at 300°C for 5 min. Microstructural observation of the alloy was conducted by optical microscopy (OM), scanning electron microscope (SEM) and transmission electron microscope (TEM). Tensile properties were measured by using flat tensile specimens with 12.6 mm gauge length, 1 mm gauge thickness and 5 mm gauge width at a strain rate of 6.4×10−4 s−1.

3. Results

3.1 Microstructure

Figure 1(a) shows the SEM micrograph of the as-cast Mg97.3Zn2.3Y0.4 alloy. As shown in Fig. 1(a), overall as-cast microstructure consists of α-Mg dendrite and interdendritic phase. There are some areas having the clusters of interdendritic particles. EDS analysis of the interdendritic phase shows that it contains mainly Mg, Zn, and Y with Mg:Zn:Y ratio (in at%) equal to 3:6:1. Diffraction pattern analysis shows that the interdendritic phase has a quasicrystalline symmetry (inset in Fig. 1(c)), which has the quasi-lattice parameter aR of 5.2 nm. It is consistent with the value of icosahedral phase reported in Mg-Zn-Y alloys.15) XRD analyses also indicate the presence of icosahedral phase in as-cast state as shown in Fig. 2. Microstructures after pre-heating at 400°C for 30 min and 12 h are shown in Figs. 3(a) and (b), respectively. After pre-heating for 30 min, dendrites present in as-cast state develop into grains. However, the overall grain structure still resembles the as-cast structure and has the interdendritic second phase particles mostly along the grain boundaries. This indicates that complete homogenization is not achieved by pre-heating for 30 min, which is evidenced by TEM analysis of the microstructure showing the formation of subgrains within Mg grains (Fig. 4(a)). It also shows that some of the interdendritic icosahedral phase present in as-cast state changes to MgZn3Y H-phase which has hexagonal structure with α = 0.918 nm and c = 0.95 nm.16) XRD analyses shown in Fig. 2 also indicate that...
interdendritic icosahedral phase present in as-cast state is being replaced by H-phase during pre-heating for 30 min. After pre-heating for 12 h, on the other hand, interdendritic particles are broken into discrete particles and as-cast dendritic structure has changed to well developed grain structure with average grain size of about 39 μm, as shown in Fig. 3(b). As shown in Fig. 4(b), Mg grains virtually do not contain dislocations and most of the interdendritic icosahedral phase particles present in as-cast state change to H-phase particles. However, the transformation of icosahedral phase into H-phase is not complete and some icosahedral phase particles still remain even after pre-heating for 12 h as shown in Fig. 2. It also shows that H-phase particles are coarser in the specimen pre-heated for 12 h than in the one pre-heated for 30 min (~0.6 μm vs. ~0.4 μm).

Figure 5 shows the development of microstructure after rolling of the alloy at 400°C with reduction of 75%. It shows that the grain structure of the specimen pre-heated for 30 min...
becomes elongated after rolling, forming a banded structure consisting of elongated Mg grains and second phase particles along the grain boundaries as shown in Fig. 5(a). It also shows that some grains appear to have been recrystallized but most of grains do not indicate the occurrence of recrystallization. In contrast to the specimen pre-heated for 30 min, the specimen pre-heated for 12 h shows the development of fine (~7 µm) and equiaxed grain structure after rolling with 75% reduction. Also the interdendritic particles become spheroidized after rolling with 75% reduction as shown in Fig. 5(b), which is totally different from the banded structure of the specimen pre-heated for 30 min. TEM analyses of the specimen pre-heated for 12 h after rolling show that there is a precipitation of additional phase, Zn3Mg2Y3 W-phase (FCC structure with a = 0.683 nm) as shown in Fig. 6. It is interesting to note that this W-phase coexists with icosahedral phase located along its perimeters. It can be thought that icosahedral phase remained after pre-heating transforms to W-phase during TMT of the alloy. However, the morphology of icosahedral phase such as its globular shape and presence along the perimeter of W-phase suggest that there might a re-precipitation of icosahedral phase during TMT. Further study is necessary to clarify the issue.

3.2 Mechanical properties

Tensile properties of the alloy in various conditions are shown in Table 1. In as-cast state, the alloy fails with very small amount of plastic deformation, typical of as-cast alloys. Pre-heating of the alloy results in large increase in elongation but also with large decrease in strength. Rolling of the alloy significantly improves its strength with no or little change in the elongation. Such improvement in the tensile properties is possibly due to the break-up of as-cast microstructure by rolling. It shows that the specimen pre-heated for 30 min and the specimen pre-heated for 12 h in as-rolled condition have essentially the similar tensile properties although they have quite different microstructure, suggesting that the morphology of second phase particles do not have a significant effect on tensile properties in as-rolled condition. However, their tensile properties become quite different after annealing. While both specimens show decrease in strength with increase in elongation after annealing as compared to those in as-rolled condition, the specimen pre-heated for 12 h shows a much larger increase in elongation than the specimen pre-heated for 30 min. On the other hand, the difference in the strength between two specimens is rather small. The tensile properties of the specimen pre-heated for 30 min after annealing are quite similar to those of AZ31 in H24 condition (partially annealed). On the other hand, specimen pre-
heated for 12 h after annealing shows quite large elongation of 27%. Larger elongation of the specimen pre-heated for 12 h than that of the specimen pre-heated for 30 min after annealing indicates the importance of morphology of second phase particles in affecting tensile properties, particularly elongation.

### 4. Discussion

It has been known that Mg-TM-RE alloy systems can produce quite interesting microstructures much different from other alloy systems.\(^{13,17}\) They can form either icosahedral phase or LPO structure phase depending on the composition. It has been reported that icosahedral phase or LPO structure forms when the ratio of RE to TM is smaller than 0.5 or larger than 0.67, respectively.\(^{13,17–19}\)

The alloys containing icosahedral phase show good combinations of strength and ductility owing to the strengthening effect of icosahedral phase and the coherent interface between icosahedral phase and Mg matrix.\(^{13,20,21}\) Lee et al. evaluated the mechanical properties of gravity cast Mg-Zn-Y alloys with different volume fractions of icosahedral phase,\(^{11,16}\) which showed that strength increased with increasing volume fraction of icosahedral phase. They also showed that the volume fraction of icosahedral phase in gravity cast Mg\(_{97}\)Zn\(_{2}\)Y\(_{0}\) alloy was 2.9%, which is much smaller than 7.0% of the present TRC alloy of the similar composition. In fact, the volume fraction of the icosahedral phase in TRC Mg\(_{97}\)Zn\(_{2}\)Y\(_{0}\) alloy is similar to that in the higher solute containing gravity cast Mg\(_{94}\)Zn\(_{4}\)Y\(_{0.7}\) alloy. It has been previously shown that TRC has beneficial effects on microstructure such as the formation of metastable phases and extension of solid solubilities due to its fast solidification rate.\(^{3,9}\) Therefore, such large volume fraction of icosahedral phase in TRC alloy can be thought to be due to the fast solidification rate of TRC. The present study shows that, however, icosahedral phase is not thermally stable and undergoes phase transformation during subsequent TMT at high temperatures. It has been shown in the present study that the icosahedral phase transforms to the H-phase after pre-heating at 400°C and there is an additional precipitation of W-phase during rolling at 400°C. Bae et al. suggested that the transformation to H-phase occurs at above 440°C in the similar alloy based on the results of thermal analyses.\(^{13,22}\) Singh et al. also reported that the transformation occurs at 460°C.\(^{16}\) The presently observed transformation of icosahedral phase into other phases at lower temperatures than the reported temperatures might be due to the presence of numerous diffusion paths in TRC alloy. TRC alloys have much finer dendritic structure than conventional ingot cast alloys, providing more diffusion paths in the former than in the latter. In fact, transformation temperature of 400°C was observed in extruded Mg\(_{97}\)Zn\(_{6}\)Y alloy,\(^{16}\) which proposed

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**Table 1 Mechanical properties of the alloys in various conditions.**

<table>
<thead>
<tr>
<th>Conditions</th>
<th>Yield strength (MPa)</th>
<th>Tensile strength (MPa)</th>
<th>Elongation (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>As-cast</td>
<td>225</td>
<td>240</td>
<td>0.4</td>
</tr>
<tr>
<td>Pre-heated for 30 h at 400°C</td>
<td>125</td>
<td>215</td>
<td>4.6</td>
</tr>
<tr>
<td>Pre-heated</td>
<td>290</td>
<td>325</td>
<td>6.4</td>
</tr>
<tr>
<td>Annealed**</td>
<td>215</td>
<td>280</td>
<td>17.2</td>
</tr>
<tr>
<td>Pre-heated for 12 h at 400°C</td>
<td>100</td>
<td>230</td>
<td>8.5</td>
</tr>
<tr>
<td>Pre-heated*</td>
<td>280</td>
<td>320</td>
<td>7.6</td>
</tr>
<tr>
<td>Annealed</td>
<td>195</td>
<td>265</td>
<td>27.3</td>
</tr>
</tbody>
</table>

*Total reduction of 75% at 400°C. **Annealed at 300°C for 5 min.

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**Fig. 6 (a) TEM micrograph of Mg\(_{97}\)Zn\(_{2}\)Y\(_{0}\) alloy after rolling at 400°C and (b) diffraction pattern of second phase particles ([011] zone axis of W-phase and 5-fold zone axis of icosahedral phase).**
that high diffusivity along grain boundaries played an important role in the transformation of icosahedral phase into other phases.

It has been shown that the degree of pre-heating has a significant effect on the microstructural evolution during hot rolling. As shown in Figs. 5(a) and 5(b), after rolling at 400°C with 75% of reduction the specimen pre-heated for 30 min shows mostly unrecrystallized elongated grain structure, while the specimen pre-heated for 12 h shows the equiaxed grain structure, indicating the occurrence of complete recrystallization in this case. The main difference between the specimens pre-heated for 30 min and pre-heated for 12 h is the degree of the relief of strain accumulated in TRC alloy during pre-heating. When the alloy is subjected to rapid solidification such as TRC, it usually contains a large amount of strain due to shrinkage of volume during solidification. The amount of strain in TRC alloy can be increased further since the alloy experiences hot deformation during TRC. When the complete development of grains occurs during pre-heating such as the case of the specimen pre-heated for 12 h, it contains essentially no strain. On the other hand, the specimen pre-heated for 30 min still contains a large amount of strain as evidenced by the incomplete development of grains. Considering that recrystallization occurs more readily in the specimen having a larger amount of accumulated strain, it is expected that after the same amount of rolling reduction, the specimen pre-heated for 30 min would show more rapid recrystallization than the one pre-heated for 12 h. However, the present result shows that complete recrystallization did not occur in the specimen pre-heated for 30 min. It suggests that other mechanisms are operating for the recrystallization of the present alloy. The important difference between the specimens pre-heated for 30 min and pre-heated for 12 h is the morphology of second phase particles. As shown in Fig. 5(a), the specimen pre-heated for 30 min has second phase particles mostly along grain boundaries. On the other hand, the particles present in the specimen pre-heated for 12 h are discrete and homogeneously distributed in the microstructure. Therefore, it can be thought that the morphology of second phase particles has a great effect on recrystallization during hot rolling. It has been shown that dynamic recrystallization during rolling can be stimulated by the presence of second phase particles.\(^{23}\)

During hot rolling process, dislocations become accumulated and tangled around second phase particles and such high density of dislocations can be the source of driving force of dynamic recrystallization. The homogeneously distributed second phase particles in the specimen pre-heated for 12 h can be more effective source of driving force than the inhomogeneously distributed second phase particles in the specimen pre-heated for 30 min.

The above-mentioned differences in the microstructure between the specimens subjected to various TMT conditions well explain the resultant differences in the mechanical properties. It shows that the specimens pre-heated for 30 min show higher strength and smaller elongation than the ones pre-heated for 12 h, regardless of TMT conditions (Table 1). As mentioned previously, the microstructure of the specimens pre-heated for 30 min is characterized by the incomplete development of grains in as-pre-heated state and incomplete recrystallization in as-rolled state. On the other hand, the specimens pre-heated for 12 h show well-developed grain structure in pre-heated state and recrystallized structure in as-rolled state. Therefore, the specimens pre-heated for 30 min have larger amounts of internal strain than the ones pre-heated for 12 h in all conditions, resulting in higher strength and smaller elongation in the former than in the latter. The only exception can be found for the specimens in pre-heated state. The higher ultimate tensile strength of the specimen pre-heated for 12 h than that of the one pre-heated for 3 h (i.e., 230MPa vs. 215MPa) is due to the larger elongation of the former than the latter. The banded microstructure of the specimen pre-heated for 30 min might also be beneficial for the strength. However, it usually results in lower ductility than the one having homogeneous distribution of second phase particles.\(^{24}\) Such effect of the morphology of second phase particles is well exemplified in the specimens after annealing. It shows that the specimen pre-heated for 12 h shows a much larger improvement in ductility after annealing at 300°C for 5 min than the one pre-heated for 30 min. The specimen pre-heated for 12 h after annealing shows yield strength of 195MPa and total elongation of more than 27%, suggesting that highly ductile Mg alloys can be developed by TRC. It should also be pointed out that the strength of the present TRC Mg\(_{97.5}Zn_{2.5}Y_{0.4}\) alloy is quite similar to that of the gravity cast Mg\(_{94.2}Zn_{4.6}Y_{0.7}\) alloy having larger amounts of Zn and Y.\(^{11}\)

5. Conclusion

Twin-roll casting process has been utilized to fabricate the sheet product of Mg\(_{97.5}Zn_{2.5}Y_{0.4}\) alloy containing icosahedral phase, and their tensile properties have been evaluated after various TMTs. TMT at 400°C transforms the icosahedral phase present in as-cast state to H-phase and W-phase. The specimen pre-heated at 400°C for 30 min has elongated grain structure with second phase particles mostly along grain boundaries. On the other hand, the specimen preheated at 400°C for 12 h has fully recrystallized equiaxed grain structure with homogeneously distributed second phase particles. Accordingly, the specimens pre-heated at 400°C for 12 h show better combinations of strength and ductility than the ones pre-heated at 400°C for 30 min.

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REFERENCES