Microstructural stability of Mg-5Al-0.4Mn-3RE alloy during annealing

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Abstract
As-cast Mg-5Al-0.4Mn-3RE magnesium alloy was prepared successfully. The microstructure and microstructural stability at 473K were investigated by light microscopy and X-ray diffraction (XRD). The results revealed that the as-cast Mg-5Al-0.4Mn-3RE alloy consists of $\alpha$ - Mg matrix, eutectic $\alpha + \gamma$ (where $\gamma$ is $\text{Mg}_{17}\text{Al}_{12}$), $\text{Al}_{11}\text{RE}_{3}$ precipitates and $\text{Al}_{10}\text{RE}_{2}\text{Mn}_{7}$ phase. After annealing at 473K for 100h and annealing at 473K for 100h after compression with a stress 280 MPa needle shape particles of $\text{Al}_{11}\text{RE}_{3}$ remained unchanged due to their relatively high melting point.

Keywords: Mg-Al-Mn-RE alloy, Microstructure, Annealing

1. Introduction
The most challenging issue for the transportation industry nowadays is the requirement of reducing fuel consumption and CO$_2$ emission. It can be fulfilled by vehicle-weight reduction and therefore it requires the development of innovative materials for lightweight design [1, 2].

Magnesium alloys are well known for their lightweight, but also offer good combination of mechanical properties and castability. That is why, the use of magnesium alloys in general, and in transport in particular, has significantly increased recently [3].

Currently most commercial magnesium alloys are based on the magnesium-aluminium system. Among them AZ91, AM60 and AM50 are the most economically attractive. Mg-Al alloys display a wide temperature spans between liquidus and solidus curves, therefore these alloys are susceptible to a casting defects including segregation. All the aluminium contents in the commercial alloys are below the maximum solid solubility limit. The equilibrium microstructure (according to magnesium-aluminium equilibrium phase diagram) for these alloys is 100% $\alpha$-magnesium, but due to non-equilibrium solidification conditions, metastable eutectic $\alpha + \gamma$ (where $\gamma$ is the intermetallic compound $\text{Mg}_{17}\text{Al}_{12}$) normally forms and is present in the as-cast microstructure in Mg-Al alloys down to about 2 wt.% Al [4]. It should be also noted that the presence of small amount of manganese in commercial magnesium-aluminium alloys causes the formation of aluminium-manganese intermetallic compounds ($\text{Al}_{12}\text{Mn}_{2}$, $\text{Al}_{3}\text{Mn}$ or $\text{AlMn}$) [5]. However, the applications of magnesium-aluminium alloys cannot expand further due to its poor mechanical properties above 393K [6, 7]. The poor elevated temperature properties of Mg-Al alloys are related to the occurrence of the low-melting $\gamma$ – $\text{Mg}_{17}\text{Al}_{12}$. In order to modify the microstructure and improve the mechanical properties of Mg-Al alloys, elements like Sb, Bi, Sr and rare earth (RE) have been...
added [8 - 10]. Rare earth elements are the most common way of improving the elevated temperature properties by modifying the precipitation of γ and formation of thermally stable precipitates of Al12RE3 phase along grain boundaries to resist the deformation by boundary sliding [6]. It has been reported that the Al12RE3 phase is unstable during creep at temperature above 423K for up to 1000h under a load of 70-80 MPa and decomposes to Al12RE compound, releasing some Al atoms [11 - 13].

Previous studies allowed to introduce successfully rare earth elements into AZ91 and AM50 magnesium alloy [14, 15]. In the present work, experimental AM50+3wt.%RE alloy and its microstructural stability during annealing was studied.

2. Experimental material and procedures

An ingot of AM50 magnesium alloy (the composition of which is listed in Table 1), was melted at 953K in an electric resistance furnace using a steel crucible. The 3 wt.% addition of rare earth elements was done in the form of cerium rich mish metal, with the composition according to attestation listed in Table 2. The melt was held for 5 minutes and mechanically stirred for 5 minutes to ensure a homogenous composition. Then, it was cast into a metal mould.

Table 1.
Chemical composition of AM50 alloy according to ASTM B93-94

<table>
<thead>
<tr>
<th>Alloy</th>
<th>Al</th>
<th>Mn</th>
<th>Zn</th>
<th>Si</th>
<th>Fe</th>
<th>Cu</th>
</tr>
</thead>
<tbody>
<tr>
<td>AM50</td>
<td>4.5-5.3</td>
<td>0.28-0.5</td>
<td>max</td>
<td>max</td>
<td>max</td>
<td>0.02</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>0.05</td>
<td>0.004</td>
<td>0.008</td>
<td></td>
</tr>
</tbody>
</table>

Mgrest

In order to determine the microstructural stability of the investigated alloy annealing was carried out at a temperature of 473K for 100h. Two samples were annealed: before and after compression with a stress 280 MPa.

Table 2.
Chemical composition of mish metal

<table>
<thead>
<tr>
<th>Chemical composition [wt. %]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ce</td>
</tr>
<tr>
<td>----</td>
</tr>
<tr>
<td>mish metal</td>
</tr>
</tbody>
</table>

The microstructure was characterized by a light microscopy (LM). A standard metallographic technique was used for sample preparation which includes wet prepolishing and polishing with different diamond pastes. Specimens were examined by a Neophot-21, Carl-Zeiss Jena microscope.

Phase constitutions of the alloy before and after annealing were analyzed by X-ray diffraction (XRD) using a Brucker D8 Advance diffractometer. CuKα X-ray radiation was used.

3. Results and discussion

Fig. 1 shows the morphology of as-cast AM50+3wt.%RE alloy. The microstructure is composed of α solid solution of alloying elements in magnesium (point 1 in Fig. 1) and small amount of binary eutectic α + γ (point 2 in Fig. 1). The previous studies indicated that volume fraction of eutectic is smaller in alloys with rare earth elements addition than in AM50 solidified at the same conditions [16]. The microstructure observations of AM50+3wt.%RE revealed also the occurrence of needle-like precipitates (point 3 in Fig. 1). Moreover, small amount of manganese caused the formation of precipitates with polygonal shape (point 4 on Fig. 1). Similarly to as-cast, in the microstructure of AM50+3wt.%RE after annealing (Figs. 2a and 2b) needle-like and polygonal precipitates occurred. Annealing and annealing after compression did not change the structure of the dominant intermetallic needle-like phase in the investigated alloy. However, in the annealed alloy no presence of eutectic was revealed.

Fig. 1. Microstructure of as-cast AM50+3wt.%RE; LM

In order to identify the existing phases in the alloys, XRD analysis was performed, the results of which are shown in Fig. 3. The diffraction lines were indexed as arising from three different phases. It can be seen that as-cast AM50+3wt.%RE (Fig. 3a) is mainly composed of α – Mg, Al12RE3 and Al12(RE)2Mn7. X-ray diffraction patterns of annealed samples are shown in Figs. 3b and 3c. In both the annealed and annealed after compression samples, the second phases present are still Al12RE3 and Al12(RE)2Mn7. It implies that Al12RE3 phase has high thermal stability at a temperature of 473K with no decomposition observed after 100h, even after compression. This relatively high thermal stability of this compound can be inferred from its high melting temperature.
No characteristic reflections of $\text{Mg}_{17}\text{Al}_{12}$ phase was observed in the X-ray diffraction pattern of all investigated samples. It indicates that the weight fraction of $\text{Mg}_{17}\text{Al}_{12}$ might be below the limit of detection.

Fig. 2. Microstructure of AM50+3wt.%RE: a) annealed at 473K 100h, b) annealed at 473K 100h after compression with a stress 280 MPa; LM

Fig. 3. X-ray diffraction patterns of AM50+3wt.%RE: a) as-cast, b) annealed at 473K 100h, c) annealed at 473K 100h after compression with a stress 280 MPa

The XRD results show that no other phases such as Mg-RE or Mg-Al-RE except from Al-RE and Al-RE-Mn are detected. To predict the possibility to form intermetallic compounds the differences in electronegativity can be used [17]. The more difference between two elements, the stronger the bonding force between these elements and the formation of a metallic compound is easier. The differences in electronegativity between Al and RE (represented by Ce, La, Nd and Pr) are larger than that between RE and Mg, as well as between Al and Mg. Because of that, during solidification of Mg-Al-RE alloy, rare earth elements preferentially react with Al to form Al$_{11}$RE$_3$ compound. Additionally, the formation of Al$_{11}$RE$_3$ phase consumes a portion of the aluminium atoms, thus reducing the precipitation of the $\text{Mg}_{17}\text{Al}_{12}$ phase.
4. Summary

The microstructure analysis of AM50+3wt.%RE was presented. The results revealed that the investigated alloy consists of α – Mg matrix, eutectic α + γ (where γ is Mg7Al3), Al6RE3Mn12 phase and precipitates of Al11RE3 compound, which is the dominant intermetallic phase in the alloy. During annealing at 473K for 100h Al11RE3 phase is thermally stable, even after compression with a 280MPa stress.

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References