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ScienceDirect

Journal of Magnesium and Alloys 2 (2014) 124–132
www.elsevier.com/journals/journal-of-magnesium-and-alloys/2213-9567

Full length article

Understanding effects of microstructural inhomogeneity on creep response – New approaches to improve the creep resistance in magnesium alloys

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Received 14 February 2014; accepted 12 March 2014
Available online 15 June 2014

Abstract

Previous investigations indicate that the creep resistance of magnesium alloys is proportional to the stability of precipitated intermetallic phases at grain boundaries. These stable intermetallic phases were considered to be effective to suppress the deformation by grain boundary sliding, leading to the improvement of creep properties. Based on this point, adding the alloying elements to form the stable intermetallics with high melting point became a popular way to develop the new creep resistant magnesium alloys. The present investigation, however, shows that the creep properties of binary Mg–Sn alloy are still poor even though the addition of Sn possibly results in the precipitation of thermal stable Mg_2Sn at grain boundaries. That means other possible mechanisms function to affect the creep response. It is finally found that the poor creep resistance is attributed to the segregation of Sn at dendritic and grain boundaries. Based on this observation, new approaches to improve the creep resistance are suggested for magnesium alloys because most currently magnesium alloys have the commonality with the Mg–Sn alloys.

Keywords: Magnesium alloy; Creep resistance; Microstructure; High temperature deformation; Mechanical properties

1. Introduction

The high demand in the automotive industry for weight savings has resulted in the great interests for magnesium alloys due to their high specific strength and low density. Some Mg–Al based alloys, such as AZ91D and AM60B, have been used in automotive products since these alloys exhibit superior die castability and a good balance of strength and ductility [1]. However, the applications of magnesium alloys are still limited. One of the problems is the restriction of creep resistance at elevated temperatures. More investigations have revealed the poor elevated temperature creep resistances of Mg–Al based alloys is due to discontinuous precipitation of $Mg_{17}Al_{12}$ (β phase) from the supersaturated α-Mg solid solution and coarsening of β in the interdendritic eutectic region at high temperatures. The β phase has a b.c.c. structure with a melting point of 437 °C and its thermal stability is low. Its hardness decreases by 50–60% when the temperature increases from 25 to 200 °C. How to suppress the formation of β phase plays a key role in improving the creep resistance. Two major approaches were taken to develop new creep-resistant magnesium alloys in the past [2–4]:

- Adding the alloying elements to remove Al by forming Al-containing intermetallics.
- Development of aluminum free magnesium alloys.

The general principle is to form the thermal stable precipitates so that the deformation by grain boundary sliding can be suppressed by these precipitates.

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Peer review under responsibility of National Engineering Research Center for Magnesium Alloys of China, Chongqing University

ELSEVIER Production and hosting by Elsevier

http://dx.doi.org/10.1016/j.jma.2014.03.003.

Recently, heterogeneity of creep deformation was observed by Dargusch et al. [5]. They observed higher creep deformation at grain boundaries than within dendrites. The same phenomenon was also investigated by Han and coworkers [6]. They explained that this inhomogeneous deformation was caused by the enhanced deformation in the primary α(Mg) phase and eutectic α(Mg) phase adjacent to the grain/dendritic boundaries. In these regions, both the stresses and homologous temperature are higher. On the basis of these observations, they put forward a new approach to develop creep-resistant magnesium alloys: adding alloying elements increasing the local homologous temperature or reducing the volume fraction of the eutectic α(Mg) phase. Unfortunately, in their paper no technique routes were given. The present paper will first report the effects of microstructural inhomogeneity on the creep response of Mg–Sn and AZ91 alloys. Based on the analysis of experimental results, new approaches to improve the creep resistance are proposed and discussed, and in the meantime, their practical availability is preliminary examined.

2. Experimental procedures

The selected alloys were Mg–Sn and AZ91 alloys. Mg–Sn is a promising system to develop new cheap creep resistant magnesium alloys by alloying with other elements [7]. The reason to select AZ91 alloy is that this alloy is the most extensively commercially used alloy at present. Table 1 lists the nominal composition of the alloys; all values are given in wt.%. Pure magnesium (99.98%, Hydro Magnesium), pure tin (99.96%, MCP HEK) and pure Al were used for the production of alloys. The alloys were cast using permanent mold casting. During the melting process a protective atmosphere of Ar + SF6 was employed. The melt was poured into a permanent steel mold (preheated up to 350 °C) and then cooled down to room temperature by air cooling.

Tensile creep tests were performed to investigate the creep response of Mg–3Sn and AZ91 alloys in the as-cast and heat-treated states. Tensile specimens with a diameter of 6 mm, gauge length of 30 mm and with M10 threads, were prepared from the as-cast material. Creep tests were performed using an ATS lever arm creep testing machine in air under a constant stress and temperature. The temperature was measured using Ni–CrNi thermocouple calibrated to an accuracy of ±3 °C.

The microstructure was examined using an optical microscope, scanning electron microscope (SEM), transmission electron microscope (TEM). The specimens were ground with the help of silicon carbide emery papers. Then they were polished using OPS containing 0.05 μm colloidal silica. These specimens were chemically etched in a solution of 8 g picric acid, 5 ml acetic acid and 10 ml distilled H_2O in 100 ml ethanol for 10 s. SEM investigations were performed using a JSM 5310, with an accelerating voltage of 15 kV. Back scattered images and energy dispersive X-rays were used for characterizations. Specimens for TEM were ground mechanically to about 70 μm and then thinned using electropolishing in a twin jet system using a solution of 5% $HClO_4$ and 95% ethanol at about −30 °C and a voltage of 40 V. TEM observations were performed on a JEOL 2000 transmission electron microscope with an energy dispersive X-ray analysis (EDX) system operating at 200 kV. X-ray diffraction (XRD) investigations were also carried out using a Siemens diffractometer operating at 40 kV and 40 mA with Cu K_α radiation. Measurements were obtained by step scanning 2θ from 20 to 120° with a step size of 0.02°. A count time of 3 s per step was used.

Table 1
Nominal compositions of investigated alloys (wt.%).

Alloy no.	Alloys	Sn	Ca	Al	Zn	Mg
1	Mg3Sn	3	0			Bal.
2	Mg5Sn	5	0			Bal.
3	Mg3Sn1Ca	3	1			Bal.
4	Mg3Sn2Ca	3	2			Bal.
5	AZ91			9	1	Bal.

3. Results and discussion

In this section, the relationship between the microstructure and creep response of Mg–Sn alloy is first presented and discussed, with an emphasis on the effect of interfacial microstructure on the creep response. Then the commonality of currently most magnesium alloys with Mg–Sn alloys is described. Based on these observations, the possible approaches to improve the creep resistance are put forward.

3.1. *Effect of interfacial microstructure on the creep response of Mg–Sn alloys*

The microstructure of Mg–Sn alloys has been described in detailed elsewhere [7,8]. Small amount of the phase Mg_2Sn with a globular shape was observed at the dendritic and grain boundaries in the as-cast Mg–3Sn alloys. The increment in the content of Sn increases the amount of the phase Mg_2Sn (Fig. 1). The subsequent ageing treatment also increases the volume fraction of Mg_2Sn phase [7]. After the addition of Ca, the phase CaMgSn forms instead of the phase Mg_2Sn.

Further microstructural investigations demonstrate that the distribution of Sn is very inhomogeneous in the binary Mg–Sn alloy. SEM observations show the existence of diffusive bright bands at the dendritic and grain boundaries (Fig. 2). These bright bands are enriched with Sn. EDS analysis indicated that the content of Sn is up to 7 wt.% (1.5 at.%) in these regions which is much higher than that in the matrix (approximately 1.2 wt.% (0.25 at.%)). Fig. 3 shows the microstructure after creep rupture for the Mg–3Sn alloy. Voids are observed at the dendritic boundaries. The cracks propagate along the dendritic boundaries, indicating that deformation by boundary sliding plays an important role in the creep of this alloy (Fig. 3(b)).

The creep life is only zero for the binary Mg–3Sn and Mg–5Sn alloys at 135 °C and a constant tensile load of 85 MPa (Table 2). The addition of Ca largely improves the creep properties of Mg–Sn alloy. The creep life increases to 358.4 h after 2 wt.% Ca was added to Mg–3Sn alloy (Table 2).