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Deformation-induced dynamic precipitation during creep in magnesium-tin alloys

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Abstract. The oversaturated solutes in the alloy matrix, which are caused by nonequilibrium phase transformation during casting or T4 treatment, can precipitate in the subsequent annealing treatments or during service at high temperatures. Under deformation the precipitation could be enhanced in comparison to conventional isothermal ageing. The present work investigates the dynamic precipitation of oversaturated Mg-Sn alloys during creep. The influence of this dynamic precipitation on creep properties is discussed. It is found that the distribution of deformation-induced precipitates is inhomogeneous. These precipitates are first formed at grain boundaries and then at deformation bands which are kinetically and thermodynamically favourable. The dynamic precipitation accompanies the volume change of phases, which influences the subsequent formation and growth of voids. Consequently, the creep behaviour also changes due to different precipitation under deformation.

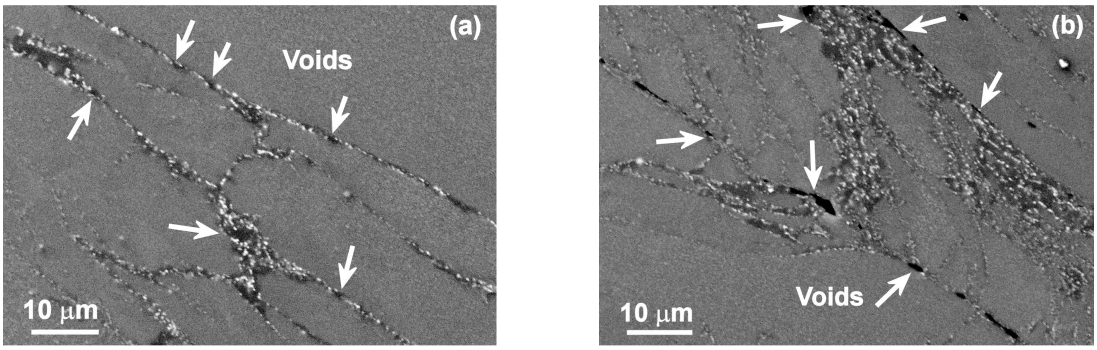
Introduction

Poor creep property at high temperatures is one of the factors to limit the extensive applications for magnesium alloys. The development of new heat-resistant magnesium alloys becomes a major research topic in the current time [1]. The principles to develop these new alloys mainly follow how to obtain thermally stable intermetallics at dendritic and grain boundaries, and how to suppress the formation of beta phase $Mg_{17}Al_{12}$ by the addition of rare earths (RE) to form Al-RE intermetallics [1, 2]. By these ways, the heat resistant magnesium alloys, such as Mg-Al-Nd, Mg-Y-Nd and Mg-Gd-Y alloys etc., were developed. However, recent investigations indicated that the thermal stability of intermetallics is not the exclusive factor to influence the creep resistance [3]. Other factors such as the segregation of primary alloying elements also affects the creep properties of magnesium alloys. The annealing treatments like T4 treatment can effectively increase the creep properties.

For the materials with T4 or underageing treatments, the second phases could re-precipitate during the subsequent creep deformation. Unlike the static precipitation, the formation of these second phases accompanies with the deformation. How the deformation influences the formation of second phases, and then how this process influences the creep response, should be an interesting investigation topic for magnesium alloys. The phenomenon of deformation-induced precipitation and its effects have been well-documented for traditional materials like steels and aluminum alloys. In contrast, the investigations on deformation-induced precipitation still received fewer attentions in magnesium alloys. Cizek et al. investigated the influence of deformation on the precipitation process in Mg-15 wt.% Gd alloys [4]. They found the deformation decreases the precipitating temperature and enhances the precipitation. Suziki and Zeng et al. investigated the creep behaviour and deformation microstructures of Mg-Y and Mg-Zn-Al-Y alloy [5, 6]. They indicated that the dynamic precipitating at dislocation lines is beneficial to increase the creep resistance, because these precipitates supply an effective barrier to the movement of dislocations. Besides the aforementioned precipitate strengthening effects, the dynamic precipitation possibly results in the volume change due to the solid phase transformations. How this volume change affects the formation of voids still remains unclear for magnesium alloys. Normally, the service temperature of the present commercially magnesium alloys is less than 200°C. It is therefore interesting to investigate how the deformation-induced precipitation influences the mechanical response at low temperatures. The present paper reports the dynamic precipitation during creep in Mg-3Sn alloy. How the dynamic precipitation proceeds and how it affects the formation of voids is discussed.

Experimental

The selected alloy was Mg-3Sn alloy (all values are given in wt.-%). Mg-Sn is a promising alloy system to develop new cheap creep resistant magnesium alloys by adding other alloying elements such as Ca [7]. Pure magnesium (99.98 %,) and pure tin (99.96 %) were used for the production of alloy. The alloy was cast using permanent mould casting. During the melting process a protective atmosphere of Ar+SF6 was employed. The melt was poured into a permanent steel mould preheated up to 350°C and then cooled down to room temperature by air cooling. The grain size is more than 300 µm. The cylindrical samples used for the compressive creep tests have a diameter of 6 mm and gauge length of 15 mm. Creep tests were performed using an ATS lever arm creep testing machine in air under a constant stress of 85 MPa and temperature of 135°C. The temperature was measured using Ni-CrNi thermocouple calibrated to an accuracy of ±3 °C. The microstructure was examined using an optical microscope and scanning electron microscope (SEM). SEM investigations were performed using a JSM 5310, with the accelerating voltage of 15 kV. Back scattered images and energy dispersive X-rays were used for characterization. The change in the sample size during isothermal treatment was measured using an Al_2O_3 tube dilatometer Netzsch Thermische Analyse DIL 402C equipped with TASC 414/3 controller. In order to suppress the precipitating during heating, a quick heating rate was taken with a value of 20°C/min.



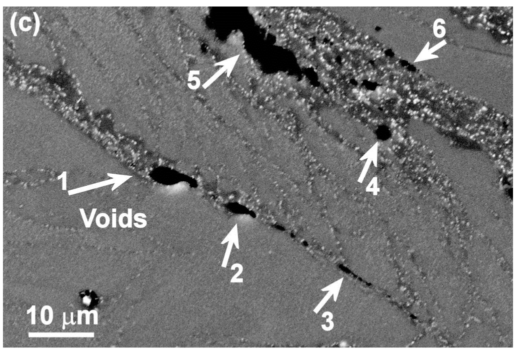


Figure 1. Microstructures for Mg-3Sn alloy with T4 treatment after creep rupture at 135°C and 85 MPa, (a) sample far from the fracture surface; (b) sample close to the fracture surface; (c) sample very close to the fracture surface, showing voids and dynamic precipitating at both grain boundaries and deformation bands.

Results and discussion

The as-cast microstructure before creep was described in Ref. [3, 7]. In the sample with T4 treatment, the deformation induced precipitation is clearly distinguished after creep deformation (Figure 1). In the regions far from the rupture surface (shear fracture surface), the precipitates are observed at the elongated grain boundaries (Figure 1 (a)). Small voids are also seen at the grain boundaries. In the regions close to the rupture surface, the deformation-induced precipitation occurs at both the grain boundaries and deformation bands (Figure 1 (b) and (c)). The size of voids increases compared with that formed in the areas far from the rupture surface. Besides the voids at the grain boundaries the voids are also found at the deformation bands, see No. 4 and 5 in Figure

1(c). It is interesting to notice that the size of particles looks larger at the grain boundaries than at the deformation bands. Based on all these observed phenomena, it can be concluded that, the deformation-induced precipitation first starts at the grain boundaries. With the deformation proceeding, these particles coarsen and the precipitation spreads over the deformation bands.

The defects such as the original boundaries, deformation bands and newly formed cracks are thermodynamically favourable sites as the nucleation position of these particles. The distribution of particles formed by deformation-induced precipitation is closely related to the deformation mechanism. For magnesium alloys they have an h.c.p crystal structure. Only two available slip systems operate at low temperatures [8]. Other slip systems become operative when temperature is more than 200°C. The deformation of magnesium is really inhomogeneous at low temperatures. An inhomogeneous distribution of particles is therefore expected formed by dynamic precipitation at low temperatures, as shown in Figure 1.

Several effects on the creep response are associated with the inhomogeneous distribution of precipitates formed by the deformation-induced precipitation. The positive effect is that, the formation of these precipitates is beneficial for hindering the movement of dislocation and the sliding of grain boundaries. The negative effects include the production of the interfacial thermal stress around the particles caused by the difference in the coefficients of thermal expansion between the particles and magnesium. This effect could be ignorable because the thermal stress can be alleviated at creep temperature (something like low temperature stress-removal annealing treatment). The final effect caused by the precipitation is the possible change of local volume (Figure 2). This effect may not only result in the production of internal stress but also enhance or suppress the formation of voids, which depends on how the local volume changes [9] In the binary Mg-Sn alloys, the precipitation of Mg_2Sn decreases the volume of specimen (Figure 2 (a)). This volume change seems to accelerate the growing of voids and is detrimental to the creep properties. During the creep, once the voids are formed these sites are also thermodynamically favourable for the nucleation of new particles (Figure 1 (c)). It can be expected the decrease in the local volume, i.e. the growth of voids, is consequently enhanced as to the precipitation of new particles and to the enhanced growth of previous particles. The following observed phenomena support the above explanation:

- Voids and cracks exist in the regions where the precipitates are clustered (Figure 1).
- No voids and cracks are observed in the precipitate-free regions.

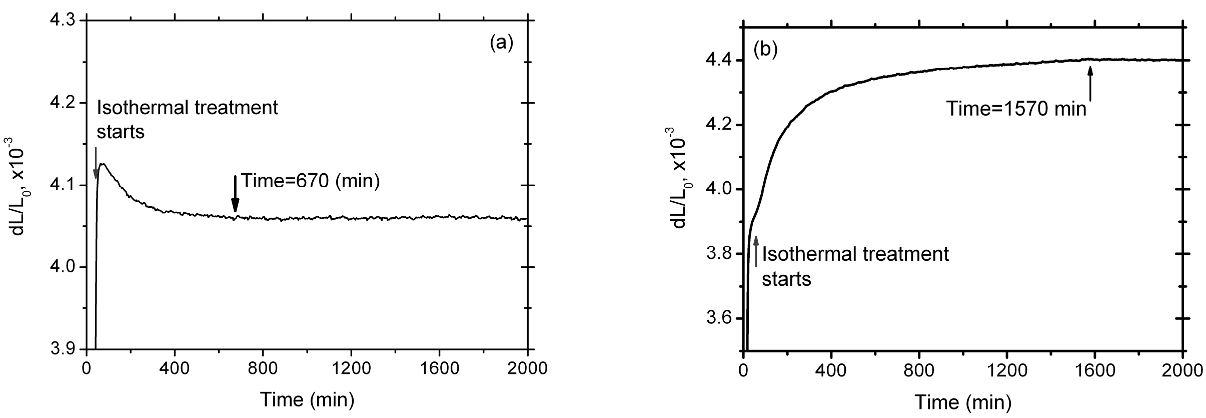


Figure 2. (a) Dimensional change during isothermal ageing (a) at 191°C for Mg-3Sn alloy with T4 treatment; (b) at 183°C for Mg-9Al alloy with T4 treatment.

Finally, it may be interesting to compare with the previous results of Mg-Al alloys and to answer why the creep properties of binary Mg-Sn alloys are comparable to or even worse than that of Mg-Al alloys (Table 1), even though in the Mg-Sn alloys more stable intermetallics Mg_2Sn is formed. As indicated in Table 1, the minimum creep rates of Mg-3Sn and Mg-5Sn alloys are comparable to that of AZ31 alloy, and less than that of AZ91 alloy. Unlike in Mg-Sn alloys, the isothermal ageing of Mg-9Al alloy leads to an increase in the volume of specimen (Figure 2). This indicates that the precipitation of beta phase $Mg_{17}Al_{12}$ increases the local volume. During creep deformation, the preferable dynamic precipitation of $Mg_{17}Al_{12}$ phase at the voids and cracks produce a compressive stress in these regions. Consequently, the compression of voids and cracks should slow their growth. The same beneficial effects of dynamic precipitation on the suppression of crack growth have also been observed in Al-Alloys [9].

Table 1. Minimum creep rates of Mg-Sn and Mg-Al alloys.

Alloy	Grain size (μm)	Creep parameters	Creep rate (s^{-1})		References
			As-cast	T4 treatment	
Mg-3Sn	>300	135°C, 85MPa, compressive creep	4.8×10^{-5}		Present
Mg-3Sn	>300	135°C, 85MPa, compressive creep		7.8×10^{-6}	Present
AZ91D	300	135°C, 85MPa, compressive creep	3.7×10^{-8}		Present
AZ31	98	150°C, 80 MPa shear creep		2.0×10^{-6}	[10]

Summary

The distribution of deformation-induced precipitates is inhomogeneous during low temperature creep. The precipitates are first formed at the grain boundaries. With the deformation increasing, they are then observed at the deformation bands inside the grains. In the sample with T4 treatment, the deformation-induced precipitation and the growth of precipitates proceed with the release of solute atoms from the oversaturation matrix. The dynamic precipitation accelerates the growth of the voids in Mg-3Sn alloys due to the decrease in the local volume. In contrast, the increase in the local volume caused by the precipitation of $Mg_{17}Al_{12}$ is helpful to suppress the formation and growth of voids in Mg-Al alloy.

References

[1] K.U. Kainer: *Magnesium Alloys and Technology* (Wiley-VCH GmbH, Weinheim, Germany 2003).

[2] A.A. Luo: International Materials Reviews 49 (2004), p. 13-30.

[3] Y.D. Huang, H. Dieringa, T. Abu Leil, N. Hort and K.U. Kainer: Scripta Mater. 58 (2008), p. 894-897.

[4] J. Cizek, I. Prochazka, B. Smola, I. Stulikova and V. Ocenasek: Journal of Alloys and Compounds 430 (2007), p. 92-96.

[5] M. Suzuki, H. Sato, K. Maruyama and H. Oikawa: Materials Science and Engineering A 252 (1998), p. 248-255.

[6] X. Zeng, H. Zou, C. Zhai and W. Ding: Materials Science and Engineering: A 424 (2006), p. 40-46.

[7] N. Hort, Y.D. Huang, T.A. Leil, P. Maier and K.U. Kainer: Advanced Engineering Materials 8 (2006), p. 359-364.

[8] M.M. Avedesian and H. Baker: *ASM Specialty Handbook: Magnesium and Magnesium Alloys* (ASM International, Materials Park, OH 1999).

[9] R. Lumley, in: Self Healing in Aluminium Alloys, edited by S. V. d. Zwaag, (2007), p. 219-254.

[10] H.-K. Kim and W.-J. Kim: Journal of Materials Science 42 (2007), p. 6171-6176.