Creep properties and microstructure of a Mg-6Al-1Nd-1.5Gd alloy at temperatures above 150 °C

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Abstract

Compressive creep behavior of a Mg-Al alloy containing a small amount of Nd and Gd (Mg-6Al-1Nd-1.5Gd) was investigated at temperatures from 150 °C to 200 °C under a constant applied stress of 90 MPa, and its microstructure before and after creep testing was compared. Results showed that steady-state creep rate of the alloy was only 1.946 × 10^{-8} /s at 150 °C, and was increased by four times and almost one order of magnitude at 175 °C and 200 °C, respectively. The microstructure of the alloy mainly consists of α-Mg, β-Mg_{17}Al_{12} phases, and Al_{3}RE phases, which were distributed both in dendrites of α-Mg and at grain boundaries originally. After creep for 120 h, more Al_{3}RE phases were aggregated at grain boundaries. The continuous β-Mg_{17}Al_{12} phase turned into dispersed dot-like or blocky particles. As the test temperature increased, the number of dislocation lines gradually increased due to the increase of creep strain. Meanwhile, dislocation tangle and dislocation pile-ups occurred near grain boundaries. However, obvious slip traces and slip lines appeared inside α-Mg dendrites at 175 °C and 200 °C, respectively, indicating that (c + a) non-basal slip system was activated, creep resistance decreased dramatically.

1. Introduction

As the lightest structural metallic materials, Mg-based alloys exhibit the advantages of low density, high specific strength and specific stiffness, good dimensional stability, damping capacity, and excellent electromagnetic shielding performance, hence have a wide application prospect in the fields of aerospace, new energy vehicles, and 5G communication [1, 2]. Mg-Al alloys are the most widely used commercial cast Mg-based alloys, and have been widely used in automobile, such as car dashboard, steering wheel, engine cradles, seat, transfer cases and many different housings due to their good castability, wide range of room temperature mechanical properties and high corrosion resistance [3, 4]. However, the creep resistance of Mg-Al alloys declines at temperatures above 120 °C due to the coarsening and softening of β-Mg_{17}Al_{12} phase (melting point is 473 °C) [5], which seriously limited their application in high-temperature components in powertrain system [6]. Therefore, it is urgent to improve the creep resistance of Mg-Al alloys.

It is reported that the addition of rare earth (RE) elements (such as Y, La, Ce, Gd and Nd) to Mg-Al alloys can significantly improve their creep resistance by forming stable Al-RE intermetallic phases instead of Mg_{17}Al_{12} phase [7–9]. Among these RE elements, Gd has a high equilibrium solid solubility in Mg, and the solid solubility decreases rapidly with decreasing temperature from 23.5 wt.% at 548 °C to 3.8 wt.% at 200 °C, thus significant improvement in properties can be achieved via solution strengthening and precipitation strengthening if a proper amount of Gd is added to Mg-Al alloys [10–12]. Nd is a kind of light rare earth element, and is an effective strengthening element for Mg-Al alloys, which can refine solidification microstructure and improve mechanical properties [13]. The presence of thermally stable Al-RE intermetallic phases can hinder both grain boundary migration and sliding during high temperature creep, thereby improving the creep resistance of Mg-Al alloys.
Microscope (precision of the displacement transducer and the temperature controller is 0.01 mm) and system, a loading device, a heating device, and a data acquisition and recording system, as shown in Figure 1.

Figure 1. Schematic diagram of the compressive creep equipment.

W Wang et al [14] found that when 1.0 wt.% Nd was added to AZ80 alloy, the grains were refined obviously, rod-shaped Al11Nd3 phase and blocky Al12Nd phase were formed, β-Mg17Al12 phases are refined and become discontinuous, and the combination properties reach an optimum consequently, although when the Nd amount increase to 1.5 wt.%, coarsening of Al11Nd3 phase will impair the mechanical properties. The content of Al11Nd3 phase increase with increasing Nd addition, and their morphology will become coarsen, which will cause the increase of hot cracking susceptibility of Mg–Al alloy due to the difference in solidification shrinkage rate between Al11Nd3 and γ-Mg [15].

Jiang et al [16] found that a combined addition of Nd and Gd to AZ80 alloys have a better strengthening effect than the addition of a single one. The addition of Gd could promote the precipitation of the block-shaped Al12Nd phase and inhibiting the precipitation of rod-shaped Al11Nd3 phase. Block-shaped Al12Gd and Al12Nd phases with high hardness can effectively hinder the movement of dislocation, thus the tensile strength, yield strength and elongation of the AZ80 alloy were significantly improved compared with the AZ80 alloys containing only Nd element. The author of present work previously investigated the mechanical properties of Mg–6Al–1Nd alloy containing 0–1.5 wt.% Gd and found that the optimal addition of Gd is 1.5 wt.%, then the tensile strength at room temperature was increased by 40.3%, creep strain and the steady-state creep rate were lowered by 40% and 39.3% than those of the Mg–6Al–1Nd alloy at 150 °C and 90 MPa, respectively [17]. However, the creep properties of Mg–6Al–1Nd–1.5Gd alloy at elevated temperatures above 150 °C need to be further studied, which are significant for the promotion and application of Mg–Al alloy in high-temperature components in powertrain system [18].

In present work, the compressive creep resistance of Mg–6Al–1Nd–1.5Gd alloy at temperature ranging from 150 °C to 200 °C was evaluated, and the microstructural change of the alloy before and after creep was investigated. The results are beneficial for understanding the deterioration mechanism of creep resistance for Mg–Al–RE alloys at temperature above 150 °C.

2. Materials and processing

Mg–6Al alloy was selected as the basic alloy, Nd and Gd were selected as the RE additives. The raw materials for this experiment were magnesium ingot (99.7 wt.%, purity), aluminum ingot (99.98 wt. % purity), Mg–30 wt.% Gd and Mg–30 wt.% Nd master alloys. A Mg–6.0Al–1.0Nd–1.5Gd (wt.%) alloy was prepared by permanent mold casting. Melting was carried out in a SG2–7.5–12A resistance furnace under RJ-2 covering flux (32%–40% KCl, 38%–46% MgCl2, 3%–5% CaF2, and 5%–8% BaCl2 in mass fraction) to prevent the oxidation and combustion of the melt. When the raw materials were melted completely at 700 °C, and the melt was held for 30 min for composition homogenization. After that, the melt was immediately poured into a preheated steel mold (200 °C).

Cylindrical specimens (Ø12 × 20 mm) for compressive creep test were cut from the obtained as-cast ingot. The compressive creep tests at elevated temperature were carried out in a creep testing machine under the applied stress of 90 MPa at 150, 175 and 200 °C. The creep testing machine consists of a temperature control system, a loading device, a heating device, and a data acquisition and recording system, as shown in figure 1. The precision of the displacement transducer and the temperature controller is 0.01 mm and ± 1 °C, respectively.

The microstructure of the alloys before and after creep tests was observed by a Scanning Electron Microscope (SEM, Zeiss-Merlin, Oberkochen, Germany) equipped with an energy dispersive spectroscopy (EDS) and a Transmission Electron Microscope (TEM, Talos F200X, FEI, USA). Among them, the highest acceleration voltage of the TEM is 200 KV, and the point resolution and information resolution are 0.25 nm and 0.12 nm, respectively. Phase identification was performed by an x-ray diffractometer (XRD, XRD-7000,
shimadzu, Japan). In addition, selected area electron diffraction (SAED) and high-resolution transmission electron microscopy (HRTEM) were used for phase characterizations. Samples for SEM observation were prepared by standard metallographic techniques and then etched with a 4 vol.% nitric acid-ethanol solution. The samples for TEM observation were cut from the as-cast ingot and the specimens after creep tests, and ground to approximately 1 mm in thickness, and polished to less than 0.1 mm, and then punched into a disk with 3 mm in diameter from the thin sample. Finally, thin foils were obtained by ion-milling technique in a high-precision ion-milling system (Gatan 691).

3. Results and discussion

3.1. Microstructure of the as-cast alloy

Figure 2 shows the SEM image of the alloy. It is seen that the microstructure mainly consists of light gray island-like phase, dark-grey network phase around the island-like phase, bright white rod-like phase and blocky phase, which are marked by A, B, C, D, respectively. The XRD pattern of the as-cast Mg-6Al-1Nd-1.5Gd alloy is presented in figure 3, which indicates that the alloy is mainly composed of $\alpha$-Mg, $\beta$-Mg$_{17}$Al$_{12}$ phase, Al$_2$Nd and Al$_2$Gd phases. The EDS analysis results are listed in table 1. According to the atomic ratios between the involved elements combined with the XRD pattern, it can be inferred that the light gray island-like phase in figure 2 (point A) is $\alpha$-Mg, and the dark gray phase (point B) around $\alpha$-Mg is $\beta$-Mg$_{17}$Al$_{12}$ phase (Mg:Al = 1.49:1 for point B, which is near to 17:12); the bright white rod-like phase (point C) and blocky phase (point D) are Al$_2$RE (Nd, Gd)

Figure 2. SEM image of the as-cast Mg-6Al-1Nd-1.5Gd alloy.

Figure 3. XRD pattern of the as-cast Mg-6Al-1Nd-1.5Gd alloy.
Al:RE = 2.17:1 for point C, and Al:RE = 2.22:1 for point D, which are all near to 2:1, which are thermally stable intermetallic phases (the melting points of Al$_2$Nd phase and Al$_2$Gd phase are 1205 °C and 1525 °C, respectively) [10, 19]. Both of Al$_2$Nd and Al$_2$Gd phases have the face-centered-cubic structure, and their lattice parameters are similar, hence Gd and Nd elements coexist in Al$_2$RE phases [16]. These Al$_2$RE phases are distributed both at grain boundaries and inside the dendrite of α-Mg.

3.2. The creep properties

Figure 4 (a) depicts the compressive creep curves of the Mg-6Al-1Nd-1.5Gd alloy performed at 150 °C, 175 °C and 200 °C. It is obtained that all the creep processes consist of primary and secondary creep stages, and no tertiary creep stage was found owing to compressive nature of the test, where necking did not take place. At the beginning of the primary creep stage (for about 5 h), the creep strain increases rapidly, and then increase slowly. After this stage, the creep strain increases almost with a linear feature, i.e. creep enters a steady state stage. The creep properties of the alloy were further evaluated by steady-state creep rate, which was defined as the slope of the creep strain-time curve in the steady state stage, and the obtained creep rate curves are shown in figure 4(b). The relationship between creep rate and creep time was also established by nonlinear fitting. It is obtained from figure 4(b) that all the creep rate curves were composed by decelerating creep stages and steady state creep stages. From figure 4, it is noticed that both the duration time of the decelerating creep stage and maximum strain increase with the increasing creep temperature. The maximum strain and the steady-state creep rates of the alloy at different temperatures are listed in table 2. It is seen that with the increase of the creep temperature, the steady-state creep rate also increases. When the test temperature increases from 150 °C to 175 °C, the maximum strain increases from 1.95% to 5.00%, and the steady-state creep rate increases from 1.946 × 10$^{-8}$/s to 7.897 × 10$^{-8}$/s, which are enhanced by about 2.56 and 4.06 times, respectively. When the creep temperature further

![Figure 4. Compressive creep curves and creep rate curves of the Mg-6Al-1Nd-1.5Gd alloy.](image-url)

| Temperature (°C) | Maximum strain (%) | Steady-state creep rate (/s) |
|------------------|--------------------|-----------------------------|
| 150              | 1.95               | 1.946 × 10$^{-8}$           |
| 175              | 5.00               | 7.897 × 10$^{-8}$           |
| 200              | 11.1               | 2.128 × 10$^{-7}$           |

| Composition analysis point | Mg (at.%) | Al (at.%) | Nd (at.%) | Gd (at.%) |
|----------------------------|-----------|-----------|-----------|-----------|
| A                          | 97.72     | 2.28      | —         | —         |
| B                          | 59.84     | 40.16     | —         | —         |
| C                          | 10.76     | 61.12     | 10.31     | 17.81     |
| D                          | 10.79     | 61.48     | 8.65      | 19.08     |

Table 1. EDS analysis results of the as-cast Mg-6Al-1Nd-1.5Gd alloy.
**Figure 5.** SEM images of the alloy after creep at 150 °C (a), 175 °C (b), and 200 °C (c).

**Figure 6.** TEM image of the β-Mg$_{17}$Al$_{12}$ phase in the alloy after creep at 200 °C and its corresponding SAED pattern (a), color mappings of Mg (b) and Al (c).
increases to 200 °C, the maximum strain and steady-state creep rate reach 11.1% and 2.128 × 10^{-7}/s, which are 5.69 and 10.94 times higher than those at 150 °C, respectively. It is clear that with the increase of creep temperature from 150 to 200 °C, the decline of the creep resistance was greater and greater.

3.3. Microstructure after creep

Figure 5 illustrates the SEM morphology of the alloys crept at different temperatures. It can be seen from figure 5 that after creep, most of the Al2RE phases aggregated at the grain boundaries of α-Mg. The aggregation of Al2RE phases at grain boundaries after creep can be due to that the equilibrium volume fraction was not reached during solidification, under a long-term creep at high temperatures, partial Gd elements solutioned in the α-Mg dendrites became active, and was precipitated at grain boundaries, combining with Al preferentially to form new Al2RE phases, hence the amount of Al2RE phases at grain boundaries seems increased somewhat.

It is well known that the poor creep resistance of Mg–Al alloys at elevated temperature is mainly due to the softening of β-Mg17Al12 phase [20, 21]. TEM image of the Mg-6Al-1Nd-1.5Gd alloy after creep at 200 °C is

![Figure 7. TEM images of the alloy after creep at 150 °C (a), 175 °C (b) and (c), 200 °C (d) and (e).](image-url)
shown in figure 6(a), it is observed that there are massive light-grey blocky phases, and its corresponding SAED pattern is given in inset. It is seen from the SAED pattern that the light-grey phase has a body-centered cubic structure with lattice parameters $a = b = c = 1.056$ nm. Figures 6(b) and (c) show the mapping of the Mg alloy. It is observed that the Al element is mainly distributed at the grain boundaries, and just corresponds to the light-grey blocky phase, hence it is inferred that the dispersed light-grey blocky phase is $\beta$-Mg$_{17}$Al$_{12}$. By comparing figure 6(a) with figure 2, it is noticed that the original network $\beta$-Mg$_{17}$Al$_{12}$ phase was broken into discontinuous blocks after the creep deformation. The morphology transition of the $\beta$-Mg$_{17}$Al$_{12}$ phase can be attributed to following two aspects: firstly, it is incoherent with $\alpha$-Mg matrix (the magnesium matrix has a hcp lattice, while Mg$_{17}$Al$_{12}$ phase has a cubic structure), so it is easy to be broken under compressive stress. Secondly, partial Al-rich regions of the $\beta$-Mg$_{17}$Al$_{12}$ phase may decompose during creep [22]. At low temperature, hard network $\beta$-Mg$_{17}$Al$_{12}$ phase plays a strengthening role in form of interconnected skeleton. However, it softens and coarsens at elevated temperatures, hence gradually lost its strengthening effect consequently.

Figure 7 illustrates the typical bright-field TEM micrographs of the alloy after creep. It is seen from figure 7(a) that a small number of dislocations appeared in $\alpha$-Mg dendrite after creep at 150 °C. When the creep temperature rises to 175 °C, slip traces with sliding trend emerged in $\alpha$-Mg dendrite (see figure 7(b)). Moreover, there were a small number of dislocations which cross and tangle with each other, as shown in figure 7(c). When the creep temperature reaches 200 °C, obvious parallel arranged slip lines appeared in $\alpha$-Mg dendrite (see figure 7(d)), indicating that $\langle c + a \rangle$ non basal slip system was activated. In addition, dense dislocation pile-ups and dislocation tangles are observed in $\alpha$-Mg dendrite near their boundaries, as shown in figure 7(e).

The plastic deformation of Mg alloys at room temperature is mainly controlled by two independent slip systems, i.e. (0001) $\langle 11 \bar{2} 0 \rangle$ and (0001) $\langle 12 \bar{1} 0 \rangle$ on basal plane [23]. With the increase of the creep temperature, the critical shear stress required for activating non-base slip system gradually decreases. In present study, the creep temperatures are lower than 225 °C, above which $\langle c + a \rangle$ non-basal slip system can be opened easily via thermal activation, and dominate the plastic deformation of polycrystalline magnesium together with basal slip [24]. The samples were exposed to high temperature and compressive stress for a long time, so the transition energy of atoms in the Mg alloy increased [25]. As a result, the non-base slip systems were activated at 200 °C. The dense dislocation pile-ups and dislocation tangles can hinder grain boundary sliding, but the activation of the non-base slip systems caused the dramatic reduction of the creep resistance at 200 °C.

4. Conclusions

(1) The steady-state creep rate and maximum strain were increased from $1.946 \times 10^{-8}$ and 1.95% to $2.128 \times 10^{-7}$ and 11.1%, which were increased by almost one order and 4.69 times when the creep temperature increased from 150 °C to 200 °C, respectively.

(2) The as-cast Mg-6Al-1Nd-1.5Gd alloy mainly consists of $\alpha$-Mg, $\beta$-Mg$_{17}$Al$_{12}$ phases, and intermetallic phase Al$_{12}$RE (Al$_2$Gd and Al$_2$Nd). The Al$_{12}$RE phases are distributed both in the $\alpha$-Mg dendrite and at the grain boundaries.

(3) More Al$_{12}$RE phases aggregated to the grain boundaries of $\alpha$-Mg after creep, the morphology of the $\beta$-Mg$_{17}$Al$_{12}$ phase changed from original network to dispersed blocks, and the strengthening effect were weakened consequently.

(4) Obvious slip traces and slip lines appeared in the $\alpha$-Mg grains of the alloy after creep at 175 °C and 200 °C, respectively. The activation of $\langle c + a \rangle$ non-basal slip system caused the dramatic reduction of the creep resistance.

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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).
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