

Effect of different artificial aging treatments on tensile creep behavior of extruded lean Mg–Al–Ca–Mn alloy

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Abstract: The effects of artificial aging (T6) on the creep resistance with tensile stresses in the range of 50–80 MPa at 175 °C were investigated for an extruded Mg–1.22Al–0.31Ca–0.44Mn (wt.%) alloy. The Guinier-Preston (G.P.) zones primarily precipitate in the sample aged at 200 °C for 1 h (T6-200°C/1h), while the Al₂Ca phases mainly precipitate in the sample aged at 275 °C for 8 h (T6-275°C/8h). The T6-200°C/1h sample exhibits excellent creep resistance, with a steady-state creep rate one order of magnitude lower than that of the T6-275°C/8h sample. The abnormally high stress exponent (~8.2) observed in the T6-200°C/1h sample is associated with the power-law breakdown mechanism. TEM analysis illuminates that the creep mechanism for the T6-200°C/1h sample is cross-slip between basal and prismatic dislocations, while the T6-275°C/8h sample exhibits a mixed mechanism of dislocation cross-slip and climb. Compared with the Al₂Ca phase, the dense G.P. zones effectively impede dislocation climb and glide during the creep process, demonstrating superior creep resistance of the T6-200°C/1h sample.

Keywords: Mg–Al–Ca–Mn alloy; tensile creep; artificial aging; cross slip

1 Introduction

The magnesium (Mg) alloys have gained considerable interest in response to the increasing need for lightweight materials in the transportation and aerospace industries. This is primarily because of their favorable specific strength and low density [1–3]. However, poor creep performance limits their high-temperature applications [4,5]. In recent years, research has focused mainly on enhancing the creep resistance of Mg-based alloys by adding rare-earth (RE) elements [6–8]. Many Mg–RE alloys with excellent creep performance have been developed, most of which involve Mg–Gd and Mg–Y based

alloys. However, the addition of RE elements increases the cost of alloys, making them unsuitable for widespread commercial applications. Therefore, the development of low-cost, heat-resistant Mg alloys is crucial. Among non-RE Mg alloys, the Mg–Al–Ca–Mn alloy system has gained widespread attention owing to its excellent creep resistance and cost-effectiveness [9–11].

Creep is a critical consideration in engineering applications in which components are subjected to prolonged mechanical stress at elevated temperatures. The principal creep mechanisms include atomic diffusion, dislocation glide, and climbing [12–15]. The evolution of the microstructure in Mg–Al–Ca–Mn alloys during the creep,

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specifically the alterations in the precipitate morphology, distribution, and phase composition, significantly impacts their mechanical properties and resistance to creep. The addition of Ca can result in the formation of thermally stable intermetallic compounds, specifically, Mg_2Ca (C14) and Al_2Ca (C15) [16]. ZHU et al [17] studied squeeze-cast Mg–Al–Ca alloys and reported that the $(Mg,Al)_2Ca$ phases improve creep resistance. SUZUKI et al [18] reported that the Al_2Ca phase in a die-cast Mg–Al–Ca alloy poses a significant obstacle to non-basal dislocation slip. Moreover, artificial aging treatments exert a notable influence on the microstructural features of these alloys, consequently affecting their creep behavior. Single-layer ordered Guinier-Preston (G.P.) zones were observed in aged Mg–0.5Al–0.3Ca (wt.%) alloy [19] and Mg–3.6Al–3.4Ca–0.3Mn (wt.%) alloy [20]. HOMMA et al [21] demonstrated that the addition of Mn significantly enhances the creep resistance of Mg–6Al–3Ca (wt.%) alloys, and both the Al_2Ca phases and spherical precipitates (Al–Mn phases) improve the creep properties. Furthermore, dynamic precipitation in the G.P. zone also contributes to the creep resistance of the alloy.

The precipitates resulting from the artificial aging treatment may have a more complex impact on creep performance. AMBERGER et al [22] found that heat treatment did not significantly affect the creep performance of AZ91 alloy with the addition of 5 wt.% Ca. ZHANG et al [23] also observed that Mg alloys exhibited superior creep resistance after overaging. Although many studies have proposed various factors that affect the creep performance of Mg–Al–Ca–Mn alloys, there is still limited research on the effects of different aging conditions on creep performance. Because precipitates can be controlled by adjusting the aging conditions, it is essential to accurately identify the distinct influence of each type of precipitate on creep resistance. Hence, the Mg–1.22Al–0.31Ca–0.44Mn (AXM100) alloy was selected as the focus of this research to analyze the effects of the G.P. zones and precipitate phases on the creep properties through different aging treatments. This study aims to fill the existing research gap concerning the creep behavior of AXM100 alloy under different aging conditions and establish the relationship between aging treatments and creep properties. The findings of this study provide valuable insights into the development of cost-

effective, novel, high-temperature, and creep-resistant magnesium alloys.

2 Experimental

The composition of the Mg–1.22Al–0.31Ca–0.44Mn (AXM100) alloy was determined using an optical spectrum analyzer (ARL 4460, Switzerland), and the results are presented in Table 1. Commercially pure Mg (99.90 wt.%) was first melted in an electric furnace at ~ 690 °C under a protective gas mixture of 99.5% CO_2 and 0.5% SF_6 . Then, pure Al (99.90 wt.%), Mg–23.5Ca (wt.%), and Mg–3.5Mn (wt.%) were added into the Mg melt. The homogenization treatment process for the AXM100 alloy ingots included treatment at 300 °C for 3 h, followed by treatment at 420 °C for 8 h, and then at 450 °C for 4 h. The billets were subsequently extruded to form plates 40 mm wide and 5 mm thick at 390 °C, with an extrusion ratio of $\sim 35:1$. The extruded sheets were subjected to solid-solution (T4) treatment at 500 °C for 0.5 h. Subsequently, the T4 samples were subjected to artificial aging (T6) at 200 and 275 °C.

Table 1 Composition of AXM100 alloy (wt.%)

Al	Ca	Mn	Fe	Si	Mg
1.22	0.31	0.44	<0.01	<0.02	Bal.

The gauge length of the tensile creep samples is 25 mm (along the extrusion direction), with a gauge width of 6.5 mm and thickness of 5 mm, as shown in Fig. 1. Tensile creep tests were conducted using an electronic creep test machine (RDL50) equipped with an environmental chamber capable of controlling the temperature to be within ± 1 °C. The samples were heated to 175 °C, held for 20 min, and then pulled under four constant applied stresses of 50, 60, 70, and 80 MPa, respectively. The creep deformation within the gauge length of the sample was measured using two symmetrically arranged grating rulers with a precision of ± 0.5 μm .

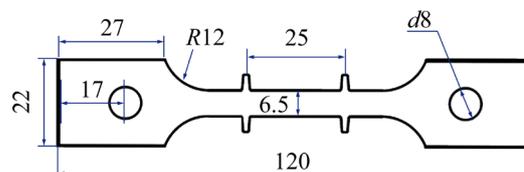


Fig. 1 Dimensions of tensile creep specimen (Unit: mm)

The Vickers microhardness of the alloy after the aging treatment was measured using a microhardness tester (HVS-1000A). The average hardness values were recorded based on at least ten measurements. Electrical conductivity was measured using a D60K digital test instrument. The microstructure was characterized using a scanning electron microscope (SEM, ZEISS Sigma 500) equipped with an EBSD (Oxford Instrument). The EBSD samples were electro-polished at 20 V for 90–120 s at $-30\text{ }^{\circ}\text{C}$. The EBSD data were analyzed using AZtec and Channel 5.0. The precipitation behavior and dislocation characterization were observed using transmission electron microscopy (TEM, JEM-2100F). Circular test samples with a thickness of approximately $80\text{ }\mu\text{m}$ and diameter of approximately 3 mm were prepared for TEM observation. Subsequently, the samples were thinned using a precision ion thinning instrument (Gatan 691), and the temperature was maintained below $-120\text{ }^{\circ}\text{C}$ using liquid nitrogen.

3 Results

3.1 Initial microstructures

SEM backscattered electron (BSE) images illustrate the phases of the as-extruded and T4-treated AXM100 alloys. Surface scans conducted using energy-dispersive X-ray spectroscopy (EDS) reveal the presence of Al–Mn and small Al–Ca

phases within the extruded sample. From Fig. 2(b), it is observed that after 0.5 h T4 treatment at $500\text{ }^{\circ}\text{C}$, nearly all the Al–Ca phases dissolve back into the Mg matrix, with the remaining phases primarily consisting of Al–Mn phases.

ZHANG et al [24] determined that the formation of G.P. zones was still evident after aging at T6 treatment ($250\text{ }^{\circ}\text{C}$, 8 h). As a result, aging at 200 and $275\text{ }^{\circ}\text{C}$ was selected to compare the effects of G.P. zones and Al_2Ca on the creep behavior. Figure 3(a) presents the Vickers hardness of the AXM100 alloy at various aging temperatures over time. The hardness decreases noticeably after T4 treatment. After heating at $200\text{ }^{\circ}\text{C}$ for 1 h, a peak-aging hardness of $\sim\text{HV } 61$ is reached, indicating a rapid aging response. The hardness remains relatively stable for 100 h. At $275\text{ }^{\circ}\text{C}$, a peak aging hardness of HV 53 is achieved at 0.5 h, which is lower than the peak hardness at $200\text{ }^{\circ}\text{C}$.

In terms of electrical conductivity, as depicted in Fig. 3(b), there is minimal variation in the electrical conductivity at $200\text{ }^{\circ}\text{C}$ with increasing aging time. However, at $275\text{ }^{\circ}\text{C}$, the electrical conductivity continues to increase with increasing aging time. When the G.P. zones precipitate and remain stable, the electrical conductivity remains relatively constant. After aging at $275\text{ }^{\circ}\text{C}$, particles precipitate from the matrix and continue to grow. This reduction in supersaturated solute atoms results in a decrease in the lattice distortion of the matrix,

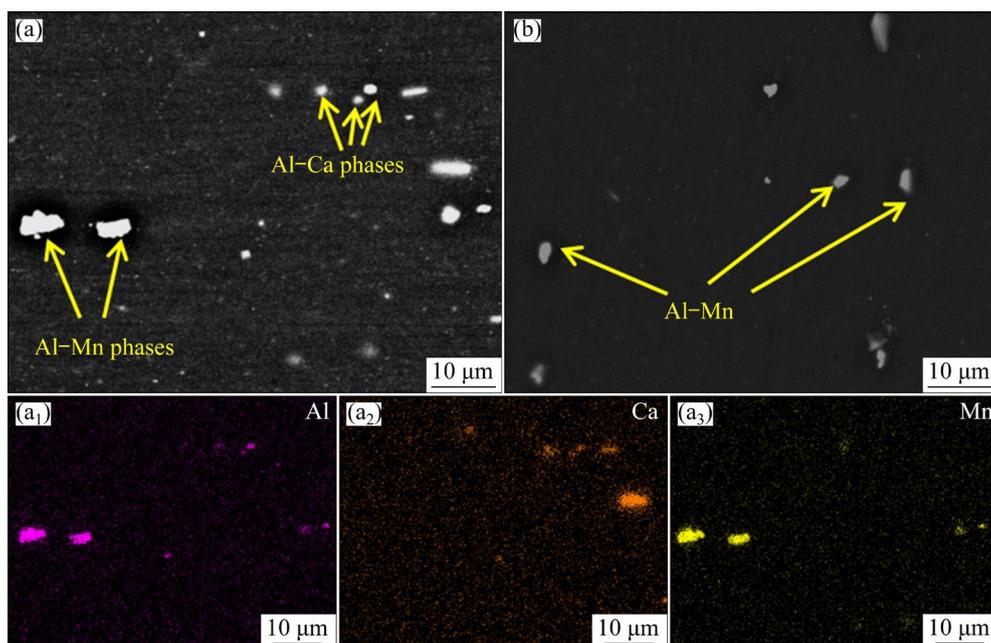


Fig. 2 SEM image and EDX mappings for extruded (a, a₁–a₃) and T4-treated (b) AXM100 alloy

leading to an increase in electrical conductivity [25,26]. The sample aged at 200 °C for 1 h is labeled as T6-200°C/1h, and the sample aged at 275 °C for 8 h is labeled as T6-275°C/8h. To investigate the influence of the G.P. zone and precipitates on the relative creep rates and rupture strains, T6-200°C/1h and T6-275°C/8h samples were chosen for further analysis.

TEM was employed to characterize the aging precipitates with different T6 treatments. Observations were made from the zone axes of $[10\bar{1}0]$ and $[\bar{2}110]$, as shown in Fig. 4. Figure 4(a) shows the presence of high dense G.P. zones through T6 (200 °C, 1 h) treatment, which are parallel

to the $(0002)_{Mg}$ basal plane. The corresponding Fast Fourier Transform (FFT) pattern in Fig. 4(b) displays four distinct continuous stripes at the $1/3$ and $2/3 \{11\bar{2}0\}$ positions (yellow arrows), indicating the presence of G.P. zones. The HRTEM image in Fig. 4(c) reveals that the G.P. zone, with a length of ~ 5 nm and a monatomic layer thickness of ~ 0.51 nm, exhibits a coherent interface with the Mg matrix. The observed characteristics of the G.P. zone align with those reported by OH-ISHI et al [27] and PING et al [28]. In Fig. 4(d), many Al_2Ca phases with an average length of ~ 60 nm and thickness of ~ 2.87 nm are dispersed along the $(0002)_{Mg}$ basal plane. The absence of stripes in Fig. 4(e) indicates

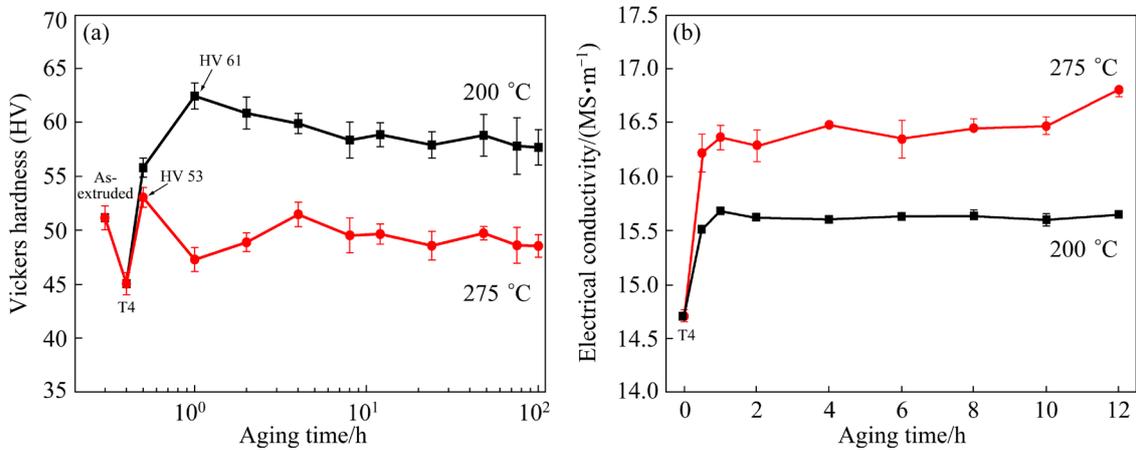


Fig. 3 Hardness (a) and electrical conductivity (b) changes of alloy aged at various temperatures

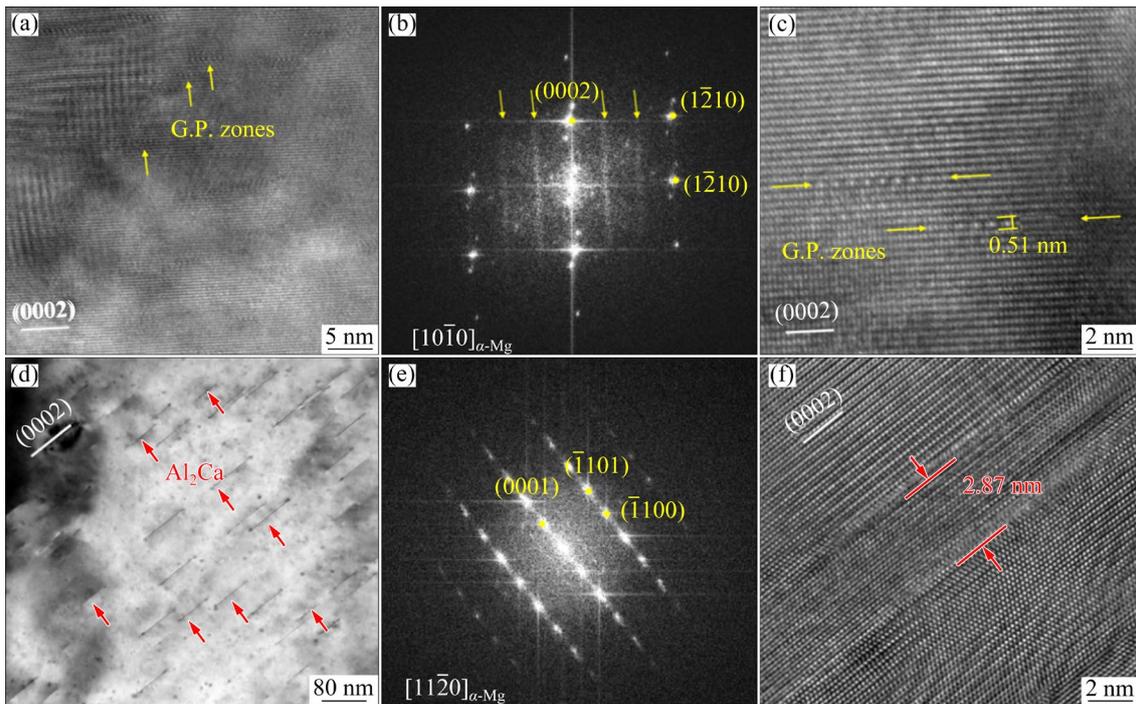


Fig. 4 Bright field (BF) TEM images (a, d), FFT patterns (b, e), and HRTEM images (c, f) of T6-200°C/1h (a-c) and T6-275°C/8h (d-f) samples

that the G.P. zones cannot maintain stability during T6 (275 °C, 8 h) treatment.

Furthermore, EBSD analysis was performed on alloys subjected to two different aging treatments, as depicted in Fig. 5. It is evident that the alloy exhibits a typical fiber basal texture, with the *c*-axes of the majority of grains being perpendicular to the ED. The grain size of the T6-200°C/1h sample is approximately 54 μm, while that of the T6-275°C/8h sample is nearly 60 μm. Both aged samples display similar grain sizes and texture strengths, indicating that these factors do not influence the creep properties of the alloy.

3.2 Tensile creep properties

Figure 6 presents the creep strain versus time curves at 175 °C with tensile stresses in range of 50–80 MPa. The G.P. zones can be stably present at 175 °C, and a stress range of 50–80 MPa is also a common research scope for low-content non-rare earth Mg alloys [14]. Tables 2 and 3 present a summary of the steady-state creep rates and the corresponding creep strains over 50 and 100 h for

AXM100 alloy under various conditions. As the stress increases, the steady-state creep rate increases. Under the same stress and temperature conditions, the T6-200°C/1h sample exhibits excellent creep resistance, with a steady-state creep rate one order of magnitude lower than that of the T6-275°C/8h sample. With the tensile stresses of 50 MPa, the creep strain over 100 h for the T6-275°C/8h sample is ~2.13%, almost ten times that of the T6-200°C/1h sample (~0.22%). The T6-275°C/8h samples with the tensile stresses of 70 and 80 MPa exhibit the third stage of creep with fractures occurring at 96 and 49 h, respectively. These results indicate that the creep resistance of the T6-200°C/1h samples is significantly superior to that of the T6-275°C/8h samples and that the presence of G.P. zones contributes to improving the creep resistance.

Furthermore, the creep resistance of the T6-200°C/1h sample exhibits certain advantages compared to commercial alloys such as AZ31, AZ61, and AZ91 [29]. CHEN et al [29] conducted tensile creep experiments on peak-aged AZ91 alloy and found that the steady-state creep rate with the tensile

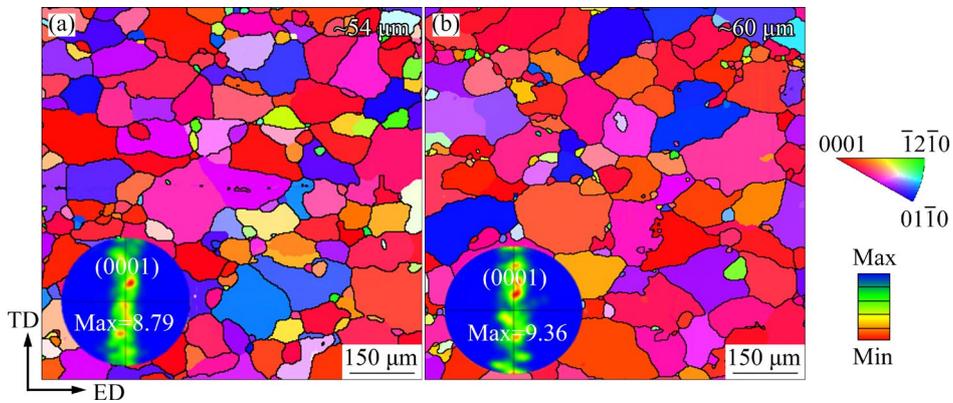


Fig. 5 Inverse pole figure of samples: (a) T6-200°C/1h; (b) T6-275°C/8h

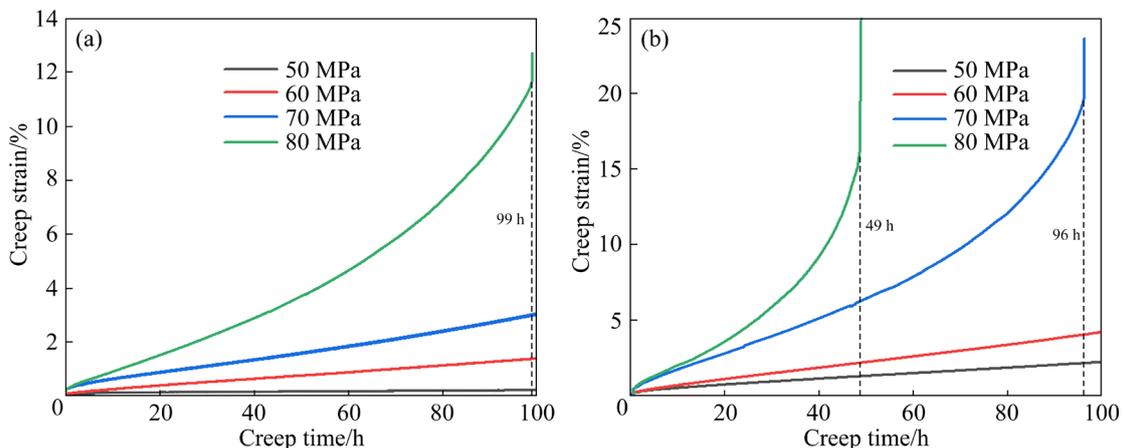


Fig. 6 Tensile creep curves of samples at 175 °C: (a) T6-200°C/1h; (b) T6-275°C/8h

Table 2 Creep data of T6-200°C/1h sample at 175 °C

σ/MPa	$\dot{\epsilon}_m/\text{s}^{-1}$	$\epsilon_{50\text{h}}/\%$	$\epsilon_{100\text{h}}/\%$	$\epsilon_{\text{fracture}}/\%$
50	3.08×10^{-9}	0.17	0.22	–
60	3.45×10^{-8}	0.76	1.39	–
70	6.51×10^{-8}	1.56	3.05	–
80	1.67×10^{-7}	3.69	–	11.70

Table 3 Creep data of T6-275°C/8h sample at 175 °C

σ/MPa	$\dot{\epsilon}_m/\text{s}^{-1}$	$\epsilon_{50\text{h}}/\%$	$\epsilon_{100\text{h}}/\%$	$\epsilon_{\text{fracture}}/\%$
50	5.30×10^{-8}	1.21	2.13	–
60	1.03×10^{-7}	2.09	4.08	–
70	3.19×10^{-7}	6.62	–	15.17
80	6.57×10^{-7}	–	–	19.74

stress of 70 MPa at 180 °C was $\sim 1.50 \times 10^{-7} \text{ s}^{-1}$. In contrast, the creep resistance of the T6-200°C/1h sample is more superior under similar stress and temperature conditions compared to the peak-aged AZ91 samples. Additionally, ZHANG et al [30] conducted creep experiments on a high-alloy content die-cast AXM90302 alloy at 150 °C and 70 MPa, where the alloy exhibited a steady-state creep rate of $\sim 1.24 \times 10^{-7} \text{ s}^{-1}$ and a creep deformation of $\sim 6.28\%$ over 100 h. In comparison, the T6-200°C/1h sample, with its lower alloying content, demonstrates a steady-state creep rate nearly an order of magnitude lower under the same stress and temperature conditions. Creep strain over 100 h is only $\sim 3.05\%$, indicating more superior creep resistance.

3.3 Creep stress exponent

The influence of stress and temperature on the steady-state creep rate ($\dot{\epsilon}_m$) can be expressed using the power-law relationship [31]:

$$\dot{\epsilon}_m = A\sigma^n \exp[-Q/(RT)] \quad (1)$$

where A is a constant that depends on the material, σ is the applied stress, n is the stress exponent, Q is the activation energy for creep, R is the molar gas constant and T is the thermodynamic temperature. A double logarithmic plot of the steady creep rates as a function of the applied stress at 175 °C is shown in Fig. 7. According to Eq. (1), the stress exponent n can be determined using the following equation [31]:

$$n = \left[\frac{\partial \ln \dot{\epsilon}_m}{\partial \ln \sigma} \right]_T \quad (2)$$

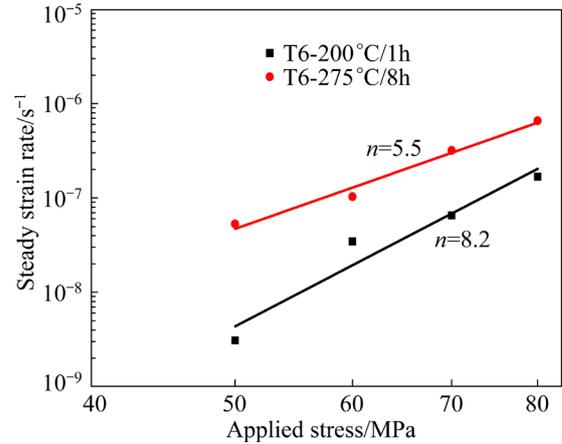


Fig. 7 Double-logarithmic plots of steady-state creep rate versus applied stress for T6-200°C/1h and T6-275°C/8h samples

The T6-200°C/1h and the T6-275°C/8h samples have the n values of ~ 8.2 and ~ 5.5 , respectively. The value of $n=1$ corresponds to diffusion based creep; when $n=2$, grain boundary sliding plays a dominant role in controlling the rate. When $n=3-7$, dislocation movements (glide and/or climb) become the primary deformation mechanisms during the creep [32,33]. Hence, the creep behavior of the T6-275°C/8h samples is primarily governed by dislocation motion. When $n>7$ in the T6-200°C/1h sample, a power-law breakdown may occur. Therefore, further analysis is required to determine the stress exponent.

SHERBY and BURKE [34] proposed a criterion for material creep power-law breakdown. The conditions under which power-law breakdown occurs are as follows:

$$\dot{\epsilon}/D > 10^{13} \text{ m}^{-2} \quad (3)$$

D is the lattice diffusion coefficient for rate-controlled creep and satisfies the following relationship [35]:

$$D = D_0 \exp[-Q_{sd}/(RT)] \quad (4)$$

where D_0 is $10^{-4} \text{ m}^2/\text{s}$, and Q_{sb} (lattice self-diffusion energy) is 135 kJ/mol [36]. The applied stress is normalized using the shear modulus, and the temperature dependence of the shear modulus of Mg can be determined by $\mu = 1.92 \times 10^4 - 8.6T$ [37]. The steady-state creep rate normalized by the activation energy for the self-diffusion of Mg is plotted against the effective stress normalized by the shear modulus in Fig. 8. All data are within the range of the power-law breakdown. Therefore, the abnormally high stress

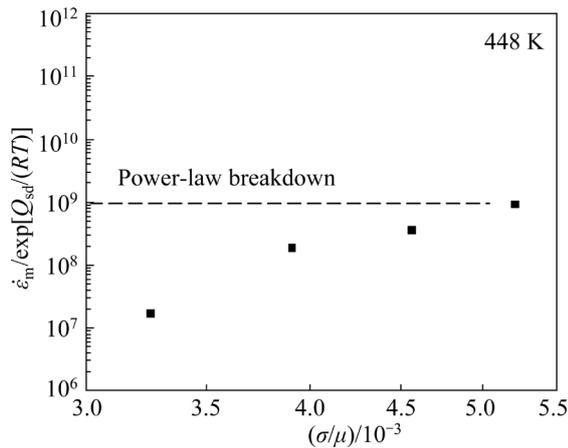


Fig. 8 Plot of steady-state creep rate normalized by activation energy for self-diffusion of Mg against stress normalized by shear modulus

exponent obtained under 448 K and 50–80 MPa conditions is attributed to the dominant role of the alloy power-law breakdown. ZHU et al [17] investigated the creep mechanism of squeeze-cast Mg–Al–Ca alloys in the range of 30–120 MPa and 423–473 K. It was found that there is a high stress exponent that corresponds to a power-law breakdown mechanism.

4 Discussion

4.1 Creep deformation mechanisms

To further elucidate the creep mechanism, TEM observations were performed to examine the microstructural evolution and dislocation patterns. Figures 9 and 10 present typical TEM images of the samples crept at 70 MPa for 50 h, with the corresponding incident beam directions being $B=[\bar{2}110]$.

With the diffraction vector $g=[01\bar{1}0]$, only $\langle a \rangle$ dislocations were detected based on the $g \cdot b=0$ criterion (Fig. 9(a)) [38,39]. The straight lines parallel to the basal plane trace represent the basal $\langle a \rangle$ dislocations, as indicated by the short red arrows. The other short segments perpendicular to the basal plane trace correspond to prismatic $\langle a \rangle$ dislocations [40]. In addition, some kinked or curved dislocation lines (in yellow) are observed, and their angles with respect to $[0002]_{Mg}$ were neither 90° nor 0° , indicating that these line segments may lie in pyramidal planes and/or exist as mixed dislocations [41]. The dark-field image (Fig. 9(b)) clearly shows a significant occurrence of basal $\langle a \rangle$ and prismatic $\langle a \rangle$ slips. With the diffraction vector $g=[0002]$, only $\langle c \rangle$

dislocations are visible, suggesting that the $\langle c \rangle$ dislocations are barely activated (Figs. 8(c, d)). It is evident that the presence of G.P. zones has an inhibitory effect on the prismatic $\langle a \rangle$ slip on pyramidal planes. Hence, for T6-200°C/1h samples, the primary creep mechanism involves the cross-slip of basal $\langle a \rangle$ and prismatic $\langle a \rangle$ dislocations. The G.P. zones are shown to inhibit $\langle c \rangle$ slip. Additionally, in Fig. 9(e), HRTEM images reveal the stable presence of G.P. zones in the T6-200°C/1h sample. There is virtually no precipitation of Al_2Ca during creep deformation. This observation indicates that the G.P. zones consistently maintain a smaller size for their monatomic layer, contributing to the improvement in the creep resistance of the alloy. Therefore, the cross-slip of screw dislocations from the basal plane to the prismatic plane may be the controlling mechanism for the power-law breakdown creep observed in the T6-200°C/1h samples.

The incident beam direction for T6-275°C/8h sample is $B=[\bar{2}110]$. Under $g=[01\bar{1}0]$, a cross-slip containing basal and prismatic dislocation is observed in Fig. 10(b) (indicated by the yellow arrows). The number of cross-slips is higher than that in the T6-200°C/1h sample, indicating that the G.P. zones exert a stronger hindrance to cross-slip than Al_2Ca phases. For the observation under $g=[01\bar{1}0]$, in Figs. 9(a, b), clusters of short slip lines are parallel to basal plane trace. These are basal $\langle a \rangle$ dislocations arranged as dislocation channels (as indicated by the green dotted rectangles), which result from the climbing of dislocations [23]. In Figs. 9(c, e), no obvious $\langle c \rangle$ dislocations are observed. Figure 10(d) shows that a large amount of Al_2Ca phases are uniformly distributed within the grains. During creep deformation, there is an increased quantity of the Al_2Ca phases. Therefore, the creep deformation in the T6-275°C/8h sample is induced by the collective mechanism involving the cross-slip of basal $\langle a \rangle$ and prismatic $\langle a \rangle$ dislocations along with the climb of basal $\langle a \rangle$ dislocations.

4.2 Influence of G.P. zones and Al_2Ca phases on creep properties

Both the T6-200°C/1h and T6-275°C/8h samples exhibit Al–Mn phases. Therefore, the differences in creep properties are primarily determined by the G.P. zones and Al_2Ca phases. In summary, at 175 °C and 50–80 MPa, the creep mechanisms for the T6-200°C/1h and T6-275°C/8h

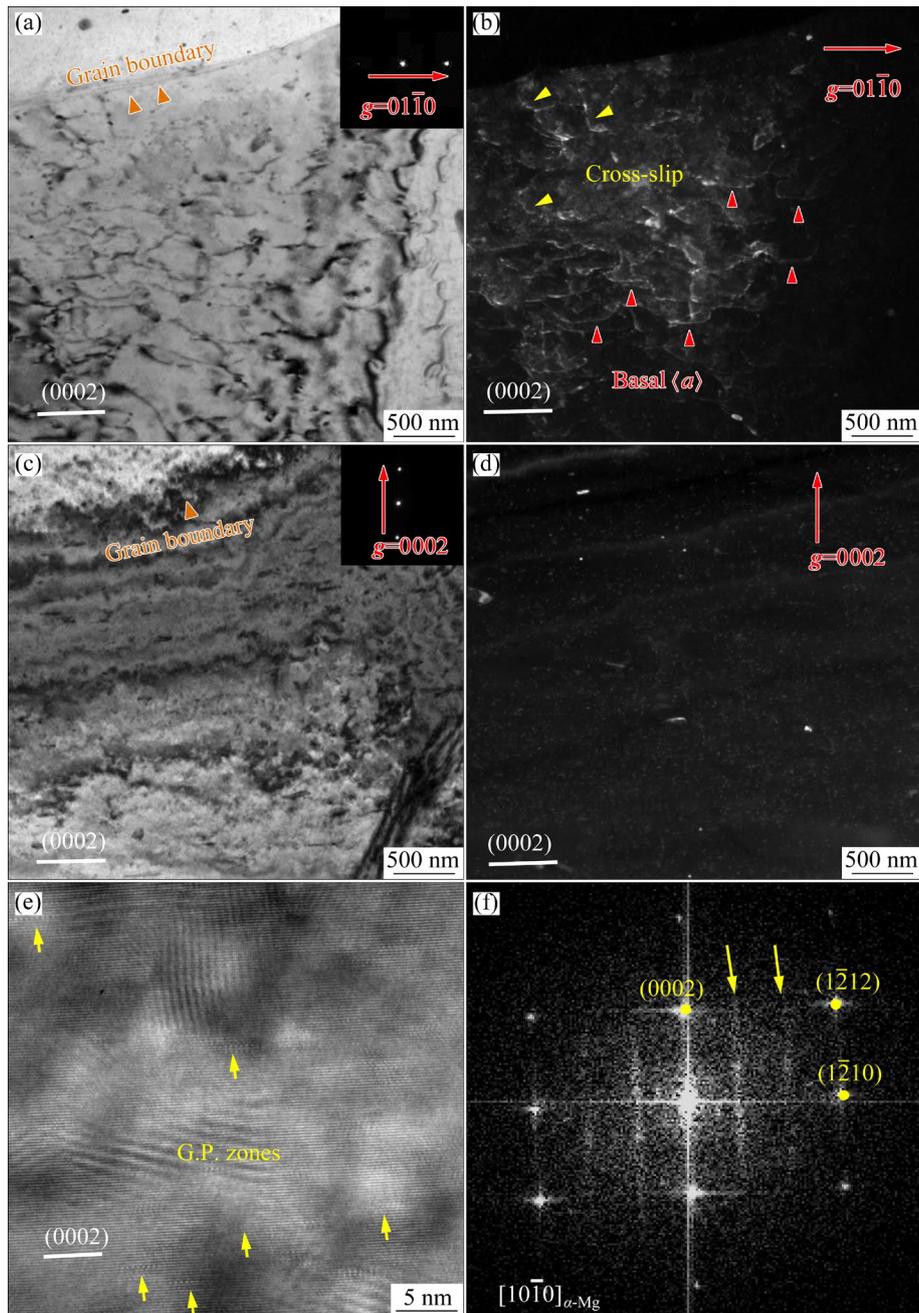


Fig. 9 Bright (a, c) and dark (b, d) field TEM images of T6-200°C/1h sample crept at 70 MPa to 50 h with incident beam direction $B=[\bar{2}110]$ and diffraction vectors $g=[01\bar{1}0]$ (a, b) and $g=[0002]$ (c, d); HRTEM image (e) and FFT pattern (f) of G.P. zones after creeping

samples differ. The power-law breakdown caused by the cross-slip of screw dislocations from the basal to prismatic plane represents the steady-state creep mechanism in the T6-200°C/1h samples, whereas in the T6-275°C/8h sample, the creep deformation is dominated by both dislocation climbing and cross-slip motion. The presence of G.P. zones and Al_2Ca phases act as effective barriers, hindering the movement of non-basal dislocations.

By comparing the degree of cross-slip observed

in Figs. 9(a) and 10(a), it can be concluded that the G.P. zones are more effective on inhibiting dislocation glide and suppressing dislocation climbing. This is also consistent with the observation of ZHANG et al [42] that the ordered G.P. zones can serve as an effective barrier for dislocations to climb and glide during creep deformation. This can be attributed to the higher density of G.P. zones, as shown in Fig. 11 (the number densities of the G.P. zones and the Al_2Ca phases before creep are

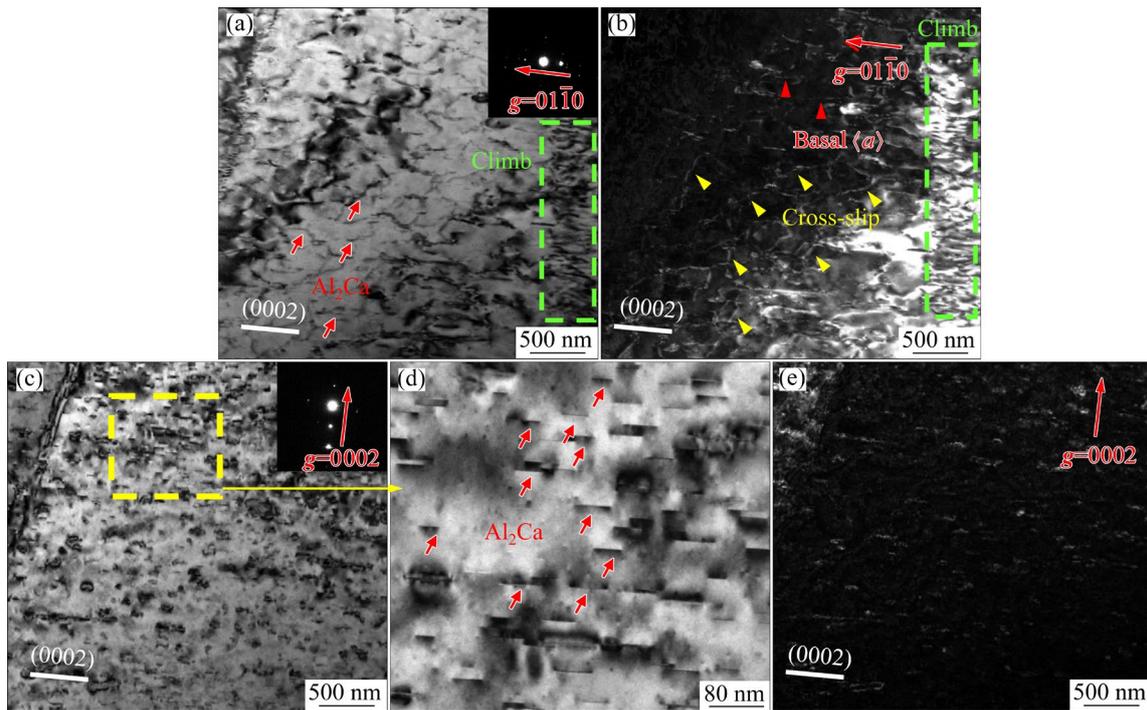


Fig. 10 Bright (a, c, d) and dark (b, e) field TEM images of T6-275°C/8h sample crept at 70 MPa to 50 h with incident beam direction $B=[\bar{2}110]$ and diffraction vector $g=[01\bar{1}0]$ (a, b) and $g=[0002]$ (c, d, e)

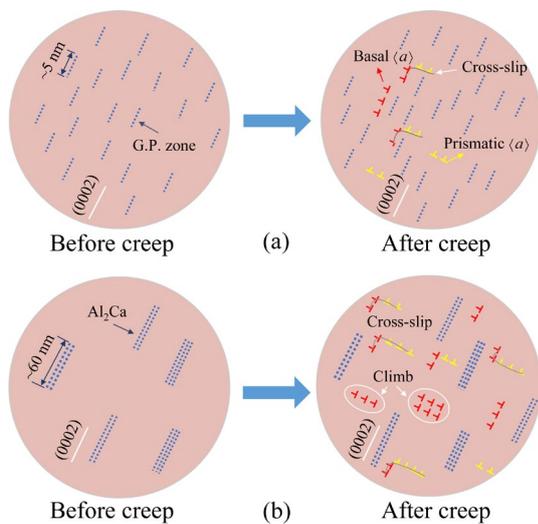


Fig. 11 Schematic diagram of creep mechanism in different samples: (a) T6-200°C/1h; (b) T6-275°C/8h

measured as $(1.4 \pm 0.3) \times 10^{23}$ and $(1.2 \pm 0.6) \times 10^{21} \text{ m}^{-3}$, respectively. After creep, the number densities are $(2.3 \pm 0.2) \times 10^{23}$ and $(8.1 \pm 0.4) \times 10^{21} \text{ m}^{-3}$ for the G.P. zones and the Al_2Ca phases, respectively). Furthermore, compared with Al_2Ca phases, the G.P. zones maintain a stable monolayer morphology. When precipitates are finer and more uniformly distributed, the average distance of the dislocation glide is reduced, effectively inhibiting dislocation motion and enhancing the creep resistance.

Consequently, it is plausible that the creep properties of alloys can be controlled through adjusting the density of high-density G.P. zones. However, obvious dislocations are observed near the Al_2Ca phase (Fig. 10). The precipitation of larger Al_2Ca phases may induce local structural heterogeneity and introduce stress gradients within the crystals. These gradients can lead to local stress concentrations, particularly at the interfaces between the precipitate and the matrix, serving as a significant driving force for dislocation climbing. Areas with high levels of stress in the local stress field may induce dislocation climbing, allowing the crystal to adapt better to the stress distribution [43]. Conversely, G.P. zones have smaller sizes and a more uniform distribution, which reduces the likelihood of localized stress concentrations and thus hinders the initiation of the dislocation climbing. This phenomenon simultaneously delays the occurrence of creep fractures. However, the interaction between the dislocations and G.P. zone requires further investigation.

5 Conclusions

(1) In addition to Al–Mn phases, the G.P. zones are the dominant precipitates in the T6-200°C/1h

sample, whereas the Al₂Ca phases are the main precipitates in the T6-275°C/8h sample.

(2) The T6-200°C/1h samples exhibit steady-state creep rates ranging from $3.08 \times 10^{-9} \text{ s}^{-1}$ to $1.67 \times 10^{-7} \text{ s}^{-1}$ in tensile stress range of 50–80 MPa at 175 °C, nearly an order of magnitude lower than the steady-state creep rates of the T6-275°C/8h sample.

(3) The stress exponents for the T6-200°C/1h sample and T6-275°C/8h sample are ~5.5 and ~8.2, respectively. The unusually high stress exponent observed in the T6-200°C/1h sample is associated with the power-law breakdown creep mechanism.

(4) The power-law breakdown creep of the T6-200°C/1h sample is primarily caused by cross-slip of screw dislocations from the basal to prismatic plane, whereas the T6-275°C/8h sample exhibits a collective mechanism involving both dislocation cross-slip and dislocation climb. Compared to the Al₂Ca phases, the dense G.P. zones can effectively inhibit dislocation climb and dislocation glide during creep deformation. Therefore, it is plausible that the creep properties of alloys can be controlled through adjusting the density of high-density G.P. zones.

CRedit authorship contribution statement

Ming-yu LI: Methodology, Validation, Data curation, Writing – Original draft; **Zhi-ping GUAN:** Methodology, Writing – Review & editing; **Jia-wang SONG:** Investigation, Validation, Supervision; **Hong-jie JIA:** Methodology, Investigation; **Pin-kui MA:** Data curation, Methodology; **Gang WANG:** Data collection, Supervision; **Wei YAN:** Experimental data supplement, Investigation; **Ming-hui WANG:** Validation, Supervision; **Zhi-gang LI:** Investigation, Writing – Review & editing.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability statement

The raw/processed data required to reproduce these findings cannot be shared at this time as the data also form part of an ongoing study.

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不同人工时效处理对挤压低含量 Mg–Al–Ca–Mn 合金 拉伸蠕变行为的影响

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摘要: 探讨了人工时效(T6)对挤压 Mg–1.22Al–0.31Ca–0.44Mn(质量分数, %)合金在 175 °C、50–80 MPa 拉应力作用下的蠕变性能的影响。在 200 °C 时效 1 h (T6-200°C/1h) 的样品中, 主要析出 Guinier-Preston (G.P.) 区, 而在 275 °C 时效 8 h (T6-275°C/8h) 的样品中, 主要析出相为 Al₂Ca。T6-200°C/1h 样品表现出优异的抗蠕变性能, 其稳态蠕变速率比 T6-275°C/8h 样品的低一个数量级。T6-200°C/1h 样品中观察到的异常高应力指数(~8.2)与幂律失效机制相关。经透射电子显微镜(TEM)分析发现, T6-200°C/1h 样品的蠕变机制由基面和柱面位错之间的交滑移主导, 而 T6-275°C/8h 样品表现出位错交滑移和攀移的混合机制。与 Al₂Ca 相相比, 密集的 G.P. 区在蠕变过程中能有效阻碍位错的攀移和滑移, T6-200°C/8h 样品表现出更优异的抗蠕变性能。

关键词: Mg–Al–Ca–Mn 合金; 拉伸蠕变; 人工时效; 交滑移

(Edited by Bing YANG)