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# Creep mechanism of Mg-Nd-Zn-Zr alloy at ambient temperature

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Abstract: Magnesium alloy's creep stress exponent and creep mechanism at the room temperature were analyzed by TEM. Relationship among strain, stress and creep time was studied. The creep rate of some mechanisms were calculated. The results show that the dislocation mechanism is possible. The deformation mechanism is dislocation slipping on basal plane, and twinning improves creep deformation.

Key words: magnesium alloy; room-temperature creep; twinning; slipping

#### **1** Introduction

Magnesium alloy is the lightest structural metallic materials of industry application. There are many merits of magnesium alloy for engineering structures, such as low-density, high specific strength and rigidity, dimensional stability, electromagnetic shielding and workability[1]. Magnesium alloys have been widely used in the fields of automotive industry, electric communication industry, aerospace industry, military and so on[2–5]. However, magnesium alloys creep at room temperature although they are loaded below the yield strength, and significant creep strain accumulates. This phenomenon has been reported over the past 50 years, but the study on its regularity and the mechanism is few[6–7].

Ambient temperature creep is often not considered as a viable failure mechanism, as creep strains at low temperatures are small, and failure due to low-temperature creep is unlikely (but for problem of dimensional stability, such creep effects can be of engineering importance)[8]. When the creep deformation occurs in a bolt and nut, it causes stress relaxation and brings very dangerous state[9]. In this paper, the creep behavior of Mg-Nd- Zn-Zr (ZM6) at room temperature was studied.

#### 2 Experimental

The material used in the study is Mg-Nd- Zn-Zr system alloy (ZM6). The chemical compositions are listed in Table 1.

Tensile tests and creep tests were performed at room temperature. A instrumentation system was built for accurately measuring creep deformation, of which the resolution can reach 0.1 µm. Tensile properties was tested on the CSS-44100 Electronic Universal Testing Machine. Deformed and undeformed structures were examined by transmission electron microscopy (TEM). TEM observation was conducted to establish the deformation mechanisms. Thin foils for TEM observation were prepared by twin jet electro polishing in a solution of 25% HNO3 and 75% methanol cooled down to  $-20^{\circ}$ C, and then low energy beam ion thinning was carried out. Once sample preparation was completed, the samples were observed by TEM using a Philips CM200.

#### **3** Results and discussion

When the stress is less than yield stress, material can also creep, and the deformation increases. Creep rate and stress exponent are two important parameters to

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**Table 1** Nominal composition of prepared alloys(mass fraction,%)

Nd	Zn	Zr	Mg
2.75	0.65	0.51	Bal.

characterize the creep deformation. The minimum creep rate of pure magnesium is about  $10^{-8}$ /s with yield stress(YS) 70%–90%, and the creep rate of AZ31 is slower because of its strengthening mechanism. Generally the creep rate of magnesium alloys is faster than  $10^{-8}$ /s at elevated temperature.

The tensile strength of ZM6 is about 250 MPa, and the yield stress is about 138 MPa. The tests were performed with constant load and yield stress of 20%, 40%, 60%, 80%, holding for a period of more than 200 h at room temperature.

Fig.1 shows creep curves for four tests conducted at an absolute temperature(*T*) of 308 K under stresses( $\sigma$ ) of 28, 55, 87 and 110 MPa, respectively. These curves show that the minimum creep stain-rate became faster with the augmentation of load, and the rates are from 10<sup>-10</sup>/s to 10<sup>-9</sup>/s. The relationship between  $\dot{\varepsilon}_{min}$  versus stress  $\sigma$  for the as-cast specimens is shown in Fig.2. Apparently, the creep exponent *n* is about 3.

The creep mechanism is different with the diversity of temperature and load. The different stress exponents are related to the corresponding creep mechanisms. The exponent *n* is 3-8 in the dislocation creep[10–12]. It is possible to use some constitutive equations to describe various creep mechanisms. It provides a helpful guide forecasting the dominant deformation mechanism at a given set of deformation conditions, i.e., temperature, stress and grain size. The constitutive equations are generally expressed as follows[13].

Nabarro-Herring creep:

$$\dot{\varepsilon} = \frac{14\Omega\omega}{kT} \frac{1}{d^2} D_1 \tag{1}$$



**Fig.1** Relationship between creep strain and time for Mg-Nd-Zn-Zr alloy at various stress and *T*=308 K



Fig.2 Relationship between minimum creep rate and stress for stress exponent

Coble creep:  $\dot{\varepsilon} = \frac{14\pi\Omega\sigma}{kT} \frac{\delta}{d^3} D_{\rm gb}$ (2)

Grain-boundary sliding (Lattice diffusion controlled):

$$\dot{\varepsilon} = 8 \times 10^6 D_1 \frac{\mu b}{kT} (\frac{b}{d})^2 (\frac{\sigma}{\mu})^2 \tag{3}$$

Grain-boundary sliding (Grain-boundary diffusion controlled):

$$\dot{\varepsilon} = 2 \times 10^5 D_{\rm gb} \, \frac{\mu b}{kT} (\frac{b}{d})^3 (\frac{\sigma}{\mu})^2 \tag{4}$$

Dislocation creep:

$$\dot{\varepsilon} = 3.83 \times 10^5 D_1 \frac{\mu b}{kT} (\frac{\sigma}{\mu})^n \tag{5}$$

where  $k=1.38 \times 10^{-23}$  J/K, is a constant; the magnitude of the Burgers vector  $b=3.21 \times 10^{-10}$  m; the shear modulus  $\mu=15.1$  GPa; the grain size  $d=70 \ \mu$ m;  $\delta=2 \ b$ ; the cubic content  $\Omega=2.3 \times 10^{-2} \text{ nm}^3$ .

The diffusion coefficient D is an important parameter constitutive equations. From Table 2 (T=308 K), it can be calculated:

$$D_{\rm gb} = 1.9 \times 10^{-18} \,\mathrm{m^2/s}$$
  
 $D_{\rm l} = 1.24 \times 10^{-27} \,\mathrm{m^2/s}$ 

From Eqs.(1)–(5), minimum creep rate can be calculated as follows.

Nabarro-Herring creep:

$$\dot{\varepsilon} = 1.9 \times 10^{-18} / s$$

Coble creep:

$$\dot{\varepsilon} = 8.46 \times 10^{-14} / s$$

Grain-boundary sliding:

$$\dot{\varepsilon} = 1 \times 10^{-14} / \text{s}$$
 (Lattice diffusion)  
 $\dot{\varepsilon} = 1.8 \times 10^{-12} / \text{s}$  (Grain-boundary diffusion)

Burgers vector/m	Pipe diffusion coefficient, $D_p/(m^2 \cdot s^{-1})$	Pre-exponential factor, ap $D_{0p}/(\text{m}^2 \cdot \text{s}^{-1})$	Grain boundary diffusion coefficient, $D_{gb}/(m^2 \cdot s^{-2})$	Pre-exponential factor, $\delta D_{0gb}/(m^3 \cdot s^{-1})$	Lattice diffusion coefficient, $D_{\rm L}/({\rm m}^2 \cdot {\rm s}^{-2})$	Pre-exponential factor, $D_{0L}/(\text{m}^2 \cdot \text{s}^{-2})$
$3.21 \times 10^{-10}$	$D_{0p} \exp(192\ 000/(RT))$	$3.0 \times 10^{-23}$	D <sub>0gb</sub> exp (-92 000/( <i>RT</i> ))	5.0×10 <sup>-12</sup>	$D_{0L} \exp(-135\ 000/(RT))$	$1.0 \times 10^{-4}$

Table 2 Material factors on magnesium[14]

Dislocation creep:

$$\dot{\varepsilon} = 1.573 \times 10^{-7} / \text{s} \quad (n=3)$$
  
 $\dot{\varepsilon} = 2.4 \times 10^{-5} / \text{s} \quad (n=2)$   
 $\dot{\varepsilon} = 1 \times 10^{-9} / \text{s} \quad (n=4)$ 

From all the above, it is revealed that  $\dot{\varepsilon}$  in the Nabarro-Herring creep, Coble creep and GBS creep is far less than the test result, so it can't be the main creep mechanism of magnesium alloys. After breeding out several mechanisms above, the main creep mechanism of ZM6 at room temperature can be determined as dislocation creep.

Fig.3 shows a typical bright field (BF) image of the microstructure in magnesium sample. In this orientation,  $B = [01\overline{1}1]$ , the projected image of such precipitated



**Fig.3** TEM micrographs of ZM6: (a) Bright field imge; (b) Selected area diffraction pattern

phase has a form of parallel epipeds, which indicates that it precipitates along  $<11\overline{2}0>$  orientation.

Fig.4 shows the microstructure of the sample that has been loaded at 308 K under 28 MPa for more than 200 h. The crystal grain contains large twin, which indicates that twinning is one of the creep mechanism in magnesium alloys at room temperature. Twinning is found in Fig.4(a).

But the number of twin crystal is few, which is only found in large grains, so twining can not contribute to the creep. The formation of Twin is related to the symmetry of the crystal. The critical shear stress of basal slipping in magnesium that belongs to hexagonal structure are lower than that of twinning [15]. But considering the symmetry is low and the primary slip system at room temperature is only two, when the grain's orientation goes against slipping, twinning is another important plastic yield. The HCP structure of simple metal and alloy often form twinning in the process of deformation.

In a large range of temperature, twinning and slipping are two concurrent deformations. Twinning often generate on  $\{10\overline{1}2\}$  crystal planes in HCP structure maganism. The deformation that caused by twinning is too small, compared with dislocation slipping, so it is only the secondary mechanism to the creep at ambient temperature, and causes less than 10% of total deformation.

In this orientation,  $B = [5\overline{1}\overline{4}3]$ , the projected image of basal slipping is line. The paralleled dislocation line in Fig.4(b) shows typical feature of basal slipping. Fig.4(c) shows that some dislocation lines have slipped to the grain boundary, which expresses the deformation mechanism, i.e. along with the process of time, and some dislocation lines arrive at the grain boundary and accumulate in the course of the following deformation. Consequently, the creep mechanism of ZM6 is dislocation slipping also with some twinning phenomenon.

## **4** Conclusions

1) The creep rate of ZM6 at room temperature is  $10^{-10}-10^{-9}$  s<sup>-1</sup> with the stress from 28 MPa to 110 MPa for Mg-Nd-Zn-Zr alloy. A log-log plot of  $\dot{\varepsilon}_{min}$  versus stress  $\sigma$  reveals the creep stress exponent is 3.

2) The creep dominant mechanism of Mg-Nd-Zn-Zr alloy at ambient temperature is dislocation slipping.



**Fig.4** TEM micrographs of ZM6 after creep at 308 K under 28 MPa for more than 200 h: (a) Deformation twins; (b) Parallel dislocation lines; (c) Dislocation lines and grain boundary; (d) Selected area diffraction pattern

### References

- MORDIKE B L, EBERT T. Magnesium-properties-applicationspotential[J]. Mater Sci Eng A, 2001, 302: 37–45.
- [2] LUO A LAN A. Recent magnesium alloy development for automotive power train application[J]. Material Science Forum, 2003, 419/422: 57–56.
- [3] TAN J C, TAN M J. Superplastic magnesium alloy for sporting and leisure equipments[C]//Proceedings of Symposium on Materials and Science in Sports. FROES F H, HAAKE S J, eds. Coronado, California TMS, 2001, 22/25: 95–104.
- [4] SHIGERU I, Y UUJI N, SHIGEHARU K, et al. Age hardening characteristics and high temperature tensile properties of Mg-Gd and Mg<sub>2</sub>Dy alloys[J]. Journal of Japan Institute of Light Metals, 1994, 44: 38.
- [5] ROKHLIN L L, NIKITINA N I. Recovery after ageing of Mg-Y and Mg-Gd alloys[J]. Journal of Alloys and Compounds, 1998, 279: 166–170.
- [6] NEERAJ T, HOU D H, DAEHN G S, MILLS M J. Phenomenological and microstructural analysis of room temperature creep in titanium alloys[J]. Acta Mater, 2000, 48: 1225–1238.
- [7] DAEHN G S. Primary creep transients due to non-uniform obstacle sizes[J]. Mater Sci Eng, A, 2001, 319/321: 765–769.
- [8] UCHIC M D, CHRZAN D C, NIX W D. Primary creep of Ni<sub>3</sub>(Al, Ta)

in the anomalous flow regime[J]. Intermetallics, 2001, 9: 963-969.

- [9] YAMADA T, KAWABATA K, SATO E, KURIBAYASHI K, JIMBO I. Presences of primary creep in various phase metals and alloys at ambient temperature[J]. Materials Science and Engineering A, 2004, 387/389: 719–722.
- [10] Watanabe H, Mukai T, Kohzu M, Tanabe S, Higashi K. Superplasticity in powder metallurgy aluminum alloys and composites[J]. Acta Mater, 1999, 47: 3753–3758.
- [11] WATANABE H, TSUTSUI H. MUKAI T, KOHZU M, TANABE S. HIGASHI K. Deformation mechanism in a coarse-grained Mg-Al-Zn alloy at elevated temperatures[J]. International Journal of Plasticity, 2001, 17(3): 387–397.
- [12] RUANO O A, WADSWORTH J, SHERBY O D. Microstructural evidence for diffusional creep in copper using atomic force microscopy[J]. Mater Sci, 1985, 20: 3735–3744.
- [13] SOMEKAWA H, HIRAI K, WATANABE H, TAKIGAWA Y, HIGASHI K. Dislocation creep behavior in Mg-Al-Zn alloys[J]. Materials Science and Engineering A, 2005, 407: 53–61.
- [14] FROST H J, ASHBY M F. Deformation mechanism map[M]. New York: Pergamon Press, 1982: 44.
- [15] Ion S E, Huphreys F J, White S H. Dynamic recrystallisation and the development of microstructure during the high temperature deformation of magnesium[J]. Acta Mater, 1982, 30: 1909–1919.

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