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Impression creep properties of a semi-solid processed magnesium—aluminum alloy containing calcium and rare earth elements

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The creep properties of a thixoformed magnesium–aluminum alloy containing calcium and rare earth elements were studied under shear modulus-normalized stresses ranging from 0.0225 to 0.035 at temperatures of 150–212 °C using the impression creep technique. Analysis of the creep mechanism based on a power-law equation indicated that pipe diffusion-controlled dislocation climb is the dominant mechanism during creep. The alloy has a better creep resistance than high-pressure die-cast magnesium–aluminum alloy.

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The good casting characteristics and room-temperature mechanical properties of AZ91 magnesium alloy, combined with other advantages of magnesium alloys, have led to the increasing application of this material in the automotive industry [1]. On the other hand, the relatively poor high-temperature creep properties of AZ91 have restricted its application to parts that operate at temperatures of <120 °C [2,3]. In a number of studies it has been claimed that the degradation of AZ91's high-temperature mechanical properties is due to the presence of massive $\beta(Mg_{17}Al_{12})$ eutectic phase in addition to the discontinuous grain boundary precipitation of the incoherent $\beta(Mg_{17}Al_{12})$ phase from the supersaturated $\alpha(Mg)$ solid solution during creep testing [4–6]. Although some studies have shown that the creep properties of high-pressure die-cast (HPDC) AZ91 alloy can be improved by adding certain alloying elements, such as Ca, Sr and La [4–7], little is known about the effects of these elements on the creep properties of thixoformed AZ91 alloy. Therefore, in the present study the effect of Ca and rare earth (RE) elements on the creep properties of thixoformed AZ91 alloy has been studied.

An ingot of AZ91 + 1RE + 1.2Ca (AZRC91) alloy with the chemical composition of 9.2 Al, 0.91 Zn, 0.21 Mn, 1.18 Ca and 0.95 La (wt.%) based misch metal

was prepared. Cylindrical samples 38 mm in diameter and 100 mm high were machined from the ingot and melted in steel crucibles at 630 °C for 30 min. The crucibles containing the liquid alloy were quenched into water at room temperature. The quenched specimens were compressed about 10% at 250 °C and partially remelted at 580 °C for 50 min. Finally, they were cast in the form of cylindrical specimens 2.2 mm in diameter and 100 mm high using a 150 ton cold chamber die-casting machine. Slices 6 mm thick were prepared from the thixoformed specimens. Creep testing was performed on the specimens using the impression creep technique at temperatures ranging from 150 to 212 °C under shear modulus-normalized stresses (σ/G) of between 0.0225 and 0.035 for 4500 s.

The shear modulus of the alloy was obtained from G (MPa) = 18,460 - 8.2T (K) [8]. Details of the impression creep testing have been reported elsewhere [4].

Metallographic investigations were carried out on the surface beneath and perpendicular to the indenter. The samples were prepared by standard grinding and polishing methods and etched with a 1% nital solution (1 ml HNO₃ and 99 ml ethylic alcohol). Microstructural characterizations were performed by scanning electron microscopy (SEM) and energy-dispersive X-ray spectrometry (EDX) using a Zeiss EVO40 microscope equipped with a Rontec spectrometer. Microhardness testing was conducted on the specimens using a 50 g load.

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Figure 1a and b show typical creep curves and creep rates of specimens crept at 212 °C under different punching stresses. According to these figures, creep curves clearly show a short primary creep followed by a secondary one. During the primary creep, the creep rate decreases with the time, whereas in the secondary creep it remains constant over the test time. According to Figure 1b, although the curves have different lengths, steadystate creep has been achieved in all of the specimens. Contrary to conventional creep test curves, tertiary creep is not seen in Figure 1a and b. Previous investigations [4] showed that despite the compressive nature of the impression creep test, creep curves of a HPDC AZ91 alloy at 212 °C under shear modulus-normalized stress values of >0.025 exhibit tertiary creep. The presence of tertiary creep was ascribed to the discontinuous precipitation of $\beta(Mg_{17}Al_{12})$ phases during the creep test, which resulted in the progressive degradation of the alloy's creep properties. Therefore, the absence of tertiary creep during the creep of the thixoformed AZRC91 alloy even under higher stress values reveals that the microstructure stability of this alloy is higher than that of HPDC AZ91 alloy.

The following power-law equation was used to identify the dominant creep mechanism [9]:

$$\stackrel{\circ}{\varepsilon} = A \left(\frac{b}{d}\right)^p \left(\frac{GbD_0}{kT}\right) \left(\frac{\sigma}{G}\right)^n \exp\left(-\frac{Q_c}{RT}\right) \tag{1}$$

where A is a constant, b is the Burgers vector length (which for Mg is equal to 3.21×10^{-10} m [10]), d is the grain size, p is the grain size exponent, n is the stress exponent, k is Boltzmann's constant, D_0 is an appropriate diffusion coefficient constant, Q_c is the creep activation energy, T is the test temperature and R is the universal gas constant.

The equivalent uniaxial creep strain rate $(\mathring{\varepsilon})$ and stress (σ) can be evaluated from the impression velocity (V_{imp}) and impression stress (σ_{imp}) using the following two semi-empirical relations [4]:

$$\sigma = \frac{4F}{c_1 \cdot \pi \varphi^2} = \frac{\sigma_{imp}}{c_1} \tag{2}$$

$$\stackrel{\circ}{\varepsilon} = \frac{dh/dt}{c_2 \varphi} = \frac{V_{imp}}{c_2 \varphi} \tag{3}$$

where c_1 and c_2 are constants, φ is the punch diameter, h is the penetration depth and F is the load applied to the punch. The values of c_1 and c_2 for both AZ91 and AZRC91 alloys are 3 and 0.5, respectively [4].

As shown in Figure 1c and d, the stress exponent, n, and creep activation energy, Q, can be obtained from the plot of $\ln(V_{imp}T/G)$ vs. $\ln(\sigma_{imp}/G)$ at a constant T, and the plot of $\ln(V_{imp}T/G)$ vs. 1/T at a constant σ_{imp}/G , respectively. Only results of tests in which steady-state creep was achieved were used for analysis.

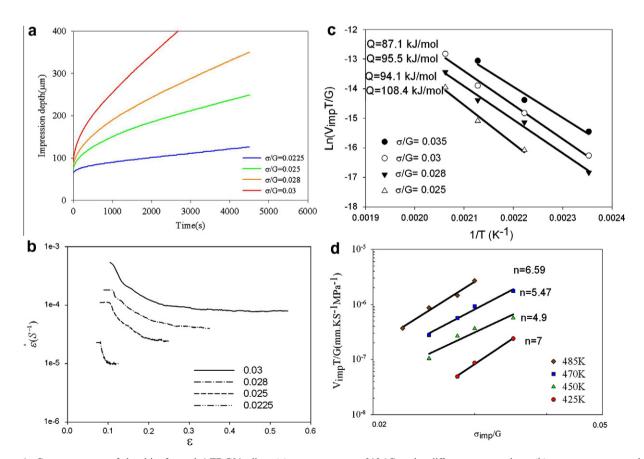


Figure 1. Creep response of the thixoformed AZRC91 alloy: (a) creep curves at 212 °C under different stress values; (b) creep rates vs. strain at 212 °C under different stress values; (c) variation of $\ln(V_{im}T/G)$ vs. 1/T for calculating the creep activation energy; (d) variation of $V_{im}T/G$ vs. σ/G for calculating the stress exponent.

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