Effect of Lamellar Spacing on Creep Strength of α -Mg/C14–Mg₂Ca Eutectic Alloy*¹

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In tensile tests, α -Mg/C14–Mg₂Ca eutectic alloy with a lamellar structure is plastically deformed above 473 K but ruptures before yielding at temperatures below 423 K. This study investigates the effect of the α /C14 interface on the creep strength of α -Mg/C14–Mg₂Ca eutectic alloy at 473 K under 40 MPa stress. The creep curves of the alloy exhibited three stages: a normal transient creep stage, minimum creep-rate stage, and accelerating stage. The minimum creep rate was proportional to the lamellar spacing, indicating that the α /C14 lamellar interface plays a creep-strengthening role. In high-resolution transmission electron microscope observations of the specimens after the creep test, α -dislocations appeared within the α -Mg lamellae and were randomly distributed on the α /C14 interface. It was deduced that the α /C14 interface presents a barrier to dislocation glide and does not annihilate and/or rearrange the dislocations caused by the creep test.

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1. Introduction

Magnesium alloys are the lightest of structural metallic materials. These alloys are increasingly used in automotive and aerospace industries, where they are instrumental in increasing fuel efficiency and thus minimizing carbon dioxide emissions.¹⁾ The widespread application of the magnesium alloys to high-temperature components, not only to room-temperature components, is essential for designing transport equipment with low weight and high fuel efficiency.^{2,3)} Pure Mg has low strength at high temperatures,^{4,5)} and the solubility of alloying elements in Mg is quite limited.⁶⁾ The thermally stable intermetallic phases, which are utilized as the precipitation–dispersion phase^{7,8)} or covering phase,^{9,10)} are indispensable to improve the high-temperature strength of heat-resistant Mg alloys.^{11,12)}

Another method for improving the high-temperature strength by using intermetallic phases is to control the microstructure so that the matrix and intermetallic phases are layered via a eutectic reaction.^{13–16)} The Mg–Al–Ca ternary system without rare-earth elements, which usually exhibits excellent nonflammability, is a promising alloy system for developing highly versatile heat-resistant magnesium alloys with excellent cost performance.^{17–21)} When the Mg–Al–Ca ternary alloys in the Mg-rich composition region are melted and cast, three kinds of eutectic reactions are possible during solidification depending on the [Ca]/[Al] ratio: (i) $L \rightarrow \alpha\text{-Mg} + \text{Al}_{12}\text{-Mg}_{17}\text{Al}_{12}$, (ii) $L \rightarrow \alpha\text{-Mg} + \text{C36-(Mg,Al)}_2\text{Ca}$, and (iii) $L \rightarrow \alpha\text{-Mg} + \text{C14-Mg}_2\text{Ca}$.²²⁾ Only third eutectic reaction occurs in the composition region with [Ca]/[Al] > 1.5, and the resultant α /C14 eutectic structure is extremely fine compared to the α /Al₁₂ and α /C36 eutectic structures.^{23,24)}

When a binary Mg–Ca hypoeutectic alloy with a composition close to the eutectic composition (Mg–

16.2 mass% Ca) is melted and then cast with a mild steel mold, the resultant α /C14 eutectic structure has a lamellar structure with a curved α /C14 interface. The lamellar spacing is a submicron size less than 1 μm .²⁵⁾ In a previous work, the crystal orientation relationship between α -Mg lamellae and the α /C14 interface in the α /C14 lamellar structure was examined, and the temperature region in which the α /C14 lamellar structure is stable in morphology was determined.²⁵⁾ The results showed that (1) the primary slip plane (0001) $_{\alpha}$ of the α -Mg lamellae was oriented toward the α /C14 interface, and (2) the α /C14 lamellar structure became increasingly coarse at temperatures above 573 K. The quantitative relationship between lamellar spacing (λ) and aging time (t) was obtained: $\lambda^2 - \lambda_0^2 = k_T t$, where λ_0 is the α /C14 lamellar spacing for the as-cast specimen, and k_T is a constant depending on aging temperature.

The strength of metallic materials with lamellar microstructures is known to increase with decreasing lamellar spacing at room temperature.^{25–29)} In contrast, the effect of λ on high-temperature creep strength has not been elucidated for Mg alloys. The objectives of this study were to clarify the following three points with regard to the binary Mg–Ca eutectic alloy with the α /C14 lamellar microstructure: (1) To clarify the temperature range in which the plastic deformation occurs and decide the temperature for the creep test; (2) to clarify quantitatively the correlation between creep strength and λ ; (3) to clarify the role of the α /C14 lamellar interface on dislocation glide during creep. To clarify the first point, tensile tests were performed for the as-cast specimen under a wide range of temperature and strain rate conditions. To clarify the second point, creep tests were performed for the alloys whose λ was controlled by the aging treatment. To clarify the third point, the dislocation substructure of the creep specimens was examined.

2. Experimental

A binary Mg–13.8 mass% Ca hypoeutectic alloy was used in this study. The Ca content of the alloy was reduced by 2.4 mass% from the eutectic composition (Mg–16.2 mass% Ca) to avoid precipitation of the brittle primary C14–Mg₂Ca

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phase. A block of the alloy of dimensions $100 \times 160 \times 20$ mm was gravity-cast at a casting temperature of 1053 K by the permanent mold-casting method with a mild steel mold in argon atmosphere using easily available starting materials of the highest purity. The as-cast specimen showed a mixed microstructure of primary α -Mg phase and α -Mg/C14-Mg₂Ca eutectic lamellae.²⁵⁾ According to the Mg-Ca binary phase diagram,⁶⁾ the weight ratios of the primary α -Mg phase and α -Mg/C14-Mg₂Ca lamellar were estimated as 16% and 84%, respectively, based on the lever rule. The α /C14 lamellar spacing, λ , in the α /C14 lamellar region for the as-cast specimen was $0.9 \mu\text{m}$.²⁵⁾ The microstructure of the alloy was almost occupied by the α /C14 lamellar structure. Therefore, this alloy is termed the α -Mg/C14-Mg₂Ca eutectic alloy hereafter in this study, and the microstructure observation was performed in the α /C14 lamellar region.

Tensile-test specimens with a gage length of 13.2 mm and a cross-sectional area of 3×1 mm were prepared by electric-discharge machining and mechanical polishing from the rectangular ingot manufactured by permanent mold-casting as mentioned earlier. Tensile tests were performed at four temperatures between 298 and 473 K at strain rates between 1.2×10^{-5} and $1.2 \times 10^{-4} \text{ s}^{-1}$. Creep test specimens with a gage length of 28 mm and a cross-sectional area of 6×3 mm were prepared from 3-mm-thick plates cut out from the rectangular ingot.^{30,31)} The creep specimens were subjected to aging treatment at 673 K for 3.6×10^4 s (10 h) and 3.6×10^5 s (100 h) to achieve $\lambda = 1.9 \mu\text{m}$ and $\lambda = 5.3 \mu\text{m}$.²⁵⁾ A creep test specimen that had undergone the aging treatment at 723 K for 3.6×10^3 s (1 h) to achieve $\lambda = 1.3 \mu\text{m}$ was also prepared. Creep tests under tension were performed in air at 473 K under a stress of 40 MPa. The current of the creep furnace was designed to stop at creep rupture to minimize the exposure time at high temperatures for the creep-ruptured specimen. During creep interruption, the sample was rapidly cooled under a load by using compressed air followed by water quenching, to preserve the dislocation substructure.

The dislocation substructure of the creep specimens was observed by high-resolution transmission electron microscopy (HRTEM). Thin foils were cut from the creep gage portion of the sample and machined to disks with a diameter of 3 mm. In the case of the creep-ruptured specimen, the foils were cut out from a distance of 5 mm from the ruptured portion. The disks were further thinned down to $90 \mu\text{m}$ by mechanical polishing, followed by dimple grinding and ion-milling, to perforate the center portion of the disks. The perforated disks were examined using a Cs-corrected scanning transmission electron microscope FEI Titan³ G2 60-300 operated at 300 kV.

3. Results and Discussion

3.1 Stress-strain curve

Figure 1 shows the stress-strain curves of the α -Mg/C14-Mg₂Ca eutectic alloy at temperatures between 373 and 473 K. For a strain rate of $1.2 \times 10^{-5} \text{ s}^{-1}$, the specimen ruptured before yielding, and plastic deformation did not occur at 373 and 423 K. In contrast, the stress increased continuously with increasing strain after yielding at 473 K, and a plastic strain exceeding 5% was evident after the maximum stress at

Fig. 1 Stress-strain curves at temperatures between 373 and 473 K for the as-cast α -Mg/C14-Mg₂Ca eutectic alloy.

Table 1 Summary of the tensile property for the as-cast α -Mg/C14-Mg₂Ca eutectic alloy.

Strain rate	298 K	373 K	423 K	473 K
$1.2 \times 10^{-4} \text{ s}^{-1}$	×	×		△
$6.0 \times 10^{-5} \text{ s}^{-1}$				△
$2.4 \times 10^{-5} \text{ s}^{-1}$	×	×		○
$1.2 \times 10^{-5} \text{ s}^{-1}$		×	×	⊙

× : ruptured before yielding.

△ : ruptured after yielding, but before the stress maxima.

○ : ruptured at the stress maxima.

⊙ : ruptured after showing the stress maxima.

132 MPa. When the strain rate was doubled to $2.4 \times 10^{-5} \text{ s}^{-1}$ at 473 K, the stress increased drastically with increasing strain after yielding, and the specimen ruptured at the maximum stress of 143 MPa.

Table 1 summarizes the tensile properties of the α -Mg/C14-Mg₂Ca eutectic alloy against temperature and strain rate. In Table 1, the tensile-test conditions under which rupture occurred before yielding are denoted as ×; the conditions under which rupture occurred before the maximum stress after yielding are marked by △; the conditions under which rupture occurred at the maximum stress and after the maximum stress are denoted by ○ and ⊙, respectively. Under six types of tensile-test conditions in the temperature range below 423 K, the specimens ruptured before yielding, and there was no plastic deformation. In contrast, plastic deformation was evident at any strain rates examined at 473 K. That is, when the strain rate was as high as $1.2 \times 10^{-4} \text{ s}^{-1}$ and $6.0 \times 10^{-5} \text{ s}^{-1}$, the specimens ruptured after yielding before exhibiting the maximum stress. The rupture strain increased with decreasing strain rate, and the specimen ruptured after the maximum stress at the lowest strain rate of $1.2 \times 10^{-5} \text{ s}^{-1}$. Thus, the above results show that for the α -Mg/C14-Mg₂Ca eutectic alloy, plastic

deformation occurred at temperatures higher than 473 K, and the lower strain rate enhanced plastic deformability.

3.2 Creep strength and lamellar spacing

From the results of the tensile tests shown in the previous section, the creep tests were performed at 473 K. Plastic deformation of the α -Mg/C14-Mg₂Ca eutectic alloy was evident at this temperature. From the stress-strain curves at a strain rate of $1.2 \times 10^{-5} \text{ s}^{-1}$ (Fig. 1), the 0.2% proof stress for the alloy was evaluated as 84 MPa at 473 K. Therefore, in this study, the applied stress of the creep tests was adopted as 40 MPa, which is less than a half of the 0.2% proof stress for the alloy at 473 K. This value was adopted to ensure excellent plastic deformability in the evaluation of the long-term creep characteristics.

To clarify the effect of the α /C14 interface on the creep strength, creep tests were performed at 473 K under a stress of 40 MPa for the α -Mg/C14-Mg₂Ca eutectic alloy aged at 673 K and 723 K to increase λ . The α /C14 lamellar structure of the alloy had stable morphology at 473 K.²⁵⁾ Figure 2 shows the creep rate-time curves for the alloy aged at 673 K for 10 and 100 h, together with the data for the as-cast specimen. The overall creep rate-time curve for every specimen shows a downward curvature from stress application until creep rupture. That is, after stress was applied, a normal transient creep was detected, and the creep rate decreased continuously with creep time. Subsequently, there was a gradual increase in the creep rate in the accelerating region that led to the creep rupture of the specimen. A well-defined steady state was barely evident. For the as-cast specimen, the creep rate decreased by more than two orders of magnitude in the transient region, and the minimum creep rate ($6.6 \times 10^{-9} \text{ s}^{-1}$) was observed at a creep time of $8.0 \times 10^5 \text{ s}$ (222 h); subsequently, creep rupture occurred at $3.6 \times 10^6 \text{ s}$ (1006 h). The creep rate-time curves of the specimens aged at 673 K for 10 and 100 h are similar to that for the as-cast specimen; meanwhile, the minimum creep

Table 2 Summary of minimum creep rate ($\dot{\epsilon}_{\min}$), rupture life (t_{rup}), and rupture strain (ϵ_{rup}) for the as-cast and aged α -Mg/C14-Mg₂Ca eutectic alloys, together with the lamellar spacing (λ). The creep tests were carried out at 473 K under a stress of 40 MPa.

Specimen	$\lambda / \mu\text{m}$	$\dot{\epsilon}_{\min} / \text{s}^{-1}$	$t_{\text{rup}} / \text{h}$	$\epsilon_{\text{rup}} (\%)$
As-cast	0.9	6.6×10^{-9}	1006	12.2
723 K/1 h aged	1.3	7.4×10^{-9}	—	—
673 K/10 h aged	1.9	1.1×10^{-8}	947	20.6
673 K/100 h aged	5.3	3.0×10^{-8}	212	8.3

rate increased, and the decrease in creep rate during the transient stage becomes less significant with longer aging time at 673 K.

The minimum creep rate ($\dot{\epsilon}_{\min}$), rupture life (t_{rup}), and rupture strain (ϵ_{rup}), and λ , for the as-cast and aged α -Mg/C14-Mg₂Ca eutectic alloys are summarized in Table 2. These values were obtained from creep tests performed at 473 K under 40 MPa stress. The creep test of the specimen aged at 723 K was interrupted immediately after showing the minimum creep rate; the results of this specimen too are included in the table. The data in Table 2 show that $\dot{\epsilon}_{\min}$ continuously increased and t_{rup} continuously decreased with increased aging time at 673 K, and λ increased from 0.9 to 5.3 μm . In addition, ϵ_{rup} was maximum at 20.6% for the specimen aged at 673 K for 10 h, and it decreased when λ was changed from the value of 1.9 μm .

Morphologically, the interface area included in a unit volume of the two-phase lamellar microstructure decreases to $1/N$, when λ of the alloy increases by a factor of N .³²⁾ Therefore, the quantitative correlation between $\dot{\epsilon}_{\min}$ and λ for the α -Mg/C14-Mg₂Ca eutectic alloy can be evaluated by using a power approximation, not a linear approximation. The $\dot{\epsilon}_{\min}$ of the α -Mg/C14-Mg₂Ca eutectic alloy that underwent creep at 473 K under 40 MPa stress is plotted against λ in double logarithmic coordinates in Fig. 3. In the figure, $\dot{\epsilon}_{\min}$ increases continuously with increasing λ , and all the four data points are on a straight line with a slope of unity. It is evident that the correlation between $\dot{\epsilon}_{\min}$ and λ can be expressed by using a power approximation. Note that the gradient of the $\dot{\epsilon}_{\min}$ - λ curve was evaluated as 0.89 by the method of least squares. Since the $\dot{\epsilon}_{\min}$ decreases continuously with decreasing λ , it is inferred that the α /C14 interface enhances the creep strength, i.e., the α /C14 interface acts as a creep strengthener. Hence, introducing the interface into a microstructure by utilizing the fine lamellar structure can be an effective way for enhancing the high-temperature creep strength of Mg alloys. In addition, a novel high-temperature strengthening mechanism was identified in this study; according to this mechanism, the interface enhances the high-temperature strength, and this phenomenon is termed interface strengthening (IFS).

The phenomenological creep equation for the α -Mg/C14-Mg₂Ca eutectic alloy should include microstructure parameters, such as colony size (d) and λ , in addition to the creep testing temperature (T) and applied stress (σ).^{33,34)} The $\dot{\epsilon}_{\min}$ is expressed as a function of σ , T , d , and λ for the alloy, as shown in eq. (1).

Fig. 2 Creep rate vs. time curves at 473 K under a stress of 40 MPa for the α -Mg/C14-Mg₂Ca eutectic alloys aged at 673 K for 10 and 100 h, together with that for the as-cast specimen.

Fig. 3 Plots of minimum creep rate vs. lamellar spacing for the α -Mg/C14-Mg₂Ca eutectic alloy, where the creep tests were carried out at 473 K under a stress of 40 MPa.

$$\dot{\epsilon}_{min} = A(\sigma/G)^n(b/d)^m(\lambda/b)^p \exp(-Q_c/RT) \quad (1)$$

where A is the material constant; G is the shear modulus; b is the length of the Burgers vector in the α -Mg lamellae; R is the gas constant; Q_c is the activation energy for creep; and n , m , and p are constants. The gradient of the $\dot{\epsilon}_{min}$ - λ curve in Fig. 3 corresponds to p in eq. (1), and the results shown in Fig. 3 suggest $p = 1$ for the α -Mg/C14-Mg₂Ca eutectic alloy.

3.3 Dislocation substructure of creep specimens

The dislocation substructure of the α -Mg/C14-Mg₂Ca eutectic alloy that underwent creep at 473 K under 40 MPa stress was investigated to clarify the role of the α /C14 interface on dislocation glide during creep. The HRTEM image of the as-cast specimen that underwent creep rupture is shown in Fig. 4(a), where the incident beam direction is $\mathbf{B} = [01\bar{1}1]_\alpha$ for the α -Mg lamellae under the multiple-beam diffraction condition. Many dislocations are observed within the α -Mg lamellae. Most dislocations are distributed uniformly inside the α -Mg lamellae, while some dislocations are aligned through the α -Mg lamellae, as indicated by white arrowheads in Fig. 4(a). In contrast, dislocations are scarce within the C14-Mg₂Ca lamellae. From these observation, it is deduced that the α /C14 interface limits the dislocations within the α -Mg lamellae.

The $\mathbf{g} \cdot \mathbf{b}$ values with an incident beam direction $\mathbf{B} = [01\bar{1}1]_\alpha$ were calculated for a reciprocal lattice vector $\mathbf{g} = 0\bar{1}12_\alpha$ (Table 3) to identify whether perfect $\mathbf{a}$, $\mathbf{a} + \mathbf{c}$, and $\mathbf{c}$ -dislocations in the hcp structure with the Burgers vector ($\mathbf{b}$) are visible. Further, $\mathbf{a}$ -dislocations with $\mathbf{b} = 1/3[11\bar{2}0]$ and $\mathbf{b} = 1/3[1\bar{2}10]$, $\mathbf{a} + \mathbf{c}$ dislocations, and $\mathbf{c}$ -dislocations are visible, whereas no $\mathbf{a}$ -dislocations with $\mathbf{b} = 1/3[2\bar{1}10]$ are visible in the case of $\mathbf{g} = 0\bar{1}12_\alpha$. Figure 4(b) shows the HRTEM image of the same field as that shown in Fig. 4(a), taken under the two-beam diffraction condition with $\mathbf{g} = 0\bar{1}12_\alpha$. The visible dislocations within the α -Mg lamellae under the multiple-beam diffraction condition in Fig. 4(a) are scarcely detected under the two-beam diffraction condition,

Fig. 4 HRTEM image, taken with $\mathbf{B} = [01\bar{1}1]_\alpha$, of the as-cast α -Mg/C14-Mg₂Ca eutectic alloy creep-ruptured at 473 K under a stress of 40 MPa under (a) multiple and (b) two-beam diffraction conditions. The dislocation alignment is indicated by white arrowheads in (a).

Table 3 The $\mathbf{g} \cdot \mathbf{b}$ invisibility criterion for perfect dislocations in the hexagonal close-packed crystals close to the $[01\bar{1}1]$ zone axis.

Mode	$\mathbf{b}$	$\mathbf{g} = 0\bar{1}12$
$\mathbf{a}$	$1/3[11\bar{2}0]$	-1
	$1/3[1\bar{2}10]$	1
	$1/3[2\bar{1}10]$	0
$\mathbf{a} + \mathbf{c}$	$1/3[11\bar{2}3]$	1
	$1/3[1\bar{2}13]$	3
	$1/3[2\bar{1}13]$	2
	$1/3[11\bar{2}\bar{3}]$	-3
	$1/3[1\bar{2}1\bar{3}]$	-1
	$1/3[2\bar{1}1\bar{3}]$	-2
$\mathbf{c}$	$[0001]$	2

as shown in Fig. 4(b). The result indicates that most dislocations operating in the α -Mg lamellae during creep for the α -Mg/C14-Mg₂Ca eutectic alloy are $\mathbf{a}$ -dislocations with $\mathbf{b} = 1/3[2\bar{1}10]$.

The HRTEM image of the α /C14 interface for the specimen with $\lambda = 1.3 \mu\text{m}$, obtained by aging at 723 K for 1 h and exhibiting creep at 473 K under 40 MPa stress, is shown in Fig. 5. The creep test was interrupted at a strain of

Fig. 5 HRTEM image, taken with $\mathbf{B} = [01\bar{1}1]_{\alpha}$, of the α /C14 lamellar interface for the 723 K/1 h-aged α -Mg/C14-Mg₂Ca eutectic alloy crept at 473 K under a stress of 40 MPa, where the creep test was interrupted at a strain of 1.3%.

1.3%, which corresponds to the strain immediately after the minimum creep rate was observed. Many dislocations are detected on the α /C14 interface, and they are not arranged in any specific manner but randomly distributed. From the results of the dislocation substructure for the creep specimens, it is inferred that the α /C14 interface presents a barrier to the α -dislocation glide within the α -Mg lamellae and does not annihilate and/or rearrange the dislocations caused by the creep test.

Grain boundaries are regarded to act as a sink of dislocations during creep deformation and diminish the creep strength; i.e., a grain boundary weakens the creep.³³⁾ In contrast, the α /C14 interface is inferred to act as a creep strengthener because it presents a barrier to dislocation glide during creep deformation and does not annihilate and/or rearrange the dislocations caused by the creep test. In future, the IFS may be systematically examined as a high-temperature strengthening mechanism for eutectic alloys based on metals other than Mg.

4. Conclusions

Tensile tests were conducted for a binary Mg–13.8 mass% Ca hypoeutectic alloy, whose microstructure is mostly occupied by the α /C14 lamellar structure. This alloy was termed α -Mg/C14-Mg₂Ca eutectic alloy in this study to clarify the temperature range in which it undergoes plastic deformation. Creep tests were performed for the as-cast and aged specimens to evaluate the effect of the α /C14 interface on creep strength, and the dislocation substructure of the studied specimens was observed by HRTEM. The following results were obtained:

- (1) In the tensile tests of the α -Mg/C14-Mg₂Ca eutectic alloy, the specimen ruptures before yielding, and no plastic deformation occurs at temperatures below 423 K. In contrast, plastic deformation occurs at temperatures above 473 K, and a lower strain rate enhances the plastic deformability.
- (2) The creep tests results of the α -Mg/C14-Mg₂Ca eutectic alloy obtained at 473 K under a stress of

40 MPa showed that the creep curves of the alloy exhibits three stages: a normal transient creep stage, a minimum creep-rate stage, and an accelerating stage that is followed by creep rupture. Since the minimum creep rate decreases continuously with decreasing lamellar spacing, the α /C14 interface is regarded to enhance the creep strength, i.e., the α /C14 interface acts as a creep strengthener.

- (3) The dislocations during creep are chiefly limited within the α -Mg lamellae for the α -Mg/C14-Mg₂Ca eutectic alloy, and most dislocations are α -dislocations with the identical Burgers vector. The dislocations are randomly distributed on the α /C14 interface. The α /C14 interface presents a barrier to the glide of α -dislocations within the α -Mg lamellae and does not annihilate and/or rearrange the α -dislocations caused by the creep test.

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