Impression creep behavior of the extruded Mg–4Zn–0.5Ca and Mg–4Zn–0.5Ca–2RE alloys

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Abstract

The creep characteristics of the extruded Mg–4Zn–0.5Ca and Mg–4Zn–0.5Ca–2RE (wt%) alloys were investigated using impression creep tests in the temperature range of 423–498 K. The creep behavior of both alloys was well described by the Garofalo hyperbolic sine relationship modified for the threshold stress concept. The stress exponent was 5 and the activation energies for creep were found to be close to that of the lattice diffusion in the α-Mg, i.e., 135 kJ mol⁻¹. Lattice diffusion-controlled climb of dislocations was thus determined as the dominant rate-controlling mechanism. At lower temperatures and higher stresses, the power-law breakdown was observed. The appearance of the threshold stress was attributed to the finely dispersed second phase particles that are present in the microstructure. Threshold stresses decreased linearly with increasing temperature. The RE-containing alloy showed higher threshold stresses and better creep resistance, due to the effect of load transfer resulting from high volume fraction of the second phases and formation of the thermally stable (MgZn)₂RE particles.

Keywords:
Mg–Zn–Ca alloys
RE addition
Impression creep
Garofalo relationship
Threshold stress

1. Introduction

As the lightest available constructional metals, magnesium and its alloys have received considerable attentions due to the increasing needs for efficient usage of natural resources and environmental issues. Magnesium cast components have found their commercial applications, while the applications of wrought magnesium alloys are still limited. The main drawbacks that prevent their widespread applications of wrought magnesium alloys are their limited formability at room temperature, and poor creep resistance at high temperatures [1,2]. However, research on the materials and processes for forming magnesium alloys have attracted much attention in recent years [3,4]. Interpretation of the high temperature deformation behavior of wrought magnesium alloys, such as AZ, ZK and ZM series, has been the subject of many studies [5–9]. Different alloy systems and various heat treatments have been examined to find out how the creep resistance of wrought magnesium alloys could be further enhanced [2,10,11]. Among various magnesium alloys, Mg–Zn based wrought alloys are of much importance due to their low cost, age hardenability, and biodegradability [2,11–14]. However, there have been limited studies reported in the literature on the creep properties and rate-controlling mechanisms of these alloys.

Creep behavior of pure metals and alloys can be explained by the well-known Dorn equation [15]

$$\dot{\varepsilon}_{\text{min}} = \frac{A G b D_0}{kT} \left( \frac{b}{G} \right)^p \left( \frac{\sigma}{G} \right)^n \exp \left( -\frac{Q}{RT} \right)$$  (1)

where $\dot{\varepsilon}_{\text{min}}$ is the minimum creep rate, $\sigma$ is the applied stress, $G$ is the shear modulus, $b$ is the Burgers vector, $k$ is the Boltzmann constant, $T$ is the absolute temperature, $d$ is the average grain size, $p$ is the inverse grain size exponent, $n$ is the stress exponent, $D_0$ is the pre-exponential constant, $Q$ is the activation energy for creep, $R$ is the universal gas constant, and $A$ is a dimensionless constant. Shear modulus $G$ is considered as a function of temperature. According to Eq. (1), stress exponent and activation energy can be determined from the experimental data using the following equations:

$$n = \left( \frac{\partial \ln \dot{\varepsilon}_{\text{min}} T/G}{\partial \ln (\sigma G)} \right)_T$$  (2)

$$Q = -k \left( \frac{\partial \ln (\dot{\varepsilon}_{\text{min}} T/G)}{\partial \ln (T/G)} \right)_G$$  (3)

In some particle-strengthened metals, however, Eq. (1) does not satisfactorily describe the high-temperature deformation behavior. In such materials, a downward curvature is observed in the curves at lower stresses in the logarithmic plots of $\dot{\varepsilon}_{\text{min}}$ against $\sigma$. In other words, the apparent stress exponent reaches very high values at the lowest levels of the applied stress. Although the
particle strengthening effect is evident at all steady-state stress levels, at lower stresses there appears a modulus-compensated stress, below which plastic deformation during creep is not measurable or is at least negligible. This stress has been termed as the “threshold stress”, $\sigma_{th}$ [16–18]. Activation energies appear to be relatively high (greater than lattice diffusion of the matrix), and the stress exponents are also high in the region where a threshold is apparent [16,19].

Threshold creep behavior is usually evident in metal–matrix composites [16,20], and in particle-strengthened metals containing either coherent precipitates or incoherent dispersoids [21,22]. Creep behavior of such materials is interpreted in terms of an effective stress ($\sigma_e$) acting on the material, defined as $\sigma - \sigma_{th}$, where $\sigma$ and $\sigma_{th}$ are the applied stress and the threshold stress, respectively. Assuming the effective stress acting on the material as $\sigma - \sigma_{th}$, Eq. (1) can be rewritten as

$$\dot{\varepsilon}_{\min} = \frac{A G b}{kT} \left( \frac{\sigma - \sigma_{th}}{G} \right)^p \exp \left( - \frac{Q}{kT} \right)$$

where $\sigma_{th}$ is the threshold stress and all other parameters are the same as in Eq. (1).

In the present study, the creep mechanisms of two extruded Mg–4Zn–0.5Ca and Mg–4Zn–0.5Ca–2RE (wt%) alloys were examined by the impression creep test, considering the threshold stress concept. The effect of RE addition on the creep resistance of the base alloy was also studied.

2. Experimental procedure

2.1. Materials and processing

Two alloys with nominal compositions of Mg–4Zn–0.5Ca and Mg–4Zn–0.5Ca–2RE (wt%) were used in this study. The alloys were prepared from pure Mg (99.80 wt%), pure Zn (99.90 wt%) and an Mg–10 wt% Ca master alloy. The RE was added in the form of Cerich micsh-metal (MM) which is a relatively cheap source of RE compared to individual rare-earth elements. The approximate composition of the MM used for alloying was 50.6 Ce, 23.2 La, 20.3 Nd and 5.9 Pr (wt%). The alloys were melted in an electrical furnace at 1023 K, in a graphite crucible under protection of a covering flux. A tilt-casting technique was used in order to minimize the melt turbulence and the casting defects. The cast billets were homogenized at 753 K for 8 h and then extruded with an extrusion ratio of 11:1 at 593 K.

2.2. Microstructural characterization

Microstructural examinations of the extruded alloys were conducted by optical and scanning electron microscopy (SEM). For this purpose, samples were prepared by mechanical grinding with SiC papers and polishing by 0.3 $\mu$m alumina paste followed by polishing on an abrasive-free polishing microcloth. Etching was carried out using an acetic picral solution (10 cc acetic acid, 4.2 g picric acid, 10 cc distilled water and 70 cc ethanol) at room temperature. Grain size measurements and determination of the area fraction of the second phases were performed using an image analyzer software.

2.3. Impression creep tests

3-mm-Thick specimens were cut by an electrodischarge wire-cut machine from the extruded bars perpendicular to the extrusion direction. Impression creep tests were conducted using a 50 kN capacity SANTAM universal tensile testing machine equipped with a three-zone split furnace. A flat-ended cylindrical punch of 2 mm diameter was used as the indenter. Details of the testing arrangement have been explained elsewhere [23] and will not be described here. Creep tests were carried out in the temperature range of 423–498 K under different applied stresses in the range of 100–850 MPa, depending on temperature and examined alloy for dwell times up to 3600 s. The load was controlled with the accuracy of $\pm$ 1.25 N and the impression depth was recorded with the resolution of $\pm$ 0.001 mm. Temperature control was achievable to a high accuracy within $\pm$ 1 K.

3. Results and discussion

3.1. Microstructural observations

The optical images of the extruded Mg–4Zn–0.5Ca and Mg–4Zn–0.5Ca–2RE alloys are shown in Fig. 1, where both alloys display a dynamically recrystallized equiaxed grain structure. It is evident that the grain size decreases with the addition of RE. Average grain size of the base ternary alloy is 4.6 ± 1.5 $\mu$m, while the RE-containing quaternary alloy has a smaller average grain size of 2.5 ± 1.3 $\mu$m. The large grains in the microstructure of the RE-containing alloy are regions that have remained unrecrystallized. Dark constituents in Fig. 1 are second phase particles that are distributed in the $\alpha$-Mg matrix. SEM micrographs of the base- and RE-containing alloys are demonstrated in Fig. 2 in the extrusion direction and normal direction (perpendicular to the extrusion direction). It is clear that the dendritic microstructure usually observed in the as-cast Mg–Zn and Mg–Zn–Ca alloys [24] has been broken during the extrusion process so that the second phase particles are mainly aligned along the extrusion direction.
be seen, there are some isolated spherical second phase particles with an average diameter of 2–3 μm, while most of the second phase particles are coarse, irregularly-shaped, and aligned in the extrusion direction. These coarse particles are more frequent in the RE-containing alloy (Fig. 2c and d). In addition, some of the second phase particles, finely distributed in the microstructure, have sub-micrometer dimensions. The volume fraction of these fine precipitates is higher in the RE-containing alloy compared to the base alloy. The area fractions of the second phase particles, calculated from the SEM images, are about 2.4% and 9.7% for the base- and RE-containing alloys, respectively. According to the EDS analysis presented in Table 1, second phase particles in the base alloy are identified as Ca$_2$Mg$_6$Zn$_3$. In the RE-containing alloy, however, RE-rich particles having a chemical composition close to that of (MgZn)$_{12}$RE are also formed.

3.2. Impression creep behavior

Typical impression creep curves of the materials tested at 448 K, and plotting impression depth against dwell time under different punching stresses in the range of 300–700 MPa are shown in Fig. 3. As can be seen, after a short primary stage each of the individual impression curves exhibit a steady-state or minimum creep rate region where depth increases linearly with time. It is also observed that increasing stress results in higher impression rates. Due to the compressive nature of loading in the impression creep test, fracture of the specimen does not occur, and hence it is obviously not possible to record a third stage of the curve, as opposed to what happens in an ordinary creep test. To ensure that the accumulated strain during a 3600 s test is sufficient for achievement of the steady-state, especially at lower stresses, further investigation of the steady-state creep rates involved performing some tests for the much longer periods of time.

![Fig. 2. SEM micrographs of Mg–4Zn–0.5Ca and Mg–4Zn–0.5Ca–2RE alloys in the extruded condition: (a and c) perpendicular and (b and d) parallel to extrusion direction.](image)

![Table 1. Average chemical composition of the particles in the tested materials obtained by EDS.](table)

<table>
<thead>
<tr>
<th>Particle</th>
<th>Element (at%)</th>
<th>Mg</th>
<th>Zn</th>
<th>Ca</th>
<th>RE</th>
</tr>
</thead>
<tbody>
<tr>
<td>α-Mg (base alloy)</td>
<td>98.6</td>
<td>1.3</td>
<td>0.1</td>
<td>-</td>
<td></td>
</tr>
<tr>
<td>α-Mg (RE-containing alloy)</td>
<td>98.5</td>
<td>1.1</td>
<td>0.2</td>
<td>0.2</td>
<td></td>
</tr>
<tr>
<td>Ca$_2$Mg$_6$Zn$_3$</td>
<td>59.8</td>
<td>24.6</td>
<td>15.6</td>
<td>-</td>
<td></td>
</tr>
<tr>
<td>(MgZn)$_{12}$RE</td>
<td>77.3</td>
<td>15.1</td>
<td>1.1</td>
<td>6.5</td>
<td></td>
</tr>
</tbody>
</table>
15,000 s and superimposing the creep curves on those obtained at the shorter period of 3600 s. Fig. 4a exhibits the results for the Mg–4Zn–0.5Ca alloy at \( T = 448 \text{ K} \) and \( \sigma_{\text{imp}} = 350 \text{ MPa} \). The comparison of the creep rates, shown in Fig. 4b, indicates that the shorter creep test yields the same minimum creep rate as the longer 15,000 s test, and thus the dwell time of 3600 s is considered to be sufficient for reaching the steady-state condition for the present alloys. It should be mentioned however that, the impression creep test has the limitation of studying creep at very low stresses, in which a reliable creep curve cannot be obtained. At low stresses, the load is not sufficiently high to overcome the initial frictional resistance of the material surface against the indenter, and thus, no data can be recorded for the impression depth. Despite this limitation, all of the recorded data are reliable and reproducible.

There are several studies which show that the impression creep data correlates well with those obtained in the conventional tensile and compressive creep [8,25,26]. Although impression creep tests usually show lower creep rates compared to tensile tests, due to the confined plastic deformation zone underneath the punch and the friction between punch and specimen during impression, the stress exponents and activation energies for creep, which dictate creep mechanisms, are almost the same. To correlate the impression and tensile creep data, the equivalent stress and strain rate can be evaluated from the impression stress under the punch (\( \sigma_{\text{imp}} \)) and the impression rate (\( V_{\text{imp}} \)) at a given load \( F \) and punch diameter \( \varphi \) as [27]

\[
\sigma = \frac{\sigma_{\text{imp}}}{c_1} = \frac{4F}{\pi \varphi^2 c_1}
\]

where \( c_1 \geq 3 \), and \( c_2 \geq 1 \) are the constants. Eqs. (5) and (6) were used to convert \( \sigma_{\text{imp}} \) and \( V_{\text{imp}} \) to the equivalent tensile creep data. The resultant \( \sigma \) and \( \dot{\varepsilon}_{\text{min}} \) values were then used to determine the stress exponent and activation energy for creep. Plots of the minimum creep rates, \( \dot{\varepsilon}_{\text{min}} \), against applied stress, \( \sigma \), in a double-logarithmic scale at \( T = 423–498 \text{ K} \), for both of the base- and RE-containing alloys are shown in Fig. 5. Stress exponents were obtained using Eq. (2) by plotting \( \dot{\varepsilon}_{\text{min}}/G \) versus \( \sigma/G \) at a constant \( T \) in a double-logarithmic scale, and the activation energy for creep was calculated according to Eq. (3) by plotting \( \dot{\varepsilon}_{\text{min}}/G \) against \( 1000/T \) at a constant \( \sigma/G \) in a semi-logarithmic scale. Shear modulus, \( G \), at each temperature was obtained using the relationship

\[
G (\text{MPa}) = \frac{19,200}{C_0 8.6^T}
\]

where \( C_0 = 19.200 \) and \( 8.6 \) is the constant. Although this relationship has been proposed for pure Mg, it has been frequently used for Mg alloys containing low levels of alloying elements [29–31]. Stress exponents and activation energies for creep are summarized in Table 2. It is obvious that stress exponents decrease with increasing temperature from 6.4 and 6.7 at 423 K to 5.0 and 4.6 at 498 K for the base- and RE-containing alloys, respectively. Apparent activation energies are also higher than that of the lattice diffusion in Mg, i.e., 135 kJ mol\(^{-1}\). Higher values of the stress exponent at lower temperatures and high values of the calculated activation energies may be due to the power–law breakdown. Power–law relationship, Eq. (1), can be used to fit the experimental data for a range of \( \sigma/G \) values up to 0.001–0.005 [32,33]. At higher normalized stresses, the power–law breaks down and the measured
strain rates are greater than the values predicted by the power-law relationship. In this work, normalized stresses in the range of $0.003 < \sigma/G < 0.018$ have been examined. Therefore, the possibility of power-law breakdown, especially at lower temperatures and higher stresses should be taken into account. For this purpose, the Garofalo hyperbolic sine relationship can be used to describe the deformation behavior for the entire experimental data.

\[ \varepsilon_{\text{min}} = A (\sinh \left( \frac{\alpha \sigma}{C} \right))^n \exp \left( \frac{-Q}{RT} \right) \]  

where $A$ and $n$ are the material-dependent parameters. At sufficiently low stresses sinh values are equal to their arguments, $(\sinh (a))^n \approx (a)^n$ (for $a < 0.8$). Therefore, Eq. (7) reduces to the conventional power-law relationship, Eq. (1). At high stresses, on the other hand, Eq. (7) can be substituted by an exponential relationship since $(\sinh (a))^n \approx (\exp (a))^n \exp (nu)$ (for $a > 1.2$) [21,34]. Thus, the hyperbolic sine relationship can describe the transition from power-law to power-law breakdown.

The downward curvature which is observed at the lower stresses of the experimental data (Fig. 5) can be rationalized by introducing the threshold stress, $\sigma$, to the Garofalo equation. The temperature- and shear modulus-compensated form of the Garofalo hyperbolic sine relationship can be used to describe the deformation behavior for the entire experimental data.

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That is observed when dislocation glide is the rate-controlling mechanism (class A alloys), \( n = 5 \), which is representative of the dislocation climb-controlled creep deformation (class M alloys), and \( n = 8 \) that is associated with the constant structure model of creep. The fitted curves are shown in Fig. 6 for the optimum value of the stress exponent \( n = 5 \). Parameters \( a \) and \( A = A' \exp(-Q/RT) \), and \( \sigma_{th} \) obtained by fitting are summarized in Table 3. Plotting \( A \) against \( 1000/T \) in a semi-logarithmic scale, as shown in Fig. 7a and b, gives the activation energies of 133.6 kJ mol\(^{-1}\) and 134.0 kJ mol\(^{-1}\) for the base- and RE-containing alloys, respectively. These values are very close to the activation energy for the lattice diffusion in pure Mg.

It is evident from Table 3 that the threshold stresses are temperature dependent and decrease by increasing the testing temperature. It is also observed that the RE-containing alloy generally possesses higher threshold stresses. Appearance of the threshold stress has been also reported in the creep of Mg–4Al–1Ca [21], die-cast Mg–8Zn–5Al–xCa [22], and Mg–3Ni–1.5Y–1.5Mm [34] magnesium alloys. In these cases, precipitation of the second phase particles has been noted as the origin of the threshold stress.

It is generally believed that in dispersion-strengthened materials with incoherent particles, where there is an attractive interaction between dislocations and particles, the strain energy of dislocations will increase by leaving the interface after completion of the climb [35,36]. Thus, detaching the dislocations from interacting particles increases the strain energy, and the need for higher stresses to detach the dislocations gives rise to the threshold stress. In the present study, the microstructures of the tested alloys, as shown in Fig. 2, are composed of equiaxed grains resulting from the dynamic recrystallization during the extrusion.

### Table 3

<table>
<thead>
<tr>
<th>Material</th>
<th>( T ) (K)</th>
<th>( a )</th>
<th>( A ) (K s(^{-1}) MPa(^{-1}))</th>
<th>( \sigma_{th} ) (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mg–4Zn–0.5Ca</td>
<td>423</td>
<td>72.0</td>
<td>( 8.900 \times 10^{-7} )</td>
<td>12.3</td>
</tr>
<tr>
<td></td>
<td>448</td>
<td>73.1</td>
<td>( 2.598 \times 10^{-6} )</td>
<td>8.6</td>
</tr>
<tr>
<td></td>
<td>473</td>
<td>91.6</td>
<td>( 5.202 \times 10^{-6} )</td>
<td>3.7</td>
</tr>
<tr>
<td></td>
<td>498</td>
<td>133.4</td>
<td>( 1.201 \times 10^{-5} )</td>
<td>0.5</td>
</tr>
<tr>
<td>Mg–4Zn–0.5Ca–2RE</td>
<td>423</td>
<td>60.1</td>
<td>( 9.977 \times 10^{-7} )</td>
<td>43.1</td>
</tr>
<tr>
<td></td>
<td>448</td>
<td>62.6</td>
<td>( 2.903 \times 10^{-6} )</td>
<td>29.0</td>
</tr>
<tr>
<td></td>
<td>473</td>
<td>73.3</td>
<td>( 7.380 \times 10^{-6} )</td>
<td>16.9</td>
</tr>
<tr>
<td></td>
<td>498</td>
<td>92.1</td>
<td>( 1.380 \times 10^{-5} )</td>
<td>1.5</td>
</tr>
</tbody>
</table>

![Fig. 7. Temperature dependence of the parameter \( A - A' \exp(-Q/RT) \) obtained by fitting Eq. (8) for (a) Mg–4Zn–0.5Ca and (b) Mg–4Zn–0.5Ca–2RE alloys.](image)

![Fig. 8. Temperature dependence of threshold stresses in (a) Mg–4Zn–0.5Ca and (b) Mg–4Zn–0.5Ca–2RE alloys.](image)
process, some coarse second phase particles, mostly aligned along the extrusion direction, and some fine precipitates. These finely dispersed precipitates may be responsible for the appearance of the threshold stress. It is reasonable to expect that the interactions of dislocations with these fine particles could give rise to the threshold stress. In the RE-containing alloy which possesses higher threshold stresses, the volume fraction of these fine particles is considerably higher (Fig. 2). This fact supports the argument that the threshold stress may be caused by these fine particles.

It is worth noting that the coarse second phase particles, which are mostly aligned along the extrusion direction, may have an extra strengthening effect similar to that of reinforcements in the composite materials. The reinforcement causes strengthening by load transfer and impeding the plastic flow of the softer matrix material. The presence of the reinforcing phase redistributes the stresses and strains within the composite and has the effect of transferring load from the matrix to the reinforcing phase. This in turn reduces the effective driving stress for creep in the matrix and results in lower creep rates for the composite material [37]. Reinforcements also hinder plastic flow of the softer matrix material. As a result of this constraint, hydrostatic stresses develop which increase the load carrying ability of the composite [38]. The factor by which the effective flow stress is reduced due to the load transfer and build-up of hydrostatic stresses depends on reinforcement volume fraction and aspect ratio as well as stress exponent $n$. The higher is the aspect ratio of the reinforcement, the higher will be the strengthening effect of the reinforcement due to the load transfer. In the present study, the better creep resistance of the RE-containing alloy can be attributed to the higher volume fraction and higher aspect ratios of the more thermally stable second phase particles (Fig. 2).

The variation of the threshold stress with temperature for the base- and RE-containing alloys is plotted in Fig. 8a and b, respectively. The values of the normalized threshold stress, $\sigma_{th}/G$, at each temperature are also shown in the same figure. Two different relationships between threshold stress and temperature have been reported in materials showing threshold creep behavior. In some studies, temperature dependence of the threshold stress has been reported to be linear [39], while some authors have reported non-linear temperature dependence of the threshold stress [40,41]. Mohamed et al. [42,43] showed that the variation of threshold stress with temperature is governed by a relationship of the Arrhenius-form

$$\frac{\sigma_{th}}{G} = B_0 \exp\left(\frac{Q_{th}}{RT}\right)$$

where $Q_{th}$ is an energy term which is associated with the process through which the moving dislocations overcome the obstacles in their glide planes. $Q_{th}$ is calculated from the slope of the plot of the $\sigma_{th}/G$ against inverse absolute temperature.

It is obvious from Fig. 8 that the relationship between threshold stress and temperature is linear. The threshold stress approaches zero at a temperature slightly higher than 500 K and disappears above this temperature. The similar phenomenon has been observed in dispersion strengthened materials. Gonzales-Doncel and Sherby [44] interpreted the disappearance of the threshold stress at high temperatures in terms of two creep processes, acting simultaneously. When the process not associated with the threshold stress dominates the other, the threshold behavior disappears. Here, the extent of the experimental data for the creep behavior at temperatures higher than 500 K is not sufficient to investigate the creep mechanism at these temperatures. Another explanation for the disappearance of the threshold stress at high temperatures was made by Rosler and Arzt [45,46]. The detachment of dislocations from interacting particles, which causes the threshold stress, is assumed to be athermal. However, as the temperature increases detachment stress decreases, and when the applied stress is lower than that, a new mechanism, i.e. the thermally activated detachment of dislocation, becomes rate-controlling that is not associated with the threshold stress. Transition from athermal to thermally activated detachment of dislocations from small incoherent particles has been reported to be the reason for the disappearance of the threshold stress at higher temperatures. In other words, at temperatures that threshold stress disappears, dislocations still interact with incoherent particles. However, the detachment process in this case is thermally activated rather than athermal. Therefore, interaction of dislocations with incoherent particles does not give rise to a threshold stress.

Fig. 9 shows the curves of $\dot{\varepsilon}_{min}T/G$ against $\sinh(\alpha(\sigma - \sigma_{th})/G)$ in a logarithmic scale for the studied materials. It can be seen that all of the experimental data points are well represented by linear fits.
with approximate slopes of 5. This indicates that the Garofalo relationship modified for the threshold stress concept is applicable to the interpretation of creep behavior of the extruded Mg–4Zn–0.5Ca and Mg–4Zn–0.5Ca–2RE alloys in the stress regime investigated.

3.3. Rate-controlling creep mechanisms

The experimental stress exponents together with the activation energy values have been frequently used to identify the mechanisms that control the deformation process. The preceding analysis of the creep behavior of the Mg–4Zn–0.5Ca and Mg–4Zn–0.5Ca–2RE alloys by fitting the modified Garofalo relationship suggested that true stress exponent of both alloys is 5 (Fig. 9) and the creep behavior is associated with a threshold stress. Values of true activation energy for creep were found to be close to the activation energy for lattice diffusion in Mg for both of the alloys. Fig. 10 shows the temperature-compensated minimum creep rate normalized to the lattice diffusion coefficient ($D_L$), Burgers vector ($b$), and shear modulus ($G$), $\dot{\varepsilon}_{\text{min}}kT/D_LbG$, plotted against normalized effective stress, in the form of sinh($\alpha(\sigma-\sigma_{th})/G$), for both of the alloys. All the data points lie along straight lines of the slopes close to 5. The linear regression coefficients are 0.982 and 0.979 for the base- and RE-containing alloys, respectively. This further confirms that the lattice diffusion-controlled climb of dislocations is the dominant deformation mechanism during creep. However, at higher stresses especially at lower temperatures power–law breaks down. It has been well established that under high stresses excess vacancies are generated by dislocation intersection processes. The concentration of vacancies under high stresses may be 3 or 4 orders of magnitude larger than the equilibrium vacancy concentration at a given temperature. Such excess vacancies will

![Fig. 10. Temperature-compensated minimum creep rate normalized to the lattice diffusion coefficient ($D_L$), Burgers vector ($b$), and shear modulus ($G$) as a function of sinh($\alpha(\sigma-\sigma_{th})/G$) at different testing temperatures for (a) Mg–4Zn–0.5Ca ($\alpha=88.0$) and (b) Mg–4Zn–0.5Ca–2RE ($\alpha=72.0$) alloys.](image)

![Fig. 11. Minimum creep rate normalized to the activation energy of the lattice diffusion ($Q_L$) as a function of sinh($\alpha(\sigma-\sigma_{th})/G$) for (a) Mg–4Zn–0.5Ca ($\alpha=88.0$) and (b) Mg–4Zn–0.5Ca–2RE ($\alpha=72.0$) alloys. The Sherby–Burke criterion for the power–law breakdown is also shown.](image)
enhance dislocation climb and therefore make creep easier when power-law breaks down [47]. Therefore, lattice diffusion-controlled climb of dislocations accelerated by excessive vacancy concentration due to high stresses is the rate-controlling mechanism at higher stresses and lower temperatures.

Values of the minimum creep rate normalized to the activation energy of lattice diffusion \( Q_\alpha \) as a function of \( \sinh (a(\sigma - \sigma_0)/G) \) are demonstrated in Fig. 11. The criterion of \( \xi_{\min}/D_0 = 10^{13} \text{ m}^{-2} \) proposed by Sherby and Burke [47], for the power-law breakdown in creep of materials, is also shown in this figure which corresponds to \( \xi_{\min} \exp(Q_\alpha/RT) = 10^3 \text{ s}^{-1} \left( D_0 = 10^{-4} \exp(-Q_\alpha/RT) \text{ m}^{-2} \right) \). According to this figure, most of the experimental points especially at lower temperatures fall in the power-law breakdown region. However, \( \alpha \) values obtained by the fitting indicate that the power-law relationship is valid in a wider region \((a(\sigma - \sigma_0)/G < 0.8)\) than predicted by this criterion.

It is worth noting that the extrusion process could completely change the creep mechanisms of the \( \text{Mg–}4\text{Zn–}0.5\text{Ca} \) and \( \text{Mg–}4\text{Zn–}0.5\text{Ca–}2\text{RE} \) alloys. The creep deformation behavior of the \( \text{Mg–}4\text{Zn–}0.5\text{Ca} \) and \( \text{Mg–}4\text{Zn–}0.5\text{Ca–}2\text{RE} \) alloys in the as-cast condition has been studied earlier [24]. Without any evidence of the threshold stress, the creep deformation of these materials in the as-cast condition exhibited two distinct regimes, depending on the applied stress level. In the low-stress regime, the stress assisted dislocation climb controlled by pipe diffusion was found to be the rate-controlling mechanism. While in the high-stress regime, with exponents higher than 11, the power-law broke down, and stress assisted dislocation climb controlled by lattice diffusion which was accelerated by excess vacancies due to high applied stresses was the rate-controlling mechanism. In contrast to the cast materials, our extruded materials show one region with one slope in the range of 4.6–5.7. There is no direct evidence for the occurrence of power-law breakdown. Therefore, possibility of power-law breakdown should be taken into account by using the Garofalo relationship modified for the threshold stress. Appearance of the threshold stress is directly related to the extrusion process, during which the fine precipitates form, giving rise to the threshold stress.

Extrusion process can also affect the creep resistance. Fig. 12 shows a comparison between the minimum creep rate data obtained in this study and those reported for \( \text{Mg–}4\text{Zn–}0.5\text{Ca} \) and \( \text{Mg–}4\text{Zn–}0.5\text{Ca–}2\text{RE} \) alloys in the as-cast condition [24] at 423 K and 473 K. The data for cast \( \text{Mg–}4\text{Zn} \) [31] and extruded ZM21 and ZEK200 [13] are also included in this figure. Cast and extruded \( \text{Mg–}4\text{Zn–}0.5\text{Ca} \) and \( \text{Mg–}4\text{Zn–}0.5\text{Ca–}2\text{RE} \) alloys have higher creep resistances than the cast \( \text{Mg–}4\text{Zn–}2\text{RE} \) alloy. This is due to the formation of thermally stable \( \text{Ca}_{0.5}\text{Mg}_{0.5}\text{Zn} \) and \( (\text{MgZn})_{12}\text{RE} \) intermetallic compounds in the \( \text{Mg–}4\text{Zn–}0.5\text{Ca} \) and \( \text{Mg–}4\text{Zn–}0.5\text{Ca–}2\text{RE} \) alloys, while in the \( \text{Mg–}4\text{Zn} \) alloy only \( \text{Mg}_{4}\text{Zn}_{7} \) phase with lower melting point and thermal stability is formed. Another point is that extrusion process could cause a decrease in the creep resistance of the \( \text{Mg–}4\text{Zn–}0.5\text{Ca} \) and \( \text{Mg–}4\text{Zn–}0.5\text{Ca–}2\text{RE} \) alloys. This is expected because the cast and extruded materials have completely different microstructural features. The skeleton morphology of the interdendritic particles in the cast materials seems to be more effective barriers against dislocation movements, than the equiaxed microstructures of the extruded materials with very small grain sizes and dispersed second phase particles. High area fraction of grain boundaries and the possibility of activation of grain boundary related deformation mechanisms at high temperatures, such as grain boundary sliding, could lead to the lower creep resistance of the extruded materials. Comparison of Fig. 12a and b supports this argument, where it reveals that by increasing the temperature, creep resistance of the extruded materials drops with a higher rate than that of the cast materials. Lower creep resistances of the extruded materials could also be related to the textural evolutions which take place during extrusion process. Detailed comparison of the high temperature mechanical properties of the cast and extruded \( \text{Mg–}4\text{Zn–}0.5\text{Ca} \) and \( \text{Mg–}4\text{Zn–}0.5\text{Ca–}2\text{RE} \) alloys is the subject of another ongoing paper. Despite the fact that extrusion process slightly decreased the creep resistance of the \( \text{Mg–}4\text{Zn–}0.5\text{Ca} \) and \( \text{Mg–}4\text{Zn–}0.5\text{Ca–}2\text{RE} \) alloys, these extruded alloys still have better creep resistance than the other wrought Mg alloys such as ZM21 and ZEK200 alloys, as observed in Fig. 12a.

### 3.4. Effect of RE addition on the creep resistance

Threshold stress is defined as a limiting stress level, below which the plastic strain is negligible. Thus, among materials that show threshold creep behavior, the one with higher threshold
stress is expected to have better creep resistance. Calculation of stress exponent and activation energy for creep showed that the addition of RE to the base alloy does not change the deformation mechanism. However, the RE-containing alloy shows not only lower strain rates at a given temperature and stress level, but also higher threshold stresses (Table 3). This means that the addition of RE enhances the creep resistance of the base alloy. Such an improvement in creep properties may be due to the higher volume fraction of the second phase particles together with the formation of the RE-rich particles. In addition, (MgZn)12RE particles have higher melting point and higher thermal stability in comparison with Ca2Mg6Zn3 particles formed in the base alloy. The load transfer effect in which part of the external load is carried by the reinforcements may also be partly responsible for the higher creep resistance of the RE-containing.

4. Conclusions

The impression creep test was used to evaluate the creep properties of the extruded Mg–4Zn–0.5Ca and Mg–4Zn–0.5Ca–2RE alloys in the temperature range of 423–498 K. The following conclusions are drawn:

1. The microstructure of the studied alloys consists of equixed recrystallized grains together with second phase particles. Average grain sizes of the base- and RE-containing alloys are 4.6 ± 1.5 and 2.5 ± 1.3 μm, respectively. In the base alloy, the second phase particles are Ca2Mg6Zn3, while in the RE-containing alloy (MgZn)12RE particles are also formed.

2. The creep behavior of the tested alloys is described by the Garofalo relationship modified for the threshold stress concept with the stress exponent of 5. At higher stresses and lower temperatures power–law breakdown occur.

3. Threshold stress decreases with increasing temperature. The RE-containing alloy exhibits higher threshold stresses. It is suggested that the observed threshold stress may be caused by the finely dispersed second phase particles that are present in the microstructure.

4. Threshold stress decreases linearly with increasing temperature up to 500 K. Disappearance of the threshold stress above this temperature may be due to the thermally activated detachment of dislocations from interacting particles.

5. The stress exponent of about 5 and activation energy close to that of lattice diffusion in Mg suggest that the lattice diffusion-controlled climb of the dislocations is the rate-controlling mechanism in both investigated alloys. At higher stresses and lower temperatures, this process is accelerated by the excessive vacancy concentrations caused by high stresses.

6. Better creep resistance of the RE-containing alloy compared to the base alloy is attributed to the higher threshold stresses, greater the load transfer effect due to the higher volume fraction of the second phase particles present in the microstructure, and formation of the thermally stable (MgZn)12RE particles.

References