Capacity for Deformation and the Evaluation of Flow Stress of Hot Extruded Mg–8Al–xRE Alloys at Elevated Temperatures

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This paper investigated the optimal deformation temperature $T_c$, and the optimal Zener-Hollomon parameter $Z_c$, of both as-extruded Mg–8Al and Mg–8Al–2RE alloys, to examine the capacity of these alloys for deformation. Additionally, an equation that involves the Zener-Hollomon parameter $Z$ was derived to evaluate the flow stress of these magnesium alloys at various temperatures and strain rates and was validated using a statistical method. These alloys were prepared by melting and casting in a vacuum induction furnace, and extruded at 633 K with a reduction ratio of 90 : 1. Tensile tests were performed at 473 to 723 K and at strain rates ranging from $8.3 \times 10^{-4}$ to $8.3 \times 10^{-1}$ s$^{-1}$. The implications of the experimental data were discussed in detail.

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1. Introduction

The cost of manufacturing thin wall magnesium alloy components by casting is rather high compared with those methods by producing complex-shaped components directly from the plates and rods. Thus, methods for manufacturing complex engineering components directly from wrought alloys must be developed. However, cold-forming of magnesium alloys is difficult because of their h.c.p. structure. Therefore, their mechanical properties must be improved to develop plastic forming technology.

A previous study$^1$ applied hot extrusion to refine grains and add rare earth elements to improve the high temperature mechanical properties of Mg-based alloys. Superplasticity of as-extruded Mg–8Al–xRE (in mass%; $x = 0, 1, 2$ or 3%) alloys has been observed at a temperature of 473 K or above, at a strain rate of $8.3 \times 10^{-4}$ s$^{-1}$. Mg–8Al alloy is well known to be more deformable at higher temperatures because secondary slip systems become available. Thus, hot deformation (such as superplastic forming) has attracted the interest of many investigators of magnesium alloys.$^2$–$^5$ The elongations and flow stresses of magnesium alloys at elevated temperatures are indispensable in investigating the hot-forming process. However, analytical data on the flow stress of magnesium alloys are scarce.

This study examines the capacity for deformation and evaluates the flow stress of both as-extruded Mg–8Al and Mg–8Al–2RE alloys at various temperatures and strain rates using the method of Takuda et al.$^9$ An equation that involves the Zener-Hollomon parameter is derived to fit the experimental data and, in this study, the equation is validated using a statistical method.

2. Experimental Procedure

The alloys used in this study were Mg–8Al (Mg–8%Al–0.62%Zn in mass%) and Mg–8Al–2RE (Mg–8%Al–0.66%Zn–2.17%RE in mass%). The alloys were prepared by melting and casting in a vacuum induction furnace in an atmosphere of Ar gas, and then extruded into sheets with a thickness of 3 mm at 633 K. The extrusion ratio was 90 : 1. Tensile tests were performed ranging from 473 to 723 K at strain rates ranging from $8.3 \times 10^{-4}$ to $8.3 \times 10^{-1}$ s$^{-1}$. The specimens took $6 \times 10^2$ s to equilibrate at the test temperatures before they were strained.

3. Results and Discussion

3.1 Optimal deformation temperature $T_c$

Figure 1 presents the elongations of both as-extruded Mg–8Al and Mg–8Al–2RE alloys at various temperatures and strain rates. The elongations of Mg–8Al and Mg–8Al–2RE alloys at 523 K and a strain rate of $8.3 \times 10^{-3}$ s$^{-1}$ are 137 and 114%, respectively, revealing superplastic elongation. Additionally, both as-extruded magnesium alloys have a unique optimal deformation temperature, $T_c$, when other parameters are held constant. At a temperature below $T_c$, elongation increases with temperature. On the other hand above $T_c$, the elongation decreases as temperature increases. This phenomenon, previously reported by the authors can be explained in two ways.$^{1,7}$

Firstly, the grain boundaries function as sites of weakness at high temperatures.$^8$ A rough method to distinguish the high temperature from the low temperature is by the equicohesive temperature. The equicohesive temperature is defined as the temperature at which the grains and grain boundaries have equal strength.$^9$ Above this temperature the grain boundary region is weaker than the grain interior. Below the equicohesive temperature the grain boundary region is stronger than the grain interior.$^9$ Based on the above, the strength of grain boundary determines the high-temperature strength of polycrystalline alloys, because the
The grain boundary is weaker than the inside of grains at elevated temperature. The $T_c$ may be close to the equicohesive temperature. Consequently, the ability of the alloys to transmit applied stress declines, causing stress concentration because the grain boundaries are further weakened when the temperature exceeds $T_c$. The effects of the above mentioned mechanism about the weakening behavior become more significant, compared with the other possible mechanisms.

Secondly, Watanabe et al. $^{11}$ examined the deformation behavior in a relatively coarse grain (17.1 μm) AZ61 magnesium alloy. They suggested that the deformation mechanism is grain boundary sliding accommodated by slip, controlled by grain boundary diffusion at low temperature and by lattice diffusion at high temperature. In other words, the diffusion process is assumed to be the rate-controlling process of the deformation mechanism in the whole investigated temperature range. It is supposed the deformation mechanism of both of the as-extruded Mg–8Al and Mg–8Al–2RE magnesium alloys are the same as that of the as-extruded AZ61 magnesium alloy due to their similar investigated temperature range and grain size of about 20 μm. Additionally, the Mg–8Al alloy is mainly strengthened by the precipitation of Al12Mg17 precipitates and the Mg–8Al–2RE magnesium alloy is mainly strengthened by both of the precipitation of Al12Mg17 precipitates and dispersion of Al17RE5 precipitates. $^{11}$ Thus the grain growth rate may increase as the temperature exceeds $T_c$, hindering the internal accommodation of the alloy, in which the diffusion of atoms and the motion of dislocations take longer paths in a larger grain and get more chance to be retarded by precipitates, and increasing the deformation resistance. The ability of the alloys to transmit applied stress declines in one way, and the deformation resistance increases in the other way. The alloys fracture when the above mentioned two phenomena are unbalanced. Therefore, the elongation is reduced as the temperature increases above $T_c$. $^{1,5}$

Further investigation shows that the $T_c$ of Mg–8Al alloy is 573 K at $8.3 \times 10^{-8}$ s$^{-1}$ and increases with strain rate, as shown in Fig. 1(a). The $T_c$ of Mg–8Al alloy is 623 K at $8.3 \times 10^{-3}$ s$^{-1}$ and exceeds 723 K at a strain rate of $8.3 \times 10^{-2}$ s$^{-1}$. Figure 1 indicates that the values of $T_c$ of both Mg–8Al and Mg–8Al–2RE alloys exhibit the same trend as strain rate changes. Interestingly, the $T_c$ of Mg–8Al–2RE alloy is much higher than that of Mg–8Al alloy under the same conditions. Additionally, the elongation of Mg–8Al–2RE alloy is also much greater than that of Mg–8Al alloy. These findings suggest that the RE is a grain boundary stabilizer of the Mg–8Al alloy. In other words, RE can retard the weakening of the boundary and the growth of the grain. In summary, at elevated temperatures, adding RE greatly improves the capacity of Mg–8Al alloy to deform.

### 3.2 Optimal Zener-Hollomon parameter $Z_c$

Figure 1 also shows that $T_c$ varies with the strain rate for each alloy. Figure 2 plots the relationship between the elongation and the temperature-compensated strain rate parameter, the Zener-Hollomon parameter, $Z$ $^{11}$

$$Z = \dot{\varepsilon} \exp\left(\frac{Q}{RT}\right)$$

where $\dot{\varepsilon}$ represents the strain rate (s$^{-1}$); $R$ is the universal gas constant (8.314 J mol$^{-1}$ K$^{-1}$); $T$ is the absolute temperature (K), and $Q$ is the apparent energy required for deformation. In this analysis, for convenience, the activation energy of the lattice diffusion of magnesium, $^{12}$ 135 kJ mol$^{-1}$ is used as the value of $Q$ for both as-extruded Mg–8Al and Mg–8Al–2RE alloys.

Figure 2 shows that the relationship between elongation and $Z$ follows the same trend as that between elongation and temperature in Fig. 1. The optimal value of the Zener-Hollomon parameter $Z_c$ for Mg–8Al alloy is $2 \times 10^9$ s$^{-1}$ at a strain rate of $8.3 \times 10^{-4}$ s$^{-1}$ or $8.3 \times 10^{-3}$ s$^{-1}$. $Z_c$ of Mg–8Al–2RE is much smaller than that of Mg–8Al under the same conditions. $Z_c$ of both magnesium alloys declines as the strain rate increases, which finding can be explained as follows. $^{11}$ When $Z$ is less than $Z_c$, the elongation decreases as $Z$ reduces, implying that the elongation decreases as the strain rate decreases or as temperature increases, because the cavities in the test alloys have more time to grow and link together as the strain rate reduces or the temperature increases, leading to a decrease in elongation.
However, when $Z$ exceeds $Z_{c}$, the elongation decreases as $Z$ increases, implying that the elongation decreases as strain rate increases or temperature decreases. This trend may be explained as follows. Superplastic alloys exhibit a sigmoidal relationship of three-stage variation on a double logarithmic scale of stress and strain rate, as indicated by several researchers. Conventional sigmoidal curve can be divided into three distinct regions from the low strain rate to the high strain rate. In region I, the strain rate sensitivity, $m$, is small. In region II, the strain rate sensitivity is $0.5 < m < 1$, the strain rate sensitivity reaches a maximum and a maximum elongation can be observed. In region III, the strain rate sensitivity decreases again.

In region II, $m$ decreases again. The curve shows a drop in the strain rate sensitivity exponent, $m$. Therefore, the alloys neck easily because the grains elongate in the stress direction and the density of dislocations inside grains increases during deformation. Consequently, the elongation is lower than expected.

Hence, $T_{c}$ is also an optimal deformation temperature when other parameters are held constant. However, some other factors, such as strain rate, must be considered for industrial applications. Accordingly, $Z_{c}$ must be discussed further.

### 3.3 High temperature flow stress

Figure 3 shows the 0.2% proof stress versus strain rate curves on a double logarithmic scale at various temperatures, obtained by the uniaxial tensile testing of both as-extruded Mg–8Al and Mg–8Al–2RE alloys. The relationship between the logarithm of the 0.2% proof stress ($\sigma$) and the logarithm of strain rate ($\dot{\varepsilon}$), for both magnesium alloys, is linear at each temperature, and is typically expressed as:

$$\sigma = K_{1}(\dot{\varepsilon})^{m}$$

However, the coefficients $K_{1}$ and $m$ (strain rate sensitivity exponent) are functions of temperature. For example, the mean value of $m$ obtained from the slope of the curves depends on the temperature, as shown in Fig. 3. Therefore, the industrial application of eq. (2) is complicated. To simplify the formula, using the Zener-Hollomon parameter $Z$ (eq. (1)).

Sellars and Tegart proposed the following relationship between the stress, the strain rate and the temperature under hot-working conditions.

$$\dot{\varepsilon} = A_1 \sinh(\alpha \sigma)^n \exp(-Q/RT)$$

Applying this formula to eq. (1) yields:

$$Z = A \sinh(\alpha \sigma)^n$$

At low stresses ($\alpha \sigma < 1.0$), eq. (4) reduces to a power relation

$$Z = A_1 \sigma^n$$

and at high stresses ($\alpha \sigma > 1.2$), it reduces to an exponential relation

$$Z = A_2 \exp(\beta \sigma)$$

where $A$, $A_1$, $A_2$, $\alpha$, $n'$ and $\beta$ are experimentally determined constants and are independent of the temperature.

Figure 4 plots the relationship between the 0.2% proof stress ($\sigma$) and the Zener-Hollomon parameter ($Z$) for both Mg–8Al and Mg–8Al–2RE alloys on a semi-log scale: $\sigma$ is linearly related to log ($Z$) for both magnesium alloys, and eq. (6) fits the test data closely. However, the last data points at $Z = 5.56 \times 10^7$ s$^{-1}$ ($Z = \dot{\varepsilon} \exp(Q/RT)$, where $\dot{\varepsilon} = 8.3 \times 10^{-3}$ s$^{-1}$, $T = 723$ K) in both of alloys seem to deviate from the eq. (6) in Fig. 4. In other words, eq. (6) is not available for the conditions: $T = 723$ K, $\dot{\varepsilon} \leq 8.3 \times 10^{-3}$ s$^{-1}$. Except these, the experimental result is consistent with the relationship between the 0.2% proof stress ($\sigma$) and the Zener-Hollomon parameter ($Z$) for AZ31 and AZ91 alloys calculated by Takuda et al. This study reveals that adding RE does not change the relationship between $\sigma$ and $Z$ for magnesium alloys of the AZ series. The method developed by Takuda et al. is applied to convert eqs. (6) to (7) to determine the constants, $A_2$, and $\beta$ in eq. (6):
\[
\sigma = (1/\beta) \ln Z - (1/\beta) \ln A_2 \\
= (1/\beta) \ln(Z/Z_0) - (1/\beta) \ln(A_2/Z_0) \\
= B \ln(Z/Z_0) + C
\]

where \(Z_0\) is the representative value of \(Z\), \(B\) is the slope of the straight lines and \(C\) is the value of \(\sigma\) at \(Z = Z_0\) in Fig. 4. \(Z_0\) is often obtained from the middle value of \(Z\). Equation (7), Fig. 4 and \(Z_0 = 2.1 \times 10^{11}\) \((s^{-1})\) together yield the following results.

\(B = 8.44\) (MPa), \(C = 71.1\) (MPa)

for Mg–8Al alloy  

\(B = 8.54\) (MPa), \(C = 75.5\) (MPa)

for Mg–8Al–2RE alloy

3.4 Validation

The 0.2% proof stresses, \(\sigma_{0.2\%}\), calculated using the above formula are compared with the measured stresses in Fig. 5 to further validate eqs. (8) and (9). A coefficient of determination, \(r^2\), is applied to show the capacity of the best-fit line to predict the actual testing data. The definition of \(r^2\) is,

\[
r^2 = SSe/(SSe + SSR)
\]

where \(SSe\) is the sum of the squares of all the residual values. In Fig. 5, the residual value is the difference between the calculated value of \(\sigma_{0.2\%}\) and the measured value of \(\sigma_{0.2\%}\) at a given Zener-Hollomon parameter (Z) value. \(SSR\) is the sum of the squares of the differences between the average of all the measured values of \(\sigma_{0.2\%}\) and the calculated values of \(\sigma_{0.2\%}\) at each \(Z\) at which a data point exists. The value of \(r^2\) is between 0 and 1. A larger \(r^2\) indicates a more accurate best-fit line for
predicting the actual test data. In Fig. 5, the $r^2$ of Mg–8Al and Mg–8Al–2RE alloys are 0.989 and 0.990, respectively. Therefore, eqs. (8) and (9) excellently fit the measured values over a wide range of stresses, and the assumed value of the activation energy $Q$ is reasonable. The preceding formulae are developed for 0.2% proof stress. The strain may have to be involved in the formula for flow stress. However, the dependence of the flow stress on the strain, such as the work-hardening, is insignificant in the investigated temperature range as shown in Fig. 6. Therefore, the flow stress during hot deformation could be evaluated approximately by the preceding formulae. This trend is similar to the conclusion of Takuda et al.61

Adding 2% RE can increase the 0.2% proof stress and the elongation of the Mg–8Al alloy by more than 51% at 573 K and 58% at 673 K at a strain rate of $8.3 \times 10^{-4}$ s$^{-1}$, respectively. However, the high-temperature flow stress of the Mg–8Al–2RE alloy does not always far exceed that of the Mg–8Al alloy over the whole investigated temperature range. The Mg–8Al–2RE alloy may be used to replace Mg–8Al alloy under high-temperature creep conditions. Consequently, not only can the Mg–8Al–2RE alloy be deformed under a stress not much greater than that required to deform the Mg–8Al alloy (such that the energies required to deform both alloys are very close), but it can also be deformed into more complicated components because it possess larger elongation than the Mg–8Al alloy. Moreover, components made of Mg–8Al–2RE are more appropriate for operating under high-temperature creep conditions than those made from Mg–8Al. Thus, given the equivalent properties of Mg–8Al and AZ80 alloys and the relative inexpensive of the RE, replacing AZ80 with Mg–8Al–2RE alloy is highly recommended.

4. Conclusions

(1) The elongations of Mg–8Al and Mg–8Al–2RE alloys at 523 K at a strain rate of $8.3 \times 10^{-3}$ s$^{-1}$ are 137% and 114%, respectively. The superplastic properties of both
magnesium alloys are very close to industrial standard of high strain rate superplasticity (HSRS), which requires that the strain rate exceeds $1 \times 10^{-2}$ s$^{-1}$ and the elongation exceeds 100%.

(2) Both magnesium alloys have a unique $T_c$ and $Z_c$. However, under the same conditions, $T_c$ of Mg–8Al–2RE alloy exceeds that of the Mg–8Al alloy, but $Z_c$ of the Mg–8Al–2RE alloy is lower than that of the Mg–8Al alloy. Therefore, RE (La-rich misch metal) is a boundary stabilizer of the Mg–8Al alloy.

(3) Adding RE does not change the relationship between $/frac{Z}{C}$ and $Z$ for magnesium alloys of the AZ series. Equation (6), $Z = A_2 \exp(B/RT)$ still fits. Consequently, the method of Takuda et al. can be used to obtain a comprehensive equation for the flow stress of both as-extruded Mg–8Al and Mg–8Al–2RE alloys at elevated temperatures. Equations that may be provide a reference for actual industrial processes are as follows.

$$
\sigma = B \ln(\frac{Z}{Z_0}) + C \\
Z = \dot{\epsilon} \exp(Q/RT) \\
R = 8.31 (J \text{ mol}^{-1} \text{K}^{-1}); \quad Q = 135 (\text{kJ mol}^{-1}) \\
Z_0 = 2.1 \times 10^{11} \text{(s}^{-1}) \\
B = 8.44 \text{(MPa)}, \quad C = 71.1 \text{(MPa)} \\
\quad \text{for Mg–8Al alloy} \\
B = 8.54 \text{(MPa)}, \quad C = 75.5 \text{(MPa)} \\
\quad \text{for Mg–8Al–2RE alloy}
$$

The equations are valid under the conditions,

$$
473 \leq T \leq 723(K) \\
8.3 \times 10^{-4} \leq \dot{\epsilon} \leq 8.3 \times 10^{-1} \text{(s}^{-1})
$$

exception for $T = 723 K$, $\dot{\epsilon} \leq 8.3 \times 10^{-3} \text{s}^{-1}$.

(4) In summary, the modified Mg–8Al–2RE Mg alloy is a possible alternative to the AZ80 alloy.

REFERENCES