A R C H I V E S O F M E T A L L U R G Y A N D M A T E R I A L S
Volume 60 2015 Issue 2
DOI: 10.1515/amm-2015-0124

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# HIGH TEMPERATURE DEFORMATION FLOW OF A ZK60A MAGNESIUM ALLOY AFTER EXTRUSION

# PŁYNIĘCIE STOPU MAGNEZU ZK60A PODCZAS WYSOKOTEMPERATUROWEGO ODKSZTAŁCENIA W TRAKCIE WYTŁACZANIA

Flow behavior of a ZK60A magnesium alloy after continuous casting and subsequent extrusion was examined in tension at a range of strain rates of $3.0\times10^{-6}$ -$1.0\times10^{-2}$ $s^{-1}$ at temperatures of 473-623K. The results demonstrated that the alloy exhibited a maximum elongation of ~250% at 523K when tested at an initial strain rate of $1.0\times10^{-5}$ $s^{-1}$ and strain rate sensitivity, $m$, of ~0.3-0.4 and the activation energy of ~94 kJ/mol were calculated under the testing conditions. The detailed investigation suggested that the high temperature flow of the ZK60A alloy having submicrometer grains demonstrates quasi-superplastic flow behavior controlled by a dislocation viscous glide process.

*Keywords*: activation energy, extrusion, flow mechanism, magnesium alloy, tensile property

## 1. Introduction

Magnesium is one of the most light-weight metals (density 1.74 g/cm$^3$) in all practical engineering materials with high specific strength, and it has outstanding mechanical properties, such as high strength and high elasticity. Therefore, in recent years the magnesium alloys have received much attention for applications in light-weight structural materials for the automobile and aerospace industries and for the portable electrical devices [1, 2]. Moreover, the magnesium is the 6$^{th}$ most abundant metal on the earth accounting for 2.5% of the crust as a mineral, and it is therefore a very abundant resource. The recycling properties of magnesium are also satisfactory and it has been called the metal which responds best to the required properties from the viewpoint of earth environment protection that humans are facing in recent years.

However, since magnesium is h.c.p. structure and the slip system at room temperature is limited to basal slip, processability is deficient and it is difficult to add any forming capability. Therefore, in the present conditions magnesium alloy products for practical use are mainly manufactured by casting. Even though, in practice, the variety of products made of casting magnesium alloys is limited due to the difficulty in deforming them into different shapes which are necessary for the structural applications. Therefore, improving the ductility and strength of magnesium alloys is the key to increasing their applications as casting products. As a consequence, extrusion of thermomechanical processing is one of the practical techniques applied to magnesium casting ingots for improved workability of magnesium alloys to date. Accordingly, the present investigation was initiated to evaluate the high temperature mechanical properties of continuous-casting ZK60A after extrusion with the aim of understanding the fundamental deformation characteristics of the ZK60A magnesium alloy.

## 2. Experimental material and procedures

A commercial grade ZK60A magnesium alloy was processed by continuous casting to have a diameter of 106 mm and the as-cast sample was subsequently extruded with an extrusion ratio of 6:1 at 573 K. The microstructure of both as-cast sample and the sample after extrusion were characterized using an equipment of electron backscatter diffraction (EBSD), EDAX Digiview-1, in a field emission scanning electron microscopy (FE-SEM), Hitachi S-4300SE. The EBSD specimens were prepared using a broad ion beam polisher, Hitachi IM-3000. The step size of the EBSD map was 1.2 $\mu$m at an accelerating voltage of 12 kV. After the raw orientation maps were collected, standard cleanup procedures were conducted to minimize the incorrectly indexed pixels and background noise using TSL OIM software.

The ZK60A alloy after continuous casting and subsequent extrusion was cut into individual billets having lengths of ~88 mm and machined for a set of tensile specimens with a gauge length of 25 mm and a diameter of 6 mm where the tensile direction is parallel to the extrusion direction. All specimens were pulled to failure in tension at temperatures of 473, 523, 573 and 623 K using a universal testing machine (MTDI, Korea) operating at a constant rate of cross-head displacement and with initial stain rates in the range from $3.0\times10^{-6}$ to $1.0\times10^{-2}s^{-1}$.

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## 3. Results and discussion

### 3.1. Microstructure

Figure 1 shows the inverse pole figure maps from electron backscatter diffraction (EBSD) analysis for the ZK60A alloy after (a) continuous casting prior to extrusion and (b) extrusion. It is apparent from Fig. 1(a) that the material after casting demonstrates typical casting microstructure consisting of randomly oriented large equiaxed grains having an average linear intercept grain size of ~100 um, and annealing twins are occasionally observed inside of grains in the as-cast ZK60A alloy. In contrast, as shown in Fig. 1(b), the extruded material demonstrates a banded structure consisting of elongated grains along the extrusion direction (marked with a black allow) due to the plastic deformation by extrusion. The extruded sample includes a significant amount of low-angle grain boundaries which were defined to have misorientation angles of less than 5° and the low-angle boundaries are denoted by white lines in Fig. 1(b). The extruded material has relatively fine grains in the size ranging from few microns to submicron which attributed to dynamic recrystallization by the significant straining during extrusion.

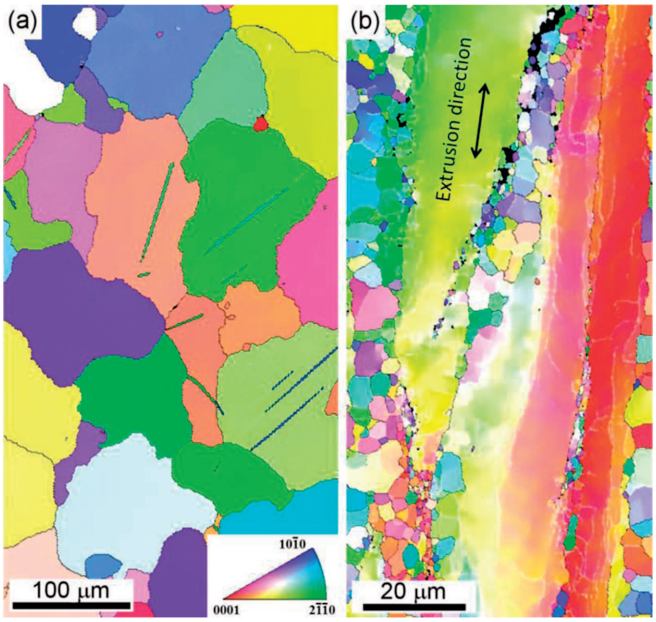
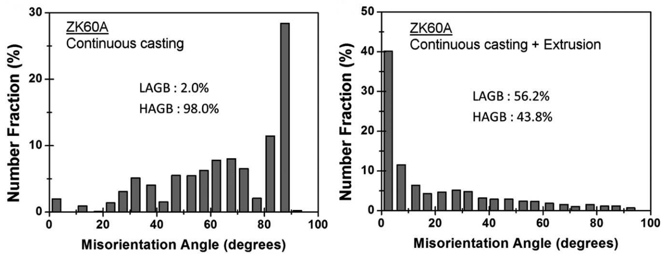



Fig. 1. EBSD inverse pole figure maps for the ZK60A alloy after (a) continuous casting and (b) extrusion



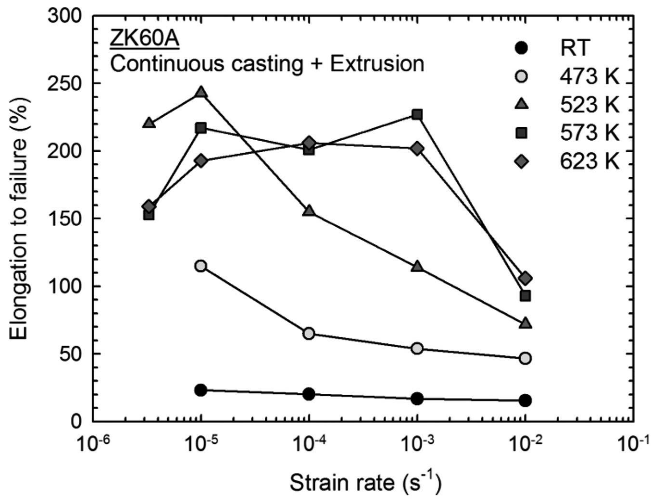

(a) (b)

Fig. 2. Distributions of the number fractions of the grain boundary misorientation angles for the ZK60A alloy after (a) continuous casting and (b) extrusion

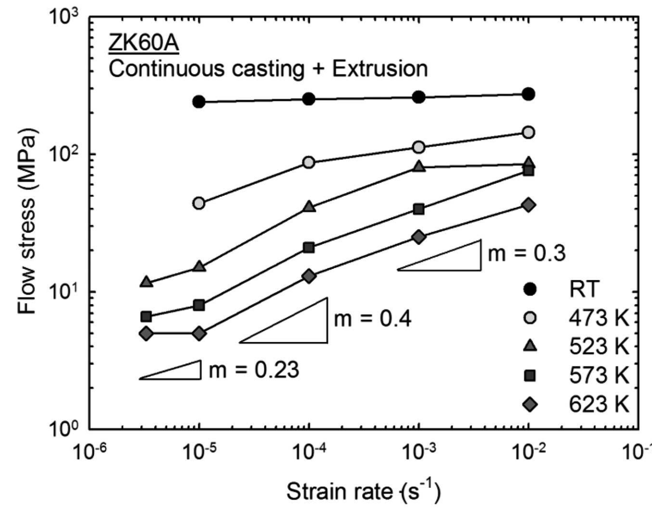
Figure 2 shows the distributions of the number fractions (in %) of the grain boundary misorientation angles for the ZK60A alloy after (a) continuous casting prior to extrusion and (b) extrusion. The data show that the fractions of high-angle grain boundaries (HAGB) were measured as 98% after casting and ~44% after extrusion. Thus, as demonstrated in Fig. 1, there is a significant change in microstructure as well as texture and grain boundary misorientations after extrusion.

### 3.2. High temperature mechanical properties

Tensile testing was conducted at 473-623 K using constant initial strain rates from $3.3\times10^{-6}$ to $1.0\times10^{-2}s^{-1}$. Using the data obtained from the specimens pulled to failure, Fig. 3 shows the variations of (a) elongation to failure versus strain rate and (b) flow stress versus strain rate for the ZK60A after continuous casting and extrusion. It should be noted that the values of 0.2% yield stress were used as flow stress in Fig. 3(b). For comparison purposes, the results in tension at room temperature (RT) are included in both plots in Fig. 3.




(a)




(b)

Fig. 3. The variations of (a) elongation to failure vs. strain rate and (b) flow stress to failure vs. strain rate for the ZK60A alloy tested at 473-623 K

Tensile testing was conducted at 473-623 K using constant initial strain rates from $3.3\times10^{-6}$ to $1.0\times10^{-2}s^{-1}$. Using the data obtained from the specimens pulled to failure, Fig. 3 shows the variations of (a) elongation to failure versus strain rate and (b) flow stress versus strain rate for the ZK60A after continuous casting and extrusion. It should be noted that the values of 0.2% yield stress were used as flow stress in Fig. 3(b). For comparison purposes, the results in tension at room temperature (RT) are included in both plots in Fig. 3.

In Fig. 3(a), a general trend of deformation flow where higher elongations are recorded at slower strain rates is observed in the ZK60A under the present testing conditions and the trend is significant when tested at lower temperatures of 473 and 523 K. The maximum recorded elongation of ~250% was observed when using an initial strain rate of $10\times10^{-5}s^{-1}$ at 523 K. However, there is a clear shift of the peak elongation to faster strain rates with increasing testing temperatures so that the relatively high elongations to failure of ~200% were observed at a faster strain rate of $1.0\times10^{-3}s^{-1}$ at higher testing temperatures of 573 and 623 K. The slope of the curves in a plot shown in Fig. 3(b) denotes strain rate sensitivity, $m$, and it was calculated as ~0.4 at strain rates of $1.0\times10^{-5}$-$1.0\times10^{-4}$ $s^{-1}$, and the lower values of ~0.2 and ~0.3 at lower than $1.0\times10^{-5}$ and $1.0\times10^{-4}$-$1.0\times10^{-2}$ $s^{-1}$, respectively, at the present testing temperatures of 523-623 K whereas slower slope, thus lower values of $m$, is measured at the lowest testing temperature of 473 K.

The measured elongations to failure in the present investigation were lower by comparison with the conventional superplastic alloys where elongations to failure are typically over 400% [5]. It is well established in practice that conventional superplasticity requires an average equiaxed grain size smaller than ~10 $\mu$m [5, 6] and it is the lower limit of grain sizes attained by extrusion in most Mg alloys. Therefore, although the calculated values of $m$ are reasonably high for a possible occurrence of superplasticity, the low ductility of the ZK60A is attributed to the large initial grain size after extrusion. It is worth noting that the strain rate sensitivity is a critical parameter in determining the total elongation to failure. Specifically, it was shown in very early work that the elongations achieved in tension increase with increasing values of $m$ [7, 8].

### 3.3. Flow mechanism and activation energy of ZK60A

In order to obtain a better understanding of the flow process of the ZK60A alloy in high temperature where diffusive flow is reasonably rapid, it is necessary to consider the dependence of flow upon the testing temperatures. When materials deform at high temperatures, the steady-state strain rate, $\dot{\varepsilon}$, is given by a relationship of the form:

$$\dot{\varepsilon} = \frac{ADGb}{kT}\left(\frac{b}{d}\right)^p\left(\frac{\sigma}{G}\right)^n \quad (1)$$

where $A$ is a dimensionless constant, $D$ is the diffusion coefficient, $G$ is the shear modulus of the material, **b** is the Burgers vector, $k$ is Boltzmann's constant, $T$ is the absolute temperature, $d$ is the grain size, $p$ is the exponent of the inverse grain size and $n$ (= $1/m$) is the stress exponent. The diffusion coefficient is given by $D_o\exp(-Q/RT)$, where $D_o$ is a frequency factor, $Q$ is the activation energy for the rate-controlling diffusive process and $R$is the gas constant. It follows from inspection of Eq. (1) that when a material has reasonably coarse grains the steady-state creep rate is defined exclusively by the values of only four terms: $A$, $Q$, $p$ and $n$.

In order to interpret the present creep date using Eq. (1), Fig. 3 was rearranged to show a logarithmic plot of tensile strain rate versus flow stress and it is shown in Fig. 4. The slopes of the best-fit lines in Fig. 4 correspond to the stress exponents for creep that are given by $n \approx 4$ at 523-623 K and $n \approx 6$ at 473 K. These values are reasonably consistent with the reciprocal of the values of $m$ under the same testing temperatures as shown in Fig. 3(b).

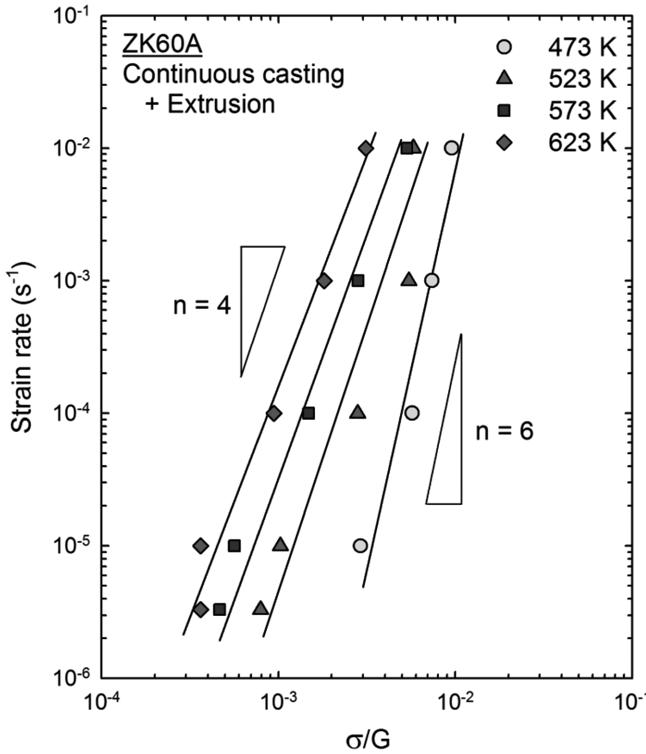


Fig. 4. A logarithmic plot of strain rate vs. flow stress for the ZK60A alloy tested at 473-623 K

It is not feasible in the present investigation to estimate the value of $p$ because all tests were conducted using samples having the consistent grain size. However, it is possible to estimate the value of the activation energy by plotting, in a semi-logarithmic form, the strain rate measured at a constant value of the normalized flow stress, $\sigma/G$, against the reciprocal of the absolute temperature, $1/T$. The shear modulus of magnesium was estimated from the following relationship [9]:

$$G = 1.66\times10^4\left\{1-0.49\left(\frac{T-300}{924}\right)\right\} \text{ [MPa]} \quad (2)$$

The result is shown in Fig. 5 with taking a constant value of $\sigma/G = 3.0\times10^{-3}$ so that the slope of the data leads to an estimated activation energy for creep of $Q \approx 94$ kJ/mol for the ZK60A under the testing conditions.

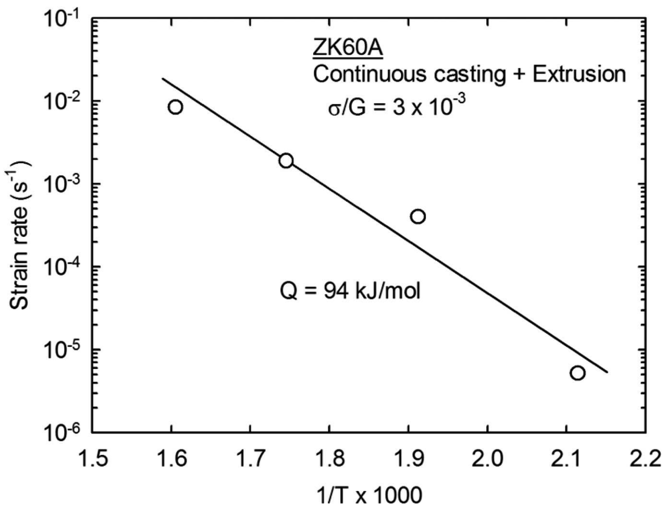



Fig. 5. Strain rate at a constant normalized flow stress vs. the reciprocal of the absolute testing temperature for estimation of the activation energy for the ZK60A alloy

This measured activation energy is significantly lower than the value of ~135 kJ/mol for self-diffusion in pure magnesium but it is in excellent agreement with the anticipated activation energy of ~92 kJ/mol for grain boundary diffusion in magnesium [9]. Nevertheless, it is unlikely that the flow of the ZK60A alloy is controlled by grain boundary diffusion because the grains in the alloy are too large to attain conventional superplastic flow and as shown in Fig. 3(b) the measured strain rate sensitivity of $m \approx 0.3$-$0.4$ is consistent with a dislocation viscous glide process rather than true superplasticity. Several solid solution alloys demonstrated $n \approx 3$ resulted in the viscous drag process controlling dislocation glide [10-13]. However, the achieved elongations to failure of ~200-250% at $1.0\times10^{-5}$-$1.0\times10^{-3}$ s$^{-1}$ is relatively high in magnesium alloys and the samples demonstrate reasonably high values of $m$ in the present testing temperature range. Thus, the level of ductility attained in the ZK60A is attributed to the advent of a quasi-superplastic type of flow and this conclusion is anticipated by the measured activation energy which is consistent with that for grain boundary diffusion.

## 4. Summary and conclusions

1. Experiments were conducted to determine the mechanical properties of a ZK60A magnesium alloy after continuous casting and subsequent extrusion. Samples were tested to failure in tension at temperatures from 473 to 623K and using initial strain rates in a range from $3.0\times10^{-6}$ to $1.0\times10^{-2}$ s$^{-1}$.
2. The alloy exhibited quasi-superplastic flow with the maximum elongation of 250% at a temperature of 523K with an initial strain rate of $1.0\times10^{-5}$ s$^{-1}$. In practice, higher testing temperatures of 573 and 623 K led to reasonably higher elongations to failure at faster strain rates up to $1.0\times10^{-3}$ s$^{-1}$.
3. The strain rate sensitivity was measured as $m \approx 0.3$-$0.4$ at strain rates of $1.0\times10^{-5}$-$1.0\times10^{-3}$ s$^{-}$1 and the activation energy for deformation flow was estimated as ~94 kJ/mol. Due to the large initial grain sizes, these results indicate the quasi-superplastic flow of the ZK60A alloy is controlled by a dislocation viscous glide mechanism.

**Acknowledgements**

This research was supported by the Basic Science Research Program through the National Research Foundation of Korea (NRF) funded by the Ministry of Science, ICT & Future Planning (MSIP) of Korean government (Grant No. 2012R1A1A1012983).

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*Received: 20 November 2014.*