Original Article

Superplasticity of a fine-grained Mg–9Gd–4Y–0.4Zr alloy evaluated using shear punch testing

Reza Alizadeh a, Reza Mahmudi a,∗, Terence G. Langdon b, c

a School of Metallurgical and Materials Engineering, University of Tehran, Tehran, Iran
b Materials Research Group, Faculty of Engineering and the Environment, University of Southampton, Southampton, United Kingdom
c Departments of Aerospace & Mechanical Engineering and Materials Science, University of Southern California, Los Angeles, United States

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ABSTRACT

The superplasticity of an extruded fine-grained Mg–9Gd–4Y–0.4Zr alloy was investigated by measuring the strain rate sensitivity using shear punch testing (SPT). Shear punch tests were conducted at shear strain rates in the range of \(3 \times 10^{-3} - 2 \times 10^{-1} \text{s}^{-1}\) and at temperatures in the range of 573–773 K. The results indicate the strain rate sensitivity, \(m\), increases from about 0.11 at 573 K to about 0.40 at 723 K and then decreases to 0.32 with a further increase in test temperature. A strain rate sensitivity of 0.40 and an activation energy of 140 kJ/mol are indicative of a superplastic deformation behavior dominated by grain boundary sliding accommodated by lattice diffusion at temperatures above 673 K.

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1. Introduction

Superplastic forming, which relies primarily on the small size of the grains, has been applied to shape magnesium alloys into complex geometries [1]. Nevertheless, Mg alloys generally suffer from poor room temperature formability because of the limited slip systems in their hexagonal close-packed (hcp) structure [2]. Furthermore, high deformation temperatures are generally unsuitable because of the unstable microstructure of fine grains, which is significantly affected by grain growth. In practice, different alloying elements have been added to Mg alloys to improve their thermal stability. For example, it was reported that the addition of gadolinium (Gd) and other rare earth elements remarkably improves the high temperature mechanical properties of magnesium alloys due to solution and precipitation hardening [3]. An excellent resistance to grain growth up to 573 K was reported for an Mg–10Gd alloy processed by high pressure torsion (HPT) [4] and in another investigation superplasticity was reported in an extruded Mg–Gd–Y–Zr alloy when testing at 723 K [5].

While most of the reports on superplasticity have used conventional tensile testing, the possibility of using more localized tests such as indentation [6], impression [7] and shear punch testing (SPT) [8] have been reported in cases where the material is only available in small amounts. In SPT, a full description of which is given elsewhere [9], the sample is clamped between two die halves and a flat cylindrical punch is driven through it. The fundamental mechanical properties, such as the shear yield stress (SYS), the ultimate shear

∗ Corresponding author.
E-mail: mahmudi@ut.ac.ir (R. Mahmudi).
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strength (USS) and the strain rate sensitivity (SRS), are readily obtained from the SPT data. Accordingly, it was the objective of this research to examine the superplasticity of an extruded Mg–Gd–Y–Zr alloy by measuring the values of the strain rate sensitivity using SPT.

2. **Experimental material and procedures**

The experiments were conducted using an Mg–9 wt.% Gd–4 wt.% Y–0.4 wt.% Zr alloy. The material was prepared from high purity Mg and Mg–30Gd, Mg–30Y and Mg–30Zr master alloys, which were melted in an electric furnace under a covering flux. Extrusion was carried out with an extrusion ratio of 19:1 at 673 K. One millimeter thick slices were then cut from the extruded bars perpendicular to the extrusion direction and these slices were ground to a thickness of about 0.7 mm. Using a Leitz optical microscope, the structures of the materials were revealed and the grain sizes and grain size distributions were measured. At least 1000 grains were considered and the grain size distributions and weighted average grain diameters were obtained using the Clemex professional image analysis program according to the ASTM E112 standard.

Shear punch tests were performed on 0.7 mm thick sample. The shear strain rates ($\dot{\gamma}$) were calculated using the following equation:

$$\dot{\gamma} = \frac{1}{2} \frac{Z}{W}$$

where $Z$ is the punch–displacement rate and $W$ is the die–punch clearance. The shear behavior of the material was investigated at different strain rates in the range of $3 \times 10^{-3}$–$2 \times 10^{-1}$ s$^{-1}$ and temperatures in the range of 573–773 K using a screw-driven MTS machine. The testing involved a shear punch fixture with a 3.175 mm diameter flat cylindrical punch and 3.225 mm diameter receiving-hole. After application of the load, the load $P$ was measured automatically as a function of the punch displacement. The data was recorded by computer in order to determine the shear stress from the relationship

$$r = \frac{P}{adt}$$

where $t$ is the specimen thickness and $d$ is the average of the punch and die hole diameters. The SPT curves were then plotted as shear stress against the normalized punch displacement.

3. **Results and discussion**

Fig. 1a shows the microstructure of the alloy in the as-extruded condition. It is apparent that the microstructure consists of equiaxed grains, implying that dynamic recrystallization has occurred during the hot extrusion process. In fine-grained materials, a uniform grain size distribution is important in achieving homogeneous mechanical properties. The grain size distribution data, collected from a number of samples, are shown in Fig. 1b. It is clear that a near-normal distribution has been achieved with an average grain size of about 6.5 µm.

Fig. 1 – (a) Optical micrograph and (b) grain size distribution of the tested alloy.

This grain size is slightly smaller than the 10 µm reported by Zhang et al. [5] who used lower extrusion ratios.

The SPT curves of the material at 723 K, obtained at different shear strain rates in the range of $3.3 \times 10^{-3}$–$1.3 \times 10^{-1}$ s$^{-1}$, are shown in Fig. 2. It can be seen that the curves consist of an elastic linear part, yielding, a work hardening region, ultimate shear strength (USS) and fracture. Also, the curves show that the strength of the material increases with increasing strain.

Fig. 2 – SPT curves of the material at different shear strain rates obtained at 723 K.
rate, indicative of a positive strain rate sensitivity index of the material.

The high-temperature tensile flow stress (σ) of materials can be related to the tensile strain rate (\(\dot{\varepsilon}\)) by a power-law relationship [10]:

\[
\frac{\ddot{\varepsilon}}{G} = \left( \frac{A b}{k} \right) \left( \frac{b}{d} \right)^p \left( \frac{\dot{\varepsilon}}{\dot{\varepsilon}_0} \right)^{1/m} \exp \left( \frac{-Q}{R T} \right)
\]  

(3)

where \(A\) is a material parameter, \(b\) is the Burgers vector, \(k\) is the Boltzmann constant, \(d\) is the grain size, \(p\) is the inverse grain size exponent, \(G\) is the shear modulus, \(m\) denotes the SRS index, \(Q\) is the deformation activation energy, \(R\) is the universal gas constant and \(T\) is the absolute temperature. This equation can be simply modified for evaluating superplastic behavior in the SPT method by replacing \(\dot{\varepsilon}\) with \(\dot{\varepsilon}\) and \(\dot{\varepsilon}_0\) with \(\dot{\varepsilon}\). This may be performed using the Von-Mises yield criterion for a state of pure shear of kinematically hardening materials, which gives \(\sigma = \sqrt{3} \varepsilon\) and \(\dot{e} = (1/\sqrt{3}) \dot{\varepsilon}\). Therefore, a modified form of Eq. (3) can be rewritten as:

\[
\frac{\ddot{\varepsilon}}{G} = \left( \frac{A' b}{k} \right) \left( \frac{b}{d} \right)^p \left( \frac{\dot{\varepsilon}}{\dot{\varepsilon}_0} \right)^{1/m} \exp \left( \frac{-Q}{R T} \right)
\]  

(4)

where \(A'\) is a material constant. Due to the constancy of \(Q\) at a given temperature, it is possible to determine the SRS index \(m\) from the relationship:

\[
m = \left( \frac{\partial \ln(\ddot{\varepsilon}/G)}{\partial \ln(\dot{\varepsilon}/G)} \right)_T
\]  

(5)

Fig. 3 shows the variations of normalized USS with the temperature compensated shear strain rate on a double logarithmic scale. The USS and shear strain rate values are normalized to the shear modulus to eliminate the changes in shear modulus with temperature. According to Eq. (5), the slope of the curves gives the corresponding SRS index, \(m\). It is apparent that the dependency of USS on strain rate is linear at 573 K with an \(m\) value of 0.11. However, the dependency becomes sigmoidal in shape with three distinct regions at higher temperatures. The \(m\) values were measured for the middle region, which shows the maximum slope. The results obtained indicate that the \(m\) value increases with increasing temperature from 0.22 at 623 K to 0.40 at 723 K and then decreases to 0.32 with a further increase in temperature. To obtain a better representation of data, the variation of \(m\) with test temperature was plotted in Fig. 4. It is now clear that the maximum strain rate sensitivity is achieved at 723 K.

The observed decrease in \(m\) value at 773 K can be related to grain growth at this temperature, as shown in Fig. 5. The average grain size after testing at this temperature is about 46 \(\mu\)m, which is significantly greater than the initial grain size of the extruded material. Although the \(m\) values are different, the observed trend in the variation of \(m\) with test temperature is the same as in the tensile test results obtained by Zhang et al. [5]. The difference may arise from different extrusion conditions and thus the initial microstructure and grain size of the samples.

According to Eq. (4), the deformation activation energy can be calculated at constant temperature-compensated shear strain rate as:

\[
Q = R \left[ \frac{1}{m} \frac{\partial \ln(\ddot{\varepsilon}/G)}{\partial (1/T)} + P \frac{\partial \ln(b/d)}{\partial (1/T)} \right]_{T/G} = Q_1 + Q_2
\]  

(6)

It is clear from this equation that the activation energy of superplastic materials, where the inverse grain size exponent is not zero and is usually considered as \(P = 2\) [11], consists from...
two separate components; $Q_1$ and $Q_2$, which correspond to the activation energies resulting from the variations of stress and grains size with temperature, respectively. Accordingly, in order to calculate the activation energy according to Eq. (6), the normalized USS and $(b/d)$ values are plotted against the reciprocal of temperature at constant temperature–compensated shear strain rates on a semi-logarithmic scale in Fig. 6. Calculations were made for region II of the sigmoidal curves in the temperature range of 673–773 K, in which the material shows superplastic flow. The average $Q_1$ and $Q_2$ values were found to be $140 \pm 5$ and $143 \pm 5$ kJ/mol, respectively. Thus, it should be noted that $Q_1$ and $Q_2$ make different contributions to the overall activation energy of superplastic materials. Specifically, $Q_1$ is related to the extent of the variations of strength of the material with temperature and thus the deformation mechanism. On the other hand, $Q_2$ reflects the severity of grain growth and thus is it related to the grain growth kinetics of the material at high temperatures, this will be discussed in more detail in future work.

To investigate the deformation mechanism of the material in the superplastic region, it is necessary to consider both the activation energy and the strain rate sensitivity. It is anticipated that the $m$ value of about 0.50 is associated with grain boundary sliding (GBS) controlled by either lattice or grain boundary diffusion [11]. Since the activation energy of deformation ($Q_1 = 140 \pm 5$ kJ/mol) is close to the magnesium lattice diffusion value of 135 kJ/mol [12], it can be concluded that GBS accommodated by lattice diffusion is the dominant deformation mechanism of the material in the superplastic region. This conclusion seems reasonable considering the fine grain microstructure of the material and the sigmoidal dependency of SRS on the shear strain rate.

4. Summary and conclusions

1. The strain rate sensitivity of the extruded Mg–9Gd–4Y–0.4Zr alloy was investigated in the temperature range of 573–773 K by SPT.
2. The $m$ value increased from about 0.11 at 573 K to about 0.40 at 723 K and then decreased to 0.32 with a further increase in test temperature.
3. According to the measured activation energy and $m$ values, lattice diffusion accommodated GBS can be considered as the dominant deformation mechanism of the material in the superplastic region.

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Conflicts of interest

The authors declare no conflicts of interest.

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