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Superplasticity of a Fine-grained Mg–1.5 wt% Gd Alloy after Severe Plastic Deformation

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Abstract: The strain rate sensitivity (SRS) of Mg–1.5 wt% Gd processed by conventional extrusion and 2 passes of simple shear extrusion (SSE) was investigated by shear punch testing. Shear punch tests were conducted at initial shear strain rates in the range of 0.003–0.3 s^{-1} and at temperatures in the range of 573–773 K. A fine-grained microstructure with an average grain size of about 2.5 µm, obtained after 2 passes of SSE, resulted in high SRS index (*m*-value) of 0.4 at 723 K. The calculated activation energy for 2 passes deformed alloy is 116 kJ/mol which is higher than the activation energy of grain boundary diffusion in magnesium (75 kJ/mol). This higher amount of activation energy can be attributed to the presence of gadolinium in this alloy. This SRS index together with an activation energy of 116 kJ/mol are indicative of a superplastic deformation behavior dominated by grain boundary sliding accommodated by grain boundary diffusion at 723 K.

Keywords: Severe plastic deformation, Simple Shear Extrusion, Superplasticity, Shear punch test.

1. Introduction

Superplasticity is the ability of certain materials to achieve large elongations of more than several hundred percent without necking prior to failure. This feature generally exists in materials with a stable, equiaxed and fine microstructure [1, 2]. The results of previous investigations on superplastic materials have shown that the strain rate sensitivity (SRS) index values of these materials generally lie in the range of 0.4–0.8 [3]. It is generally accepted that superplastic flow is a diffusion-controlled process, which needs high testing temperatures above ~ 0.5 T_m [1]. It is also well known that severe plastic deformation (SPD) techniques such as equal channel angular pressing (ECAP), high pressure torsion (HPT) and simple shear extrusion (SSE) can result in significant grain refinement in metallic materials. The reported results show that the fine- and/or ultrafine-grained structures obtained by these deformation routes can provide superplastic behavior [4].

Fine-grained magnesium alloys normally exhibit poor thermal stability. Therefore, high deformation temperatures can lead to severe grain growth in the microstructure of these alloys [5]. This drawback can, however, be addressed by the addition of different alloying elements, which are capable of forming thermally stable second phase particles. One of these elements is gadolinium (Gd), in which its beneficial effects on improving the high temperature mechanical properties of magnesium alloys have already been demonstrated [6]. Over the recent years, there has been a considerable interest in evaluating the microstructure and mechanical properties of cast and wrought Mg–Gd alloys [7–9]. These investigations have shown that the addition of Gd affects the recrystallization phenomenon, resulting in a microstructure that is very different from those of other wrought Mg alloys. It is also reported that Mg–Gd alloys show very good ductility even at low temperatures [10, 11]. Stanford et al. [12–14] evaluated different aspects

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of Mg–Gd binary alloys having different amounts of Gd. They reported that Mg–1.5 wt% Gd alloy shows the maximum ductility changes among the binary Mg–Gd alloys, in comparison with pure Mg.

Tensile test is the most common method to assess the superplastic behavior of materials [15, 16]. In most of the SPD methods, the samples are small and in some cases the material is only available in small amounts. Hence, considering the possibility of using localized tests for evaluating the superplastic behavior of materials is of some interest. There are some reports on using localized tests such as indentation [17], impression [18] and shear punch testing (SPT) [19,20] to investigate the superplastic behavior of metals. The full description of SPT method and its applicability to Mg alloys has been described elsewhere [21]. The fundamental mechanical properties such as shear yield stress (SYS), ultimate shear strength (USS), and strain rate sensitivity (SRS) can readily be obtained from the SPT data.

The novel SPD technique of SSE was first proposed by Pardis and Ebrahimi in 2009. The details of the testing arrangement and die design are explained elsewhere [22] and is only briefly described here. SSE is based on pressing the specimen through a direct channel with a specific shape. During the SSE process, the specimen is deformed gradually while its cross-section area remains constant. The extent of plastic deformation per pass of SSE depends on the maximum distortion angle (α) in the middle of the channel and the level of applied back-pressure. The theoretical strain is equal to 1.15 in each pass of the SSE process with $\alpha = 45^\circ$ [22]. Although the improvement in the room-temperature mechanical properties of pure Mg after SSE has already been considered, the superplastic behavior of SSEed Mg alloys has not been studied before. Therefore, it is the aim of this research to examine the superplasticity of extruded and SSEed Mg–1.5 wt% Gd by measuring the values of the strain rate sensitivity index using SPT.

2. Experimental procedure

The material used in this study was an Mg–1.5 wt% Gd alloy. High purity Mg (99.95%) and an Mg–30%Gd master alloy were employed to prepare the material. These were melted in a graphite crucible placed in an electrical furnace, under the Foseco Magrex 36 covering flux to protect magnesium from oxidation. The cast billets of 42 mm diameter and 120 mm length were homogenized at 733 K for 6 h and extruded to 11 mm × 11 mm bars at 653 K. The extrusion ratio was about 11.5 and the ram velocity was 5 mm min^{-1}. The SSE billets having nominal dimensions of 10 mm × 10 mm × 30 mm were machined from the extruded bars. All samples were pressed at 553 K using an SSE die having a maximum distortion angle of $\alpha = 45^o$. Repetitive pressing of the same sample was carried out with route C, in which the specimen is rotated 90^o about the extrusion axis between consecutive passes. This route was applied because it has been demonstrated to be the most effective one in achieving the homogenous refined microstructures [23]. Samples were wrapped with PTFE tape to reduce friction, before being pressed at a speed of 6 mm min^{-1} for 2 passes.

One millimeter thick slices were cut from the extruded and SSEed bars perpendicular to the extrusion direction and ground to a thickness of about 0.7 mm. A Leitz optical microscope was used for microstructural examinations of the cross-sections parallel to the pressing direction of the extruded and SSEed billets. For optical observations, the specimens were polished with 0.3 μm alumina paste, followed by polishing on an abrasive-free micro-cloth. Etching was carried out using Nital 5% solution at room temperature. The grain size distribution of SSEed samples was measured using the Clemex professional image analysis program according to the ASTM E112 standard.

Shear punch were performed on 0.7 mm thick samples in the temperature range of 573–773 K and shear strain rate range of 0.003–0.3 s^{-1}. During the test, the load P was measured automatically as a function of the punch displacement. In order to calculate the shear strain rate ($\dot{\gamma}$) and shear stress (τ), the following equations were used

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

$$\tau = \frac{P}{\pi d t} \quad (2)$$

where $\dot{Z}$ is the punch-displacement rate, W is the die-punch clearance, t is the specimen thickness and d is the average diameter of the punch and die hole. It should be noted that the testing involved a shear punch fixture with a 3.175 mm diameter flat cylindrical punch and 3.225 mm diameter receiving-hole.

3. Results and Discussion

Figure 1 shows the microstructure of the tested alloy in as-cast and extruded conditions. The microstructure of the as-cast alloy consists of very large columnar grains, implying that the addition of Gd has no significant grain refinement effect in the as-cast condition, for which a grain size of about 1000 μm is obtained after Gd addition. In contrast, the microstructure of the deformed material shows contains some patches of unrecrystallized grains surrounded by very fine recrystallized grains with an approximate grain size of about 3.5 μm. The unrecrystallized grains have appeared as well-defined coarse patches, which are aligned almost parallel to the extrusion direction. The large size mismatch between Gd and Mg atoms results in segregating Gd atoms around grain boundaries in the Mg–Gd alloys. Therefore, there is a great solute drag effect on grain boundary motion in these alloys, which can retard the recrystallization phenomenon [24]. This kind of microstructure has also been reported in other researches on Mg–Gd binary alloys [12, 25].

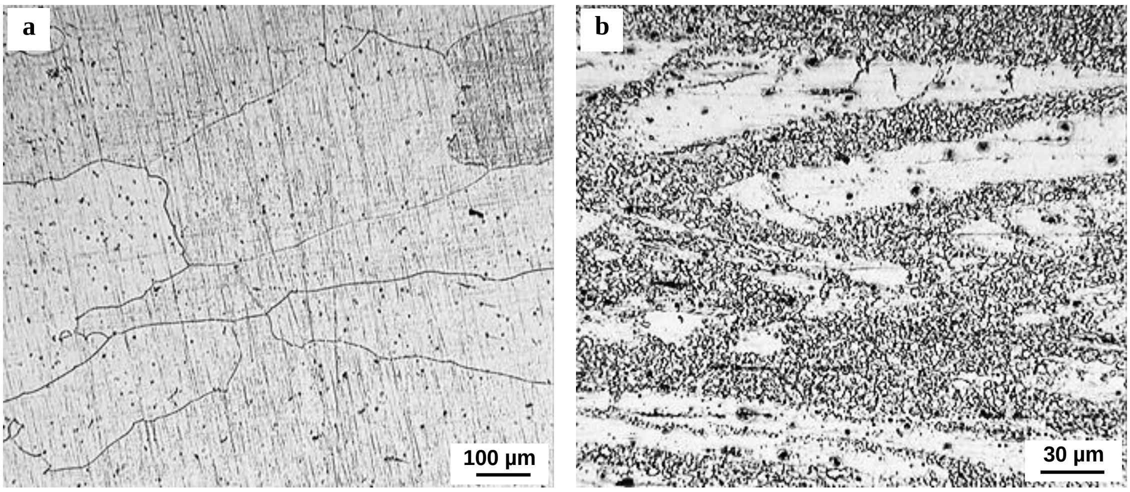

Fig. 1. Optical micrographs of (a) as-cast, and (b) extruded condition, parallel to the extrusion direction.

The microstructure of the material after 2 passes of SSE is depicted in Fig. 2a. It is evident that straining at high temperature has decreased the volume fraction of unrecrystallized patches and increased the degree of recrystallization in deformed samples. The grain size distribution data, collected from a number of samples, are shown in Fig. 2b. It is clear that a near-normal distribution has been achieved with an average grain size of about 2.5 μm and a low standard deviation of 0.8 μm after 2 SSE passes. It is believed that this kind of uniform grain size distribution is important in achieving homogeneous mechanical properties in the deformed samples.

Typical SPT curves of the material, plotted as shear stress (τ) against normalized punch displacement ($\delta = h/t$, where h is the displacement), are depicted in both extruded condition and after 2 SSE passes at different shear strain rates and 723 K in Fig. 3. All the curves consist of an elastic linear part, yielding, a work hardening region, ultimate shear strength (USS), and fracture. It can be observed that the strength of the material improved with increasing the strain rate in both conditions. This trend indicates a positive strain rate sensitivity index of Mg–1.5Gd. The obtained results also show that despite having a more uniform and refined microstructure, the USS of SSEed material is much lower than that of extruded condition at a given constant shear strain rate. For instance, the USS of SSEed sample is about 20% lower than that of the extruded material at 1.7×10^{-2} s^{-1}. This behavior is one of the main characteristics of superplastic materials [3, 20].

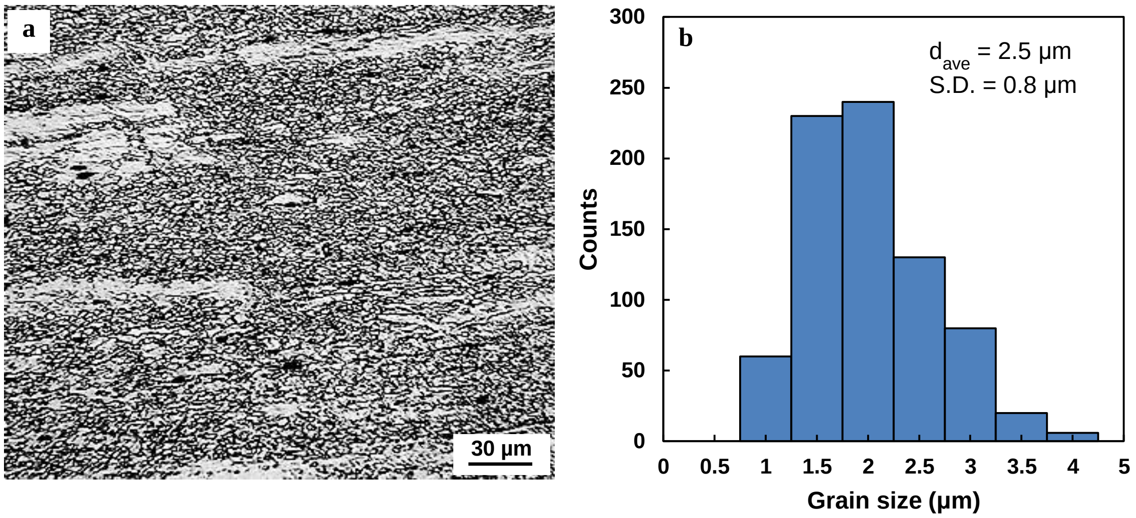

Fig. 2. (a) Optical micrograph, and (b) size distribution of the recrystallized grains after 2 passes of SSE on the sections parallel to the extrusion direction.

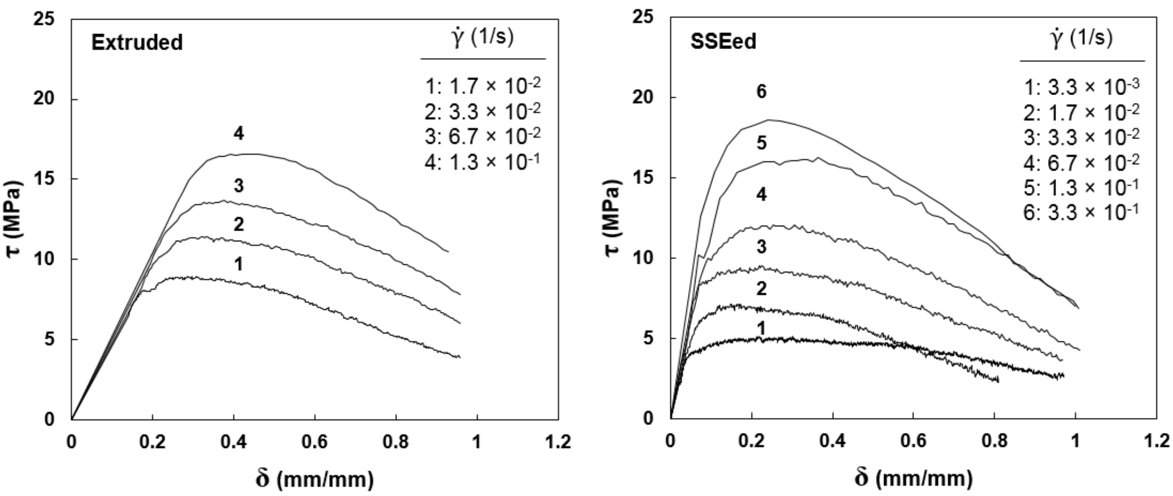

Fig. 3. SPT curves of the material at different strain rates obtained at 723 K.

In general, the constitutive equation to describe the relationship between high temperature tensile flow stress (σ) and strain rate ($\dot{\varepsilon}$) can be expressed by a power-law equation as below [25]

$$\dot{\varepsilon} = \frac{AGb}{kT}\left(\frac{b}{d}\right)^{p}\left(\frac{\sigma}{G}\right)^{1/m} D_0 \exp\left(-Q/_{RT}\right) \quad (3)$$

where *A* is a dimensionless constant, *G* is the shear modulus, *b* is the burgers vector, *d* is the grain size, *k* is Boltzmann's constant, *p* is the inverse grain size exponent, D_0 is the pre-exponential factor for diffusion, *R* is the universal gas constant, *T* is the absolute temperature and *Q* is the activation energy for the deformation, which depends on the rate controlling process. This equation can be simply modified to evaluate high temperature behavior in the SPT method by replacing σ and $\dot{\varepsilon}$ with τ and $\dot{\gamma}$, respectively. In this regard, the Von-Mises yield criterion for the state of pure shear of kinematically hardening materials, which gives $\sigma = \sqrt{3}\tau$ and $\varepsilon = \left(\frac{1}{\sqrt{3}}\right)\gamma$, can be used. Therefore, the modified form of Eq. (3) can be rewritten in the form of

$$\left(\frac{\dot{\gamma}T}{G}\right) = \left(\frac{A' D_0 b}{k}\right)\left(\frac{b}{d}\right)^p \left(\frac{\tau}{G}\right)^{1/m} \exp\left(-Q/_{RT}\right) \tag{4}$$

where A' is a material constant. Due to the constancy of *Q* at a given temperature, the SRS index (*m*) can be obtained from the following relationship

$$m = \left(\frac{\partial \ln(\tau/G)}{\partial \ln(\dot{\gamma}T/G)}\right)_T \tag{5}$$

It should be noted that the temperature dependence of shear modulus for magnesium can be estimated from the following equation [26]

$$G = (1.92 \times 10^4 - 8.6T)\ \text{MPa} \tag{6}$$

where *T* is the testing temperature in Kelvin.

The variations of normalized USS with the temperature-compensated shear strain rate is plotted on a double-logarithmic scale for the extruded and SSEed alloy in Fig. 4. 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 about 0.08 in both conditions. This low *m*-value may be attributed to the grain boundary and dislocation segregation and low diffusivity of the RE atoms such as Gd in Mg [14, 24]. It is clear that increasing the testing temperatures up to 723 K results in enhancement of *m*- values in all tested samples. It is obvious that the dependency of USS on strain rate become sigmoidal at 723 K in the samples after two SSE passes. The results of the SSEed samples indicate that the *m*-value increases with increasing temperature from 0.23 at 623 K to 0.40 at 723 K and then decreases to 0.25 with a further increase in temperature. The more uniform microstructure of the SSEed material led to higher amount of *m*-values compared with that of the extruded material.

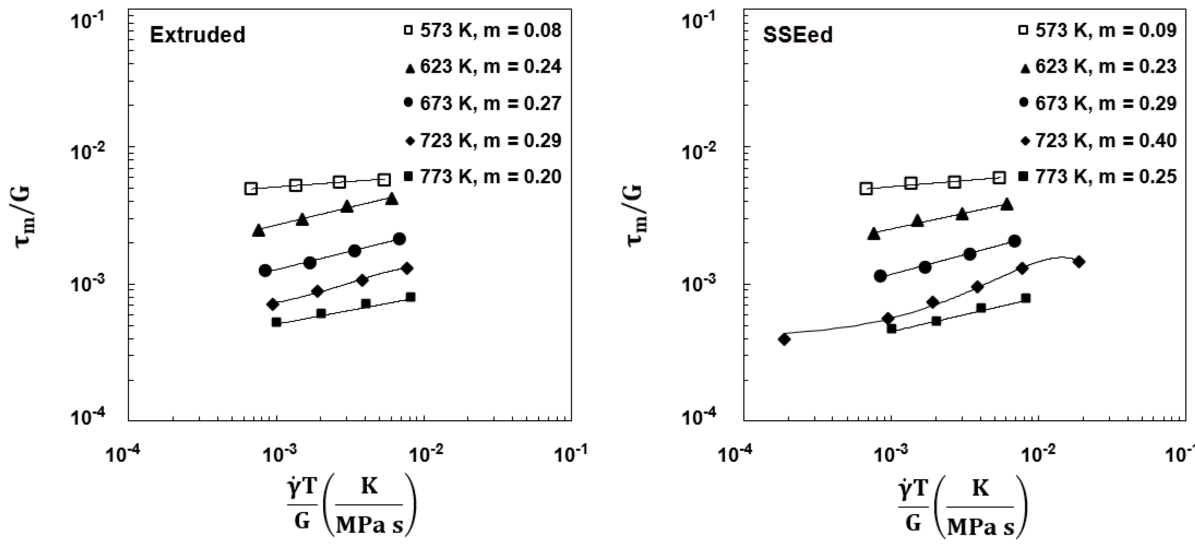

Fig. 4. Normalized USS of the material as a function of temperature-compensated shear strain rate in extruded condition and after 2 SSE passes.

Figure 5 shows the variation of *m*-values with test temperature for the SSEed material. This curve clearly shows that the maximum strain rate sensitivity of the SSEed material is achieved at 723 K. The drop in the *m*-value at the highest test temperature of 773 K can be related to the grain growth phenomenon at this temperature. This is shown in Fig. 6, where the microstructures of the SSEed alloy are compared after SPT at 723 and 773 K. The average grain size after testing at 773 K is about 46 µm, which is significantly greater than the grain size of 20 µm after testing at 723 K.

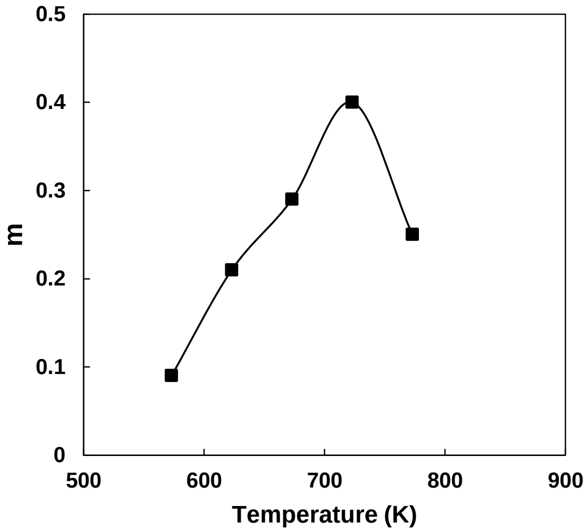

Fig. 5. The variation of m value with test temperature of SSEed material.

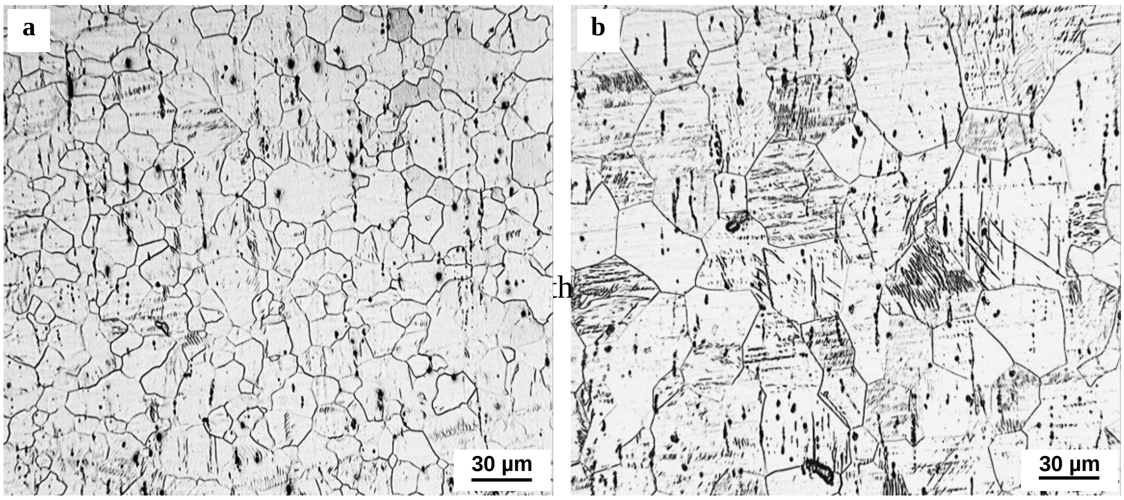

Fig. 6. Optical micrographs of SSEed material after SPT at (a) 723, and (b) 773 K.

The difference in the strain-rate sensitivity is attributed to different predominant mechanisms, which may operate at a constant temperature. In order to have a better understanding of the deformation mechanism, determination of the activation energy (*Q*) could be useful. According to Eq. (4), the activation energy can be calculated at a constant temperature-compensated shear strain rate as

$$Q = R\left[\frac{1}{m}\frac{\partial \ln(\tau/G)}{\partial \ln(1/T)} + p\frac{\partial \ln(b/d)}{\partial \ln(1/T)}\right]_{\dot{\gamma}T/G} = Q_1 + Q_2 \tag{7}$$

where Q_1 and Q_2 correspond the activation energies resulting from the variations of stress and grains size with temperature, respectively. It has been reported that in hot deformation of materials with large grain size, there is no dependence on grain size, and thus, p = 0. However, the inverse grain size exponent is not zero and is usually considered as p = 2 for superplastic materials [28]. It should be noted that the contributions of Q_1 and Q_2 to the overall activation energy of superplastic materials are different. As it mentioned before, 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 intensity of grain growth and thus is related to the grain growth kinetics. Therefore, in the following the variations of Q_1 will be discussed.

The variations of normalized USS with temperature at constant temperature-compensated shear strain rates are plotted in Fig. 7. The calculated value of Q_1 parameter, determines the controlling diffusion mechanism during the deformation. As can be seen, the activation energy of deformation, Q_1, decreased from 180 kJ/mol in the extruded condition to 116 kJ/mol after 2 SSE passes. In comparison with the activation energy of grain boundary diffusion in magnesium (75 kJ/mol), the higher value of Q_1 may be attributed to the presence of gadolinium in these alloys [29,30]. It has been suggested that the *m*-value of about 0.50 is associated with grain boundary sliding (GBS) controlled by either lattice or grain boundary diffusion [28]. Based on the *m*-value of 0.4 and Q_1 of 116 kJ/mol, it is suggested that the observed superplastic behavior in the Mg–1.5Gd alloy at 723 K after processing by 2 passes of SSE takes place with the deformation mechanism of GBS controlled by grain boundary diffusion.

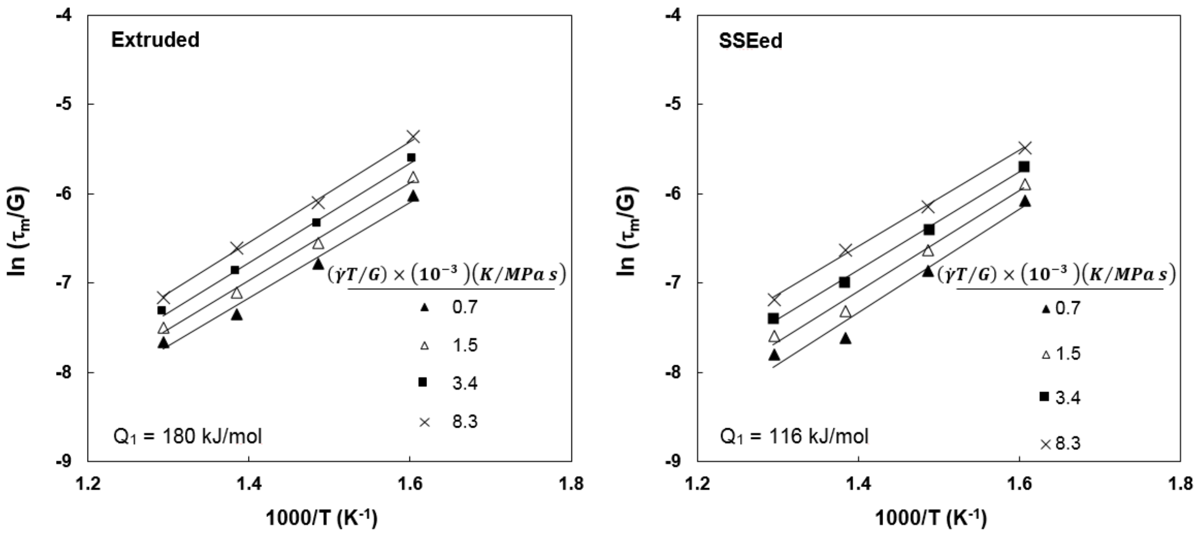

Fig. 7. Temperature dependence of normalized τ_m values at constant temperature-compensated shear strain rates.

4. Conclusion

The strain rate sensitivity of the extruded and SSEed Mg–1.5Gd alloy were investigated in the temperature range of 573–773 K by SPT. The following conclusions are drawn from the present research work:

1- In the extruded condition, the *m*-value increased from about 0.08 at 573 K to about 0.29 at 723 K and then decreased to 0.2 with a further increase in the test temperature.

2- The uniform and fine microstructure of the SSEed samples increased the *m*-value to about 0.4 at 723 K.

3- According to the measured activation energy and *m*-value, grain boundary diffusion accommodated GBS can be considered as the dominant deformation mechanism of the material in the superplastic region.

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سوپرپلاستیسیته آلیاژ ریز دانه Mg-1.5Gd پس از تغییر شکل شدید پلاستیک

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چکیده: میزان حساسیت به نرخ کرنش آلیاژ دوتایی Mg-1.5 wt% Gd فرآوری شده به روش اکستروژن و پس از دو مرحله فرآیند اکستروژن برشی ساده (SSE) مورد بررسی قرار گرفت. برای این منظور، از روش موضعی آزمون پانچ برشی در محدوده‌ی نرخ کرنش اولیه‌ی $0.003\text{-}0.3\ s^{-1}$ و محدوده‌ی دمایی 573-773 K استفاده شد. بررسی‌های ریز ساختاری نشان داد که آلیاژ مورد مطالعه پس از دو مرحله تغییر شکل به روش SSE، حاوی دانه‌های ریز با اندازه‌ای حدود 2.5 μm است. همچنین ساختار ریزدانه‌ی این آلیاژ منجر به دستیابی به ضریب حساسیت به نرخ کرنش بالایی در حدود 0.4 در دمای 723 K شد. به منظور ارزیابی دقیق‌تر مکانیزم حاکم بر تغییر شکل در آلیاژ Mg-1.5Gd، انرژی فعال سازی لازم برای تغییر شکل محاسبه شد. میزان انرژی فعال سازی برای آلیاژ فرآوری شده به روش SSE معادل 116 kJ/mol بوده که از میزان گزارش شده برای نفوذ مرزدانه‌ای در منیزیم بالاتر است. بالاتر بودن انرژی فعال سازی می‌تواند ناشی از حضور عنصر گادولینیم در ساختار آلیاژ مورد بررسی باشد. میزان ضریب حساسیت به نرخ کرنش معادل 0.4 به همراه انرژی فعال سازی در حدود 116 kJ/mol می‌تواند رفتار سوپرپلاستیک با مکانیزم لغزش مرزدانه‌ای کنترل شده با نفوذ مرزدانه‌ای اتم‌های منیزیم را در دمای 723 K پس از دو مرحله تغییر شکل به روش SSE تأیید کند.

کلمات کلیدی: تغییر شکل پلاستیک شدید، اکستروژن برشی ساده، سوپرپلاستیک، آزمون پانچ برشی