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Effect of thermomechanical parameters on dynamically recrystallized grain size of AZ91 magnesium alloy

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ABSTRACT

Dynamic recrystallization of the AZ91 alloy was studied by conducting hot compression tests at temperature range of 325–400 °C and strain rate of 0.001–1 s^{-1}. The influence of the hot deformation variables on flow stress as well as recrystallized grain size was investigated. The results showed that by decreasing temperature and increasing strain rate, flow stress increases while dynamically recrystallized grain size decreases. A power-law relation developed between the characteristic peak strain and Zener–Hollomon parameter and the exponent was determined as 0.17. Besides, the linear regression between the Zener–Hollomon parameter and dynamically recrystallized grain size developed another power law equation, with a stress exponent equivalent to −0.13.

1. Introduction

One of the most important characteristics of Mg alloys is high ratio of strength to weight. This is why there is a high demand on the application of these alloys in the transportation industries to reduce the fuel consumption and to save energy [1,2]. However, due to the high production cost of Mg alloys and relatively poor workability, in comparison to other engineering alloys, their application is more restricted [1–3]. To overcome poor formability of Mg alloys at room temperature, almost all deformation processes actually are performed at high temperatures. On the other hands, hot deformation is usually accompanied by the softening phenomena, i.e. dynamic recovery (DRV) and dynamic recrystallization (DRX). It should be mentioned that the occurrence of softening processes leads to decrease in flow stress and therefore improvement of formability and often to grain refinement. Since the value of stacking fault energy in Mg alloys is relatively high (γ = 125 mJ/m^2) [3], it is expected that during deformation at high temperatures, DRV takes place readily. On the contrary, the dominant restoration phenomenon is apparently DRX [3]. The unexpected occurrence of DRX in Mg alloys is attributed to the lack of easily activated slip systems rather than the high stacking-fault energy [3,4]. It is also interesting that DRX in Mg alloys usually proceeds discontinuously and is associated with the formation of a necklace structure [5]. In fact, during hot deformation, local bulging of grain boundaries leads to the formation of strain free volumes introducing new DRX grains. The driving force for the bulging process is provided from the different stored energies or dislocation densities at both sides of the moving boundary. Dislocation density is a function of flow stress during hot deformation process [4]. Derby showed that an inverse relation between DRX grain size and flow stress exists for a wide range of metals including Mg alloys [6]. Since flow stress is a function of temperature and strain rate, a relation between the DRX grain size and the hot deformation variables can be figured. A number of recent studies have dealt with the hot deformation microstructure of wrought Mg alloys, however there is little data considering the relation between DRX grain size and hot working regime of cast Mg alloys.

This paper is therefore devoted to investigate the influence of thermomechanical parameters on dynamically recrystallized grain size of Mg alloy AZ91 during hot deformation.

2. Methods and materials

An ingot of AZ91 alloy having chemical composition of Al 9.2%, Zn 0.8%, Mn 0.22%, Si 0.077% and balanced Mg was used in this research. Cylindrical specimens 10 and 15 mm respectively in diameter and height were machined for the hot compression test. In order to dissolve β ($Mg_{17}Al_{12}$) precipitates and to homogenize the structure, the samples were held in furnace at 420 °C for 24 h [7]. Fig. 1 shows the microstructure after homogenization process, containing a matrix of coarse and equiaxed α-Mg and distributed small β precipitates at grain boundaries.

Hot compression tests were performed using a Zwick/Roll machine equipped by holding furnace with ±5 °C accuracy, in a temperature range of 325–400 °C under constant ram speed of 0.6–600 mm/min. After soaking for 3 min at test temperature, hot compression was performed up to strain of 0.5. To reduce friction, the

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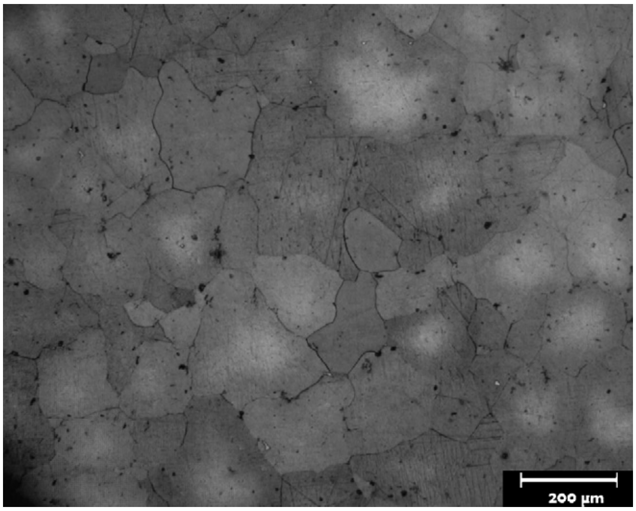

Fig. 1. Microstructure of homogenized specimen used as the starting material.

contact surfaces were wrapped by Teflon layers. After testing, the specimens were water quenched within 5 s to preserve the hot deformation microstructure. The hot deformed specimens were cut parallel to the compression direction and after etching in Acetic-Picral were metallographically examined by optical microscopy as well as Leica Cambridge S360 scanning electron microscope. The average grain size of upper right quarter part of samples was measured by the planimetric (Jeffries') method.

3. Results and discussion

True stress–strain curves of deformed samples at different deformation regimes are shown in Fig. 2. As usual, at a given strain rate, by increasing the strain we have seen that the flow stress firstly increases up to a maximum and then decreases to a steady state. The flow softening beyond the peak stress is attributed to DRX as well as coarsening of β precipitates [8–10]. As expected, peak stress increases with strain rate and decreases with deformation temperature. The influence of strain rate and temperature under hot working conditions is often incorporated in the temperature compensated strain rate parameter which is also known as Zener–Hollomon parameter, given by [11–13]:

$$Z = \dot{\varepsilon} \exp\left(\frac{Q}{RT}\right) = A[\sinh(\alpha\sigma_p)]^n, \tag{1}$$

where σ_p denotes peak stress, n is stress exponent, A and α are material constants. The value for α may be chosen in a way that parallel lines would be obtained from the curve log $\dot{\varepsilon}$ vs. log [sinh($\alpha\sigma$)]. In the present work, the value of α was obtained equal to 0.051 MPa^{-1} to make the best-fit of the experimental data, Fig. 3. The results of Z value vs. peak stress are plotted in Fig. 4. Obtaining a single line for the present experimental data indicates that the chosen value for α fits well with the experimental results. Moreover, the average slope gives the value of n as 2.2, which is in a good agreement with the values of 1.84 reported by Mwembela et al. [14] and 1.73 by Ravi Kumar et al. [10]. It is worth to note that the value of α is identical for all the cases and equal to 0.052; the difference can be attributed to some differences in the alloying contents, the amount of precipitates and the grain orientations [10,15,16].

It is worth mentioning that at a constant strain rate the strain corresponding to the peak stress, ε_p decreases with temperature. It also increases with increasing strain rate at a given deformation temperature. To study the influence of temperature and strain rate on ε_p, its variations with Z parameter were plotted in Fig. 5.

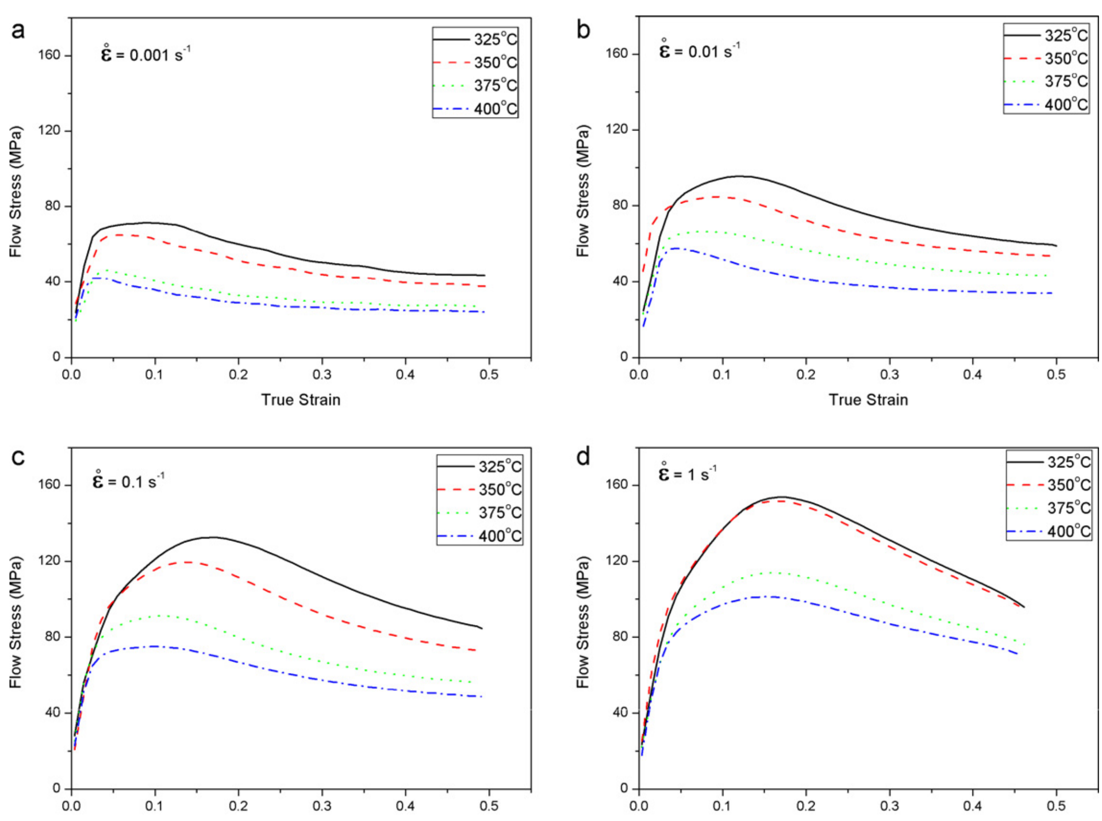

Fig. 2. True stress–strain curves of deformed samples at (A) 0.001 s^{-1}, (B) 0.01 s^{-1}, (C) 0.1 s^{-1} and (D) 1 s^{-1} and at temperature range of 325–400 °C.

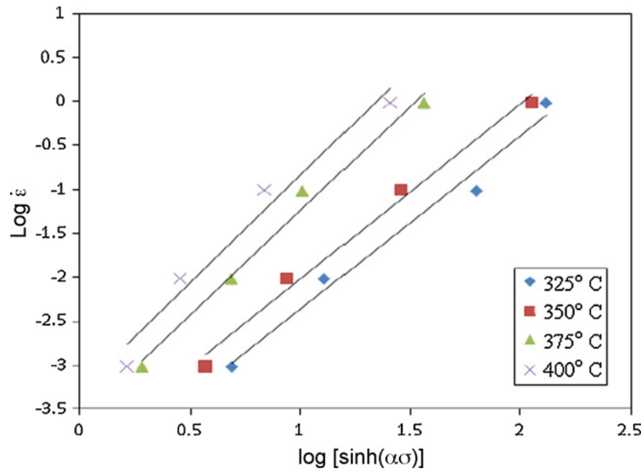

Fig. 3. Relationship between log $\dot{\varepsilon}$ and log [sinh($\alpha\sigma$)] in hot compression tests.

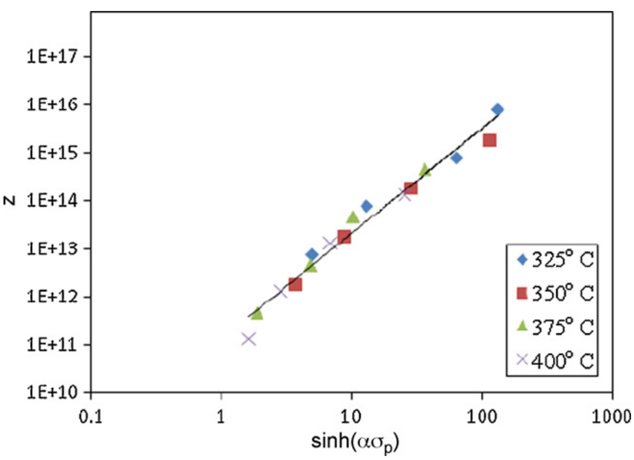

Fig. 4. Relationship between Zener–Hollomon parameter and peak stress in hot compression tests.

According to the results in Fig. 5, the following equation can fit the empirical data:

$$\varepsilon_p = (KZ)^q \tag{2}$$

with q = 0.17; q was reported to be 0.29 by Ravi Kumar et al. in case of using hot torsion test [10]. Some parameters like the amount of fine β precipitates cause an increasing in the q parameter. This means increasing pick strain and therefore an increasing in work hardening. Moreover, some other parameters such as coarse grains and precipitates lead to a decreasing in the q value.

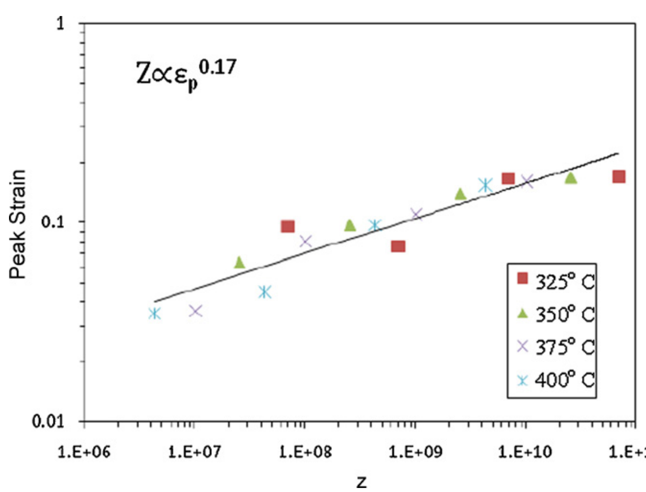

Fig. 5. Relationship between Zener–Hollomon parameter and peak strain in hot compression tests.

Fig. 6 illustrates the microstructure of the specimens hot deformed at 325–400 °C and 0.1 s^{-1}. It can be observed that, compared to the primary microstructure grain size, Fig. 1, the average grain size is significantly decreased. It is also evident that increase in the deformation temperature leads to an increase in the average grain size, Fig. 6A–D.

Microstructures of the specimens hot deformed at 325–400 °C and 0.001 s^{-1} are shown in Fig. 7. It is clearly seen that, compared to Fig. 6 at constant temperature, by decreasing the strain rate, the average grain size decreases. It is well known that the major reason for grain refinement during hot deformation process is the occurrence of dynamic recrystallization [4].

The resulting grain size values after hot deformation are shown in Table 1. Noticeably, a rational relationship between grain size and hot deformation parameters, i.e. temperature and strain rate, can be detected. For this purpose, the dynamically recrystallized grain size was plotted against Zener–Hollomon parameter in Fig. 8. The figure shows that the size of the dynamically recrystallized grains decreases with increasing the value of the Z parameter, i.e. the dislocation density in the deformed sample. On the other hand, it is well known that increase in dislocation density actually influences the nucleation rate rather than the growth rate of recrystallized grains [6,16,17]. The inverse relationship between DRX grain size and dislocation density generated through hot deformation can be represented by the following exponential relationship with Z:

$$d_{\mathrm{DRX}} = BZ^{-p} \tag{3}$$

Here, d_{DRX} is average dynamically recrystallized grain size and B and p are constants. From Fig. 8, the values of 498 μm s$^{-0.13}$ and 0.13 could be obtained for B and p, respectively. According to the linear relationship between dynamically recrystallized grain size and Z parameters, it can be concluded that, in the studied temperature range, the mechanism of hot deformation process is substantially the same, in agreement with previous researches [18].

As it was mentioned before, the obtained value of p, exponent of Z parameter in Eq. (3), in the present study is different from the results of previous researches. Higashi and co-workers [18] obtained a value of p equal to 0.52 for extrusion of AZ91 alloy. A value of 0.33 has reported, on the other hand, for AZ31 and AZ91 by Watanabe et al. [19]. Beer and co-workers declared a value of p equal to 0.11 for cast AZ31 specimens [20]. The controversy over the p-value in Mg alloys cannot be clearly addressed. However, it can be associated with the following factors:

(1) The difference is due to variations in the value of apparent activation energy, Q, which is inherited by the Z parameter. In this research the value of calculated Q is 182 kJ/mol, which is significantly higher than previous reported data, 162.5 kJ/mol [20] and 135 kJ/mol [19]. However, it should be mentioned that, based on the mathematical equations, the variations in the calculated Q value does not significantly affect the p value.
(2) Another important factor is the cooling rate after the hot deformation test. Watanabe et al. cooled down his samples in air after deformation process [19]. Barnett [21] demonstrated that for obtaining an appropriate microstructure, cooling rate is a crucial parameters. If cooling rate is sufficiently low, grain growth occurs and, for low Z-values (higher temperatures and lower strain rates), larger grains develop and therefore p value increases.
(3) The sample condition before testing also has a great influence on the recrystallized grain size. Researches by Watanabe et al. [19] and Barnett et al. [20] showed that DRX grain size in a cast specimen is larger than that of a wrought one. The dif-

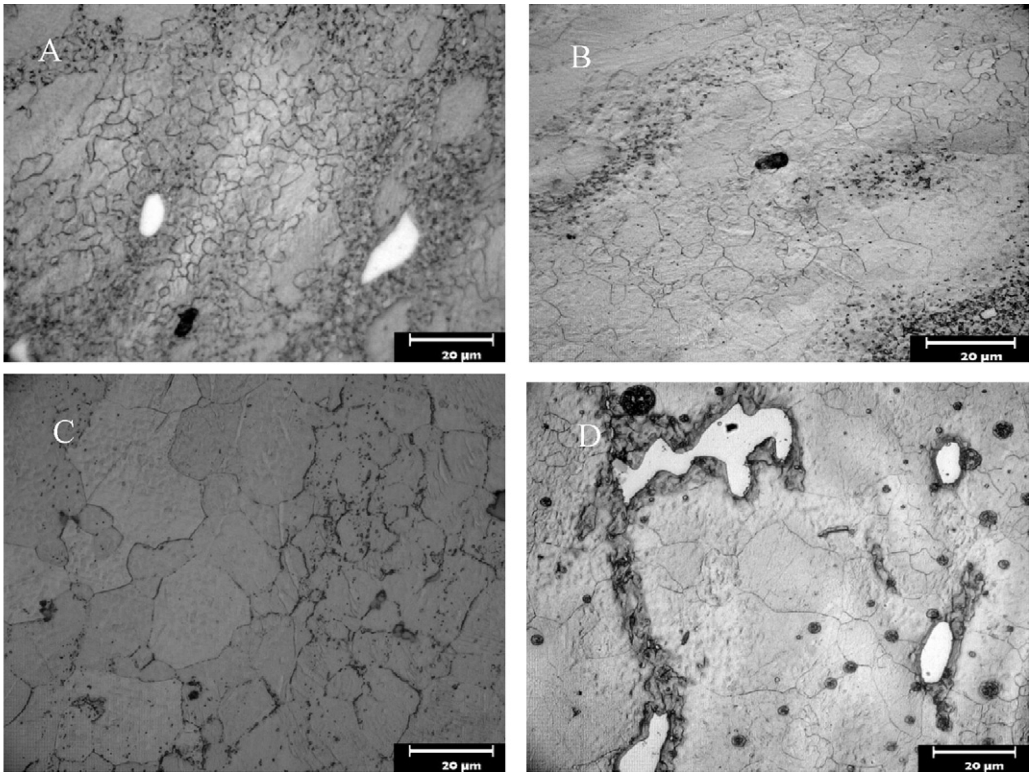

Fig. 6. Microstructure obtained after hot deformation under a strain rate of 0.1 s^{-1} and temperatures of (A) 325 °C, (B) 350 °C, (C) 375 °C and (D) 400 °C.

ference is more significant at low Z values. In the fine-grained wrought material used in current research, a high volume fraction of grain boundaries as appropriate places for initiation of DRX resulted in a very fine final microstructure.

(4) Formation of β precipitates during preheating and hot deformation can considerably affect *p*-value. Research by Li et al. proved that the grain size of cast AZ91 extruded at 270 °C is strongly dependent on the amount of β phase precipitates

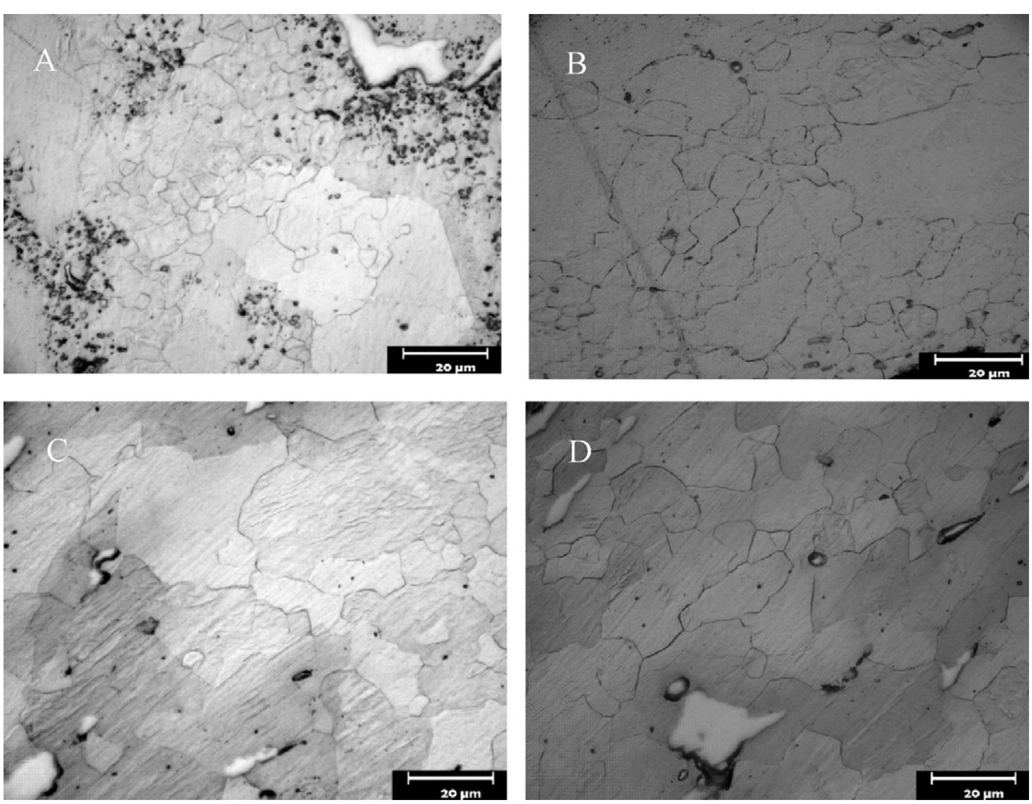

Fig. 7. Microstructure obtained after hot deformation under a strain rate of 0.001 s^{-1} and temperatures of (A) 325 °C, (B) 350 °C, (C) 375 °C and (D) 400 °C.

Table 1
Dynamically recrystallized grain size, d_{DRX} (μm), measured by Jeffries' method at various temperatures and strain rates.

Deformation temperature (°C)	Strain rate (s^{-1})			
	1	0.1	0.01	0.001
325	3	4	5.5	7
350	6.5	6.5	8	11
375	11	11	12	14
400	10	10.5	18	17

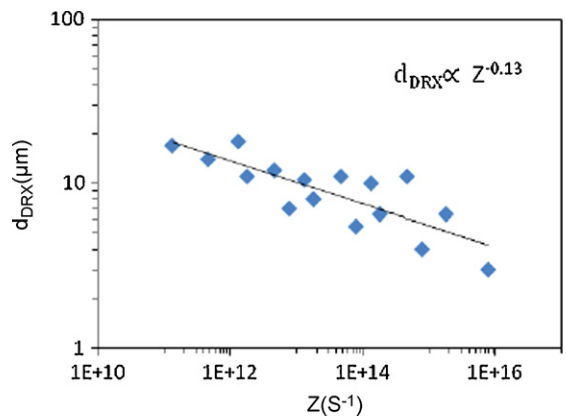

Fig. 8. Variation of dynamically recrystallized grain size with Zener–Hollomon parametrer.

[22]. The results of studies by Xu et al. [23] also indicated that recrystallized grains only formed at the vicinity of individual β precipitates and primary grain boundaries. Therefore, fine β precipitates contribute in pinning recrystallized grain boundaries and restricting their growth, leading to reduction of recrystallized grain size [22,23]. It should be mentioned that a decrease in temperature and an increase in strain rate lead to increasing the amount of dynamic precipitation. As a result, finer recrystallized grains are obtained.

Fig. 9 exhibits the individual influence of strain rate and temperature on dynamically recrystallized grain size. At constant temperature (dotted lines), by increasing strain rate, the recrystallized grain size decreases and also at constant strain rate (solid lines) the recrystallized grain size increases as the temperature is increased. It is evidence that the slope of dotted lines is greater than that of the solid lines which indicates that the recrystallized grain size is more dependent on temperature variations due to dislocation density and precipitates morphology. On the other hand, an increasing in dislocation density due to increase in strain rates lead to a reduced DRV. But an increase in temperature not only accelerates DRV which partially consumes the stored energy and hinders DRX but also the grain size will grow up. On the other hand, the fine precipitates of β-phase contribute in pinning of the recrystallized grain boundaries and therefore restrict their growth. The SEM image of compressed specimens at 350 °C and 0.1 s^{-1} in Fig. 10 clearly shows fine β precipitates. The fine β particles distributed at the newly-formed dynamically recrystallized grain boundaries confirms the previous research results [17–21]. In addition, Xu et al. [23] also confirmed that the size and morphology of β phase depend on temperature rather than on strain rate. In other words, by decreasing in temperature, not only the amount of fine β phase precipitates at the vicinity of primary grains increase but also the mechanism of grain boundary pinning becomes dominant.

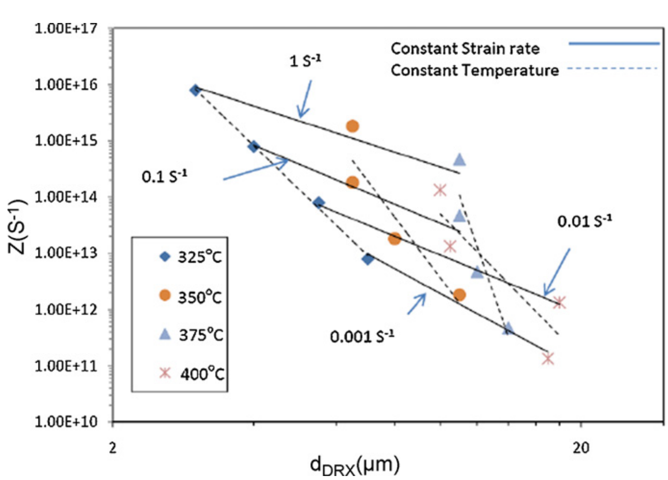

Fig. 9. Influence of temperature and strain rate on dynamically recrystallized grain size.

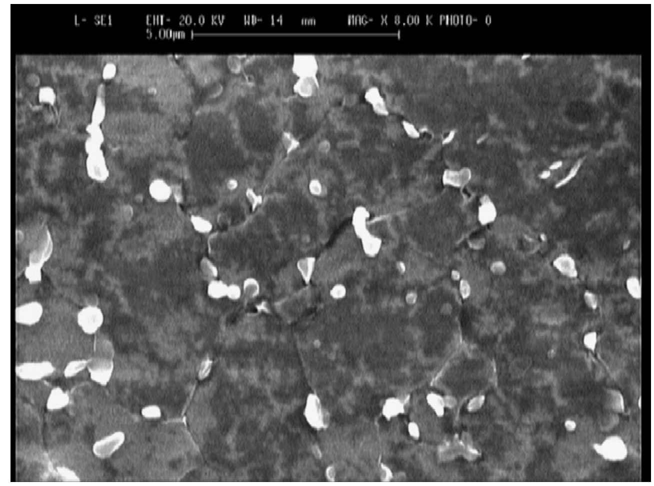

Fig. 10. SEM micrographs obtained after hot compression at 350 °C and 0.1 s^{-1}.

4. Conclusions

The influence of thermomechanical parameters on flow stress as well as dynamically recrystallized grain size of Mg alloy AZ91 during hot deformation was investigated by performing hot compression test and microstructural observation. The following conclusions can be drawn:

(1) A power-law relation can be developed between the characteristic peak strain and Zener–Hollomon parameter and the exponent is determined to be 0.17.
(2) At constant hot deformation temperature, the dynamically recrystallized grain size decreases by increase in strain rate.
(3) Increase in deformation temperature form 325 to 400 °C causes increase in dynamically recrystallized grain size.
(4) A linear relationship between recrystallized grain size and Zener–Hollomon parameter, Z was obtained. By increase in Z value, the recrystallized grain size decreases.
(5) The recrystallized grain size depends strongly upon the thermomechanical parameters, temperature and strain rate. In particular, the effect of temperature is more influential than that of strain rate.

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