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ScienceDirect
Trans. Nonferrous Met. Soc. China 22(2012) 1329−1335
Transactions of Nonferrous Metals Society of China
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Deformation mechanism and softening effect of extruded AZ31 magnesium alloy sheet at moderate temperatures

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Received 4 July 2011; accepted 5 January 2012

Abstract: The flow stress behavior of extruded AZ31 magnesium alloy sheet was investigated by means of compression tests at temperatures between 473 and 523 K and strain rates ranging from 0.001 to 1.0 s^{-1}. The deformation activation energy of the sheet in extrusion direction (ED) was calculated, and the relationship between the softening effect and deformation mechanism was elucidated by optical microscopy and transmission electron microscopy. The results show that when the extruded AZ31 magnesium alloy samples were compressed at moderate temperatures in ED direction, the deformation activation energy is 174.18 kJ/mol, which means that dynamic recrystallization (DRX) is the main softening effect and is controlled by cross slip of thermal active dislocation. Dislocation slip is the main deformation mechanism in moderate-temperature deformation process except twinning. The main DRX effect at moderate temperatures can be considered to be continuous dynamic recrystallization accommodated with twinning DRX.

Key words: AZ31 magnesium alloy; deformation mechanism; active energy; dynamic recrystallization

1 Introduction

Due to the high specific stiffness, superior specific strength, good damping property and light weight, the research on wrought Mg alloys has surged in recent years [1−9]. The deformation temperature range of Mg alloys can be divided into three levels: low temperature zone (below 473 K), moderate temperature zone (473−573 K) and high temperature zone (higher than 573 K) [1]. Compared with high temperature zone, the deformation of Mg alloys below 573 K can effectively save energy, simplify deformation technology and produce special products [2,10−12]. For example, the magnesium alloy sheet with thickness less than 1 mm can not be produced by hot rolling, but only by cold rolling [10−12]. Therefore, it is necessary to investigate the deformation mechanisms and softening effect at moderate temperatures.

In recent years, most reports have been focused on the deformation behavior of wrought magnesium alloy AZ31 at high temperatures [2−4]. However, some reports have partly shown the relationship between deformation mechanisms and softening effect of Mg alloys at low deformation temperatures [13,14]. GALIYEV et al [13] suggested that twin lamellas grew gradually and the fine grains appeared in the inner of twins at low temperatures for ZK60 Mg alloy, which can be named twinning dynamic recrystallization (TDRX). The occurrence of TDRX was possibly ascribed to twinning, basal slip and (*a*+*c*) dislocation slip. For as-cast AZ31 Mg alloy, when the deformation temperature was 453 K, lots of serrated twinning and grain boundaries were observed, which means that dynamic recovery (DRV) appeared [14]. With increasing temperature, the serrated twinning and grain boundaries can gradually convert to fine dynamic recrystallization (DRX) grains due to the effect of dislocation slip. However, it remains uncertain whether the interpretation of deformation mechanisms, softening effect and microstructural evolution behavior at high or low temperatures are suitable for the alloy deformed at moderate temperatures. The active energy that can reflect deformation mechanisms and softening effect in moderate temperatures for Mg alloys is also unclear.

Foundation item: Project (50804015) supported by the National Natural Science Foundation of China; Project (GJJ11162) supported by the Youth Science Foundation of Jiangxi Educational Committee, China; Project (EA201001035) supported by the Doctor Startup Foundation of Nanchang Hangkong University, China

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DOI: 10.1016/S1003-6326(11)61322-8

In this study, the flow stress behavior of extruded AZ31 magnesium alloy sheet at temperatures between 423 and 523 K was investigated. In order to study the deformation mechanism at moderate temperatures, the deformation activation energy of the sample was calculated. Optical microscopy (OM) and transmission electron microscopy (TEM) were employed to expose the microstructural evolution during the deformation of AZ31 magnesium alloy and to elucidate the relationship between softening effect and deformation mechanisms.

2 Experimental

The material used in this study was as-extruded AZ31 Mg alloy with composition as listed in Table 1. The manufacturing route of Mg alloy sheet can be simply described as casting and extrusion. At first, the raw material was smelted in crucible resistance furnace and RJ21 was used as the smelting protection agent. After smelting, steel mold was used in casting process to obtain a cylinder ingot. Then, a smooth ingot with dimensions of *d*160 mm×200 mm was obtained by removing the oxide layer and impurity using turning.

Table 1 Chemical composition of AZ31 magnesium alloy (mass fraction, %)

Al	Zn	Mn	Fe
3.13	0.87	0.44	<0.002

Cu	Ca	Mg
<1.00	<0.03	Bal.

After casting, the ingot endured homogenization treatment at 673 K for 10 h to eliminate the segregation and inhomogeneity of chemical content in grains. Then, the ingot was extruded with 12250 N horizontal extruder at an extrusion ratio of 17.8:1 and extrusion speed of 60 mm/min. In the extrusion process, graphite and engine oil were used as lubricant. Before extrusion, the temperatures of die cavity and extruded mold were determined at 553 and 613 K, respectively. Then, the annealing treatment at 573 K for 30 min was used to reduce the inner stress of the sheet. The casting and extrusion process refers to the national standard GB/T 5156—2003 as illustrated in Refs. [2,15]. By the extrusion process, the surface of the extruded sheet turned smooth, without cracks and defects. Finally, the round compressive specimens (*d*6 mm×9 mm) were prepared using electrical-discharge machining from the as-extruded alloy sheet (10 mm in height) both along extrusion direction (ED) and transverse direction (TD), as shown in Fig. 1.

In order to investigate the constitutive relationship and deformation mechanism of the extruded AZ31 Mg alloy sheet at moderate temperatures, compression at 473−573 K and strain rate of 1−10^{-3} s^{-1} was also conformed by Gleebel−1500 and the microstructure was observed by HITACHI H800 TEM. Then, the active energy along ED was calculated. After testing, the surfaces of all the specimens were ground with 2000 grit SiC paper and then polished by standard metallographic techniques. For optical microscopy observation (OM), specimens were etched for 10 s in a solution of 19 mL water, 20 mL acetic acid, 1 mL nitric acid and 60 mL ethylene.

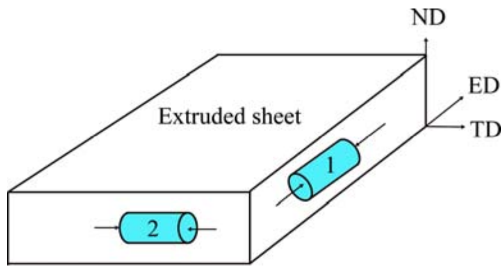

Fig. 1 Schematic diagram of extruded AZ31 magnesium alloy sheet

3 Results and discussion

3.1 Flow stress and active energy

The true stress—strain curves of the samples following extrusion direction at 473−573 K and strain rate of 1−10^{-3}s^{-1} are shown in Fig. 2. It can be seen that the deformation behavior shows obvious strain rate sensitivity at moderate temperatures. With the increase of strain rate, the peak stress also gradually increases. When the flow stress reaches the peak stress, softening and hardening effect both occur and obtain balance. Then, with the deformation pursuing, flow stress decreases and gradually reaches a stable flow state.

In order to understand the deformation mechanism at moderate temperatures, the active energy in temperature range of 473−573 K was calculated. After measuring the experimental data of different materials in plastic deformation, it is found that in the low stress level, the relationship between stress σ and strain rate $\dot{\varepsilon}$ can be described according to Ref. [16] as:

$$\dot{\varepsilon} = A_1 \sigma^{n_1} \exp\left(\frac{-Q}{RT}\right) \tag{1}$$

where A_1 and n_1 are constants independent of deformation temperature; T is thermodynamic temperature; Q is the active energy which is the critical parameter and reflects the difficult degree of hot deformation process; and R is the mole gas constant. In the high stress level, both A_1 and n_1 are full with power exponent relationship and can be expressed as [17,18]:

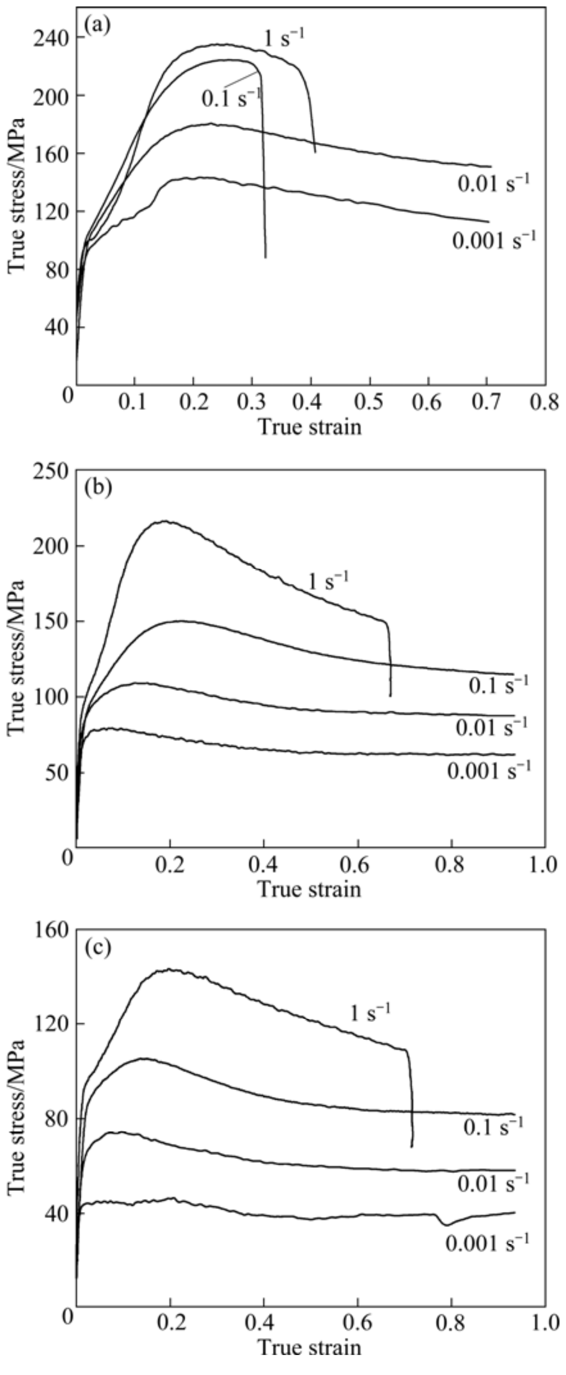

Fig. 2 True stress—strain curves of samples deformed along ED at different temperatures: (a) 473 K; (b) 523 K; (c) 573 K

$$\dot{\varepsilon} = A_2 \exp(\beta\sigma) \exp\left(\frac{-Q}{RT}\right) \tag{2}$$

where A_2 and β are constants independent of deformation temperature.

In Eqs. (1) and (2), the dynamic balance between strain softening and hardening effect is described, which is similar with stable creep deformation. Similarly, SELLARS et al [18] utilized the hyperbolic-sine correct Arrhenius relationship to describe the stable state deformation behavior as:

$$\dot{\varepsilon} = A(\sinh \alpha\sigma)^n \exp\left(\frac{-Q}{RT}\right) \tag{3}$$

where n is the stress exponent and α is a constant, which is full with $n_1=\beta/\alpha$.

Comparing Eqs. (1), (2) and (3), in the low stress level ($\alpha\sigma$<0.8), Eq. (3) is close to Eq. (1), which shows the exponent relationship. Otherwise, in the high stress level (0.8≤$\alpha\sigma$<1.2), Eq. (3) is close to Eq. (2), which reflects the power exponent relationship. The constants α, β and n_1 are full with $\alpha=\beta/n_1$. Therefore, α and n_1 can be obtained by experimental data in different stress levels. Most results show that Eq. (3) could better reflects the common hot deformation behavior.

Equations (1) and (2) can be described with logarithm relationship respectively as:

$$\ln\dot{\varepsilon} = \ln A_1 - \frac{Q}{RT} + n_1 \ln\sigma \tag{4}$$

$$\ln\dot{\varepsilon} = \ln A_2 - \frac{Q}{RT} + \beta\sigma \tag{5}$$

Figures 3(a) and (b) show the direct and logarithm relationship between strain rate and peak stress of the extruded sheet at moderate temperatures, respectively. By calculating Eqs. (5) and (4), the slopes of the curves in Figs. 3 (a) and (b) could be obtained as β and n_1,

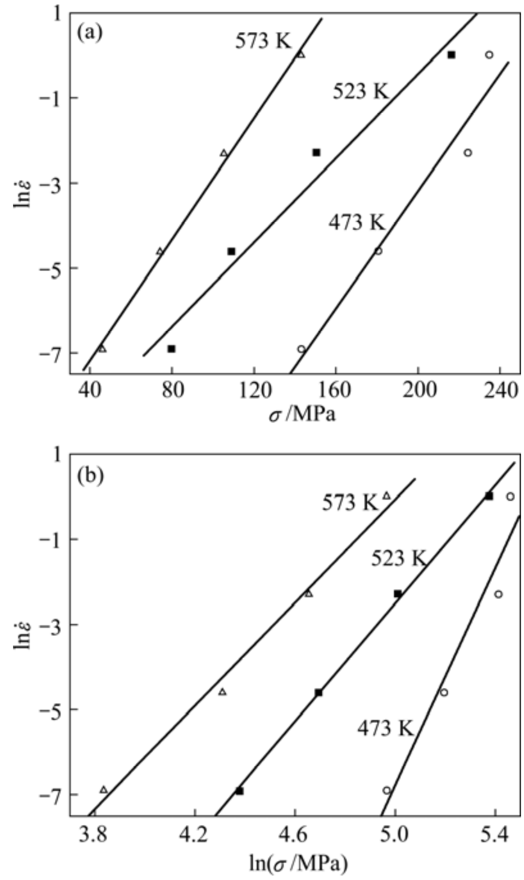

Fig. 3 Relationship between strain rate and peak stress at moderate temperatures: (a) $\ln\dot{\varepsilon}$ and σ; (b) $\ln\dot{\varepsilon}$ and $\ln\sigma$

respectively. After linearity regression of least two-multiplication method, n_1 and β can be thought the average values of the slopes in Figs. 3(b) and (a), respectively. Then, α can be obtained by $\alpha=\beta/n_1$. The two slope values are charged with linear analysis and then it can be obtained that n_1=8.57 and β=0.0631. By the formula $\alpha=\beta/n_1$, α is calculated to be 0.0074.

Equation (3) can be transformed to

$$\frac{Q}{RT}=\ln A-\ln\dot{\varepsilon}+n\ln[\sinh(\alpha\sigma)] \tag{6}$$

The differential coefficient of $1/T$ in Eq. (6) can be calculated and the formula related to Q value can be obtained as:

$$Q=R\frac{\partial\ln[\sinh(\alpha\sigma)]}{\partial(1/T)}\left|\frac{\partial\ln\dot{\varepsilon}}{\dot{\varepsilon}\,\partial\ln[\sinh(\alpha\sigma)]}\right|_T \tag{7}$$

From Eq. (7), it could be found that if Q is not related to temperature, the relationship between $\ln[\sinh(\alpha\sigma)]$ and $1/T$ shows linear. Therefore, S, defined as the value equal to $\partial\ln[\sinh(\alpha\sigma)]/[\partial(1/T)]$, can be thought the slope of the line related to $\ln[\sinh(\alpha\sigma)]$ and $1/T$; n can be thought the slope of the line related to $\ln\dot{\varepsilon}-\ln[\sinh(\alpha\sigma)]$. The relationship between $1000/T$ and $\ln[\sinh(\alpha\sigma)]$ is shown in Fig. 4, which can be obtained from the peak stress and temperature. After linearity regression of least two-multiplication method, S could be obtained as the average value of the slopes for four lines. S and n are calculated to be 3.589 and 5.845, respectively.

The active energy can be obtained as:

$$Q=RnS \tag{8}$$

Therefore, the value of Q can be calculated as:

$$Q=RnS=174.18\ \text{kJ/mol}$$

PRASAD and RAO [19] calculated the active energy of rolled AZ31 sheet compressed at 573−823 K as 180 kJ/mol in transverse direction and 168 kJ/mol in rolling direction, respectively. Otherwise, dynamic recrystallization and dynamic recovery controlled by cross slip are thought to be the main softening mechanisms when the active energy is between 168 and 180 kJ/mol. In this study, the active energy of 174.18 kJ/mol which is much higher than the lattice self-diffusion active energy 135 kJ/mol is located in this rage from 168 to 180 kJ/mol. Therefore, it can be thought that the softening mechanism is dynamic recrystallization controlled by thermal active dislocation induced cross slip when compressed at 473−573 K along ED direction.

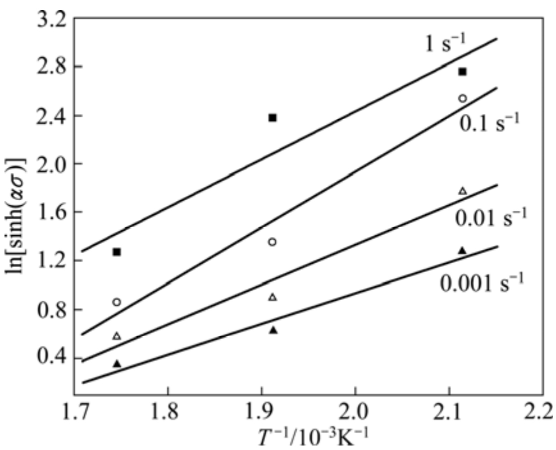

Fig. 4 Relationship between $1000/T$ and $\ln[\sinh(\alpha\sigma)]$

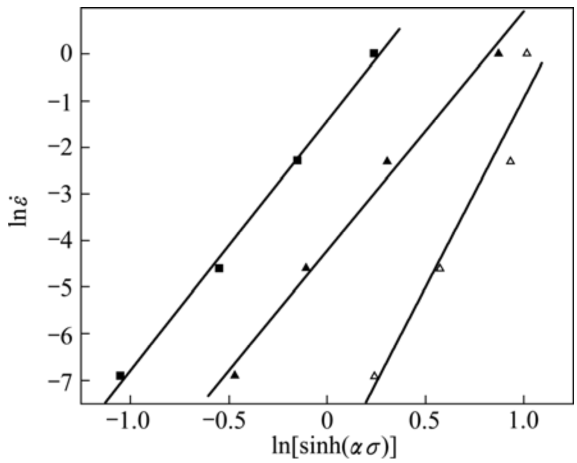

Fig. 5 Relationship between $\ln\dot{\varepsilon}$ and $\ln[\sinh(\alpha\sigma)]$

3.2 Microstructure evolution

Figure 6(a) shows the microstructure of the sample compressed along ED at strain rate of 10^{-2}s^{-1} at 373 K. It can be found that if the deformation temperature is lower than the moderate temperature zone, the grains become spindly due to compressed stress. Lots of twins occur in the grains and some twins intersect with each other. In most researches, twinning and basal slip are considered the main deformation mechanisms for Mg alloy in low temperature zone, while the occurrence of cross slip is limited due to the limitation of critical shear stress (CRSS) [1−6]. However, with the temperature increasing to 523 K, the grains are greatly fined, as shown in Fig. 6 (b). Due to the occurrence of obvious DRX, the coarse grains are replaced by fine grains and the average size is reduced to about 27 μm. Only a small mount of twins could be observed in Fig. 6(b) because the value of CRSS for twinning is higher than that for slip with increasing temperature. Therefore, twinning could not become the main mechanism at moderate temperatures.

The microstructures of the samples compressed at 473 K at strain rates of 10^{-2} and $10^{-3}\ \text{s}^{-1}$ are shown in Figs. 6(c) and (d), respectively. It can be seen that a great

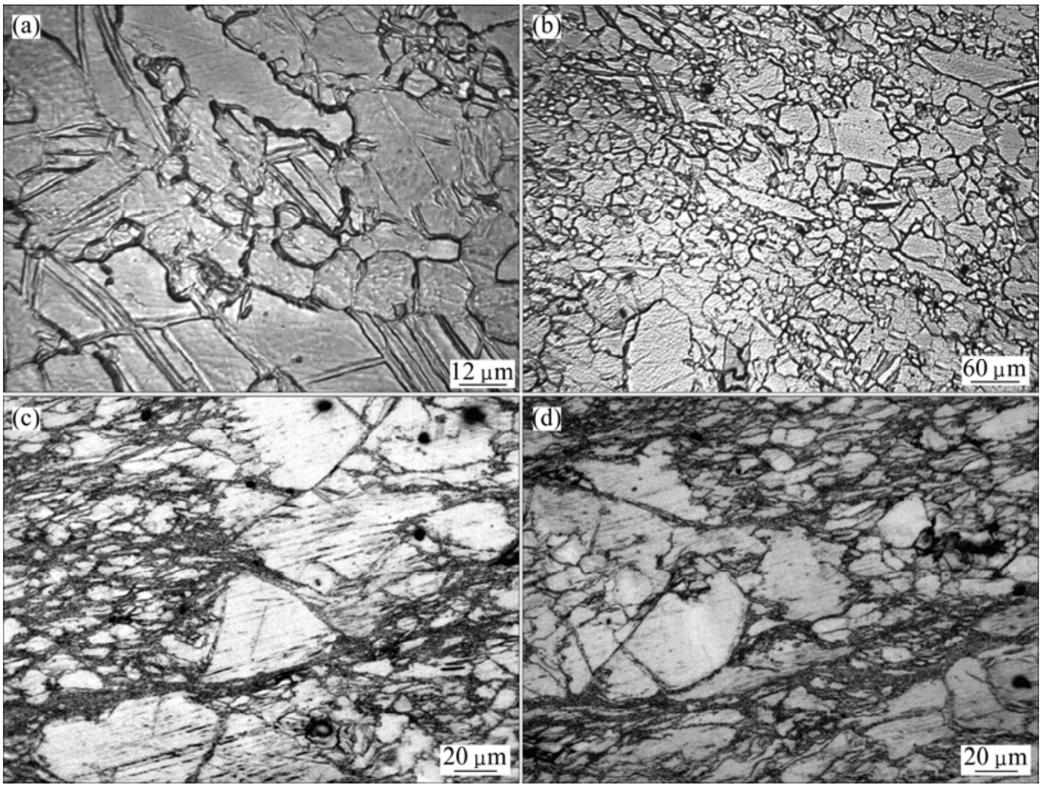

Fig. 6 OM images showing samples compressed along ED and TD at moderate temperatures and different strain rates: (a) 373 K, $10^{-2}s^{-1}$, ED; (b) 523 K, $10^{-3}s^{-1}$, ED; (c) 473 K, $10^{-2}s^{-1}$, TD; (d) 473 K, $10^{-3}s^{-1}$, TD

amount of refined grains with grain size smaller than 5 μm appear after compression and seem like necklace around the coarse grains. Twins in the grains also become recrystallized grain chains after compression. The necklace-like fine grains are thought to be the typical phenomena of continuous DRX (CDRX) [20−22]. Therefore, CDRX are the main DRX characterization in the deformation process which agrees to the results by LIU et al [23] that CDRX mechanism mainly occurs in moderate temperature zone for common Mg alloy and is caused by the cross slip of dislocations. Otherwise, it can be also seen that in Figs. 6(c) and (d), the twins are few and mainly appear in coarse grains. Fine grains also appear in the inner of twins and form recrystallized grain chains belonging to the typical TDRX.

The softening mechanism inclined to DRX for Mg alloy at moderate temperatures can be credited to two reasons [2, 20−23]. First, due to the low stacking fault energy for Mg alloy, it is difficult to congregate the extending dislocation together. Therefore, the slipping and climbing of dislocation are limited and the occurrence velocity of DRV is deferred. Second, compared with Al alloy, the diffusion velocity of grain boundary for Mg alloy is high and the accumulated dislocation near the sub-boundaries would be successfully absorbed. As a result, the occurrence of DRX is accelerated and the recombination of dislocations produced by cross slip and climb could lead to the production of low-angle grain boundary net. The continuous absorption of dislocations in the low-angle grain boundary results in the production of new refined grains, as shown in Fig. 7(a). The continuous absorption of dislocation occurs in the low-angle grain boundary of the sample compressed along TD at moderate temperatures, as shown in Fig. 7(b). In Fig. 7(c), a refined grain with average size of about 0.35 μm is observed and a serial of dislocation lines are hindered and absorbed by the boundaries of the refined grains, as shown in Fig. 7(d).

Figure 8 shows the TEM images of the sample compressed along ED at 473 K and strain rate of $10^{-2}\,s^{-1}$. It can be seen that twinning also has occurred in moderate temperature zone and the average transverse size for the spindly twins is about 0.52 μm. As the main deformation mechanism can reduce inner stress, dislocation slipping plays a significant role in moderate temperature zone. As shown in Fig. 8, a number of dislocation lines which intersect and twist with each other are hindered by twinning boundaries and directly result in the strengthening effect in the compression process. The intersection of dislocation lines indicates that both basal slip and non-basal slip which are difficult to start in low temperature zone due to the limitation of CRSS are excited in moderate temperature zone.

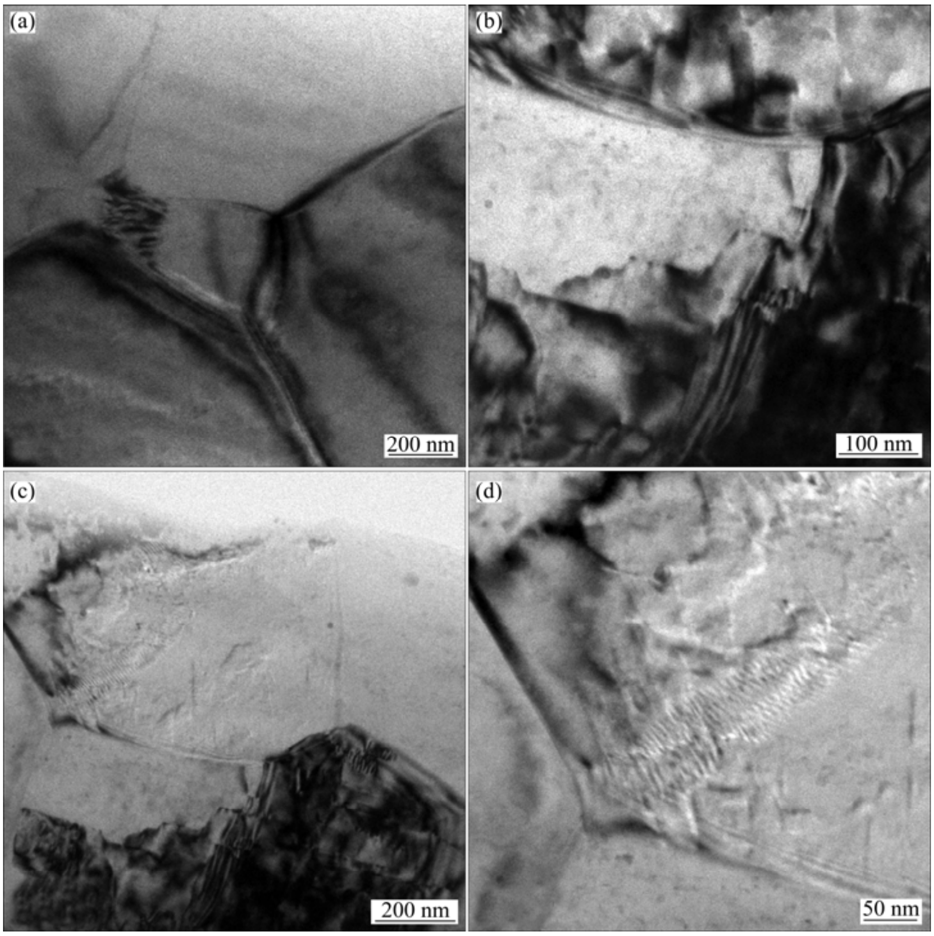

Fig. 7 TEM images of samples deformed along TD at 523 K and strain rate of $10^{-2}s^{-1}$: (a) Recrystallized grain; (b) Low-angle boundary; (c) Refined grains; (d) Dislocation lines hindered by grain boundary

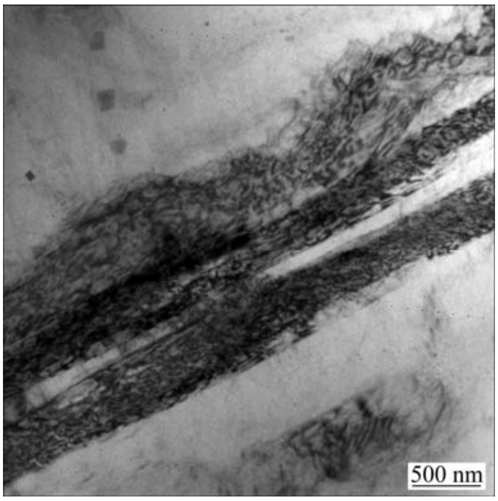

Fig. 8 TEM image of sample compressed along ED at 473 K and $10^{-2}s^{-1}$

4 Conclusions

1) When compressed at 473−573 K along ED direction, the deformation active energy of AZ31 Mg alloy is 174.18 kJ/mol, which means that DRX is the main softening mechanism and is controlled by cross slipping of the thermal active dislocation. Continuous absorption of dislocations in low-angle grain boundary leads to the occurrence of new refined grains.

2) Dislocation slip is the main deformation mechanism in moderate temperature deformation process, and twinning partly hinders the movement of dislocation. The main DRX effect at moderate temperatures can be considered CDRX accommodated with TDRX.

Acknowledgement

The authors would thank Dr. FAN J C in Sweden Royal Institute of Technology for valuable discussion and recommendation.

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AZ31 镁合金挤压板材的中温变形机理及软化机制

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摘 要：研究 AZ31 镁合金挤压板材在 473~523 K 的温度范围内。应变速率 0.001~1.0 s^{-1} 压缩时的流变应力行为，计算板材沿挤压方向压缩时的激活能，并结合光学显微镜和透射电子显微镜探讨合金软化机制和变形机理之间的联系。结果表明，在中温下沿挤压方向压缩时，AZ31 挤压态镁合金的变形激活能为 174.18 kJ/mol。这说明，由热激活位错交滑移所控制的动态再结晶是合金中温变形的主要软化机制。位错滑移是中温变形的主要变形机理，而孪生的作用则不大。其主要的动态再结晶机制为持续动态再结晶，并伴随少量的孪生动态再结晶。

关键词：AZ31 镁合金；变形机理；激活能；动态再结晶

(Edited by FANG Jing-hua)