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Plastic deformation behaviors and dynamic recrystallization mechanisms of ZK60 magnesium alloy

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Hot deformation behaviors of a ZK60 magnesium alloy were investigated by hot compressive tests on a Gleeble-1500 thermal simulation test machine at temperatures ranging from 473 K to 723 K and strain rates of $0.01 \sim 1s^{-1}$. Microstructures evolution of the alloy as well as dynamic recrystallization mechanisms at different strains with the strain rate $0.1s^{-1}$ at 623 K were examined under optical microscope (OM) and transmission electron microscope (TEM). The relationships between flow stress, strain rate and deformation temperature were analyzed, and the deformation activation energy at elevated temperatures (573~723 K) were calculated. The results indicated that under the present deformation conditions, dynamic recrystallization happened in the alloy and fine recrystallized grains tended to form ductile shear zones under the large strain condition; and there were different dynamic recrystallization mechanisms at different strains; within the temperature range from 573 K to 723 K, deformation activation energy changed with the deformation temperature and strain rate.

Key Words : Wrought magnesium alloy; Dynamic recrystallization; Plastic deformation; Thermal deformation simulation

1. Introduction

Magnesium alloys are the lightest engineering materials with high specific strengths and superior electromagnetic shielding capability [1]. They have been increasingly used for lightweight structural and functional parts in the automotive and electronic industries [2]. Owing to the hexagonal close packed structure, it is difficult for magnesium alloys to make plastic deformation at room temperature, by which the development of magnesium alloys is restricted greatly, especially for wrought magnesium alloys. Thus most parts of magnesium alloys are produced mainly by die casting [3].

Received December 31, 2005

Accepted for Publication August 30, 2006 ©2006 Soc. Mater. Eng. Resour. Japan

However, Mg and Mg alloys possess low stacking fault energy, which makes it easy that dynamic recrystallization (DRX) operates during the deformation at elevated temperatures. So DRX is a potential way to improve the plasticity and refine the structures of Mg alloys, which will make it possible for Mg alloys to process large deformation. ZK60 magnesium alloy is a kind of new model wrought magnesium alloys with good prospects. It can get high strength and superior ductility by the press working and heat treating methods. The literatures [4, 5] emphasized the capacity and behaviors of superplasticity of ZK60. But there were few studies under different deformation conditions, especially at high strain rates. The present studies on DRX of Mg alloys also lie mainly in the superplasticity field, and the correlative study when the deformation process at high strain rates was scarce. Even if in the literature [5], strain rate was merely limited at $0.0025s^{-1}$ and the study emphasized particularly on deformation mechanisms.

In this research, hot deformation behaviors of ZK60 alloy were investigated at an extensive strain rate and temperature range. Its DRX characteristic and mechanisms were still probed into. The research results will be in expectation of guiding hot-working process to a reasonable choice and providing a physical experimental groundwork for numerical simulation aiming at working course of ZK60.

2. Experiment methods

The magnesium alloy of Mg-5.3Zn-0.8Zr (wt%) was prepared by ordinary ingot metallurgy method. The as-cast material was subsequently homogenized at 673 K for 18 h. Compression samples, with a height to diameter ratio of 1.5, were cut from the homogenized material. Hot compressive deformation simulation was performed on Gleeble-1500 simulator in a range of the deformation temperatures from 473 to 723 K. Four typical strain rates on 0.01, 0.1, 0.5 and $1s^{-1}$ were chosen. The samples were quenched within 0.5s after testing so as to retain the developed microstructure. The microstructures of the alloy were observed by using Polyvar-MET optical microscope and JEOL-JEX transmission electron microscope (TEM).

3. Results and discussion

3.1 True stress-strain behaviors

The true stress-strain behaviors of the alloy with various strain rates at different deformation temperatures are shown in Figure 1. It shows that all the flow stresses showed in the experiments increase to a maximum value at first and then decrease to attain a 34

steady state. Such flow behavior is characteristic for dynamic recrystallization (DRX), and it often points out that DRX has happened in the process of deformation.

As shown in Figure 1, the maximum value of flow stress and its corresponding strain value increases with decrease of deformation temperature and increase of strain rate. Both deformation temperature and strain rate affect the process of DRX. We will discuss these in combination with the analysis of the microstructures of the alloy.

3.2 Microstructures evolution and dynamic recrystallization mechanisms

The evolution of recrystallized microstructure and DRX mechanisms of the alloy at 623 K and 0.1s^{-1} were examined by optical microscopy and TEM. Microstructure evolutions of the alloy deformed by different strains are shown in Figure 2.

At the beginning of the deformation process ($\varepsilon = 0.12$, Figure 2a), the grain boundaries appear serrated but no grain refinement has taken place. From the corresponding true stress-strain curve we can see that under this condition, the true stress exhibits an inclination to rise but its increasing rate decreases, which reveals that dynamic recovery is the main cause of the stress softening effect. When the stress approaches the maximum value ($\varepsilon = 0.24$, Figure 2b), original grains have been elongated by deformation and a small amount of fine recrystallized grains start to nucleate along grain boundaries and at triple junctions. The original grains formed near grain boundaries at a high strain value ($\varepsilon = 0.60$).

Correspondingly, the true stress-strain curve inclines to fall down, which shows that the stress-softening effect is more powerful than the strain hardening effect. With a higher strain value ($\varepsilon = 0.93$), a few original grains remain with an appropriately oblate rhomboid shape, and the area of dynamic recrystallization becomes considerable when the decreasing rate of the true stress-strain curve is too obscure to perceive, which means that the stress softening effect and the strain hardening effect are about to strike a new balance.

The microstructures and the DRX mechanisms of ZK60 magnesium alloy at different strain stages were further studied by TEM. Figure 3a shows that sparse dislocations appear in some grain when the value of ε is 0.12. Although these dislocations at least belong to two slip systems, most of them belong to a certain slip system. What is more, there are obvious corrugated slip bands in some grains (Figure 3b), which reveals that the deformation is dominated by a certain kind of slip systems and accompanied by cross-slip. Because of this, it is difficult to form a network configuration of dislocations in grains even with a moderate increase in strain.

Under the same condition ($\varepsilon = 0.12$), However, dislocation density is higher in the vicinity of the grain boundaries than in the grains. Figure 3c reveals that cellular structures appear in the vicinity of the gain boundaries. It is clear that dislocations have been absorbed by cell walls in some grains while there is no dislocation in some other areas, which suggests that dynamic recovery has happened. But the dynamic recovery is inhibited by



Figure 1 True stress-strain behaviors of ZK60 magnesium alloy with various strain rate at different temperatures: (a) $\dot{\varepsilon} = 0.01s^{-1}$, (b) $\dot{\varepsilon} = 0.1s^{-1}$, (c) $\dot{\varepsilon} = 0.5s^{-1}$, (d) $\dot{\varepsilon} = 1s^{-1}$.

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Figure 2 Microstructures under different strains of ZK60 magnesium alloy deformed at 623 K and 0.1s⁻¹: (a) $\varepsilon = 0.12$, (b) $\varepsilon = 0.24$, (c) $\varepsilon = 0.60$, (d) $\varepsilon = 0.93$.



Figure 3 Dislocation structures of ZK60 magnesium alloy under a strain of 0.12 deformed at 623 K and 0.1s⁻¹: (a, b) within the grain interior, (c) in the neighborhood of the grain boundary.

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the thick cell walls and a large amount of dislocations remain in the cellular structures. Due to the fact that the very widely extended dislocations resulted from a low stacking fault energy value of Mg, it leads to the consequences that dislocations can hardly be free from the nodal points and the dislocation network can also difficultly be neutralized by unlike dislocations through cross-slip and climbing. Thus the dynamic recovery is inadequate and the surplus stored energy can stimulate dynamic recrystallization.

An important microstructure evolution, taking place as the strain value increasing from 0.12 to 0.24, is that dynamic recrystallization happens prioritily at bowed grain boundaries, which may be inferred from the absence of arcuate grain boundaries in Figure 2b. During the plastic deformation of a polycrystalline material, grain boundaries will act as obstacles to impede the motion of dislocations. When the density of dislocation going through a grain boundary exceeds its absorption capacity or when the process of lattice dislocation absorption requires an incubation time, excessive lattice dislocations will pile up and subsequently generate a local stress concentration on the grain boundary, which leads to a non-equilibrium state of the grain boundary. If dislocation density is somehow different at both sides of the grain boundary, there will be a driving force stimulating a unidirectional movement of grain boundary, therefore paving the way for grain boundary bowing. The area enclosed by bowed boundaries has a low density of dislocation, which is likely to form nucleus of dynamic recrystallization. It is also called as strain induced crystal boundary migration mechanism. Figure 4 clearly shows the nucleus formed at the bowed grain boundary by dynamic recrystallization are growing up.

The distorted region in the vicinity of the grain boundary gets thicker as the strain increases ($\varepsilon = 0.60$), thus there are more subgrain formed near grain boundary by dynamic recovery, some of which are growing up to high-angle boundaries that are easy to move. Tang R.Z. etc. [6] had reported that such subgrains will perform as crystal nucleus of dynamic recrystallization and grow up. They pointed out that there are two ways of subgrain growth. One of which is subgrain growth by incorporation of adjacent subgrains, which is commonly observed in the metal characterized

by a high value of the stacking fault energy. Another way is subgrain growth by the movement of subgrain boundaries, which is commonly observed in the metal characterized by a low value of the stacking fault energy. Generally speaking, magnesium and its alloy are characterized by a low value of the stacking fault energy and thus it seems that their mechanism of dynamic recrystallization conforms to the latter.

But the existence of the former mechanism was also observed by TEM (Figure 5), which is related to essence of the process of subgrain incorporation. Two subgrains merge into one larger sub-grain by the process of dislocation climbing happening near their low angle grain boundaries. At the mean time, the orientation of the two original subgrains becomes the same by diffusion of atoms and rearrangement of their positions. Because the boundary of the larger subgrain annihilates more dislocations, it is likely to convert into a high angle grain boundary. Therefore, the mechanism of subgrain incorporation is closely related to dislocation climbing. It is generally agreed that dislocation climb takes place easier in metals obtaining a higher stacking fault energy under the same deformation condition, which causes the difference among subgrain growth mechanisms of metals having different stacking fault energy. However, Galiyev, etc [5], pointed out that the deformation mechanism of magnesium alloy at a high temperature concludes basal slipping, non-basal slipping and cross slipping of dislocation, but also concludes dislocation climbing. Therefore, the nucleus of dynamic recrystallization can be formed by the former mechanism when ZK60 alloy is deformed at 623 K.

The amount of fine recrystallized grains formed at the distortion region in the vicinity of grain boundaries becomes larger as the strain value increases to 0.93. At this time, the important character is that these fine recrystallized grains tend to form ductile shear zones, which was explained by Iron, etc. [7]. Given that original sizes of grains in the experiment are relatively larger, the author revised the model by combining with the microstructures evolution (Figure 2c, d) of the grains as shown by Figure 6. It is clear that the ductile shear zone is the very reason why the shape of the remained original grains tends to be approximately oblate rhombus.

Thus, deformation is easier to develop along these ductile shear zones and stress concentration will exist there. Therefore, the dynamic recrystallization mechanism will be even more complicated under a larger extent of deformation.



Figure 4 Dynamic recrystallization nucleus formed at arcuate grain boundary in ZK60 magnesium alloy under a strain of 0.24 deformed at 623 K and 0.1s⁻¹. The white arrow A and B indicate the location of arcuate grain boundary. The arrow C indicates the location of subgrain boundary and the direction of subgrain boundary migration.



Figure 5 TEM micrograph showing the merging of two neighbor subgrains in a specimen under a strain of 0.60 deformed at 623 K and 0.1s⁻¹.

It had been found by TEM that there were many prolonged substructures or grains when strain value reaches 0.93 (Figure 7a, b). As described above, these prolonged substructures are likely to form in ductile shear zones and they will develop into strip-shaped subgrains, whose growing speed is quicker than the subgrain incorporation mentioned before. The main cause of such phenomenon is [8] that the boundary tension of the long strip-shaped subgrain boundary impels its inclination to rotate to balance the force and thus converts into a high angle boundary more quickly. Figure 7a, b reveal that higher dislocation density in the vicinity of the prolonged structure, which suggests a higher stored energy, accelerates the growth of the subgrain. What is more, shear deformation zones were detected by TEM at a high strain value (Figure 7c). The high stored energy of the shear zones stimulates a quick nucleation. The variety of the grains orientations in the shear zones gives rise to differently oriented nucleus and thus also enhances the formation of high angle boundaries, which is propitious to the growth of subgrains. Figure 7d just shows that the nucleus of dynamic recrystallization formed near the edge of shear zones are growing up in the shear deformation zones.

3.3 Constitutive analysis and parameters calculation

It is clear that there is a relationship among flow stress, strain rate and deformation temperature of ZK60 magnesium alloy. Therefore, it is necessary to make it clear in order to know the plastic deformation behaviors of the alloy at a high temperature and pave the way for numerical simulation.

In the constitutive analysis, the effects of temperature and strain rate on flow stress have been adequately expressed by the following equation [9, 10]:



Figure 6 Schematic showing the model for the formation of 'ductile shear zones' during the deformation of ZK60 magnesium alloy at 623 K and 0.1s⁴.



Figure 7 TEM micrograph showing the microstructures in ZK60 magnesium alloy strained to 0.93 at 623 K and 0.1s⁻¹: (a,b) prolonged substructures or grains, (c) shear deformation zones, (d) the recrystallization nucleus formed near the edge of shear zones.

$$4[\sinh(\alpha\sigma)]^{n} = \dot{\varepsilon}\exp(Q/RT) = Z$$
(1)

where A, α and *n* are all constants, $\dot{\varepsilon}$ is strain rate and *Z* is Zener-Hollomon parameter combining the two control variables through an Arrhenius equation with activation energy *Q*. Then the *Q* can be expressed as:

$$Q = R \frac{\partial \ln \dot{\varepsilon}}{\partial \ln [\sinh(\alpha \sigma)]} \bigg|_{\mathrm{T}} \frac{\partial \ln [\sinh(\alpha \sigma)]}{\partial (\mathrm{I/T})} \bigg|_{\dot{\varepsilon}}$$
(2)

In order to calculate Q, ln $\dot{\varepsilon}$ is plotted versus ln $[\sinh(\alpha\sigma)]$ as shown in Figure 8a and ln $[\sinh(\alpha\sigma)]$ versus 1/T as shown in Figure 8b.

The relationship of Q with the temperature and strain rate is therefore found as shown in Figure 9. The deformation activation energy increases as temperature rising and its range increased is larger and larger. Especially when the temperature is above 673 K, the deformation activation energy raises with the temperature rapidly; while the behavior of Q is complicated: when $\dot{\varepsilon} < 0.1 \text{s}^{-1}$, Q increases as temperature rising, but Q decreases as temperature rising when $\dot{\varepsilon} > 0.1 \text{s}^{-1}$.

The fact that Q has an obvious increase with temperatures above 673 K is paid an attention. The nucleation and the growth of nucleus of dynamic recrystallization consume a great many of dislocations and the process will be strengthened as temperature rising, which leads to the increase of deformation activation

energy because latent dislocation sources are reduced and hard to be initiated resulting from a large consumption of dislocations and relaxed stress concentration. Thus the deformation activation energy is increased rapidly after 673 K because of the participation of dislocation climbing.

4 Conclusions

In summary, under the present deformation conditions, dynamic recrystallization happened in ZK60 magnesium alloy and fine recrystallized grains tended to form 'ductile shear zones' under large strain condition. The model for the formation of 'ductile shear zones' proposed in this paper preferably explained why the morphology of remained original grains tended to be approximately oblate rhombus with strain increasing. There were different dynamic recrystallization mechanisms at different strains. Strain induced crystal boundary migration mechanism, subgrains merging mechanism and the mechanisms by prolonged substructure's quick growth and quick nucleation in shear deformation zones operated under different stains. Within the range temperature from 573 K to 723 K, deformation activation energy increased with deformation temperature increasing, and rapidly increasing of the deformation activation energy after 673 K related to the action of dislocation climbing over this temperature.



Figure 8 (a) ln $\dot{\varepsilon}$ as a function of ln [sinh($\alpha \sigma$)] at different temperatures, (b) ln [sinh($\alpha \sigma$)] as a function of reciprocal T at different $\dot{\varepsilon}$.



Figure 9 Apparent activation energy of alloy as a function of temperature at different strain rates.

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