Article ID: 1003-6326(2004)02-0306-05

Dynamic recrystallization of AZ91 magnesium alloy during compression deformation at elevated temperature ①

LI Shu-bo(李淑波), WANG Yan-qiu(王艳秋), ZHENG Ming-yi(郑明毅), WU Kun(吴 昆)
(School of Materials Science and Engineering, Harbin Institute of Technology, Harbin 150001, China)

Abstract: High temperature compressive tests of AZ91 Mg alloy were carried out at 573-723 K and strain rates of 0.001-1 s^{-1}. The microstructures of as-compressed samples were observed by optical microscopy and transmission electron microscopy (TEM), and the microhardness was also tested. It is shown that with the increase of temperature or the decrease of strain rate, the flow stress decreases, at the same time the dynamic recrystallization (DRX) of the alloy is more noticeable. The microstructures reveal that continuous dynamic recrystallization, which develops through conversion of low-angle grain boundaries into high-angle boundaries, occurs preferentially at the grain boundary.

Key words: AZ91 magnesium alloy; high temperature compression; continuous dynamic recrystallization
CLC number: TG 146.22 **Document code:** A

1 INTRODUCTION

Softening and strain hardening take place at the same time during thermal deformation in metal materials. The former depends on dynamic recrystallization or dynamic recovery in the process of deformation. The dislocations in metals with high stacking fault energy (SFE) are easy to cross-slip, thus in these materials dynamic recovery is generally considered to be the main softening mechanism during hot deformation[1]. On the other hand, the dislocations in the metals with low SFE are difficult to cross-slip, and dynamic recrystallization (DRX) may occur in some cases. Magnesium possesses high SFE, so dynamic recovery should take place during its hot deformation. But in recent years, some researchers have discovered the phenomenon of DRX during the hot deformation in magnesium alloys such as AZ31[2-5], ZK60[6, 7] and other magnesium alloys[8-10]. In the present work, we studied the behavior of compressive deformation at elevated temperature of AZ91 magnesium alloy, observed the microstructure of this alloy compressed at different temperatures and different strain rates, and analyzed the mechanism of DRX in detail.

2 EXPERIMENTAL

The as-cast AZ91 magnesium alloy used in the present study has a main chemical composition of Mg-(8.5-9)Al-(0.45-0.9)Zn (mass fraction, %). This material has an average grain size of 75 μm determined by the linear intercept method[11]. Cylindrical samples, 9 mm in length and 6 mm in diameter, were fabricated by line-cutter.

In order to determine the solidus temperature of AZ91 alloy, the DSC curves were tested by STA449C, at the temperature ranging from 298 K to 873 K and at the velocity of 50 K/min. According to the DSC curves, the solidus temperature was proved to be 768 K.

The conditions of compressive tests were selected at temperatures ranging from 573 K to 723 K with span of 50 K, and at strain rates of 0.001, 0.01, 0.05, 0.1 and 1 s^{-1} respectively. The samples were compressed to 55% length of the initial samples.

The samples were compressed on thermal simulation testing machine with an automatic data collecting system. All the samples were firstly heated to the desired temperatures with a velocity of 10 K/s, followed by a 60 s holding time. In order to preserve the microstructures obtained by high temperature compression, at the specified strain or at fracture, all specimens were immediately unloaded and quenched in water after compression. Samples for microstructure observation were sectioned parallel to the direction of compression. Samples observed by optical microscopy were gritted by emery paper, polished mechanically using diamond paste and then etched in aqueous oxalic acid (4 g oxalic acid + 96 mL H_2O). The fine interior microstructure of grains was examined under TEM. Specimens for TEM examinations were prepared by first sectioning the sample to about 0.3 mm thickness foil, polishing to 40 μm followed

① **Received date:** 2003-08-14; **Accepted date:** 2003-12-02
Correspondence: WU Kun, Professor, PhD; Tel: +86-451-86418729; E-mail: wukun@hit.edu.cn

by punching of 3 mm diameter disks and thinning by ion-beam. The microhardness of samples compressed in different conditions was measured.

3 RESULTS AND DISCUSSION

3.1 Flow behavior of high temperature compression

The true stress —strain curves for AZ91 magnesium alloy at different deformation temperatures and constant strain rate (0.1 s^{-1}) are shown in Fig. 1(a), and Fig. 1(b) corresponds to flow curves at different strain rates and constant temperature (673 K). From Fig. 1(a) we can see that the flow stress increases rapidly to the maximum and then decreases to attain a steady state. That is to say, the flow curve exhibits high peak stress. Such flow behavior shows that the high temperature compression of magnesium alloy with high SFE may be accompanied by DRX or dynamic recovery. At the same time we can also find that the strain corresponding to the peak stress decreases with the increase of temperature. The reason is that the higher temperature is advantageous to occurrence of DRX or dynamic recovery. So high temperature results in occurrence of DRX or dynamic recovery at lower strain. Fig. 1(b) shows that when the strain rate ranges from 0.05 s^{-1} to 1 s^{-1}, every curve has a stress peak, that is, the shape of the true stress —strain curve isn't affected by the value of the strain rate. With the increase of strain rate, the peak stress and corresponding strain increase. It is suggested that the lower strain rate is also advantageous to occurrence of DRX or dynamic recovery. However, the flow curve obtained at strain rate of 0.001 s^{-1} exhibits several stress peaks, which makes this curve appear undulate. The reason is that the density of dislocations rises at a low rate, so the work hardening of the alloy must proceed further after DRX or dynamic recovery in AZ91 magnesium alloy, so that the nucleation of DRX can occur once more. Then in this case, DRX and work hardening occur alternately, and the flow curve exhibits more than one peak.

The dependence of microhardness on temperature and strain rate in AZ91 is shown in Figs. 2(a) and (b) respectively. Fig. 2(a), corresponding to the test condition of different temperatures and constant strain rate, discloses that with the increase of temperature, the microhardness of AZ91 alloy decreases. This phenomenon results from occurrence of DRX during compression. The condition of higher temperature, which leads to more thorough DRX, would decrease the microhardness. In Fig. 2(b) corresponding to the test condition of different strain rates and constant temperature, we find that when the strain rate ranges from 0.001 s^{-1} to 0.01 s^{-1}, the microhardness increases rapidly with the increase of strain rate. However, when the strain rate exceeds 0.01 s^{-1}, the microhardness increases slowly. Because DRX develops fully at the lower strain rate, microhardness would decrease with the decrease of strain rate. That is to say, when the strain rate increases, DRX cannot develop thoroughly and the effect of softening is weakened.

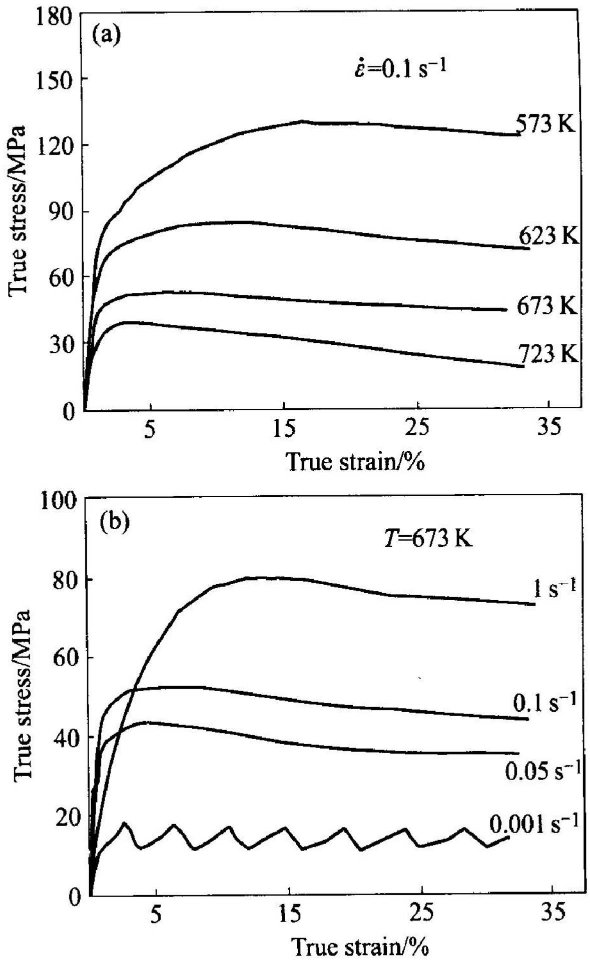

Fig. 1 True stress —strain curves of compression deformation

3.2 Analysis of microstructure

3.2.1 Initial structure

The initial microstructure of as-cast AZ91 magnesium alloy (shown in Fig. 3) consists of two phases: one phase is solid solution of Al and Zn in Mg (α-phase), the other is $Mg_{17}Al_{12}$ intermetallics (β-phase) which distribute discontinuously at the grain boundary of α-phase and form a network around the α-matrix. The size of the equiaxial initial grain is approximately 75 μm.

3.2.2 Microstructure after compression

The temperature of deformation will affect the microstructure remarkably (shown in Fig. 4). At lower temperature (573 K), the grains were elongated perpendicular to the direction of compression, then the grain boundary becomes illegible and looks like

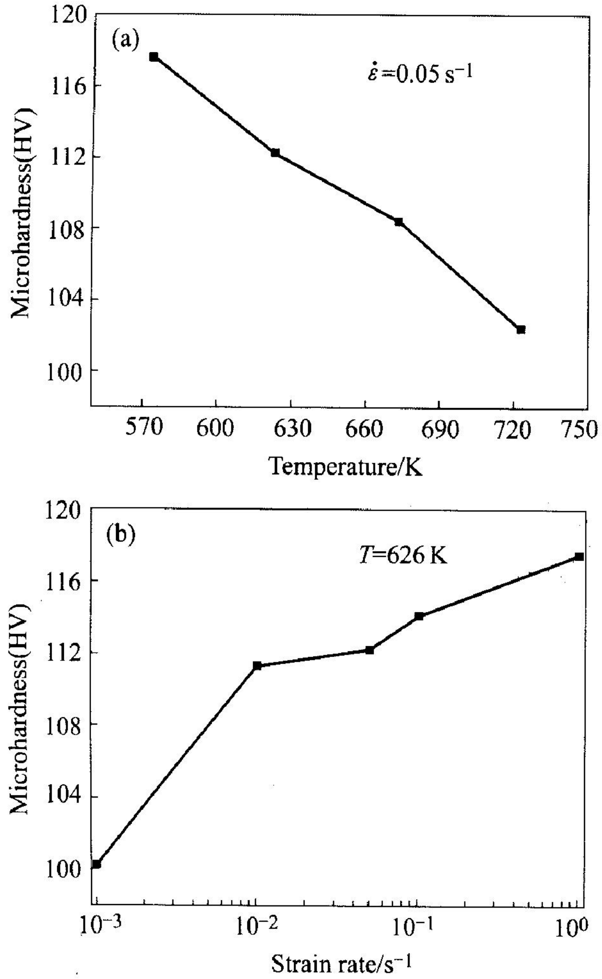

Fig. 2 Dependence of microhardness on temperature and strain rate in AZ91 magnesium alloy

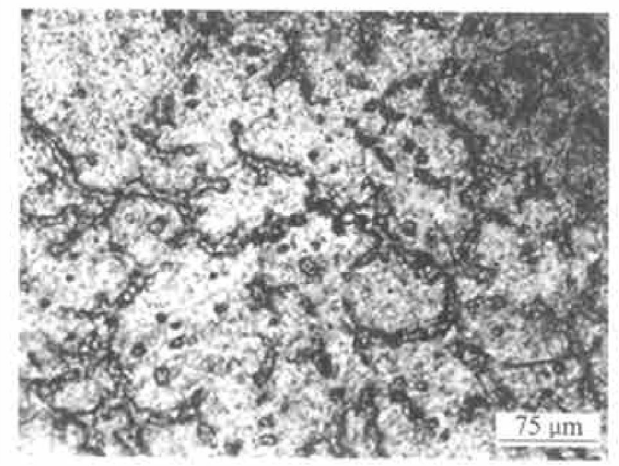

Fig. 3 Initial microstructure of as-cast AZ91 magnesium alloy

fiber-shaped stripe (Fig. 4(a)). When the temperature increases to 673 K, not only the elongated initial grain but the equiaxial grain of DRX can be observed (Fig. 4(b)).

The diffusion of atoms is necessary for nucleation and nucleus growth of recrystallization. Recrystallization is easy to occur in magnesium alloy due to the high diffusion rate of Mg atoms[12]. But the re-

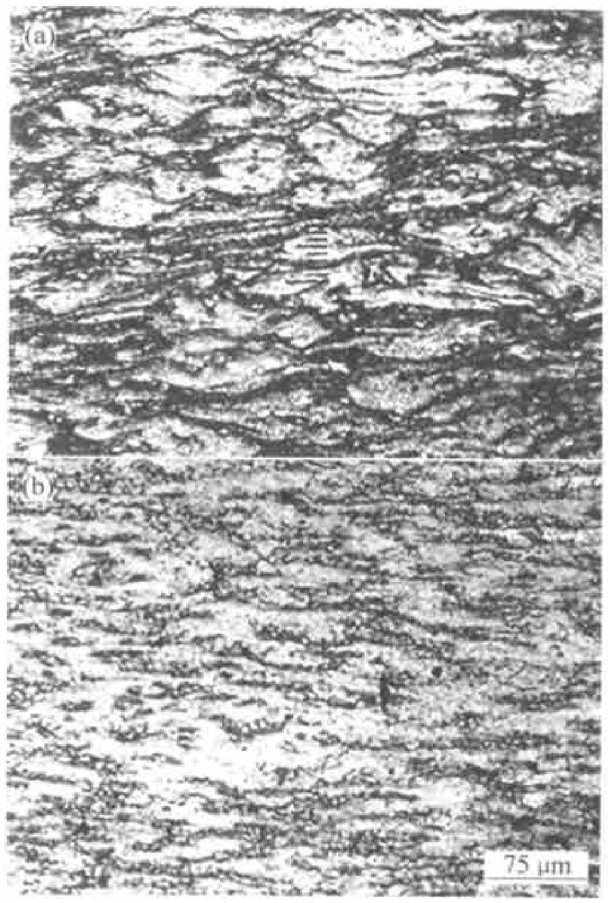

Fig. 4 Optical micrographs of Mg-alloy AZ91 compressed at different temperatures and constant strain rate of 0.05 s^{-1}
(a) —573 K; (b) —673 K

crystallization can't occur unless the temperature is high enough to activate atoms and make them remove. Therefore, the phenomenon of DRX can't be observed at 573 K. The reason may be either that the size of grain of DRX is too small to be observed, or that DRX doesn't occur at this temperature at all. However, when the temperature reaches 673 K, because of stimulated thermal activation, the abilities for diffusion of atoms, cross-slip of dislocations and migration of grain boundary are enhanced, and all these conditions are beneficial to nucleation and nucleus growth of DRX.

The microstructures of magnesium alloy compressed at different strain rates but at the same temperature show that DRX becomes more evident with the decrease of strain rate (Fig. 5). XUE et al[13] reported that before nucleation and nucleus growth, a certain incubation period is necessary for occurrence of DRX. At constant temperature, the higher strain rate means the shorter time for deformation, therefore the time isn't enough for nucleus to nucleate and grow. While at lower strain rates, there is enough time for DRX of alloy to proceed. This is coincident with the phenomenon that the stress-peak comes early with the decrease of strain rate. In Figs. 4 and 5, we also observe that nuclei of DRX preferentially form at

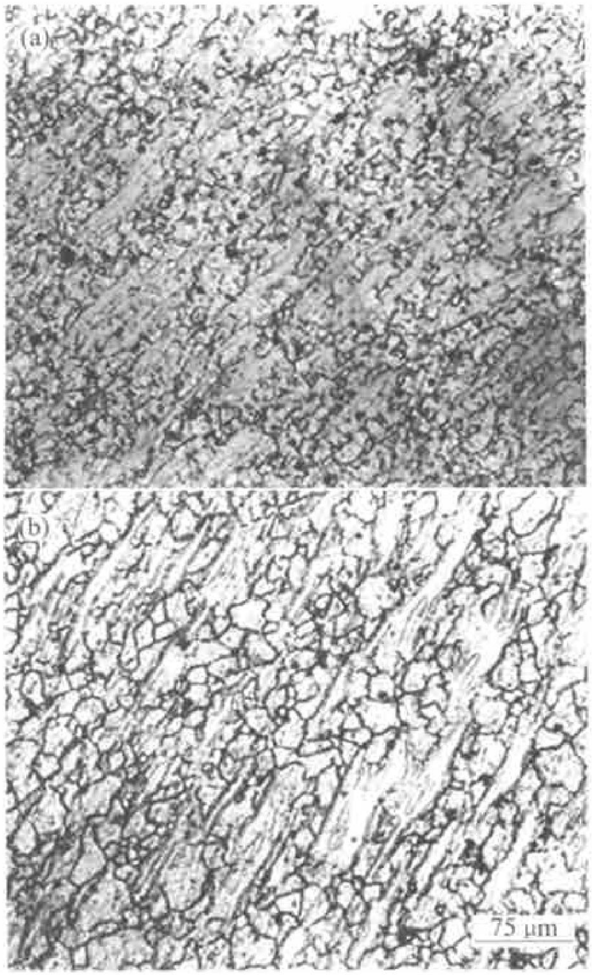

Fig. 5 Optical micrographs of Mg-alloy AZ91 compressed at different strain rates and constant temperature of 723 K
(a) —0.1 s^{-1}; (b) —0.01 s^{-1}

original grain boundaries, which coincides with the result in Ref. [3].

The microstructure observed by optical microscope shows that the DRX occurs when compression deformation is applied to AZ91 alloy, and that the phenomenon of DRX becomes more evident with increasing temperature and/or decreasing strain rate. It could be validated that the occurrence of peak stress in the true stress—strain curves and the variation of the strain corresponding to the peak stress with the variation of deformation temperature and strain rate are both involved with the DRX in AZ91 alloy.

TEM studies show that continuous DRX takes place in magnesium alloy during compression (Fig. 6). As depicted in Fig. 6(a), a lot of tangled dislocations form cellular substructures. At the boundaries of cells, besides the tangled dislocations, there are also some dislocation walls that consist of many parallel dislocations with the same sign. At the same time, we can observe some equiaxial dynamic recrystallized grains with grain boundary misorientation (Fig. 6 (b)). And in the interior of dynamic recrystallized grains, substructures formed by plenty of dislocation can be seen. The occurrence of dislocation substructures indicates that dynamic recovery has taken place in magnesium alloy when being compressed. The subgrain structure forms through the conversion of dislocation substructures into subgrain boundaries. The grain interior consists of a lot of dislocations and these dislocations further form substructures. All these phenomena depicted above reveal that when AZ91 magnesium alloy is compressed, dynamic recovery takes place, then subgrain structure form via the movement of dislocations in recovery structures. Dislocations pile up at subgrain boundaries, which results in progressive increase in subgrain boundary misorientation, until the formation of new grain boundary. This is so-called continuous recrystallization[14]. The continuous recrystallized grains occur preferentially in the vicinity of original boundaries where there are much more dislocations than interior of grain. With the development of deformation continuous recrystallization expands to grain interior. Some researches have previously indicated that continuous recrystallization would take place during the course of high temperature tensile test in magnesium alloy[15]. Continuous recrystallization is generally considered as a recovery-dominated process. Therefore continuous recrystallization occurs through gradual increase in grain boundary misorientation and conversion of low-angle

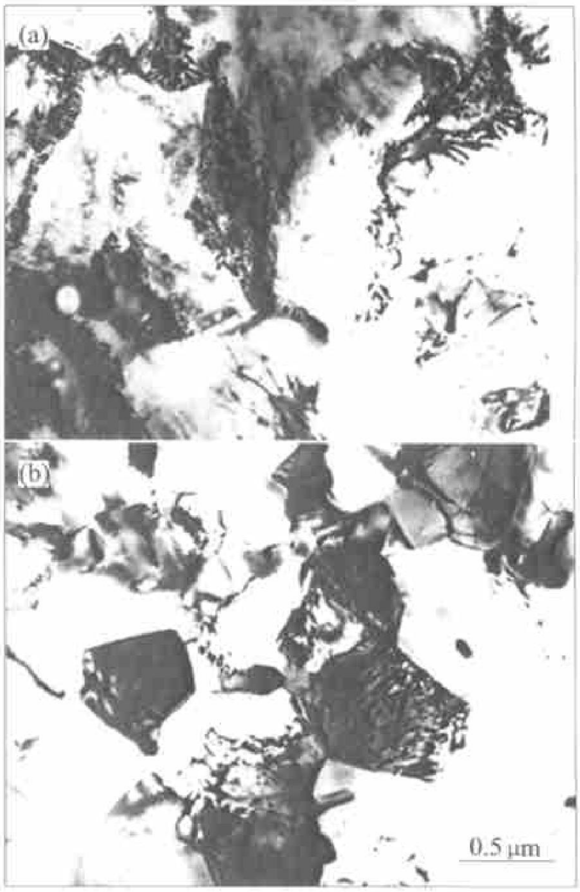

Fig. 6 TEM micrographs of recovery and recrystallization in AZ91 magnesium alloy under test conditions of 573 K and initial strain rate of 0.1 s^{-1}

grain boundaries into high-angle boundaries.

In AZ91 magnesium alloy studied in the present work, DRX takes place preferentially in the vicinity of original boundaries during compression (Fig. 4(b)). In the course of plastic deformation in polycrystal materials, grain boundary will prevent dislocations from moving, and these dislocations will pile up and subsequently generate a local stress concentration at the grain boundary. In order to further reduce the stress concentration, the pile-up dislocations in the vicinity of grain boundaries rearrange themselves to form groups of low-angle boundaries and dislocation cell structures (Fig. 6(a)), which leads to the development of dynamic recrystallization grains in the vicinity of original boundaries.

Dynamic recrystallization can be classified into either continuous or discontinuous recrystallization. In general, during continuous recrystallization, dislocations will remain in the recrystallized grains. In the present work, dislocations can be seen in both the interior and the boundaries of recrystallized grains, from which we can conclude that the DRX phenomenon in AZ91 magnesium alloy during high temperature compression is attributed to continuous dynamic recrystallization developing through conversion of low-angle grain boundaries into high-angle boundaries.

4 CONCLUSIONS

1) During high temperature compression, the true stress—strain curves exhibit stress peaks. This is attributed to the occurrence of dynamic recrystallization (DRX) or dynamic recovery.

2) Optical microscopy shows that DRX occurs preferentially in the vicinity of original boundaries, and that higher temperature or lower strain rate is advantageous to occurrence of DRX.

3) TEM examinations confirm that the DRX phenomenon in AZ91 magnesium alloy during high temperature compression is attributed to continuous dynamic recrystallization that involves the conversion of low-angle grain boundaries into high-angle boundaries.

REFERENCES

[1] LIN Jun-pin, CHENG Jing-wei. Dynamic recrystallization during hot torsion of Al-2Mg alloy[J]. The Chinese Journal of Nonferrous Metals, 1999, 9(3): 510-514. (in Chinese)

[2] Tan J C, Tan M J. Dynamic continuous recrystallization characteristics in two stage deformation of Mg-3Al-1Zn alloy sheet [J]. Materials Science and Engineering, 2003, A339(1-2): 124-132.

[3] Myshlyaev M M. Twinning, dynamic recovery and recrystallization in hot worked Mg-Al-Zn alloy[J]. Materials Science and Engineering, 2002, A337(1-2): 121-133.

[4] Yang Xu-yue, Miura Hiromi, Sakai Taku. Dynamic evolution of fine grain structure during hot deformation of an AZ31 magnesium alloy[J]. Light Metals, 2002, 52(7): 318-323. (in Japanese)

[5] Tan J C, Tan M J. Superplasticity and grain boundary sliding characteristics in two stage deformation of Mg-3Al-1Zn alloy sheet[J]. Materials Science and Engineering, 2003, A339(1-2): 81-89.

[6] Galiyev A, Kaibyshev R, Gottstein G. Correlation of plastic deformation and dynamic recrystallization in magnesium alloy ZK60[J]. Acta Mater, 2001, 49(7): 1199-1207.

[7] Bussiba A, Artzy A Ben, Shtechman A, et al. Grain refinement of AZ31 and ZK60 Mg alloys—towards superplasticity studies[J]. Materials Science and Engineering, 2001, A302(1): 56-62.

[8] Ion S E, Homphreys F J, Whice S H. Dynamic recrystallization and the development of microstructure during the high temperature deformation of magnesium[J]. Acta Metall, 1982, 30(10): 1909-1919.

[9] YU Kun, LI Wen-xian, WANG Ri-chu, et al. Hot compressive deformation simulation of Mg-5.6Zn-0.8Nd alloy at elevated temperatures [J]. Acta Metallurgica Sinica, 2003, 39(5): 492-498. (in Chinese)

[10] McQueen H J. Comment on "On the generation of thixotropic structures during melting of Mg-9% Al-1% Zn alloy" by F. Czerwinski[J]. Scripta Materialia, 2003, 49(9): 917-920.

[11] Annual Book of ASTM Standards, Section 3: 03.01, E112-96[S]. Philadelphia, PA: American Society of Testing and Materials, 1998, 229.

[12] Mabuchi M, Higashi K. Strengthening mechanisms of Mg-Si alloys[J]. Acta Mater, 1996, 44(11): 4611-4618.

[13] XUE Ke-min, LI Ping, LÜ Yan. Microstructure of Ti-15-3 alloy after high temperature deformation[J]. Journal of Harbin Institute of Technology, 2002, 34(3): 385-388. (in Chinese)

[14] TIAN Wen-Tong, LUO Shou-jing, ZHANG Guang-an. Yield stress of LC4 alloy in semi-solid state[J]. The Chinese Journal of Nonferrous Metals, 2002, 12(1): 82-86. (in Chinese)

[15] Mohri T, Mlbuchi M. Microstructural evolution and superplasticity of rolled Mg-9Al-Zn[J]. Materials Science and Engineering, 2001, A290(1-2): 139-144.

(Edited by PENG Chao-qun)