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Interaction of crack with twins in hot-rolling AZ31 magnesium alloys during in-situ TEM straining experiments

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Abstract: In-situ straining experiments in transmission electron microscopy were performed to investigate the propagation of crack, twinning and interaction of crack with twins. Lots of micro-twins were observed in the deformation band near the tip of crack. The stress is relaxed by the formation of micro-twins and the densely developed micro-twins prevent the growth of crack from the deformation band, subsequently the cracks extend into adjacent grains across grain boundaries, and grow along cleavage plane. Multiple twins are observed in hot-rolling AZ31 magnesium alloys, when cracks propagate into twins through twin grain boundaries(GBs), the direction of growth deviates. It is concluded that twins, especially multiple twins effectively baffle the development of crack.

Key words: multiple twins; in-situ TEM experiments; crack; AZ31 Mg alloys

1 Introduction

Magnesium alloys with low-density (1.7 g/cm^3), high specific strength, high stiffness and high elastic modulus ratio, are a research focus field in recent years. Early magnesium alloys have been used mainly in the aerospace field, but now are widely used in automobile industry[1−3].

The ductility of magnesium alloys at room temperature is poor. Useful methods to improve the ductility, such as elevated temperature, fine grain size, and solute additions, have been carried out[4−7]. All of these can be used to activate non-basal slip. However, the common twinning modes provide for deformation along the *C*-axis and thus can be expected to compete with any non-basal slip that has a Burgers vector with a component in the *C*-direction[6−7]. A number of investigations show the favourable effect of twinning on the ductility of hcp metal. In the hot-rolling AZ31 magnesium alloys, there exist plenty of twins. It is significant to further study microstructure evolution in the process of tensile deformation, twin generation, interaction of twins with crack for in-depth understanding the deformation mechanism of wrought magnesium alloys in room temperature and exploring ways to improve ductility.

The slip in magnesium alloy has been investigated by COURET and CAILLARD[8] using in-situ tension device in TEM. In this study, an in-situ transmission electron microscopy tension apparatus was employed to study twins, twinning and interaction of crack with twins in wrought magnesium alloys AZ31. The purpose of the present study is to highlight the role that deformation twinning plays in the tension deformation. It is thereby hoped that this will be helpful to further understanding the deformation mechanisms of the alloys, initiation of crack, and the interaction of twins and crack.

2 Experimental

The wrought alloy AZ31 was used as the experimental material with the following composition of Mg-3Al, 0.8Zn, 0.4Mn (in mass fraction, %). The alloy ingot molded at 730 ℃ was placed in a resistance furnace

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for solution treatment for 8 h, and then extruded into a sheet at 400 ℃ employing a 1 250 t horizontal extruder. Subsequently, the material was rolled into plate with a thickness of 0.8 mm.

The foils for in-situ tensile test in TEM were thinned by conventional twin jet polishing technique using an electrolyte ingredient of 11.2 g dehydrite, 5.3 g lithium chloride, 100 mL ethylene glycol monobutyl ether and 500 mL methanol at −20 to −30 ℃. In order to wipe off the oxide layer, foils were polished with a GANTAN 691 Ion Polishing System at 2.5 kV for 3−5 min[9]. A HITACHI H-800 transmission electron microscope with the in-situ tensile apparatus at 100 kV was employed for the morphology observation.

3 Results and analysis

3.1 Microstructure after rolling

According to Fig.1, after multiple pass hot rolling, a lot of deformation twins appear in the alloys. As shown in Fig.1(a), two twins intersect each other, and a high density of dislocations is observed within both twins; in Fig.1(b), multiple parallel twins are observed, and within twin *A*, dislocations tangle forms small-angle grain boundaries. The high density of dislocations in twins can promote dynamic recrystallization and refine grains[10−11].

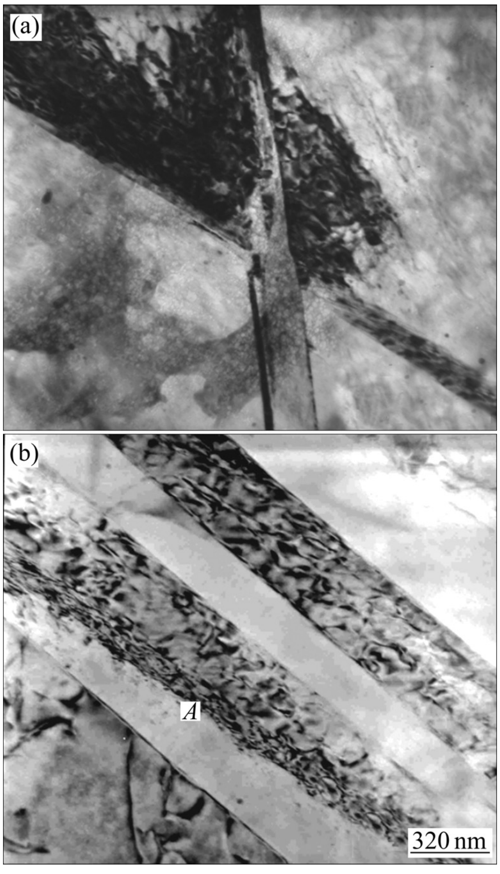

Fig.1 Twins existing in alloy after rolling

Due to HCP structure, there are a few slip systems activated during tensile deformation, *a* and *c* types dislocation, as well as (*a*+*c*) dislocation activated on both basal and non-basal planes[12−16]. Therefore, the poor ductility of magnesium alloys is attributed to the highly anisotropic dislocation slip behavior. Under the different diffraction conditions, the dislocation configuration near the $\{10\bar{1}2\}$ twin boundaries is shown in Fig.2. The Burgers vectors of dislocations may be determined according to contrast analysis. In $\boldsymbol{g}=01\bar{1}0$ operation reflections (Fig.2(a)), the dislocations are visible, but in $\boldsymbol{g}=\bar{1}101$ (Fig.2(b)) and $\boldsymbol{g}=\bar{1}102$ (Fig.2(c)), they are out of contrast. According to the $\boldsymbol{g}\cdot\boldsymbol{b}=0$ invisibility criterion, the dislocations have Burger vectors of $\boldsymbol{b}=1/3[1\bar{2}10]$.

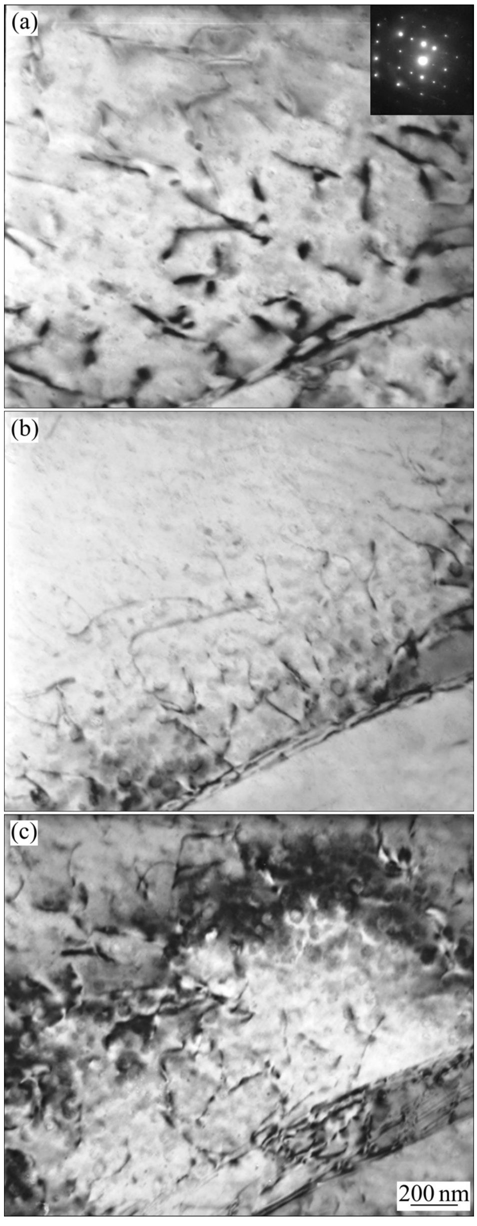

Fig.2 Dislocations configuration near $\{10\bar{1}2\}$ twin boundaries: (a) $\boldsymbol{g}=01\bar{1}0$; (b) $\boldsymbol{g}=\bar{1}101$; (c) $\boldsymbol{g}=\bar{1}102$

3.2 Extension of crack and twinning

During the tensile test in TEM, it could be observed that with stress increasing, the cracks were initiated and grew up in the foils. As shown in Fig.3(a), a crack extends forwards and intersects with a twin, and the existence of twin boundaries make the direction of crack propagation deviate. The crack within twins is quite straight, which indicates that cracks within twins extend along the cleavage plane. After the crack traverses the twin, the direction of propagation is perpendicular to the tensile stress.

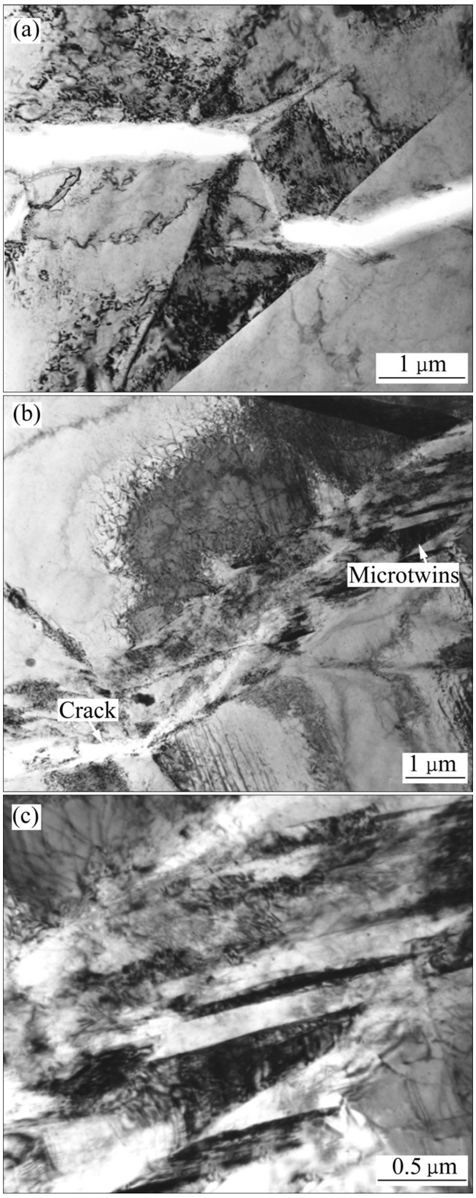

Fig.3 Extension of cracks and twinning under tension stress

Lots of micro-twins are observed at the tip of crack, as shown in Figs.3(b) and (c). As the micro-twins appear, the stress near the tip of crack will be relaxed, and the deformation of alloy increases, which retards the expansion of crack. In some cases, the direction of crack propagation changes, even not extends forwards and some small cracks are initiated in the other parts of the foil.

The generation of micro-twins near the tip of crack shows that, on one hand, the local stress concentrates near the tip of crack in in-situ TEM experiments, on the other hand, the generation of micro-twins relaxes the stress, coordinates the deformation of alloys and retards the expansion of crack forwards.

3.3 Extension of crack and fracture

As shown in Fig.4(a), with the increasing of tension stress, magnesium alloys will fracture. The initiation and growth of crack generally occur in the transgranular, and intergranular fracture, as shown in Fig.4(b), can hardly be observed, which indicates that the failure of the alloys is generally the models of transgranular fracture.

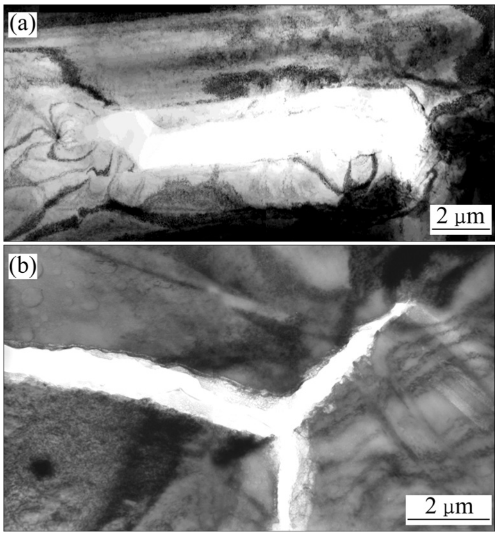

Fig.4 Fracture models under tension stress

4 Results and discussion

Deformation mechanisms of magnesium alloys are only $\langle a \rangle$ and $\langle a+c \rangle$ dislocation basal and non-basal slip. In order to fulfill Von Mises criterion, twinning occurs. When dislocations slip are insufficient to guarantee the uniformity of deformation, with the concentrated stress rising, twinning appears and coordinates the plastic deformation. It can be deduced that as an additional deformation mechanism, twinning can improve the HCP structure metal material deformation at room temperature. Under tension stress, if the orientation of grains is unfavorable to dislocations slip, twinning will occur to coordinate deformation. Although twin boundaries prevent dislocations movement, twinning makes grain orientation change slightly, which can activate dislocations slip in the twins. As shown in Fig.1(b), dislocations tangle in twin A, and small angle grain boundaries come into being. A large number of dislocations in twins can serve as dynamic recrystallization nuclei, which extend dynamic recrystallization temperature range, and are propitious to refine grains.

For metal material, due to stress concentration, near the tip of the cracks there exists high local stress, once the local stress exceeds the yield strength of materials, plastic deformation occurs inevitably. In the crack tip there exists plastic zone where crack extension must first pass through. The energy of crack propagation consume mainly on the plastic deformation. In in situ TEM tensile tests, the foils are at a plane stress state, σ_z=0, τ_{xy}=τ_{yz}=0, and only σ_x, σ_y, τ_{xy} three stress components are applied on the sample planes. The crack is an open type, and the applied stress is perpendicular to the crack plane. Under normal stress, the crack tip is open, the propagating direction is perpendicular to the stress, and the local stress near the crack tip is

$$\sigma_{ij}=\frac{k_1}{(2\pi r)^{1/2}}f_{ij}(\theta)$$

In the course of crack expansion, due to the local stress near the crack tip, the dislocations slip is first actived in the deformation zone. When the dislocations slip is insufficient to coordinate deformation, the large number of micro-twins arise, which absorb the energy of crack propagation, relax stress concentration, and coordinate deformation.

Investigation to dislocations in the vicinity of twin boundaries shows that the deformation mechanisms of the alloy are mainly basal slip. As the basal slip is incoordinate to the uniformity of alloy deformation, the concentration of stress induces twinning, which coordinates the emergence of deformation. It can be deduced that as an additional deformation mechanism, twinning can increase the deformation of the HCP structure metal material, and improve its plasticity at room temperature. Because of the concentration of stress, plenty of micro-twins will appear near the tip of crack, which effectively relaxes stress, and coordinates the deformation.

5 Conclusions

1) $\langle a\rangle$ dislocations slip in the basal planes plays main roles for magnesium alloys deformation. If the dislocations slip is hampered, twinning will occur to coordinate deformation. Under tensile stress in in-situ TEM tests, near the tip of crack, large amounts of micro-twins can be observed, which indicates the emergence of stress concentration. They coordinate alloys deformation, relax the stress, absorb the potential energy of crack expansion, retard the expansion of crack and improve the plasticity of the material.

2) The presence of twins prolongs the path of crack, and retard the growth of the crack.

3) The failure of wrought magnesium alloys AZ31 under the tensile stress in in-situ TEM test generally exhibits the models of transgranular fracture.

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