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FORMATION OF NANO-SCALE TWINS AND LOW ANGLE GRAIN BOUNDARIES DURING FRACTURE OF A FINE GRAINED MAGNESIUM ALLOYS

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Abstract

Fine grained magnesium alloys show high fracture toughness, associated with void formation. Detailed microstructural evolution during a fracture toughness test of a fine grained Mg-Zn alloy has been studied here by transmission electron microscopy (TEM). Focused ion beam (FIB) technique was used for obtaining samples near crack. Initially, subgrain structures form ahead of the crack tip, after which $\{10\bar{1}1\}$ type twins of width of about 400nm form. They further twin by $\{10\bar{1}2\}$ twinning. Subsequent twinning occurs at a finer scale near the crack, forming configurations of $\{10\bar{1}1\}$-$\{10\bar{1}2\}$ double twins and low angle boundaries. The scale of the twin domains became progressively finer to less than 50nm. In absence of enough dislocations to pile up causing fracture, deformation continues to occur by the twinning.

Introduction

Fracture toughness in an important criteria in deciding the use of a material for structural application. Fracture toughness of magnesium alloys are known to be low [1]. Formation of $\{10\bar{1}2\}$ type of twins at the beginning of the deformation [2, 3] is a cause of low fracture toughness. These twins become the crack propagation route [4]. However, grain refinement of magnesium alloys can reduce the formation of twins [5]. Consequently, the fracture mechanism changes from brittle to ductile. Ductile fracture is associated with void formation. Thus the fracture toughness of magnesium alloys can be increased by grain refinement [6, 7].

In a single crystal strained in tension parallel to the basal plane, fracture was found to occur primarily by twinning, identified to be $\{10\bar{1}1\}$-$\{10\bar{1}2\}$ double twinning [8]. Reduction of a single crystal along its c-axis is accommodated by twinning on $\{10\bar{1}1\}$ and retwinning on $\{10\bar{1}2\}$, followed by a basal shear [9]. Compression perpendicular to unconstrained c-axis activates $\{10\bar{1}2\}$ twinning [10].

Ductile fracture and high fracture toughness in magnesium alloys are associated with blunt crack tip of fracture. We reported on a high fracture toughness in a fine grained magnesium alloy and the deformation structures associated with it [11]. From the partly tested and tested to fracture samples of a three point bending specimen, samples for transmission electron microscopy (TEM) were cut out from specific sites by focused ion beam (FIB). Details of these investigations are reported here, in order to understand the fracture mechanism in a fine grained magnesium alloy.

Experimental Procedure

A Mg-2.4at%Zn alloy was cast and then extruded at 473K. The extruded alloy had grain size in the range of 1-3μm. Nano-sized precipitates of rounded shape were dispersed inside the grains. Details of initial microstructure and dimpled fracture surface showing ductile character are reported elsewhere [12]. Three-point bending specimens of length 10mm and width 5mm were machined from the extruded bar and a precrack inserted by fatigue. TEM specimens were sampled from specific sites by FIB. A specimen was cut ahead of the crack after an interrupted test. Another was selected from under a void ahead of the crack, and a third from under the crack. These specimens were observed by high resolution TEM (HRTEM) using a JEOL 4000EX microscope operated at 400 kV.

Results

Fig. 1(a) shows a micrograph from the sample taken ahead of the crack tip, in an interrupted fracture toughness test. A large strain-like contrast

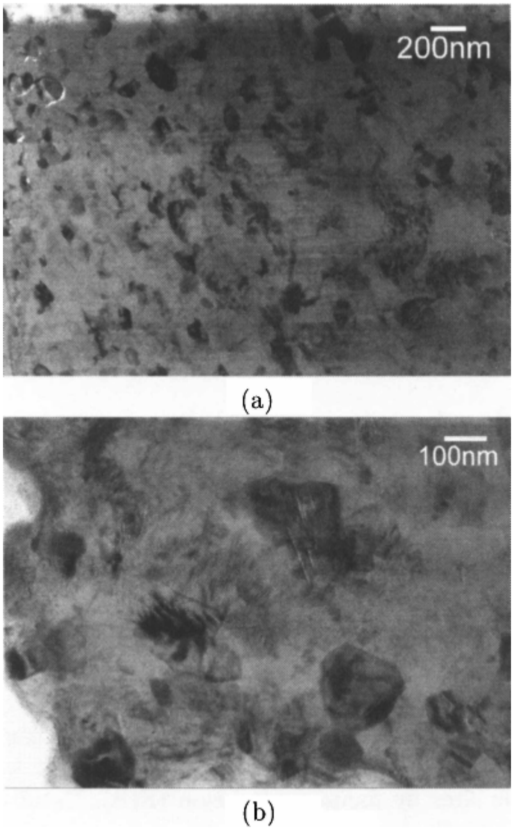


Figure 1: Bright field electron micrographs showing grain and deformation structures (a) ahead of the crack tip and (b) bottom of a void.

is observed, especially in the right of the micrograph. There are dark particles of 100-150nm in size, which are Mg-Zn binary phase believed to be of the monoclinic Mg_4Zn_7 phase. Some apparently recrystallized grains of size less then 200nm are also observed.

In the sample taken from the bottom of a void, Fig. 1(b), large strains are also present. These strained regions are more localized than in the sample taken ahead of the crack. Some recrystallized grains are observed too.

Fig. 2 shows the deformation structure just below a crack. A part of the crack surface is visible in the upper right corner. Based on the deformation structures, three regions could be identified in this sample - away from the crack surface is a region marked I, in which parallel twins, two marked by arrowheads, are identified. From diffraction patterns, these twins were identified to be $\{10\bar{1}1\}$ type twins. The width of these twin bands is similar, of about 400nm. A large strain contrast is evident in this region. In the region marked II, elongated domains parallel (in the direction marked by an arrow) to the crack surface. These domains have sharp boundaries. Closest to the crack surface is the region marked III, in which nano-grains of size about 50nm occur.

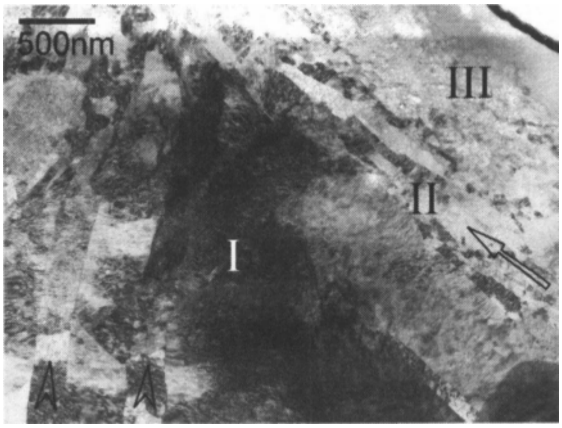
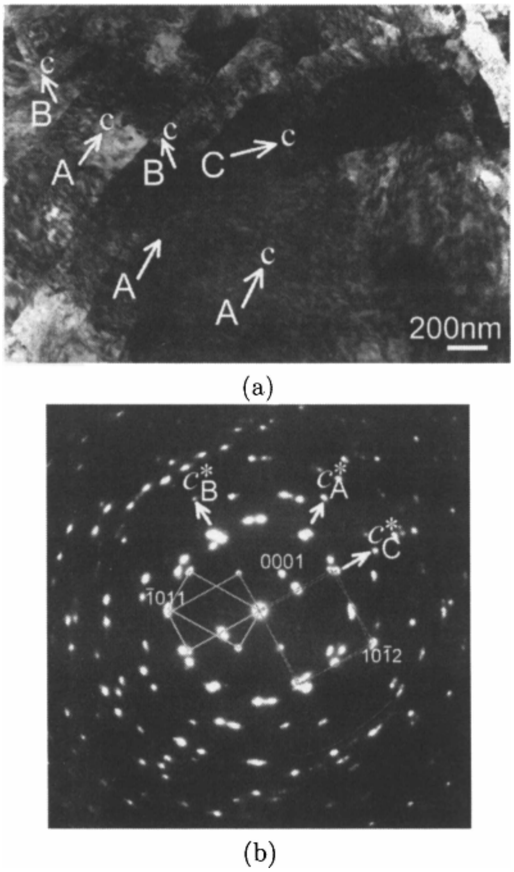


Figure 2: Bright field electron micrographs showing a region just below the crack surface.

The $\{10\bar{1}1\}$ twins parallel to each other observed in Fig. 2 twin further, to make double twins $\{10\bar{1}1\}$-$\{10\bar{1}2\}$. An example is shown in Fig. 3. All domains in this micrograph are in zone axis $\langle 11\bar{2}0\rangle$. These domains are rotated with respect to each other about this zone axis. The direction of the c axis in each domain is marked by an arrow. Diffraction patterns showed that the domains marked 'A' and 'B' are $\{10\bar{1}1\}$ type twins (angle between the c^* axes of about 56°). In between, a third twin marked 'C' is observed. 'C' makes a $\{10\bar{1}2\}$ twin with 'B' (angle between the c^* axes of about 86°). Thus A, B and C make a double twin. This is observed in the composite diffraction of Fig. 3(b). c^* axes of all the three twin domains are marked in this diffraction pattern. There is a coincidence of $\{10\bar{1}1\}$ spots of A and B, confirming a $\{10\bar{1}1\}$ type twin. A $\{10\bar{1}2\}$ of domains B and C coincide, making a $\{10\bar{1}2\}$ type twin.

Towards the crack surface, the twins multiply and become finer. Boundaries between the domains are low angle boundaries (LAB) or twins. Double twin configurations were often observed. The long domains in the region II were similarly oriented with



(a)

(b)

Figure 3: Bright field electron micrographs showing grain and deformation structures (a) ahead of the crack tip and (b) bottom of a void.

respect to the neighboring domains. This structure became finer in the region III, in which the domain size was about 50 nm in size. In this region, the boundaries between the domains were no longer planar, even if a twin orientation occurred. A domained region is shown in Fig. 4, in which all domains are oriented along one of their $\langle 11\bar{2}0\rangle$ axes. The domains are marked M to S. Strained regions within the domains are often observed. A LAB exists between M and N, on which a regular arrangement of dislocation contrast is visible. In this case, the LAB is formed by 0001 planes. LABs in other orientations were also observed, such as between the domains M and R.

Fig. 5 shows two LABs from the domains in Fig. 4. The LAB between N and M resembles a classic LAB with an array of dislocation contrasts marked by while arrows. This boundary is made up by the basal planes in the two domains. The angle of the boundary is 6°, which can be precisely measured in the FFT. Another LAB shown in Fig. 5(c,d) is, in some part, perpendicular to the basal planes of M. The basal planes in these two domains M and R make an angle 8°.

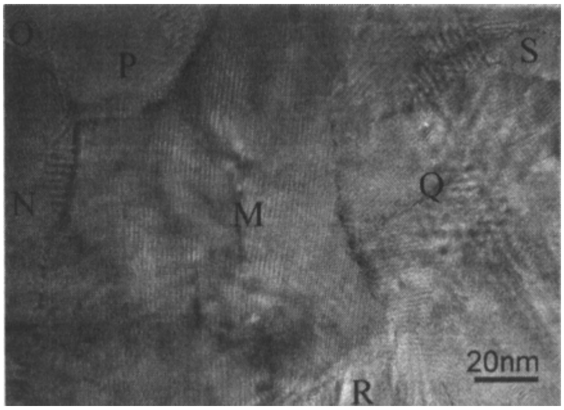


Figure 4: A lattice image showing nano-domains close to the crack surface.

Fig. 6(a) shows the interfaces between M, O and P in detail. A Fast Fourier Transform (FFT) of the domains M and O (Fig. 6(b) shows that they form a $\{10\bar{1}1\}$ type twin. The coincident $\{10\bar{1}1\}$ spots from the two domains are marked by circles in (b). The boundary between the two domains is sharp, planar and symmetrically oriented along the common $\{10\bar{1}1\}$ plane, as a twin boundary. A section of the interface between domains M and Q is shown in Fig. 6(c). The FFT in (d) indicates that these two domains form a $\{10\bar{1}1\}$ type twin. The shown section of the interface is roughly planar, but on the (0001) plane of M.

Inside domain Q is embedded another domain S, shown in detail in Fig. 6(e). The accompanying FFT in (f) shows them to be related by $\{10\bar{1}2\}$ type twinning (coincident $\{10\bar{1}2\}$ spots are encircled in (f)). The lower interface is roughly parallel to the common $\{10\bar{1}2\}$ plane; however, the upper part is highly curved.

Discussion

In a coarse grained magnesium alloy ($> 20\mu$m), the dominant deformation mechanism is twinning, which also become crack propagation route, resulting in a brittle fracture. The dominant deformation

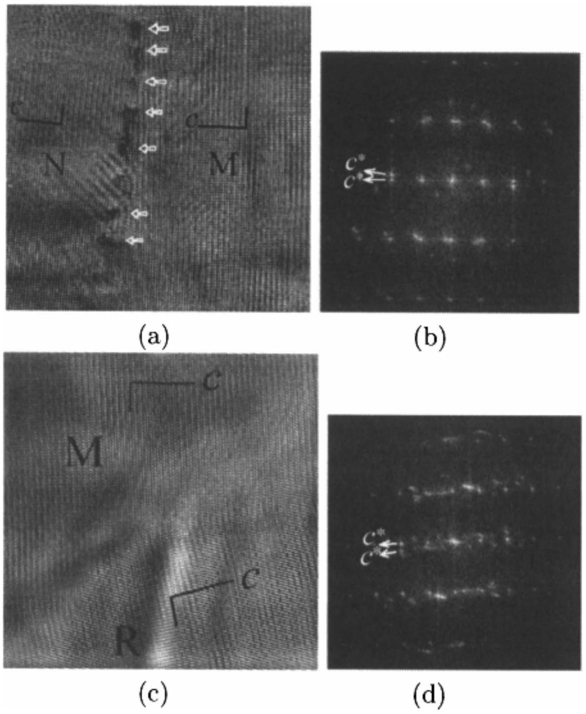


Figure 5: Two examples of low angle boundaries shown in Fig. 4 and their Fast Fourier Transforms. A low angle boundary between domains M and N (a,b) and between domains M and R (c,d).

mechanism in a fine grained alloy is dislocation slip. In the beginning of deformation, subgrain structures form ahead of the crack tip, instead of twins. The crack tip blunts because no crack propagation features such as twins exist. This causes high fracture toughness. In addition, voids are nucleated due to stress, by cracking of particles/precipitates or interfaces. Nano-twins are formed due to operation of large stresses around the voids [11].

The slip activity which results in formation of sub-grain like structure, in turn, raises the stress for twin formation. At large stresses required for fracture, formation of $\{10\bar{1}1\}$ type twins occurs. However, it is known that the $\{10\bar{1}1\}$ twins generate substantial stress in the matrix, which are then relieved by formation of $\{10\bar{1}1\}$-$\{10\bar{1}2\}$ double twins.

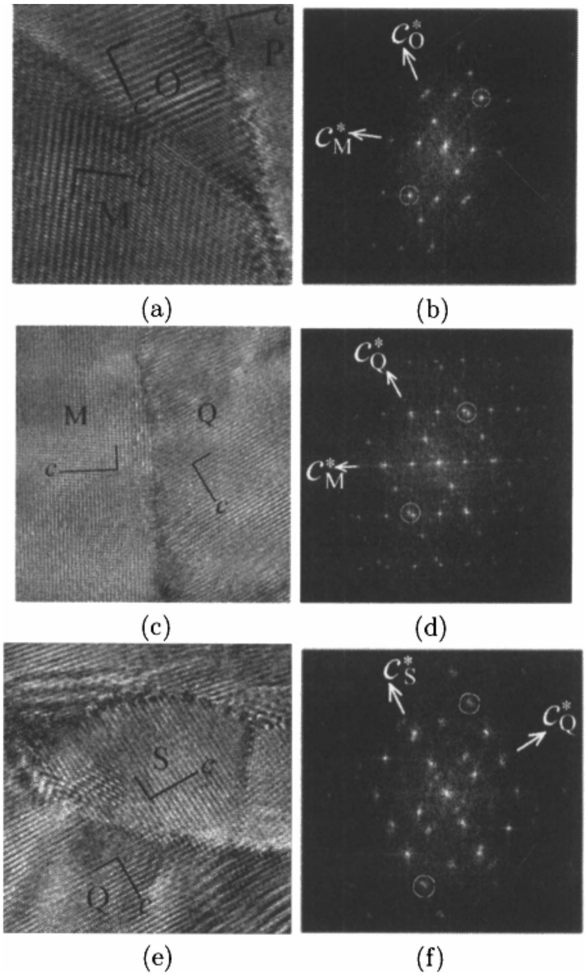


Figure 6: Some twin boundaries from the domains shown in Fig. 4 and their Fast Fourier Transforms. (a,b) $\{10\bar{1}1\}$ type twin between domains M and Q. (c,d) $\{10\bar{1}1\}$ type twin between domains M and O. (e,f) $\{10\bar{1}2\}$ type twin between domains Q and S.

Our earlier study showed that fracture in a fine grained magnesium alloy occurred by twinning, followed by slipping which caused accumulation of stresses on the twin boundaries [13]. In the present study, however, the domain size are so small that there is not enough slip activity to accumulate stresses on the boundaries. Therefore, deformation progresses by further twinning. To put this in another perspective, continuous twinning is able to relieve the stress necessary for fracture. In this, the role of LABs is crucial. LABs sustain the stresses formed between the various configurations of the twins.

Conclusions

Fracture mechanism during fracture toughness test has been investigated in a fine-grained Mg-Zn alloy with transmission electron microscopy by sampling specimens from selected sites by focused ion beam.

It was concluded that

1. Ahead of the crack, only subgrains formed, instead of twins. This gives rise to a ductile fracture, instead of a brittle one observed in large grained alloys.

2. At high stresses, $\{10\bar{1}1\}$ type twins formed, with width of about 400nm. These twins further twinned to form $\{10\bar{1}1\}$-$\{10\bar{1}2\}$ double twins.

3. In absence of sufficient slip activity in the subgrains/twins, deformation proceeded by further twinning, leading to smaller domains of 50nm.

4. Thus, before forming a crack, a microstructure consisting of nano-domains related to each other by twinning or low angle boundaries is formed.

References

[1] H. Somekawa, A. Singh, T. Mukai, "High fracture toughness of extruded Mg-Zn-Y alloy by the synergitic effect of grain refinement and dispersion of quasicrystalline phase", *Scripta Metallurgica* 56 (2007) 1091.

[2] H. Yoshinaga, R. Horiuchi, "", *Mater. Trans. JIM* 4 (1963) 1.

[3] J. Koike, Metall. "Enhanced deformation mechanisms by anisotropic plasticity in polycrystalline Mg alloys at room temperature", *Mater. Trans.* 36A (2005) 1689.

[4] H. Somekawa, A. Singh, T. Mukai, "Fracture mechanism of a coase-grained magnesium alloy during fracture toughness testing", *Phil. Mag. Lett.* 89 (2009) 2.

[5] M. A. Meyers, O. Vohringer, V. A. Lubarda, "The onest of twinning in metals; A constitutive description", *Acta metallurgica* 49 (2001) 4025.

[6] H. Somekawa, T. Mukai, "Effect of grain refinement on fracture toughness in extruded pure magnesium", *Scripta Metallurgica* 53 (2005) 1059.

[7] H. Somekawa, H. S. Kim, A. Singh, T. Mukai, "Effect of precipitate shapes on fracture toughness in extruded Mg-Zn-Zr magnesium alloys", *J. Mater. Res.* 22 (2007) 965.

[8] W.H. Hartt and R.E. Reed-Hill, Trans. TMS-AIME 239 (1967) 1511.

[9] B.C. Wonsiewics and W.A. Backofen, Trans. TMS-AIME 239 (1967) 1422.

[10] E.W. Kelly and W.F. Hosford, Jr., Trans. TMS-AIME 242 (1968) 5.

[11] H. Somekawa, K. Nakajima, A. Singh, T. Mukai, Philo. Mag. Lett. 90 (2010) 831.

[12] H. Somekwa, Y. Osawa, A. Singth, T. Mukai, J. Mater. Res.23 (2008) 1128.

[13] V. Gartnerova, A. Singh, A. Jager and T. Mukai, "Deformation behaviour of ultra-fine-grained Mg-0.3at%Al alloy in compression" (to be communicated)