Scripta Materialia 142 (2017) 50–54

Contents lists available at ScienceDirect

Scripta Materialia

journal homepage: www.elsevier.com/locate/scriptamat

Regular article

Crack propagation along grain boundaries and twins in Mg and Mg–0.3 at.%Y alloy during in-situ straining in transmission electron microscope

Dudekula Althaf Basha, Hidetoshi Somekawa, Alok Singh*

Research Center for Structural Materials, National Institute for Materials Science, Sengen 1-2-1, Tsukuba 305-0047, Japan

ARTICLE INFO

Article history:
Received 7 June 2017
Received in revised form 13 July 2017
Accepted 14 August 2017
Available online xxxx

Keywords:
Magnesium alloys
Transmission electron microscopy (TEM)
Crack propagation
Twinning
Grain boundary segregation

ABSTRACT

In-situ TEM imaging has been carried out on thin foils of pure Mg and Mg-0.3at.%Y alloy under tension, to study crack nucleation and propagation. During propagation along grain boundaries, twin nucleation occurred from the crack tip. In case of pure Mg, the crack did not propagate along the twin boundaries but continued along the grain boundaries. However, in case of Mg-0.3at.%Y alloy, in which the grain boundaries were segregated with yttrium, the crack propagated along newly formed twin boundaries nucleated at the crack tip. This directly demonstrates the effect of alloying on the fracture behavior.

Magnesium alloys, which have a very high specific strength, have large potential to be used as structural materials. With their excellent specific strength and stiffness, machinability, dimensional stability and recycling capability, they are attractive for many applications in automobile, aviation, electronic and communication industries [1–3]. The hexagonal close-packed (hcp) structured magnesium alloys exhibit low ductility at room temperature (RT) as well as plastic deformation anisotropy owing to large differences between the activation energies of the different slip systems [4–7]. At room temperature (RT), predominantly basal and prismatic slips of $\langle a \rangle$ type dislocations are activated. The $\langle c + a \rangle$ slip has a high critical resolved shear stress (CRSS), and therefore it is difficult to activate at RT. Therefore twinning is an essential deformation mode to accommodate dislocation plasticity and strains in the $\langle c \rangle$ direction. Of the two types of twins frequently observed in magnesium alloys, the $\{10\bar{1}2\}\langle 10\bar{1}\bar{1} \rangle$ type results in tension in the $\langle c \rangle$ direction and the $\{10\bar{1}1\}\langle 10\bar{1}2 \rangle$ type causes a contraction in the $\langle c \rangle$ direction [8–12]. The former type is activated more readily.

Ductility and toughness are also related to twinning. Twin boundaries can become propagation path of crack, limiting the ductility [13,14]. Reducing the grain size has blunting effect on the crack tip by creation of nano-structures, including nano-twins, at the tip [15,16]. Addition of alloying elements can change the fracture toughness by solid solution strengthening and grain boundary segregation [17]. It has also been shown that coherent twin boundaries, low-angle boundaries and coincident site lattice (CSL) boundaries show higher fracture resistance [18–21]. Deformation twinning can be nucleated near or away from crack tip during its propagation due to the accumulation of high local stress under high strain rates and low temperatures [22,23].

However, it is extremely difficult to attribute the bulk failure of a specimen to the crack nucleation and propagation along twin boundary without direct observation. Nucleation of twins in grain interior from the crack tip during crack propagation has been reported through in-situ TEM imaging, such as in molybdenum [22]. In-situ TEM studied on magnesium alloys have been mostly on single crystals, such as in pillar forms [23,24] or as thin films [25,26], which do not show the effect of microstructure, such as grain boundaries. Direct experimental observation of twin nucleation from a grain boundary during crack propagation is challenging. In this report, for the first time, the nucleation of twin from the grain boundary during crack propagation has been shown through in-situ TEM tensile study in pure Mg and Mg-0.3at. %Y alloy specimens. The crack takes different paths in these two alloys, depending on the relative energies of grain boundaries and twins. Although not completely representative of the bulk material due to its low thickness, these experiments can give valuable insights into deformation and failure mechanism.

* Corresponding author.
E-mail address: alok.singh@nims.go.jp (A. Singh).

http://dx.doi.org/10.1016/j.scriptamat.2017.08.023
1359-6462/

Pure Mg and Mg-0.3at.%Y alloys were produced by casting, and then solution treated at a temperature of 773 K for 7.2 ks. The solution-treated alloys were extruded into a plate shape at 673 K, with a reduction ratio of 19, followed by annealing at 623 K for 172.8 ks. Specimens with dimensions 3.5 mm in length, 1.5 mm wide and 70 μm thick were prepared by cutting using a low speed saw equipped with diamond coated blade and grinding. The length was cut along the extrusion direction, which became the tensile axis during straining. These specimens were then ion milled to perforation, for observation in TEM. A Gatan single-tilt in-situ straining TEM holder was used for carrying out the experiments, performed on a JEOL 2000FX microscope. Sample geometry and calculated stresses are detailed in the Supplementary section. Post mortem ex-situ TEM experiments were conducted on a FEI-Tecnai G2 F30 microscope operating at 300 kV. Microstructure observations were performed both in conventional CTEM and in STEM mode with a probe size of 2 nm. Energy dispersive X-ray spectroscopy (EDS) was performed in STEM mode. The misorientations among neighboring grains were determined as described in the Supplementary section. Five samples each of pure Mg and Mg-Y alloy were attempted. Among other factors, successful observations depended on low thickness of the sample over a large area through which the crack occurred. In-situ observations were confirmed by ex-situ examinations in other areas of the cracked sample.

Fig. 1 (a–g) shows in-situ BF TEM images of nucleation and propagation of a crack inside a pure magnesium thin foil under tensile loading. The applied tension direction is indicated by block arrows in Fig. 1a. When the tension was applied, in the beginning, a deformation twin immediately appeared in one of the grains, grain-5, as shown in Fig. 1a (grain notations are shown in supplementary section). Since this twin extended across the grain from its upper boundary with grain-2 to its lower boundary with grain-6, it is difficult to know its exact nucleation point. Later, from ex-situ TEM diffraction studies by loading the sample in a double tilt holder, the crystallographic misorientations between grain-2 and grain-5, and between grain-5 and grain-6, among various grains were found, tabulated and shown in supplementary as Table S1. The upper grain boundary and lower grain boundaries to which the ends of the twin are attached were characterized as high angle grain boundaries.

As the tensile stress was increased, a crack appeared at the grain boundaries as shown in Fig. 1b. Fig. 1b and c in-situ TEM images are acquired under same stress levels at slightly different tilts in order to distinguish the grains more clearly by varying diffraction contrast. The numbering of grains is shown in Fig. 1c. The grain boundaries on which the crack occurs have been characterized from ex-situ TEM diffraction studies (Table S1 in supplementary). The grain boundary between grain-1 and grain-8 with misorientation angle of 15.73° $[27\ \bar{4}\ \bar{23}\ 6]$ is nearly a low angle grain boundary. The other grain boundaries have been characterized as high angle grain boundaries. From this, it can be said that the crack can propagate along both low and high angle grain boundaries.

Further increase in tensile load leads to crack propagation to the left and right of the twin along the grain boundaries, as seen in Fig. 1d. However, the crack did not propagate along the twin boundary, which could be due to geometric restrictions with respect to the applied stress direction or/and twin boundary energy being smaller than the grain boundary energy, which in turn leads to the increased resistance to fracture along the twin boundary. The crack tip towards the left became blunt while the crack tip propagating towards the right is still sharper. This could be due to thickness variation along the crack propagation route.

On further increase in tensile load, crack propagating to the right side reached the specimen hole region as shown in Fig. 1e. Meanwhile, another crack nucleated at the grain corners as shown in the left bottom side of Fig. 1e. The crack propagating to the left then turned its propagation direction towards the newly generated crack, intragranularly through grain 5. Ex-situ TEM diffraction analysis showed that the crack propagation direction through the grain interior is parallel to the (0002) basal planes (see supplementary). This can be due to low Schmid factor for crack propagation on basal plane with easy basal slip. Fig. 1f shows that, on further increase in tensile load, the crack propagation through grain 5 is completed, and the crack started extending intergranularly

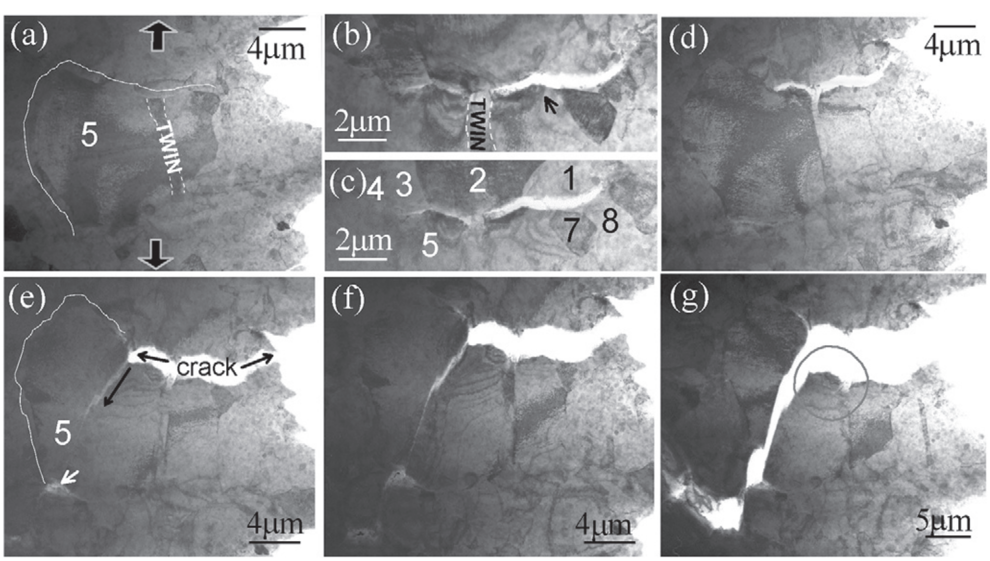

Fig. 1. In-situ TEM imaging of a crack propagation behavior in pure magnesium specimen under tension. (a)The applied tension direction is indicated by block arrows. Deformation twin formed in one of the grains, indicated with dashed lines. Solid line outlines the grain boundary. (b) An arrow mark showing crack initiation along grain boundaries. (c) An image acquired at slightly different tilt from that in (b) in order to distinguish the grains clearly. (d) Further crack propagation along the grain boundaries. (e) Crack propagating to the left has deflected towards a newly generated crack at a grain corner (towards left bottom). (f) The crack propagation through the grain-5 is complete. (g) Top and bottom part of the specimen are separated with further applied strain. Grains are numbered (see Supplementary section).