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In situ analysis of multi-twin morphology and growth using synchrotron polychromatic X-ray microdiffraction

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Abstract: Synchrotron polychromatic X-ray microdiffraction (micro-XRD) was applied to study in situ deformation twinning of commercially AZ31 (Mg−3Al−1Zn) strip subjected to uniaxial tension. The morphology and growth of twins were analyzed in situ under the load level from 64 to 73 MPa. The X-ray microdiffraction data, collected on beamline 12.3.2 at the Advanced Light Source, were then used to map an area of 396 μm × 200 μm within the region of interest. The experimental set-up and X-ray diffraction microscopy with a depth resolution allow the position and orientation of each illuminated grain to be determined at the submicron size. A list of parent grains sorted by crystallographic orientation were selected to examine their twinning behavior. The results depict twin variant selection, local misorientation fluctuation and mosaic spread for multi-twins within the same parent grain. As load increases, the amplitude of misorientation fluctuation along twin trace keeps increasing. This is attributable to the accumulation of geometrically necessary dislocations.

Key words: synchrotron polychromatic X-ray; deformation twinning; in situ analysis; local misorientation; geometrically necessary dislocations

1 Introduction

Magnesium alloy, known as one of the lightest structural metallic materials, is getting more attractive in applications for the automotive and aerospace industries due to its low density and high specific strength. For the hexagonal-close-packed (HCP) structure aggregates, improved understanding of twinning is needed to enhance performance and give automotive makers confidence to use the metal more widely. Magnesium fails via mechanisms that involve deformation twinning. Unlike dislocation slipping process that is thermally activated function of temperature and strain rate, twinning process is stressfully activated and less sensitive to two factors above [1].

Post-mortem analysis of deformed structures has shed important light on the complexity of deformation twinning behaviors in HCP metals [2,3]. However, to understand twinning, in-situ characterization techniques are required to capture the onset and growth of twins, because a detectable drop in the volume fraction of twins is often observed with the release of load [4]. In-situ neutron diffraction has been widely employed to reveal important features of the average deformation behavior of embedded grains in metals deforming by twinning, and this has mostly been done for magnesium alloy [5]. However, more dedicated experimental study is required to investigate the yielding on the micro scale. MURÁNSKY et al [6] using in-situ neutron diffraction coupled with elasto-plastic self-consistent model demonstrated that critical resolved shear stress for the onset of twinning is found to exceed that for basal slip by a factor of 2−6 times. Therefore, it is implied that what happens prior to twin's onset and during twin's growth was "micro" yielding and hardening due to dislocation slip activities.

In line with these demands, the recent advances in synchrotron X-ray microdiffraction methods provide a new opportunity to nondestructively study the microtexture with sub-grain resolution [7]. The emerging 3DXRD crystal microscopes use synchrotron X-ray sources and advanced X-ray optics to probe polycrystalline materials with submicron X-ray beams.

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Three dimensional distributions of the local crystalline phase, orientation (texture) and elastic strain tensor and geometrically necessary dislocation (GND) density tensor distributions can be measured with submicron resolution in all directions. Utilizing monochromatic high-energy 3DXRD measurements and grain reconstruction algorithms, AYDINER et al [8] presented the full stress tensor and its evolution in a growing deformation twin and corresponding parent grain. An alternative methodology known as polychromatic micro-XRD offers superior resolution for assessing intragranular microstructural information [9,10]. BALOGH et al [11] measured the strain gradients across a grain containing twins located in the bulk of a polycrystalline AZ31 sample, using the technique of different-aperture X-ray microscopy (DAXM) in polychromatic mode. ODDERSHEDE et al [12] measured the stress field around an evolving crack in tensile deformed Magnesium AZ31 using three dimensional X-ray diffraction. The insights into the spatial variant in material response and mechanical field localization gained from above direct measurement can lead to the development of more physically based predictive models.

In polycrystalline metallic aggregates, the nucleation and propagation of a twin produces a considerable shift of local elastic-plastic behavior of parent grain and needs to accommodate lattice rotations within twin. ASHBY [13] first pointed out the fundamental difference between homogeneous plastic deformation that can be accommodated by an arbitrary distribution of statistically stored dislocations (SSDs) and non-homogeneous plastic deformation that requires the particular distributions of GNDs to accommodate plastic strain gradients. The introduction of GNDs, in addition to the inherently random SSDs, results in increased hardening of the material. Twin formation and GND density strongly depend on initial microstructure and load directions [14]. PROUST et al [15] proposed an upgraded visco-plastic self-consistent model explicitly describing hardening in twin via GNDs and a directional Hall-Petch mechanism.

The modeling efforts need to be validated by experiment. From the experiments performed on a polycrystalline AZ31 sample, the objective of the present work is to provide a direct assessment of the micro-orientation and GNDs distribution within ongoing twins furnished from polychromatic micro-XRD.

2 Experimental

2.1 Experimental procedure

The material used is commercial AZ31 alloy sheet, rolled to a thickness of 1 mm and processed under half hard conditions. The tensile samples, prepared in accordance with ASTM E8 with a gauge length of 16 mm, were annealed at 400 °C for 1 h to reduce the residual stresses developed during fabrication. This heat treatment developed an equiaxed microstructure with an average grain size of 30 μm. It is confirmed by EBSD experiment that the initial texture of the rolled sheet is the conventional one in which most of the c-axis was aligned toward the normal direction, and that the initial microstructure was free of twin boundaries.

To perform in situ uniaxial tensile loading experiments, a deformation stage, previously used for micro-XRD studies, was utilized [16]. The loading system provided accurate control and feedback of the loading parameters necessary to re-create the macroscopic stress–strain curve. The tensile experiment was carried out under displacement control at the rate of 0.016 mm/s.

Figure 1 shows the schematic of the X-ray Laue-diffraction 3D microscope. The AZ31 magnesium sample was fixed in a tension jig. The tension direction was parallel to the rolling direction and in the tilt angle of 30° to the beam incident direction. A set of fixed right handed sample coordinates, of which the z-axis was

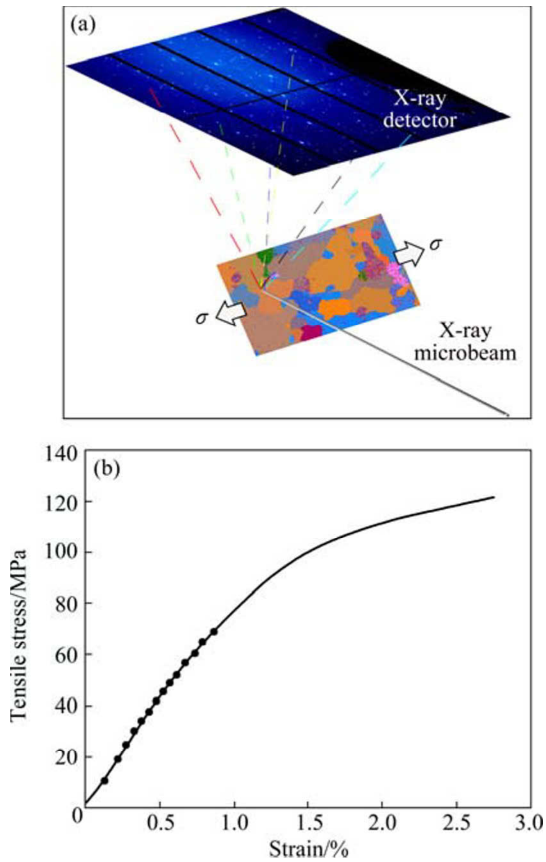

Fig. 1 Schematic of X-ray Laue-diffraction 3D microscope (a) and stress−strain curve for alloy (b) [16] (Points in Fig. 1(b) are indicative of macroscopic load at which micro-XRD maps were collected)