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In-situ analysis of grain rotation and lattice strain within a magnesium polycrystal based on synchrotron polychromatic X-ray diffraction technique: (I) prior to twin

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ABSTRACT

Hexagonal-close-packed structure aggregates exhibit complicated deformation behaviors, involving different slips and twinning. Synchrotron polychromatic X-ray microdiffraction (micro-XRD) was utilized to study in situ an extruded Mg-3Al-1Zn strip subjected to uniaxial tension. The evolution of grain rotation and lattice strain was analyzed under the load levels from 12 to 73 MPa. The micro-XRD data was used to map an area of 396 × 200 μm within the region of interest. The experimental set-up and X-ray diffraction microscopy in two dimensions allow the morphology, orientation and strain of the target grain to be determined at the submicron size. Results depict local orientation fluctuation, lattice strain evolution, slips system and elastic modulus within the same grain. As the applied load increases, the grain's rotation is accelerated between 46 MPa and 51 MPa at which level of load the grain-scale plastic deformation is activated. The predominantly slip modes prior to twin are identified as the combination of $\overrightarrow{b1}$ = (0002) [11$\bar{2}$0] and $\overrightarrow{b3}$ = (0002) [$\bar{2}$110]. During the inspection, all reflection planes displayed an onset of micro yielding at the macro load level of ~38 MPa. In this work, we confirm that magnesium is nearly elastic isotropic.

1. Introduction

The plastic deformation of a polycrystalline material is heterogeneous at the level of grains and subgrains. Because local stress and strain differ from those averaged values at macro-scale, deformation heterogeneities should account for the rotation of different micro-volumes (Bettles and Barnett, 2012). For the hexagonal-close-packed (HCP) structure aggregates, deformation twins lead to a dramatic crystallographic lattice rotation though small contribution to shear strain (Al-Samman et al., 2010). Unlike dislocation slipping process that is thermally activated function of temperature and strain rate, twinning process is stressfully activated (Abdolvand and Wilkinson, 2016). But the twin's nucleation often is pioneered by dislocation slide (Capolungo et al., 2009).

Recently, Muránsky and Barnett demonstrated that critical resolved shear stresses (CRSS) for the onset of twinning during compressing ZM20 Mg alloy along extrusion direction is found to exceed that for basal slip by a factor of 2–6 times (Muránsky et al., 2010). It is implied that the 'micro' yielding due to dislocation slip activities occurred prior to twin's onset. Therefore, an improved understanding of dislocation slide prior to twin provides an access to the insight of the role of dislocation in twin nucleation and growth. X-ray micro-diffraction is a particularly exciting application compared with alternative probes of crystalline phase, orientation, and strain, as it offers better strain resolution, competitive or superior spatial resolution in thick samples, and the ability to probe below the sample surface. Aydiner et al. (2009) using 80.7 keV synchrotron X-Rays in situ investigated the evolution of stress in individual grains and twins in AZ31 alloy aggregate. But the technique relies on "Ω" rotations about the loading axis to collect scatter signal. Wu et al. (2016) using in situ synchrotron X-ray microbeam diffraction to capture twinning-detwinning process, the "twinning-like" lattice reorientation process (Liu et al., 2014) within an individual grain inside a rolled Mg alloy.

The broadband polychromatic X-ray microdiffraction makes it possible to distinguish the diffraction patterns related to different grains (or subgrains) with a typical probe volume of the order of few μm^3 inside a polycrystal. Recently, Lynch et al. developed a new "single-shot" approach to measure and correct for the different points of origin of the diffracting grains. The approach realized a ten-fold reduction in data collection times (Lynch et al., 2014). The objective of the present

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work is use the approach for a direct assessment of the micro-orientation and lattice strain in an embedded grain within Mg alloy aggregate prior to twin.

2. Methodologies

2.1. Experimental procedure

The material used is commercial AZ31 extruded alloy. A tensile sample was cut from an extruded rod with tensile direction perpendicular to the extrusion direction. This combination of materials texture and loading axis leads to the different tendencies of grains for tensile twins, since the c-axis of grains can be oriented in any angles with the load axis. Thus, the deformation micro-mechanism prior to twinning is accessible by selecting a target grain with the crystal orientation concerned. After one hour in a vacuum-tube furnace at 400 °C, the furnace was turned off and allowed to air cool. The annealing process eliminated the residual stresses and produced an averaged grain size up to ~30 μm.

Synchrotron X-ray micro-diffraction measurements were conducted on beam line 12.3.2 at the Advanced Light Source (Kunz et al., 2009). The beamline utilizes an orthogonal pair of Kirkpatrick-Baez mirrors to focus polychromatic X-rays to a sub-micron spot (0.8 μm × 0.8 μm) with a penetration depth of ~500–600 μm. Prior to data collection, a Laue patter (LP) collected from a strain free single crystal Si was used to precisely calibrate the experimental geometry that includes the sample-to-detector distance, the detector tilt angles, and the central channel position of the detector. To ensure the exactly the same positions on the specimen throughout the loading, the polycrystalline AZ31 sample was electrochemically polished and a series of fiducial markers in the form of micro-scale indents were introduced on the surface to define the region of interest (Lynch et al., 2012).

After the region of interest was located, in-situ analysis was conducted at totally 15 applied load levels from 12 MPa to 73 MPa. The nominal load of 12 MPa was applied to the tensile sample in order to remove any mechanical instability in the sample gripping system. At each load level, the sample was translated across the beam in two directions with the step lengths of 12 μm and 20 μm respectively, generating an two dimensional map from a specific region with 340 steps (34 × 10). Each step took 1 s for exposure. At each step increment the polychromatic Laue data was collected with the MAR133 CCD area X-ray charge coupled detector, which produced one raw image.

Fig. 1 shows the schematic of the X-ray Laue-diffraction 3D microscopy. The right-handed Cartesian laboratory coordinate system $X_LY_LZ_L$ is defined such that the Y_L-direction is anti-parallel to the direction of the incoming beam, and that the tension direction is parallel to X_L-direction. A set of right handed sample coordinates $X_SY_SZ_S$ was fixed on the AZ31 Mg sample. The Z_S-axis was defined normal to the sample surface and the X_S-axis within the sample surface and perpendicular to X-ray beam direction, was established for crystal orientation representation. For a perfectly aligned apparatus, the laboratory X_L-direction is parallel to the sample X_S-direction, and the two coordinate systems are related by a rotation of 30° about the common X_L- or X_S-direction. The detected raw image is located in a 2D coordinate system X_DY_D. Its size is 1043 pixels × 981 pixels.

2.2. Data analysis

X-ray microdiffraction analysis Software (XMAS) developed by (Tamura et al., 2003, 2009) is used to analyze the x-ray microdiffraction images. The diffraction peak positions were determined by fitting two-dimensional Gaussian to individual crystalline reflections. The position of fitted maximum intensity was viewed as actual center of the spot. Fig. 2 illustrates the smart approach for peak finding and fitting procedure used in XMAS. The irregular shape spot and its 3D distribution are showed in Fig. 2(a–b). When the threshold value and

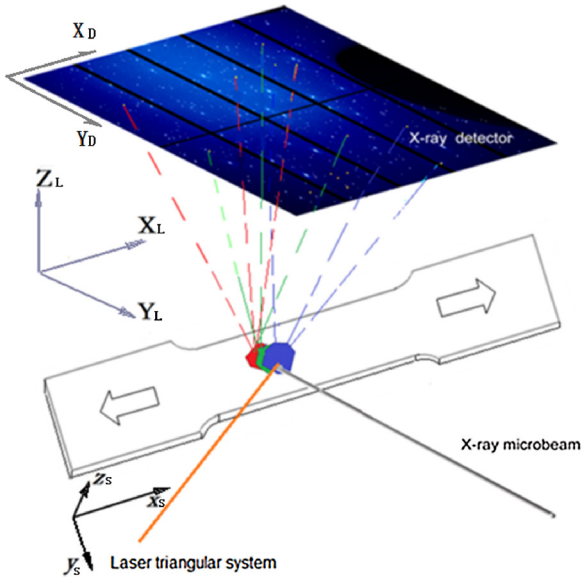

Fig. 1. Schematic of the X-ray Laue-diffraction 3D microscope. Micro-XRD diffraction pattern was collected from multi grains in different depth along incident direction.

window size were set to 20000 and 9 × 9 pixels, as shown in Fig. 2(c), only one peak position was fitted at (729.30, 849.85). When the value was set to 6000 with the window size being equal, as shown in Fig. 2(d), two peaks were determined. When the double window size was adopted with the threshold value being 20000, the fitted position was (729.59, 850.26) in Fig. 2(e). It is noted that the approach used in XMAS is sensitive to Laue spot's shape and size. Accordingly, the small and sharp Laue spots were selected in this study, which allow these patterns allows to be accurately indexed.

All LPs of grains at the nominal load of 12 MPa were automatically indexed with hexagonal lattice parameters ($a_o = b_o = 0.3209$, $c_o = 0.5210$, $\alpha_o = \beta_o = 90°$, $\gamma_o = 120°$). The crystallographic indexation of the target grain at 12 MPa presents in Appendix A in Supplementary material. The reason why the grain was selected is that its orientation under uniaxial tensile load along Xs direction readily leads to basal slips prior to twins. The analysis results of XMAS are described by incident position, crystal orientation, strain, diffraction intensities for indexed (*hkl*) Laue reflections. As the load increased, the spread of the diffraction spots were recorded at the area detector that signifies the onset of plastic deformation.

XMAS can automatically index points on different layers up to six grains in depth. For example, seeing Appendix B in Supplementary material, when the X-ray incident position is (9938 μm, −5870 μm) in sample coordinate system, six crystals were deconvoluted from the raw image of LP by the approach of multigrain indexation. To study grain rotation and lattice strain by analyzing micro-XRD data, the first step was to select the target grain. As long as the target grain is indexed, one obtains its orientation matrix including 9 elements. If the orientation matrices of indexed points matched with the matrix of target grain (i.e. the deviations of all corresponding elements were below 0.02), one affirms that the points belong to the domain of target grain. Therefore, the orientation matrix can be regarded as the unique characteristic. The details about orientation matrix are explained in Appendix C in Supplementary material. In the approach above, 33 grains are indexed in the area of interest beneath the surface ~200 μm. Fig. 3 show the orientation of 33 grains within the scanning area at 12 MPa. The indexing result indicates a loose (0001) fiber texture in the tensile sample cut from an extrusion rod. The target grain's orientation is in the red circle. The reason for targeting this grain is that it produces twins and that it possesses a high Schmid Factor for basal slip, making dislocation