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X-ray microbeam measurements of long-range internal stresses in commercial-purity aluminum processed by multiple passes of equal-channel angular pressing

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X-ray microbeam diffraction was used to measure long-range internal stresses (LRISs) in the grain/subgrain interiors of commercial-purity aluminum processed by equal-channel angular pressing for up to eight passes. The LRIS values at +4.9° off the axial (pressing) direction show only a slight increase with increasing numbers of passes. The normalized stress remains approximately constant at ~0.10 of the flow stress.

Keywords: X-ray diffraction; Aluminum; Equal-channel angular pressing; Long-range internal stresses; Severe plastic deformation

Severe plastic deformation (SPD) has been a popular method for producing materials with refined grain sizes [1–3]. This grain refinement leads to improved mechanical properties including hardness, yield strength and fracture toughness. One attractive SPD technique is equal-channel angular pressing (ECAP) [4–6], which produces relatively homogeneous refined grain-sizes in bulk material. Grain refinement occurs primarily via the formation of geometrically necessary boundaries [7]. These boundaries may be nonequilibrium and have many emanating or extrinsic dislocations [8,9]. Thus, these boundaries may also be sources for the relatively high long-range internal stresses (LRISs) observed by Alhajeri et al. [10] for aluminum 1050. Recent work by the authors examined the LRISs in the subgrain/grain interiors of the Alhajeri et al. aluminum specimens using X-ray microbeam diffraction at a synchrotron [11]. The results of the LRIS evaluation of the subgrain/grain walls and their interiors were consistent with the composite model [24], at least for one pass of ECAP. The original composite model [24] describes "hard" high dislocation density regions (e.g. cell walls, etc.) and "soft" regions of low dislocation density (e.g. cell interiors). This creates regions of high and low stress as the "composite" is strained compatibly. In practice, the stresses are also consistent with the summation of the stress fields of the dislocations. The model is reviewed in detail in Ref. [19]. In this report, a microbeam diffraction evaluation of LRISs using ECAP AA1050 processed for multiple passes is now presented as a continuation of the earlier work [11]. Commercial-purity aluminum was utilized both for relevance to commercial applications and because it is less subject to dynamic recrystallization [12].

The internal elastic strains of the low dislocation density regions within the grains/subgrains of ECAP AA1050 along the pressing direction for 1, 2, 4 and 8 passes were measured using microbeam X-ray diffraction at beam line 34ID-E of the Advanced Photon Source (APS) at Argonne National Laboratory. Only a single reflection could be measured from each grain, providing information on the elastic strain in just a single direction with respect to the sample geometry. Obtaining complete strain tensors may be possible by measuring multiple reflections but that is beyond the scope of the current study.

The ECAP AA1050 composition, machining and other experimental procedures are identical to those used in our

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previous study [11]. Ideally, the ECAP die imposes approximately +0.88 principal plastic strain for each pass along the +22.5° direction, −0.88 plastic strain along the −67.5° direction off the pressing direction and about zero plastic strain along the pressing direction [13] after a single pass and in the plane deformed by the extrusion axis as illustrated in Figure 1. The ECAP die imposes roughly +0.19 plastic strain, or about 21% of the maximum plastic strain for our measurement direction (about +5° off the pressing axis) after a single ECAP pass. The ECAP AA1050 samples were processed via route B_C, with samples rotated by 90° between each pass [14–16]. Samples subjected to 1, 2, 4 and 8 passes were examined.

The powder diffraction measurement procedures were identical to those described earlier [11]. Lattice parameters were measured using X-ray powder diffraction for the as-received (pre-ECAP) condition and after 1, 2, 4 and 8 passes (P). The lattice parameter for the as-received AA1050 was 4.05000(10) Å, for 1P: 4.05020(10) Å, for 2P: 4.05010(10) Å, for 4P: 4.05010(10) Å and for 8P: 4.05010(10) Å. These lattice parameters show little variation between the number of passes with all values lying within one standard deviation of 4.05010 Å, which suggests that these samples exhibit negligible residual stresses on length scales that are large compared to dislocation microstructures. The lattice parameter obtained from the as-received AA1050 sample was used as the baseline a_o. We note that this lattice parameter is slightly different from the pure Al lattice parameter of 4.04950(15) Å [17] that is generally used in other AA1050 LRIS studies. The difference in lattice parameter from pure Al and 1050 Al multiplied by Young's modulus translates to (an equivalent strain) error of $+10^{-4}$, which is significant. The radii of Al, Si and Fe are 0.118, 0.111 and 0.156 nm, respectively. The composition of AA1050 is 0.25% Si, 0.4% Fe. Using a simple weighted average and hard sphere model, at least a 10^{-4} increase in a_0 is expected. As mentioned above, we find that using a (powder) diffraction measurement on the 11-BM beamline at the APS, the average lattice parameter is constant (over a beam diameter of 3 mm) for all specimen passes. Thus, there is no evidence of macroscopic residual stresses in ECAP AA1050. Macroscopic residual stresses, however, were observed in separate work on ECAP AA6005.

X-ray microbeam diffraction measurements on beamline 34-ID-E at the APS were performed identically to those reported previously [11], with a microbeam cross-section of approximately 500 nm × 500 nm. The {5 3 1} reflections were used to obtain lattice spacings for each of the samples. Lattice spacings measured by the X-ray microbeams were obtained from relatively low dislocation density areas within the grain/subgrain interiors that produce sharp diffraction spots on the detector. The measured lattice spacings were then compared to powder-diffraction-based lattice parameters to calculate elastic strains within these regions. Lattice parameter measurements in high dislocation density regions (cell walls) were made on strained (0.30 strain) Cu in an earlier work by the authors [23]. There, the walls are diffuse and less well-defined, and X-ray peak positions could be reasonably assessed. However, in ECAP, the cell walls/grain boundaries had much higher dislocation densities that precluded identifying well-defined X-ray peaks and corresponding lattice parameters. Within the cell interiors there were higher dislocation density regions with more X-ray scattering, and lower dislocation density regions with less X-ray scattering. The lower dislocation density regions were associated with better-defined peaks and thus easier strain or lattice parameter measurements. We chose to measure the better-defined peaks of the cell interiors.

After 1, 2, 4 and 8 ECAP passes, the microstructure of a similar Al specimen exhibits grains with boundaries of mixed high and low misorientation angles [7] as is typical for SPD [18]. After 1 pass, about 15–20% of the boundaries are high-angle grain boundaries (HAGBs) having misorientations >15° according to transmission electron microscopy (TEM) (rather than electron backscatter diffraction that may not detect all low-angle boundaries). Similarly, after 2 passes the HAGB fraction is ~25%, after 4 passes it is ~37% and after 8 passes it is ~70% [7]. According to TEM analysis, the average grain/subgrain size formed by high- and low-angle grain boundaries is about 1 μm for 2 passes, 900 nm for 4 passes, and 680 nm for 8 passes [10].

In earlier work on deformed Cu [23], it was possible to directly measure the elastic strains within the high dislocation density cell walls. Here, the sharp grain boundaries of ECAP AA1050 provided too little scattered intensity distributed over too large an area of the detector to allow direct strain measurements from these boundaries. Within

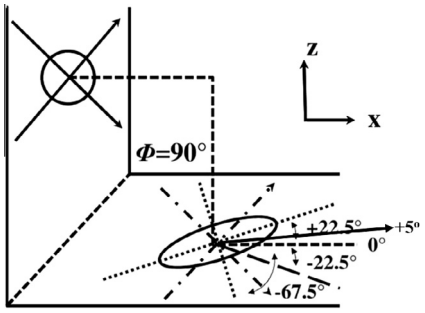

Figure 1. A circle is deformed into an ellipse after ECAP processing. The long axis (+22.5°) of the ellipse indicates the maximum tensile plastic strain. Strain along the pressing axis (0°) is theoretically zero [13]. Strain measurements are made along the +5° direction.

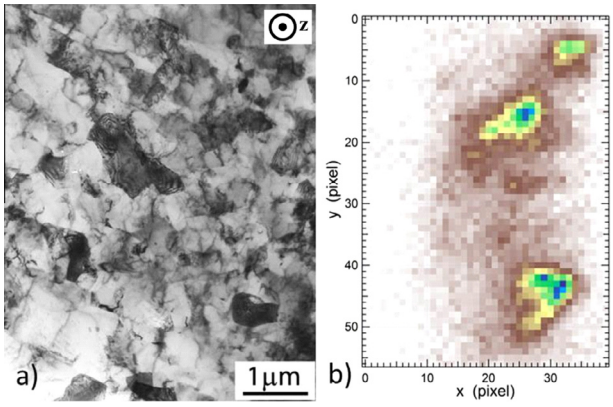

Figure 2. (a) TEM image of ECAP AA1050 after 2 passes. (b) False-color image of the energy-integrated diffracted intensity from an individual grain/subgrain in a 2-pass sample. The peaks are from low-dislocation-density regions and the smeared intensity is from walls or cell interiors with a relatively high dislocation density, (b) is taken from earlier work on the same alloy [11].