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Investigation of plastic yielding in near-micrometer grain size aluminum using synchrotron microdiffraction

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Abstract. The onset of plastic yielding has been studied in samples of aluminum with near-micrometer average grain size using in situ tensile loading combined with synchrotron radiation. A white-beam Laue 3D micro-diffraction technique was used allowing the collection of diffraction signal from a volume with a resolution of 1 μm in all three dimensions. The orientation data obtained from the micro-diffraction are used to estimate the local variation in geometrically necessary dislocation density, and correction factors are suggested for voxels where neighbors are missing in either one or two dimensions. The results demonstrate a considerable heterogeneity in deformation in the early stages of plastic deformation.

1. Introduction

The process by which a polycrystalline metal undergoes plastic yielding, i.e. the transition from elastic to plastic behavior, is of fundamental importance for the understanding of mechanical properties, and yet there are many aspects of this transition that are still only poorly understood [1,2]. The interest in understanding this elastic-plastic transition has intensified in recent years for a number of reasons. These include the observation that even in FCC metals a distinct yield drop is developed for grain sizes in the near-micrometer regime [3,4] (indicating a transition in the way in which plastic strain percolates through a loaded sample), and enhanced combinations of ductility and mechanical strength in samples with a heterogeneous grain size distribution [5].

An alternative parameter that can be used is the geometrically necessary dislocation (GND) density \( \rho_{\text{GND}} \) [7,8]. Although this is not a direct measure of local plastic strain (for a given stress state different crystal orientations deformed to the same plastic strain will have different \( \rho_{\text{GND}} \) values), increases in the GND density at a very low strain are a clear indicator of the transition from elastic to plastic deformation.
The GND density is calculated from local lattice curvature [7,8] and therefore requires spatially resolved measurements of lattice orientation. For study of plastic yielding a technique for measuring crystal orientations with high angular resolution is therefore required, where additionally these measurements should be taken in the bulk with sufficient spatial resolution to allow determination of the local lattice curvature in 3D. These requirements are all satisfied by the X-ray synchrotron facility at beamline 34-ID-E of the Advanced Photon Source, Argonne National Laboratory, where spatially resolved 3D orientation measurements with sub-micrometer spatial resolution can be achieved by use of the differential aperture X-ray microscopy (DAXM) technique [9].

For such an experiment the material for examination should have ideally a fine grain size (to allow observation of a large number of grains while using a high orientation mapping resolution), and be in a fully recrystallized condition (to allow plastic yielding to be studied in an initially deformation-free material). It has been demonstrated elsewhere that these requirements can be achieved in samples prepared by spark plasma sintering (SPS) [4,10,11], where it is possible to achieve an average grain size in the near-micrometer grain size regime. For the present experiment samples with a 5.2 μm average grain size are used, as this grain size is sufficiently large to still exhibit classical yielding behavior, but is fine enough to allow a large number of grains to be mapped using DAXM.

![Figure 1. Reconstructed 3D grain maps of the 5 μm sample at strains of (a) 0% and (b) 0.80%. The coloring scheme is based on the Euler angles of each indexed voxel. The mapped volume extends from the sample surface (S) into the interior of the sample](image)

2. Experimental

The SPS technique was used to prepare samples of Al with an average grain size of 5.2 μm. Disks of diameter 20 mm and height 6 mm were sintered from spherical Al powder (average particle size of 5.7 μm) following the procedure described by Le et al. [4]. Dog-bone shape tensile samples were cut from the as-sintered disks by electron-discharge machining with gauge width, length and thickness of 1.8, 12 and 0.6 mm, respectively. Prior to tensile deformation, the specimens were manually ground to 4000-grit silicon carbide paper, and then electrochemically polished in a 10% perchloric acid and 90% alcohol solution to achieve a flat polished surface free of mechanical damage and residual deformation.

For the synchrotron measurements, a polychromatic X-ray beam with energies in the range of 7 - 30 keV was focused using two non-dispersive Kirkpatrick-Baez mirrors, producing a beam of near-Lorentzian profile with a full width at half maximum of ~ 0.3 μm. At the beginning of the experiment DAXM was used to map a volume of dimensions 51 × 5 × 155 μm³ using a step size of 1 μm along all axes. The volume was located near the center of the gauge length, extending from the sample surface to the sample interior.

For the tensile deformation a purpose-built push-to-pull stage was used, with a nominal strain resolution of ~ 0.05% [12]. The tensile stage was equipped also with a load cell to record the instantaneous force during tensile loading. Full details of the experimental set-up are available in [13]. The specimen was sequentially loaded to three strains of 0.15%, 0.30% and 0.80%. After each deformation step the position of volume of interest was first re-established using a cross-correlation technique based on the white beam Laue patterns [13], after which DAXM was used to map the crystal orientations within the volume, in each case using a step-size of 1 μm in all three dimensions.
3. Results and discussion

Figure 1 shows Euler maps of the investigated volume before and after deformation to a strain of 0.80\%.

The agreement between the two reconstructed volumes demonstrates successful tracking of a fixed bulk volume, while small differences of colors for the grains in figure 1b qualitatively show development of small rotations inside each grain. The lattice orientation measurement at each voxel can further be used to determine the GND density using either a simple approach based on local kernel average misorientation [14], or a more detailed approach where all dislocations present can be described by the local dislocation density tensor

\[ \alpha_{ij} = -\delta_{ij} \frac{\partial \beta_{ij}^{el}}{\partial x_k} = -\delta_{ij} \frac{\partial (e_{ij}^{el} + \omega_{ijkl})}{\partial x_k} \]  

(1)

where \( \epsilon \) is the Levi-Civita symbol, \( \beta_{ij}^{el} \) is the elastic distortion tensor, which is the sum of the elastic strain tensor \( e_{ij}^{el} \) and the lattice rotation tensor \( \omega_{ijkl} \).

The latter term in equation 2 is given by the lattice rotation vector \( \theta_m = -\epsilon_{mij} \omega_{ij} / 2 \). With the introduction of the lattice curvature tensor

\[ \kappa_{ij} = \frac{\partial \theta_i}{\partial x_j} \approx \frac{\Delta \theta_i}{\Delta x_j} \]  

(2)

and assuming elastic strain is negligible, the relationship between dislocation density tensor and lattice curvature can be established by combining equations (1–3) as

\[ \alpha_{ij} = \kappa_{ij} - \delta_{ij} \kappa_{kk} \]  

(4)

where \( \delta_{ij} \) is the Kronecker delta, and the scalar measure of GND density is taken as the entrywise one-norm of the dislocation density tensor

\[ \rho_{GND} \approx \frac{1}{b} \| \kappa \|_1 = \frac{1}{b} \sum_{j} \sum_{i} |\kappa_{ij}| \]  

(5)

For conventional EBSD, since the out-of-plane curvature cannot be measured, the calculated GND density is always an underestimate. One advantage of 3DXRM is it allows the orientation measurement along the missing dimension, and the full dislocation density tensor as well as the GND density can therefore be directly determined. Another advantage of our present data compared to EBSD is a very high angular resolution in orientation determination is accessible, estimated in the present experiment as 0.02°, which also enhances the accuracy of GND density measurement.

Figure 2. Histograms showing the scaling factor required to compensate for the absence of one or two neighbors in GND density calculations for the 0.80 % strain step. The distributions for the two cases are approximately of Rayleigh distribution shape, with mean values within the 95% confidence interval of 1.45 and 3.14, respectively. A small fraction of voxels with scaling factor larger than 10 is regarded as noise and neglected in calculation of average values.

Nevertheless, even in the 3D data set not every voxel has a full set of 3D neighbors, as some voxels remain unindexed due to low diffraction pattern quality, and some voxels lie either at the end of the examined volume or adjacent to a grain (or subgrain) boundary. As such there are still some places in the data set where neighbors along three positive axes are missing in one dimension, and places where neighbors are missing in two dimensions. To provide an estimation of the effect of such cases on the calculated dislocation density a simulation has been carried out where first the GND density was
calculated for all voxels with a full set of neighbors in all three dimensions. Next, for each of these voxels the lattice curvatures along one or two dimensions are set to zero, randomly chosen among the three possible directions for each voxel. Finally, comparing the values allows the difference between the 3D value and the reduced dimension value to be determined.

The results are summarized in figure 2. The mean value within a 95% confidence interval for the one neighbor missing case is 1.45; for the two neighbors missing case the value is 3.14. The mean values are quite close to 1.5 and 3, the reciprocals of which are the inverse ratios of the number of non-zero lattice curvature elements in the GND tensor for each case (6 and 3 out of 9), implying that on average the missing gradient is of similar magnitude as the measured gradient and thus can be extrapolated. For 2D GND density calculations based on EBSD data a correction factor of 3.6 has previously been suggested [15], which as for this study was concluded via simulation. It should be noted that for the present data set, similar distributions and mean correction values for one or two missing neighbors were obtained on data for this sample at other strains, where different sets of randomly chosen missing curvature components were tested.

![Figure 3](image)

**Figure 3.** Maps showing the GND density at each voxel in the center layer of the investigated volume before deformation: (a) full 3D data; (b) 3D data after the application of a smoothing filter; (c) GND density calculated considering the center layer as a 2D data set (also with smoothing filter applied). Black and red lines indicate misorientations of 10° and 0.2°, respectively; white lines show features identified as subgrains. The sample surface is at the top of each image.

Based on the above discussion, the scaling factors of 3 and 1.5 were chosen for the GND density correction for all voxels with missing neighbors in the data set. This calculation was carried out within each grain. More precisely, first a grain detection algorithm was applied to undeformed data, using a grain boundary definition of 0.2°, and then a refinement algorithm used to find any subgrains within these grains down to a misorientation angle of less than 0.02°. The determined grain structure (including subgrains) was then mapped on to the data for the deformed samples, so that the voxels inside each grain at each deformation step were identified. For each grain, at each strain, the GND density was calculated for all voxels inside the grain. Note that this is different to the method usually used for GND calculation for EBSD data for example, where an upper cut-off angle is used to ensure that lattice curvature is not calculated across a grain boundary. As the present data set is of high angular resolution, by nature of synchrotron X-ray microscopy, and as the goal is tracking the evolution of GND density, this approach
has the advantage of allowing tracking of GND evolution even within volumes identified as subgrains in the initial material.

An example GND density map, showing the center layer of the undeformed material, is given in figure 3a. The low values confirm the recrystallized nature of most grains, though evidence of residual strain, as well as some subgrains, albeit corresponding to very low misorientation angles, is seen, similar to observations of recrystallized Al examined using dark-field X-ray microscopy [16]. Somewhat higher values are seen near the sample surface. As it is expected that the DAXM data quality decreases with increasing depth it is concluded that these higher values are related to the difficulty in removal of all surface damage from sample preparation when a high orientation resolution is used. The variation in GND density is seen more clearly in figure 3b, where 3 × 3 × 3 neighbor smoothing filter has been used.

As it is not always possible to make crystal orientation measurements in 3D, it is also of interest to compare the present 3D results with those that would be obtained if only 2D were available at the same spatial and angular resolution. For this the crystal orientation measurements for the center layer of the mapped volume in the undeformed material were extracted and then the GND density calculated treating the layer as a 2D plane, rather than a 3D volume, applying the same correction factors as described above. The result is shown in figure 3c. A similar overall pattern in the spatial distribution of the GND content is seen, although locally high values in the 2D calculation are typically larger than in the 3D case. This can be understood by the fact that for this sample high in-plane (2D) lattice curvature is in most cases associated with lower out of plane lattice curvature, and the value calculated at each pixel (or voxel in 3D) is the average of the lattice curvature at each position.

![Figure 4](image)

**Figure 4.** Evolution of 3D GND density within each identified grain/subgrain during tensile deformation showing here the center layer of the investigated volume: (a) 0%, (b) 0.15%, (c) 0.30%, and (d) 0.80% deformation. The sample surface is at the top of each image.

Maps showing the evolution of GND density for the volume corresponding to the center layer of the initially mapped volume (calculated using the 3D data set and with smoothing) are shown in figure 4. Only moderate changes are seen at strains of 0.15% and 0.30%, where the average GND density ($\rho_{\text{av}}$) increases to from $5.9 \times 10^{12}$ m$^{-2}$ in the undeformed sample to $\rho_{\text{av}} = 6.2 \times 10^{12}$ m$^{-2}$ and $6.5 \times 10^{12}$ m$^{-2}$ in samples deformed to strains of 0.15% and 0.30%, respectively. Closer inspection of the data at 0.30% strain shows obvious increases in GND density inside some grains, particularly near grain boundaries, while in a few grains no obvious change in GND density can be seen. At a strain of 0.80% a significant
increase in GND density is seen ($\rho_v = 10.2 \times 10^{12} \text{ m}^{-2}$), with a clear heterogeneity in the GND density both between grains and within grains. Preliminary analysis suggests the presence of clusters of volumes with higher GND density providing some evidence of percolation of plastic strain, which will be further analyzed in future work. It is interesting also to note that even at this strain, some larger grains still show a relatively low GND density.

4. Conclusions
A study has been carried out to track the onset of plastic strain in 3D during in situ tensile deformation of aluminum with an average grain size of 5 μm using X-ray synchrotron measurements. The following conclusions from this work can be made:

- The initial sample, produced by spark plasma sintering has a low dislocation density and is in a nearly fully-recrystallized condition. High energy X-ray synchrotron radiation allows the detection of subgrains in the sample down to a misorientation angle of 0.02°.
- The 3D nature of the data provided by DAXM allows calculation of all nine elements of the GND density tensor. For voxels with missing neighbors, average scaling factors are calculated by simulation as 3 and 1.5 for 1D and 2D restricted data, respectively.
- The onset of plastic strain, as indicated by increase in GND density can be tracked using the DAXM technique. Heterogeneity in the onset of plastic deformation is obvious at strains nominally both in the elastic and plastic regimes (based on a conventional 0.2% yield point definition).

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