3D X-ray Diffraction Microscopy of Grain Boundaries

Wenjun Liu, Gene E. Ice, Wenge Yang, Jon Z. Tischler, and Bennett C. Larson

Oak Ridge National Laboratory, Oak Ridge TN 37831

Abstract

The 3D X-ray crystal structural microscope is a new nondestructive tool for the three-dimensional characterization of mesoscopic materials structure. A prototype microscope is installed on beamline 34-ID at the Advanced Photon Source, which has a routine spatial resolution of approximately 0.5 µm x 0.5 µm and can probe tens to hundreds of microns below a sample surface depending on the composition of the sample. Here we report initial results from an emerging new method for grain boundary characterization, with unprecedented sensitivity to grain boundary misorientation of a tenth of a milliradian and with detailed new information about grain boundary surfaces in three-dimensions. This new approach is certain to address long-standing questions about grain boundary networks in materials and provides quantitative tests of grain boundary models.

Statistics on CSL Boundaries

<table>
<thead>
<tr>
<th>Type</th>
<th>Σ</th>
<th>%</th>
<th>#</th>
<th>Total</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>2</td>
<td>91</td>
<td>10</td>
<td>101</td>
</tr>
<tr>
<td>2</td>
<td>1</td>
<td>8</td>
<td>10</td>
<td>18</td>
</tr>
<tr>
<td>3</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
</tr>
<tr>
<td>4</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
</tr>
<tr>
<td>5</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
</tr>
<tr>
<td>6</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
</tr>
<tr>
<td>7</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
</tr>
<tr>
<td>8</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
</tr>
</tbody>
</table>

Open questions:
1. How and why do measured CSLs deviate from ideal model as Σ type increases?
2. Are there residual strains imposed near the deviated CSL boundaries?
3. Is difference of CSLs near or below sample surface?
4. ………

Schematic of 3-D X-ray Diffraction Microscope

Experimental Hutch 34ID-E

differential aperture (wire scan)
CCD
sample
K-B

Data Analysis

Depth Resolved Pattern

Three Dimensional Morphology of Triple Junction

Sample surface
Rotation angles:
A-B: 16.572°
B-C: 12.907°
C-A: 5.5382°