3D X-ray Diffraction Microscopy of Grain Boundaries

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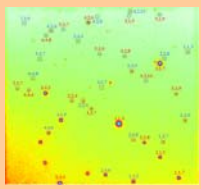
Abstract

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 0.5x0.5 x 1 mm3 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.

ref: B. C. Larson et al., "Three-dimensional X-Ray Structural Microscopy with Submicrometer Resolution", Nature 415 887-890 (21 Feb 2002).

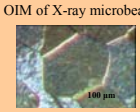
2D Orientation Imaging Microscopy (OIM)

(Without differential-aperture depth profiling)



Indexed white beam Laue pattern

OIM of X-ray microbeam

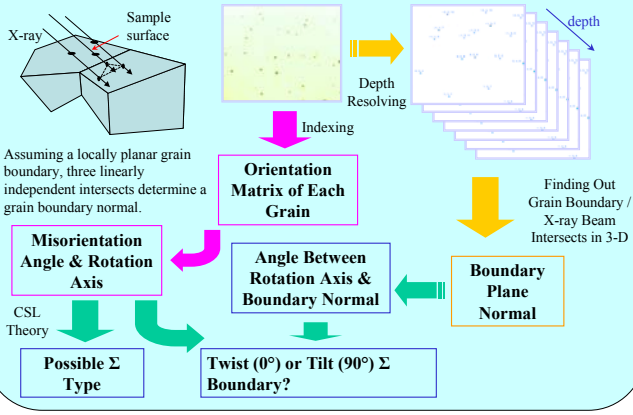


Optical microscopy

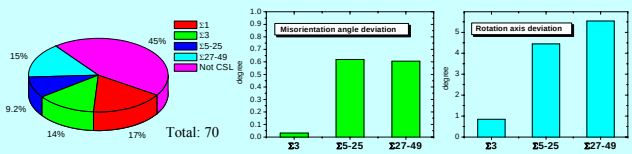
Comparing with OIM of Electron Back-Scattering Microscopy (EBSD):

- ***Phase discrimination***
  spatial resolution to
  0.3 µm x 0.4 µm
  EBSD: ~ 0.1µm
- ***Orientation < 0.01° (~ 1 x 10^{-4})***
  EBSD: ~ 0.1 – 1.0°
- ***Strain resolution ~1 x 10^{-4}***

3-D Study of Grain Boundary Types



Statistics on CSL Boundaries



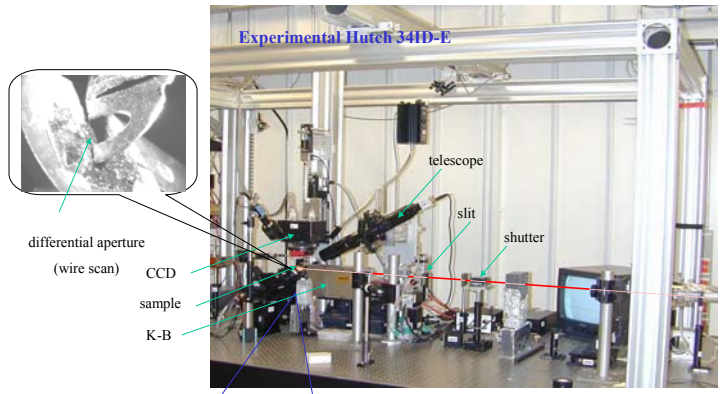
No	Sigma type	Rotation Angle (degree)	Rotation angle off (degree)	Rotation Axis (RAX)	Rotation axis off (degree)	Boundary Normal (BN) in bi-crystal	Angle between RAX – BN (degree)	
B2	Σ21b	44.40	0.011	2, 1, 1	2.945	1.00, 0.32, 0.30 / 0.69, 1.00, 0.17	86.3	Tilt
B6	Σ47b	43.66	0.800	3, 2, 0	6.114	1.00, 0.07, 0.53 /1.00, 0.87, 0.31	74.6	Tilt
B10	Σ37c	50.57	0.139	1, 1, 1	4.551	0.08, 1.00, 0.26 /1.00, 0.12, 0.68	57.6	
B34	Σ1	(6.16°)				0.00, 1.00, 0.17 / 0.04, 0.27, 1.00	88.3	
A57	Σ3	60.00	0.014	1, 1, 1	0.019	1.00, 0.11, 0.02 / 0.32, 1.00, 0.87	86.4	Tilt
A314	Σ3	60.00	0.010	1, 1, 1	0.027	0.28, 0.31, 1.00 /0.36, 0.37, 1.00	2.4	Twist
.	.	.	.	.	.	.	.	.

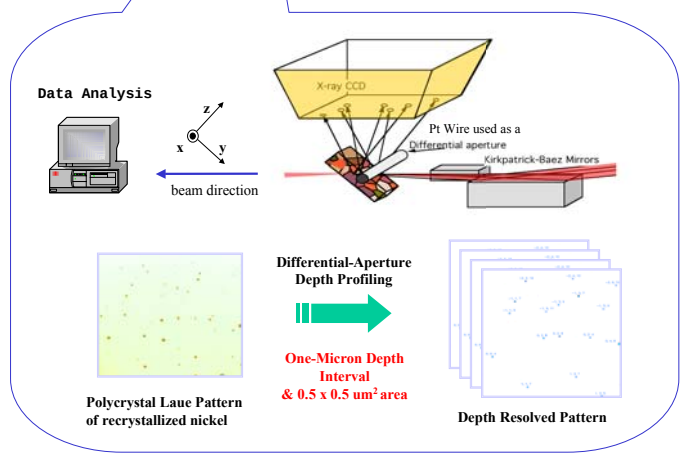
NOTE: Σ1 = low angle boundary < 15°

Open questions:

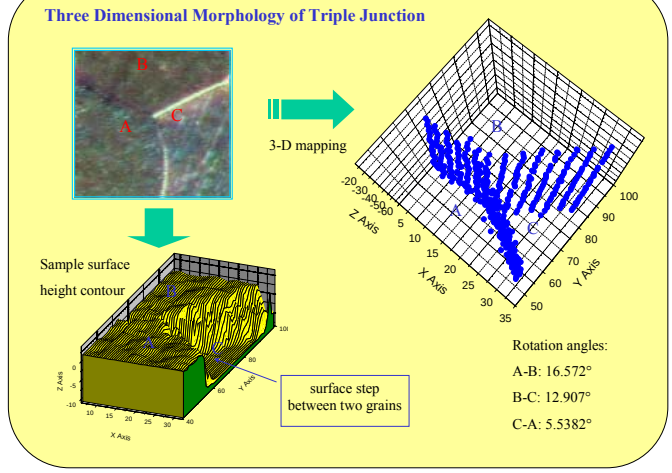
1. *How and why measured CSLs deviate from ideal model as Σ type increases?*
2. *Are there residual strains imposed near the deviated CSL boundaries?*
3. *Any difference of CSLs near or below sample surface?*
4. ……

Schematic of 3-D X-ray Diffraction Microscope





Three Dimensional Morphology of Triple Junction



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