Characterizing the Internal Grain Boundary Network Structure of Polycrystals: The Current State of the Art and Opportunities for High Energy X-ray Diffraction Microscopy

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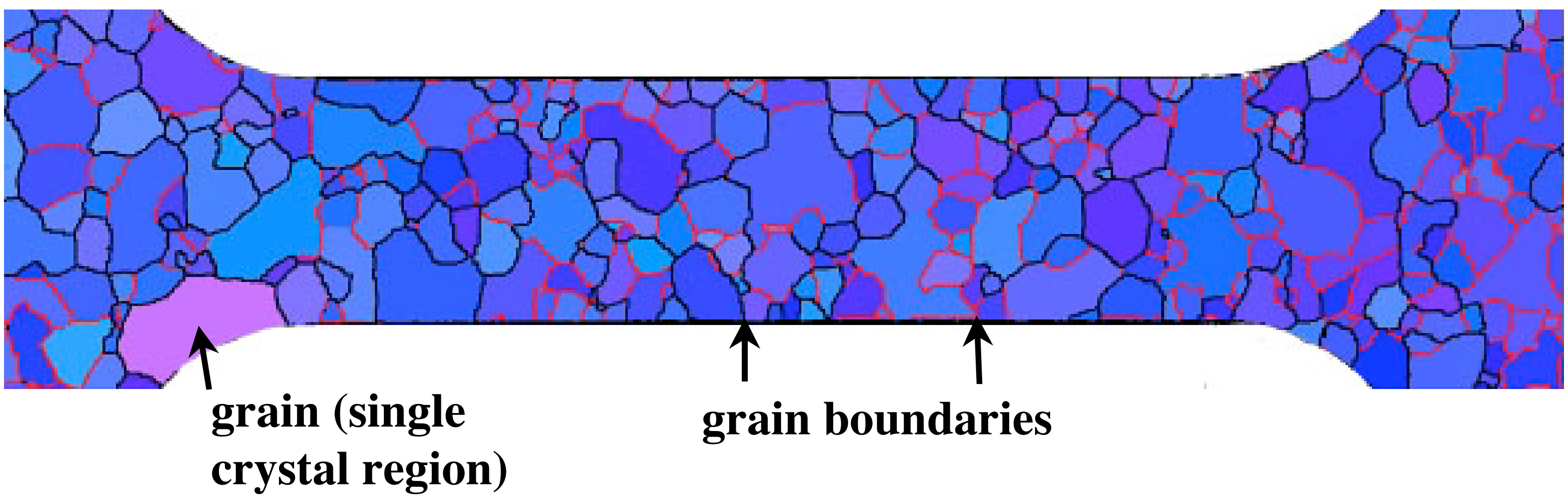
The Grain Boundary Network

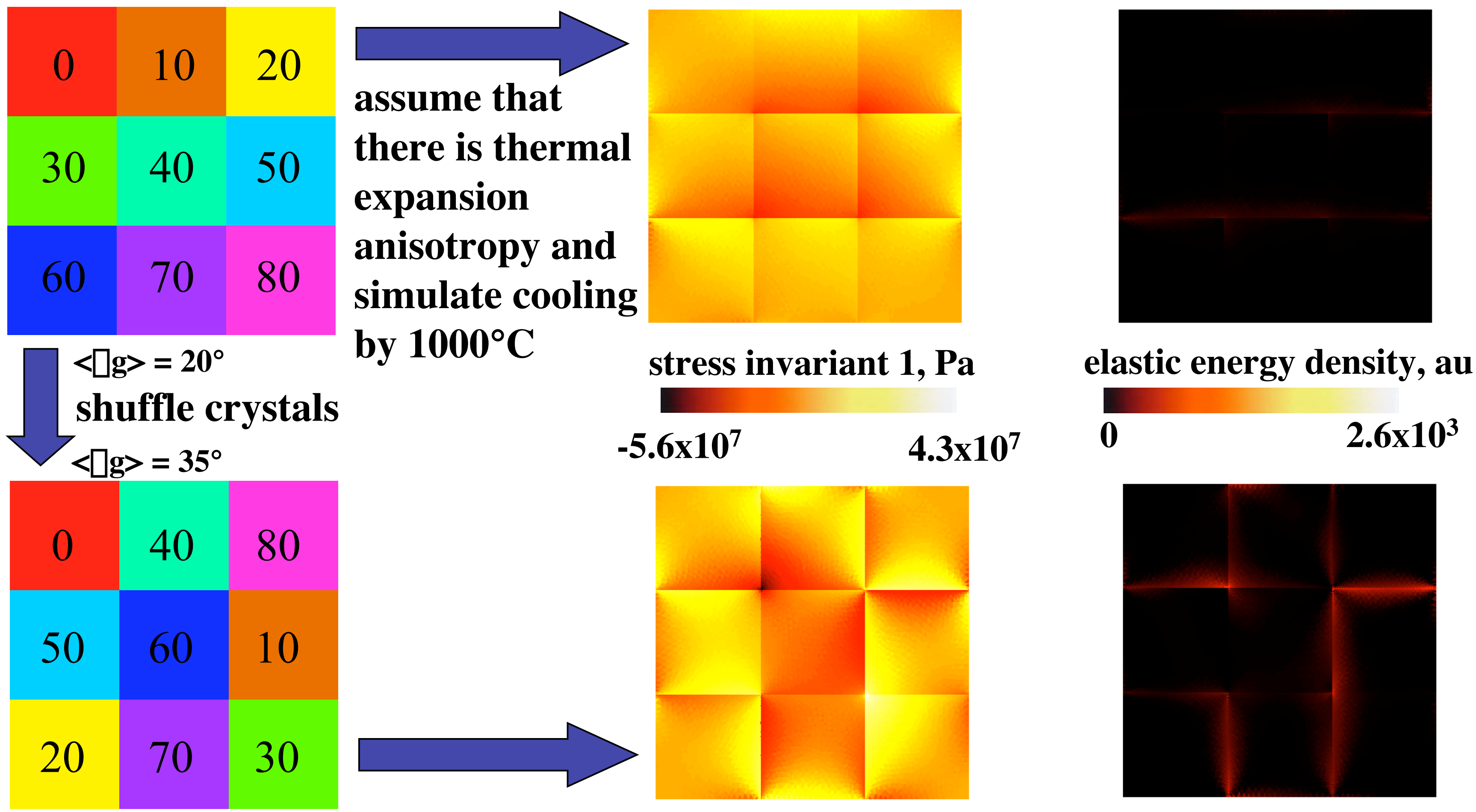
Three dimensional network of connected interfaces within polycrystalline solids



• Usually observed in two-dimensional sections.
• The different "types" of interfaces can be distinguished, but this is not necessarily common practice.

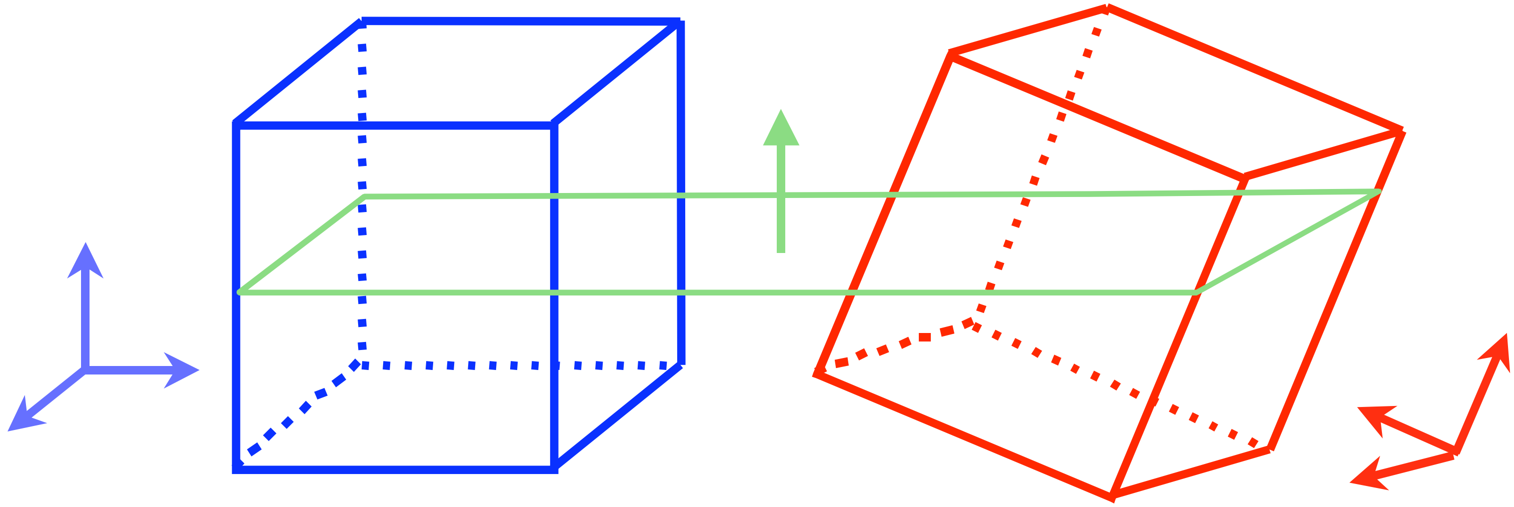
Why is this structure important?

The Structure of the Grain Boundary Network Influences Properties



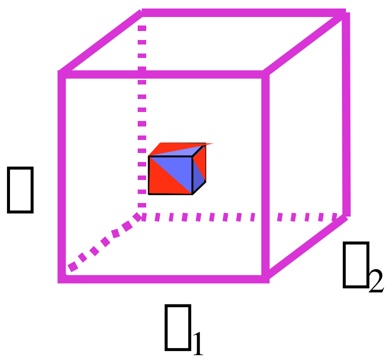
Calculated fracture strength of upper polycrystal is 60% greater than the lower one.

Classifying Grain Boundaries

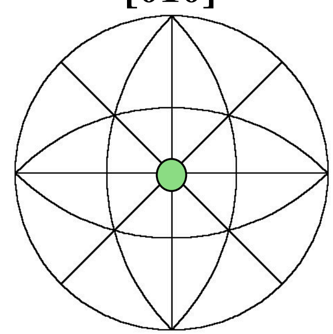


Lattice Misorientation

Boundary Plane



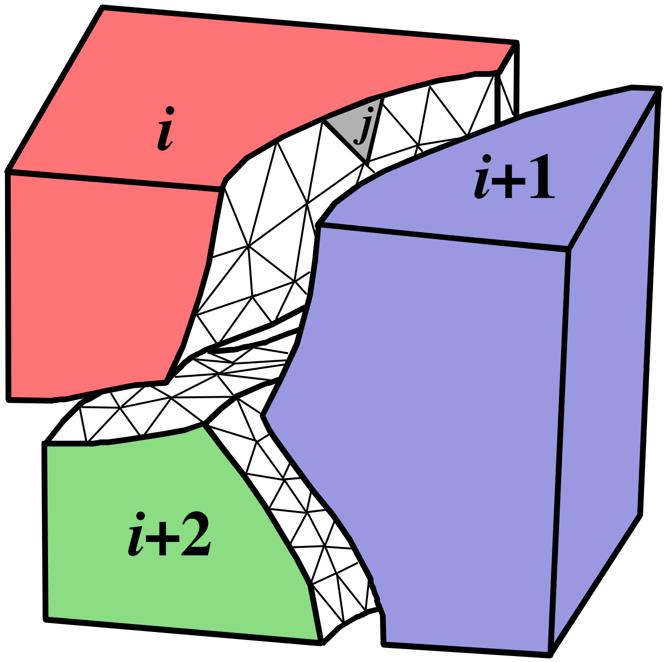
three Euler angles

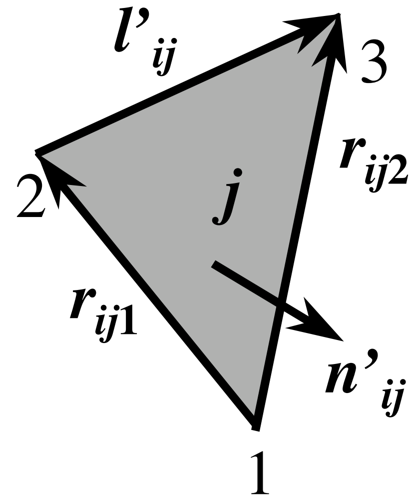


Two spherical angles

There are five macroscopically observable grain boundary parameters

Five parameter grain boundary character distribution





Three parameters for the misorientation: $\Delta g_{i,i+1}$

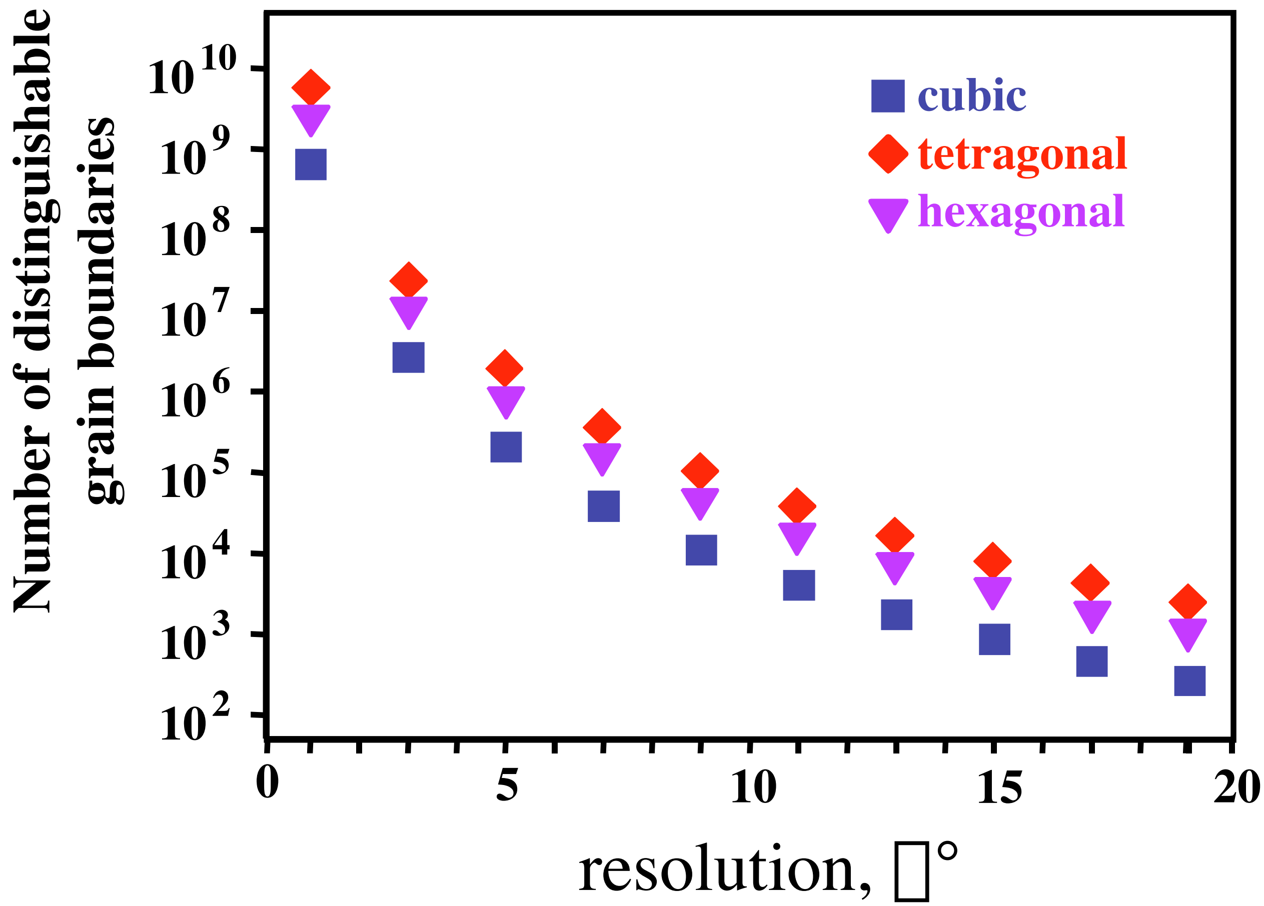
Two parameters for the orientation: $\mathbf{n}_{ij}$

Grain boundary character distribution: $\lambda(\Delta g, \mathbf{n})$, a normalized area measured in MRD

G.S. Rohrer et al., Zeitschrift fur Metallkunde, 95 (2004) 197-214

The number of boundaries in this space is large

$$N = \frac{2\square \bullet \square \bullet 2\square \bullet 2\square \bullet \square}{2 \bullet 2 \bullet P \bullet P \bullet \square^5}$$



G.S. Rohrer et al., Zeitschrift fur Metallkunde, 95 (2004) 197-214

Measuring Grain Boundary Plane Texture

Transmission Electron Microscopy

Goodhew, Yan, Balluffi, Acta Met. **35** (1978) 557 (just one example.)

Polarized Light Microscopy

Heilbronner and Pauli, J. Struct. Geo. **15** (1993) 369.

Orientation imaging microscopy (in SEM) with serial sections

Randle and Davies, Ultramicroscopy **90** (2002) 153.

Saylor et al. Acta Mater. **51** (2003) 3663

Stereology

Saylor et al., Metall. Mater. Trans., **35A** (2004) 1981.

Larsen and Adams, Metall. Mater. Trans., **35A** (2004) 1991.

Three Dimensional X-Ray Diffraction Microscopy

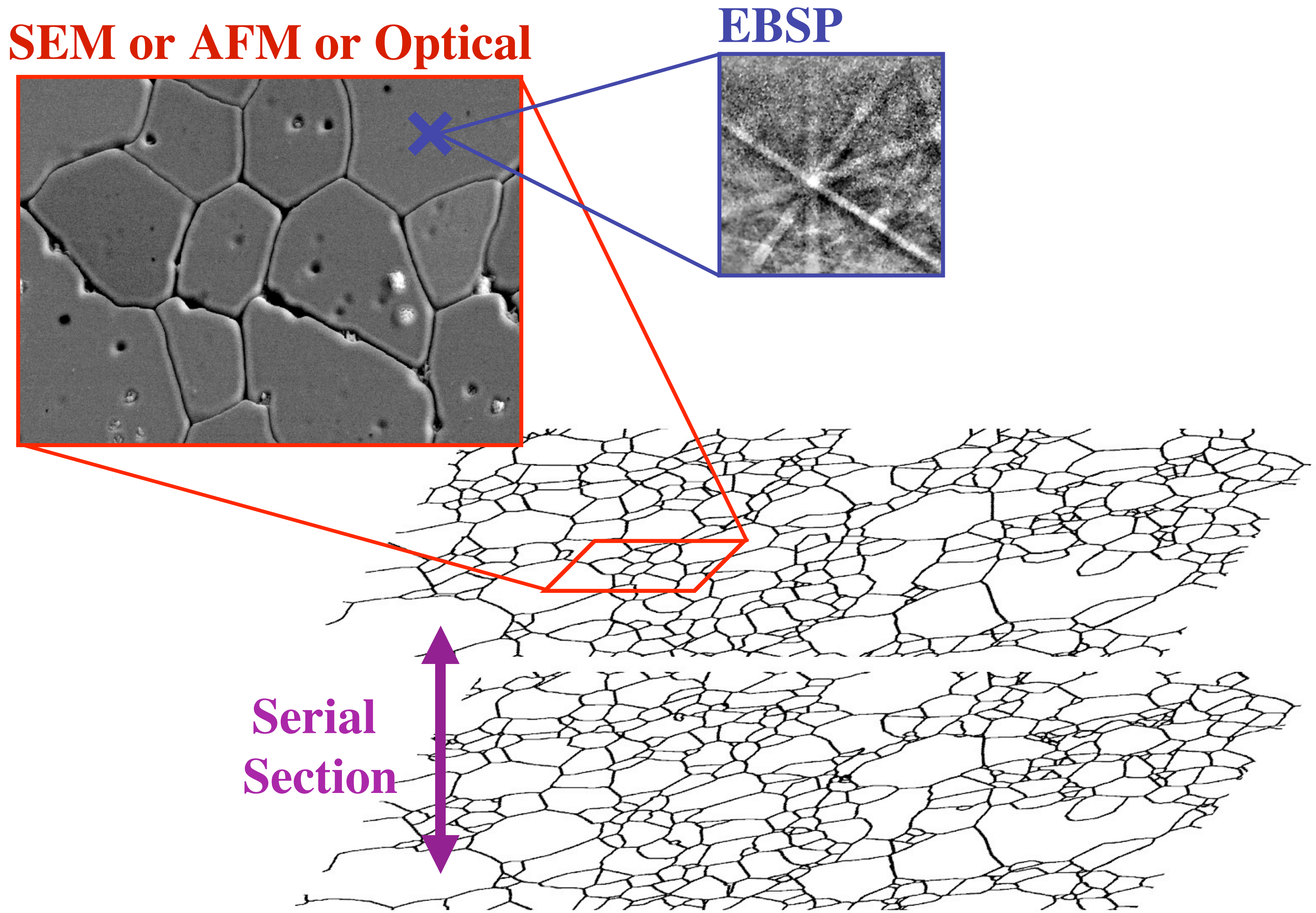
Poulsen et al., J. Appl. Cryst. **34** (2001) 751.

Schmidt et al. Science **305** (2004) 229.

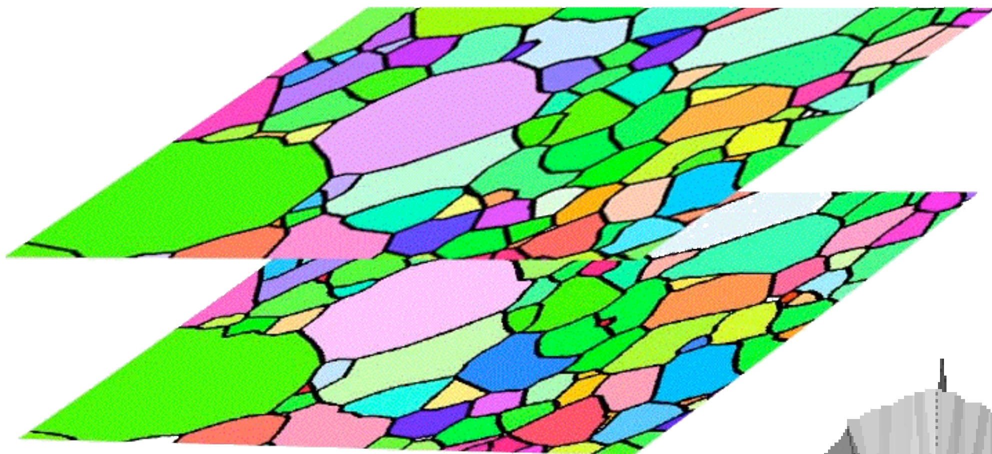
Differential Aperture X-ray Microscope

Larson et al. Nature **415** (2002) 887.

Measuring the Five Parameter Distribution



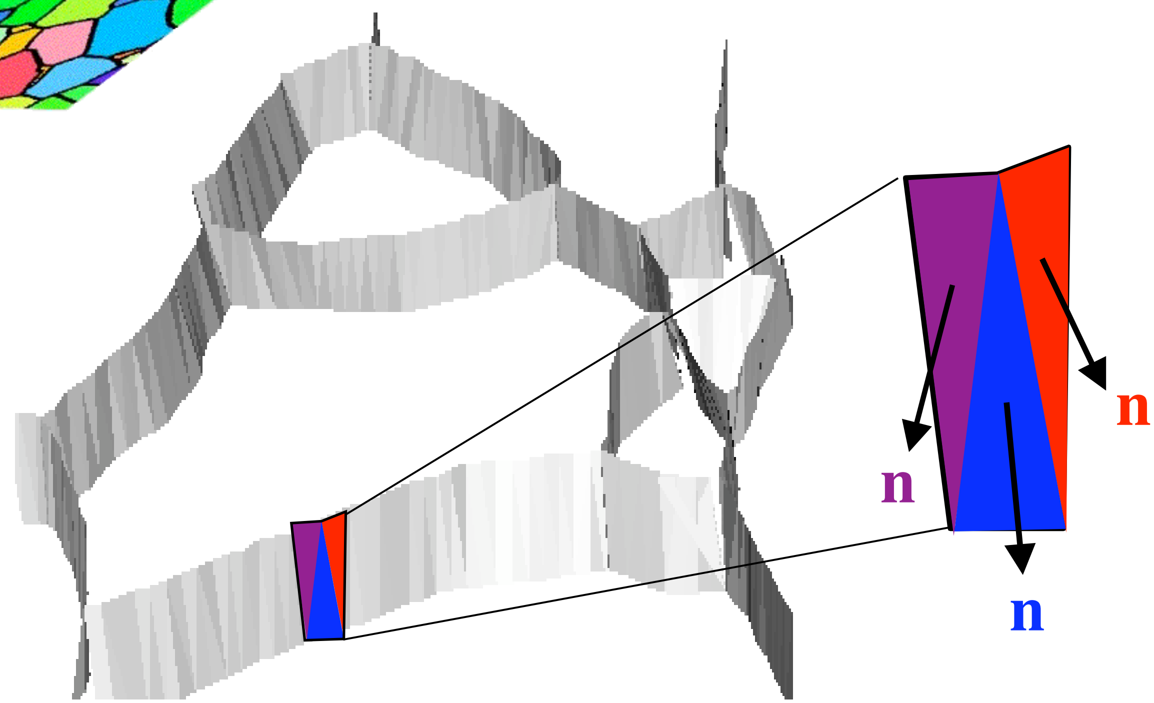
Measuring the five boundary parameters



Record high resolution EBSP maps on two adjacent layers.

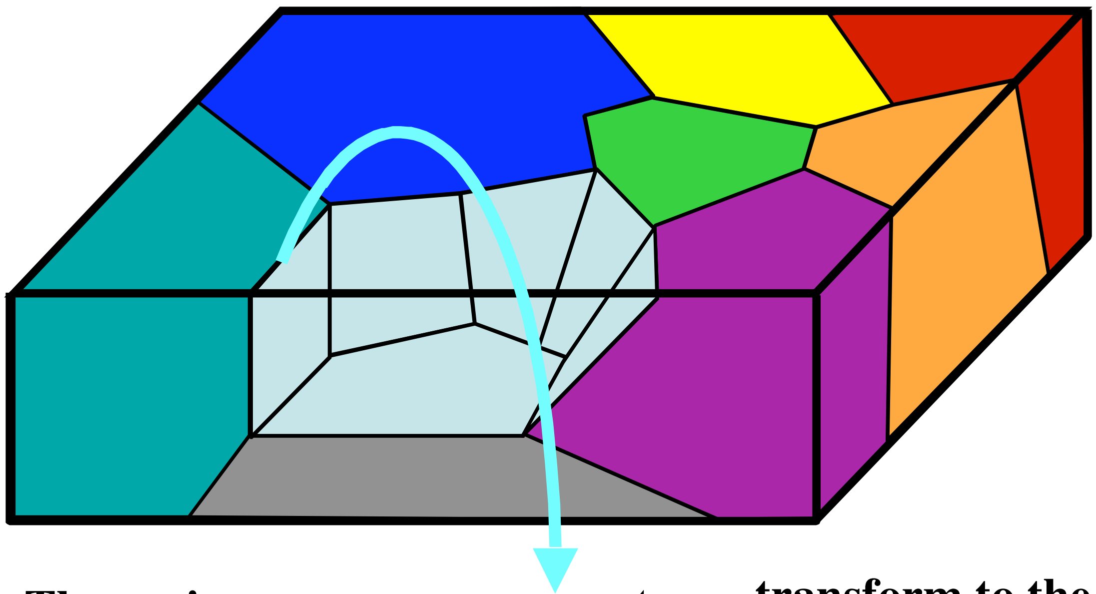
Assume triangular planes connect boundary segments on the two layers.

□g and **n** can be specified for each triangular segment



Saylor, Morawiec, Rohrer, Acta Mater. 51 (2003) 3663

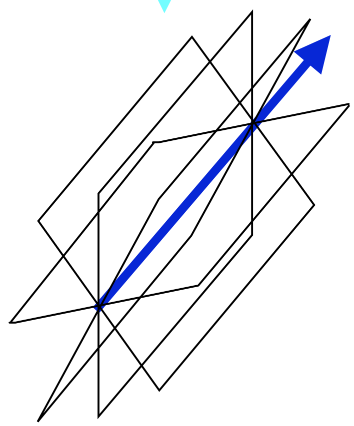
Stereology for measuring Δg and n



The probability that the correct plane is in the zone is 1.

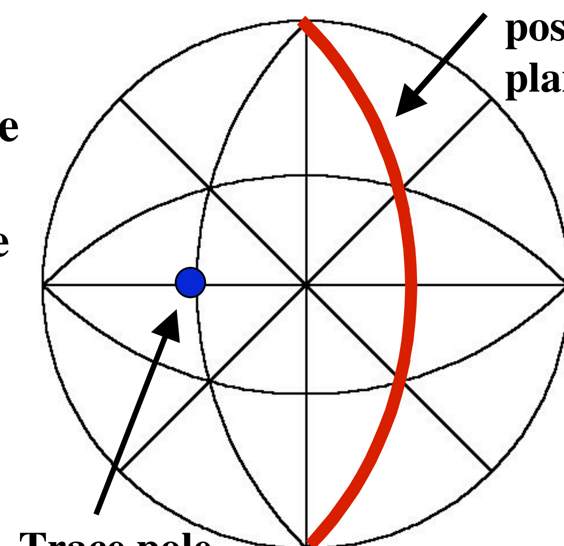
The probability that all planes are sampled is < 1.

The grain boundary surface trace is the zone axis of the possible boundary planes.



transform to the misorientation reference frame

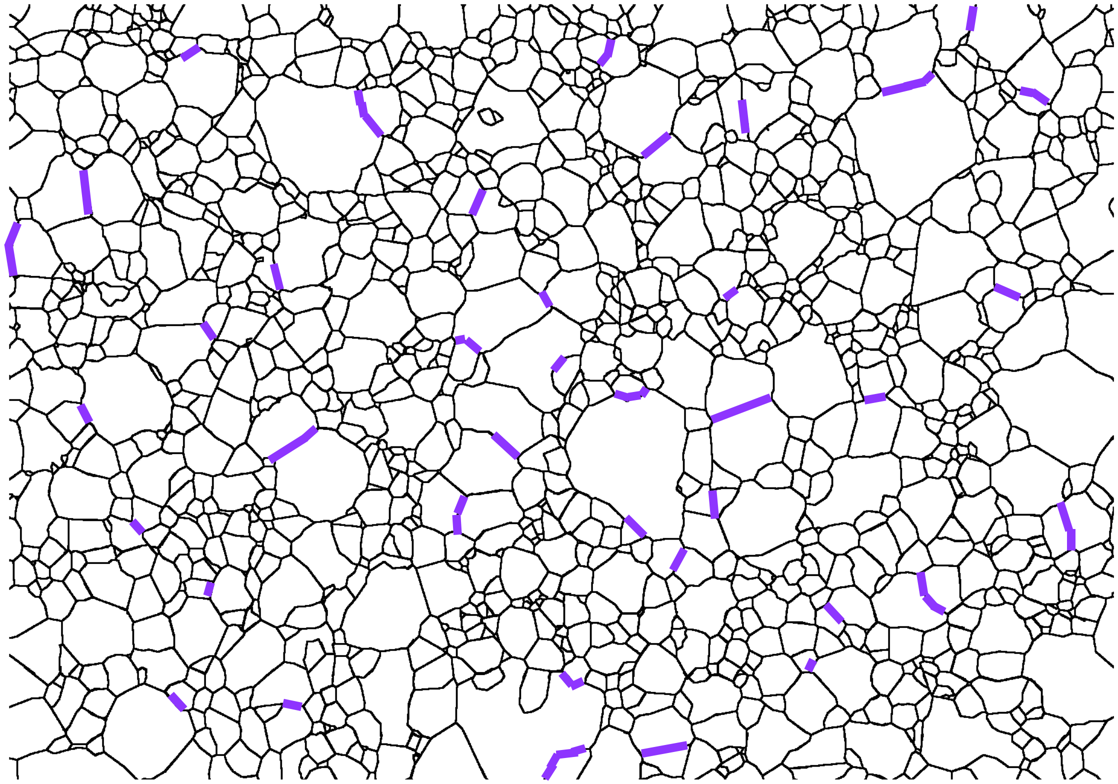
Δg



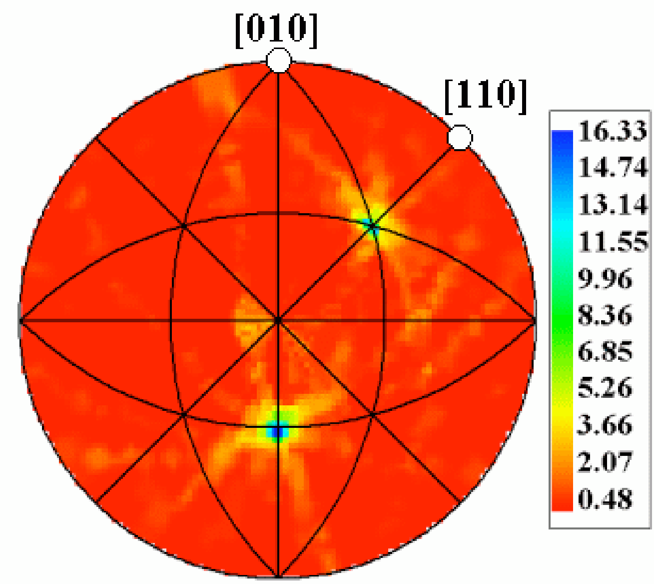
Saylor et al., Metall. Mater. Trans., 35A (2004) 1981.

Illustration of Boundary Stereology

Grain boundary traces in sample reference frame



All planes in the zone of trace, in the misorientation frame (at a fixed Δg)



N = 100 and … subtract

The background of accumulated false signals must then be subtracted.

- **The result is a representation of the true distribution of grain boundary planes at each misorientation.**
- **A continuous distribution requires roughly 2000 traces for each Δg**

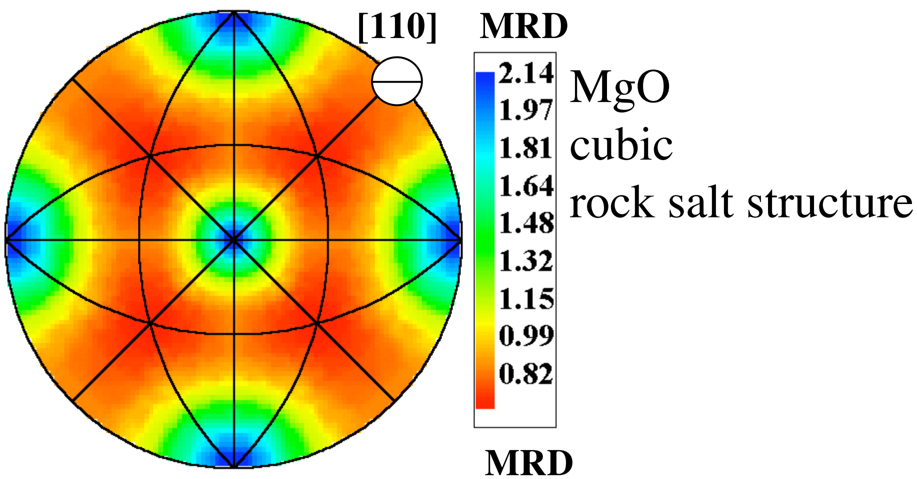
What have we learned about the grain boundary character distribution?

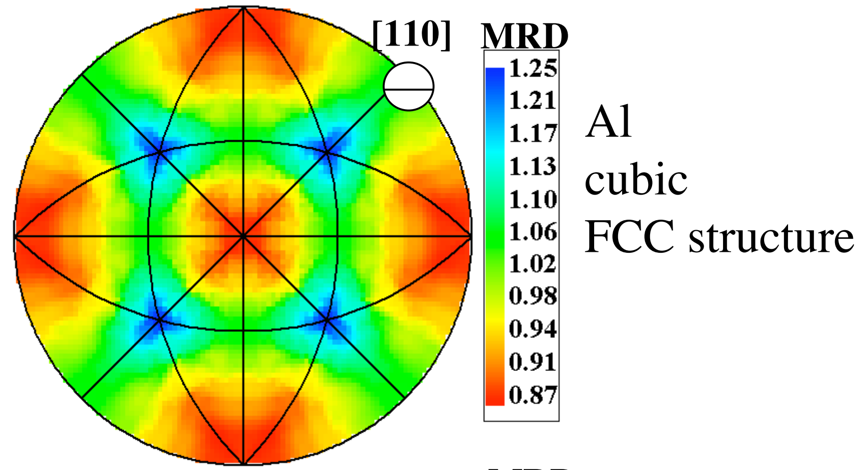
1. In a wide range of materials, grain boundary planes are textured to favor low index planes that are low energy free surfaces.

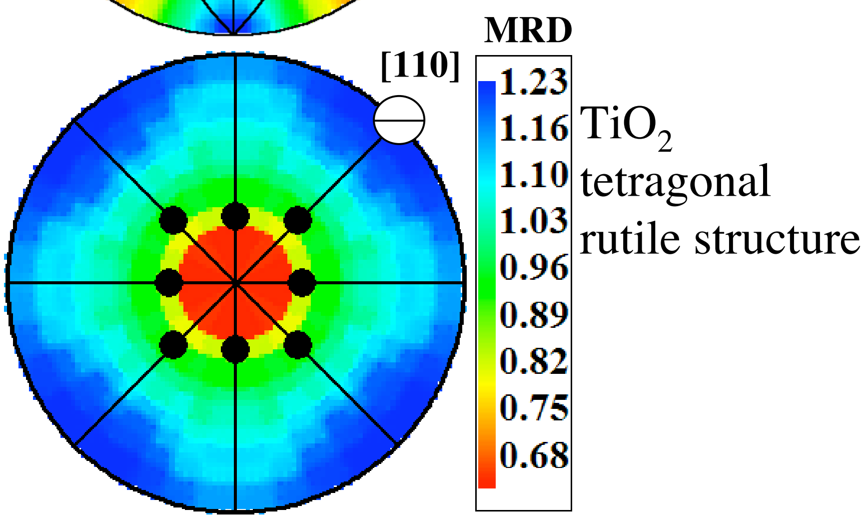
2. Grain boundary configurations in the five parameter space occur with a frequency that is inversely related to their energy. Boundaries formed from low surface energy planes have low energy.

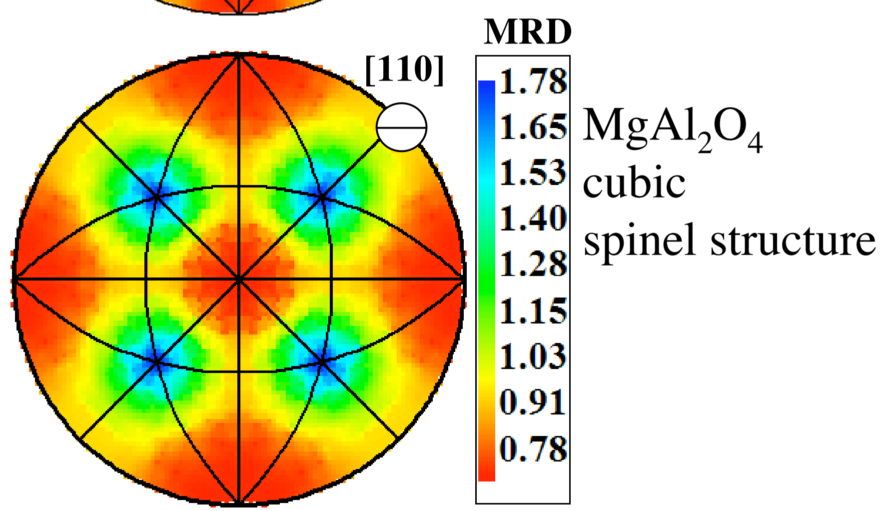
3. There is a steady state grain boundary character distribution that is predictable based on rules for curvature driven motion.

Grain boundary planes favor low index, low energy planes









Saylor et al., Acta Mater. 51 (2003) 3663
Saylor et al., J. Amer. Ceram. Soc., 87 (2004) 724
Saylor et al., Acta Mater. 52 (2004) 3649

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Grain boundary energy and population

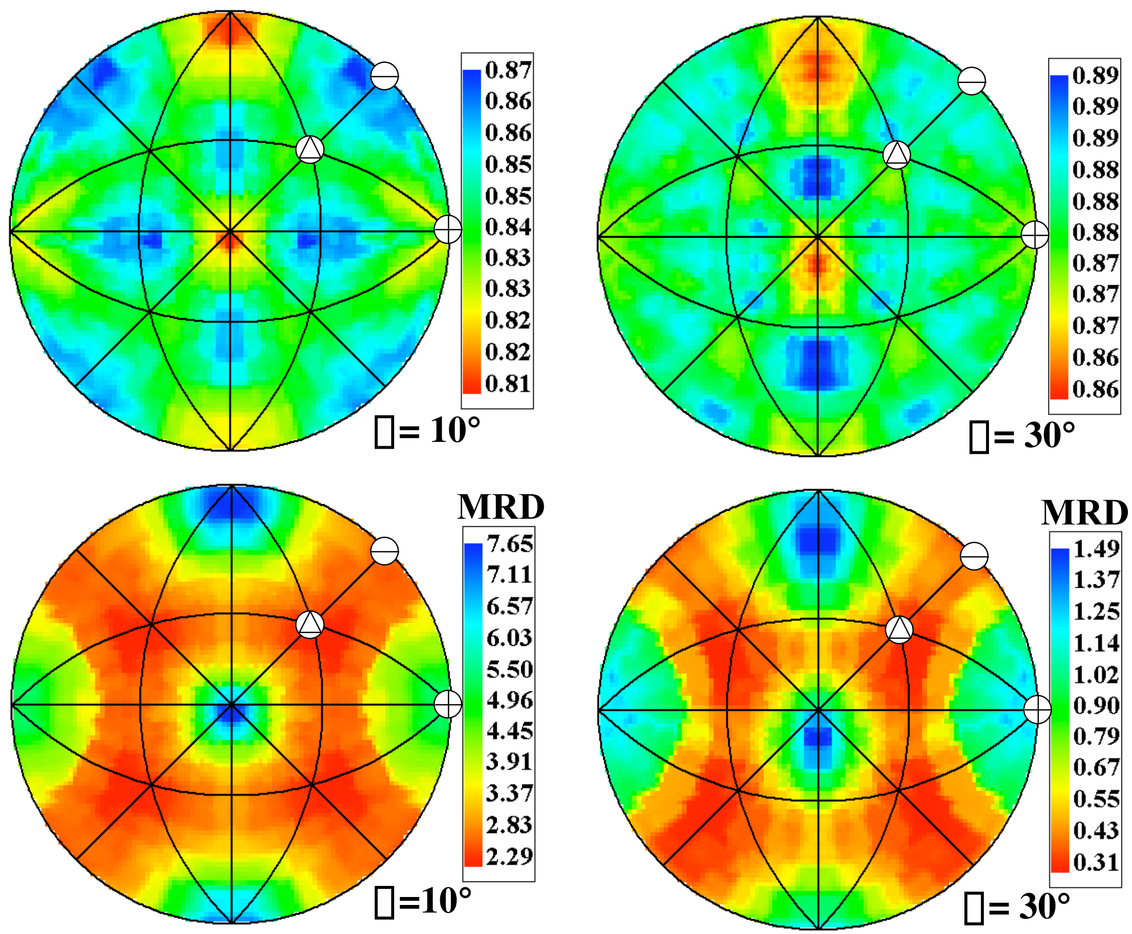
[100] misorientations in MgO

Grain boundary energy

□(**n|□/[100]**)

Grain boundary distribution

□(**n|□/[100]**)

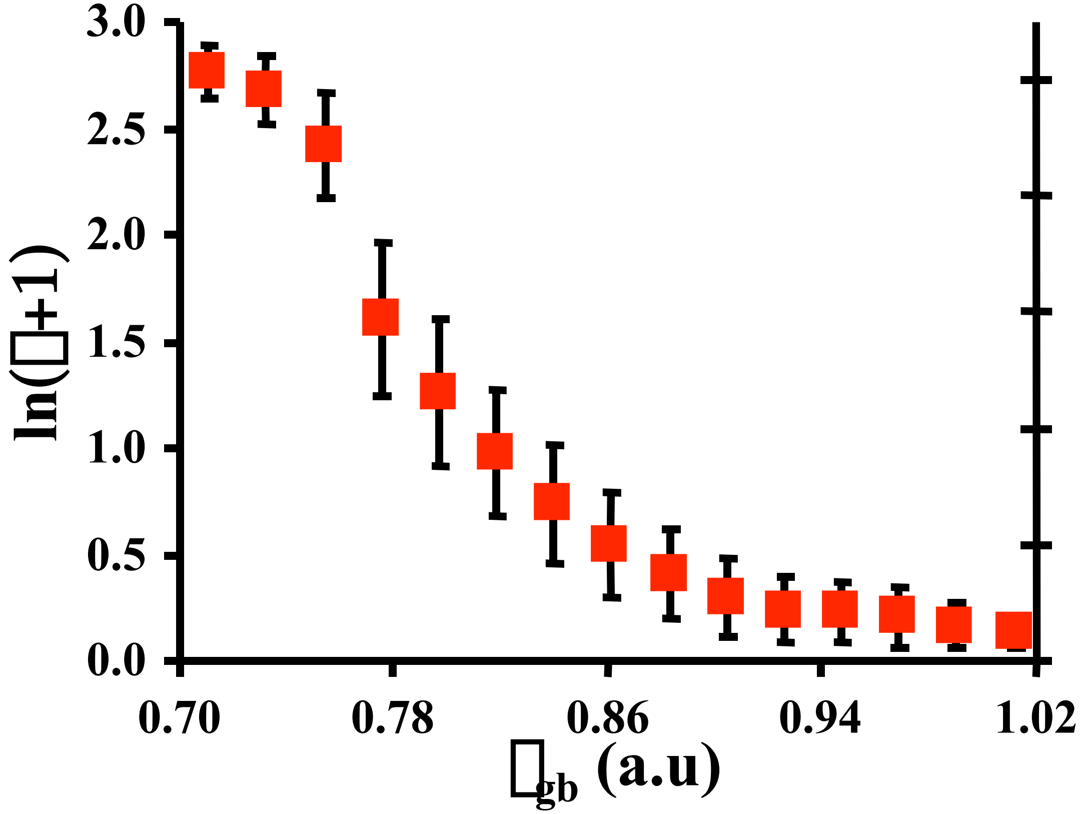


Population and Energy are Inversely correlated

Saylor, Morawiec, Rohrer, Acta Mater. 51 (2003) 3675

Grain boundary energy and population

For all grain boundaries in MgO

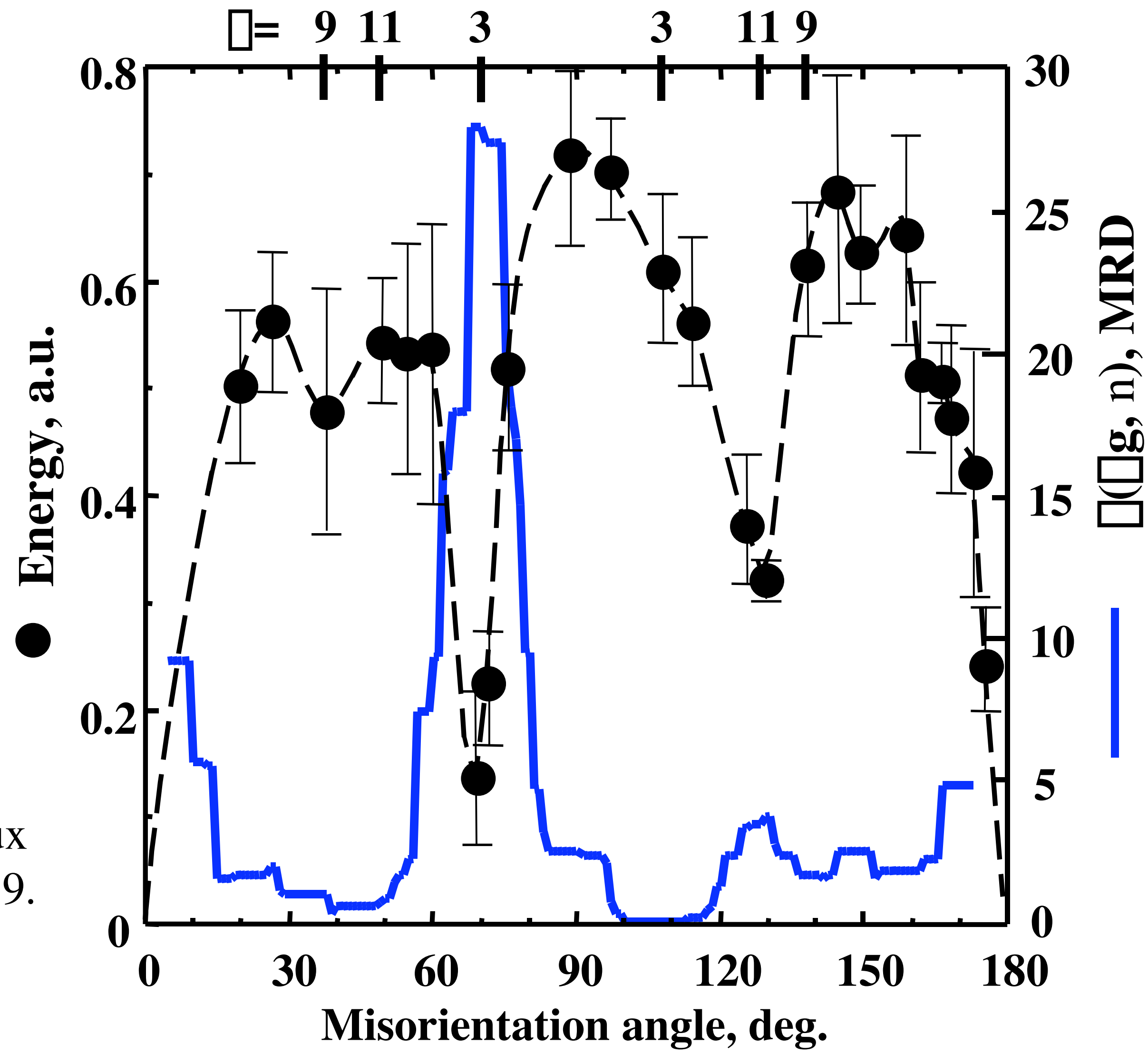


Population and Energy are Inversely correlated

Saylor, Morawiec, Rohrer, Acta Mater. 51 (2003) 3675

Boundary energy and population in Al

Symmetric [110] tilt boundaries



Energies:

G.C. Hasson and C. Goux
Scripta Met. 5 (1971) 889.

Saylor et al., Acta Mater. 52 (2004) 3649

Principal Results

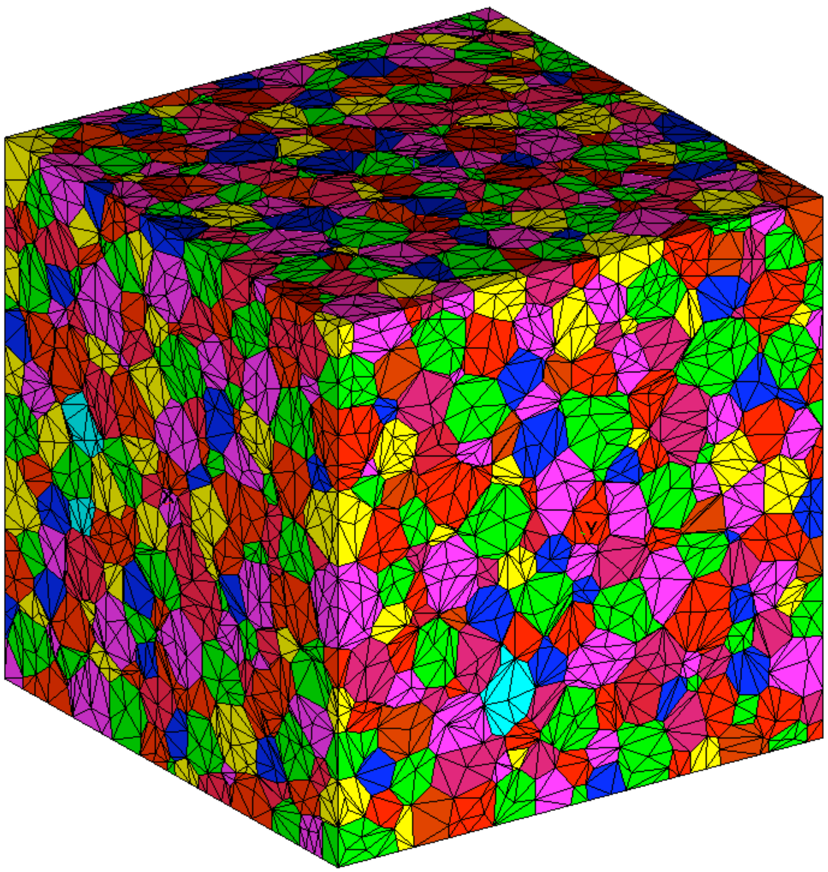
1. In a wide range of materials, grain boundary planes are textured to favor low index planes that are low energy free surfaces.

2. Grain boundary configurations in the five parameter space occur with a frequency that is inversely related to their energy. Boundaries formed from low surface energy planes have low energy.

3. There is a steady state grain boundary character distribution that is predictable based on rules for curvature driven motion.

Grain 3D Simulations

A.P. Kuprat: SIAM J. Sci. Comput. 22 (2000) 535.
Gradient weighted moving finite elements (LANL)



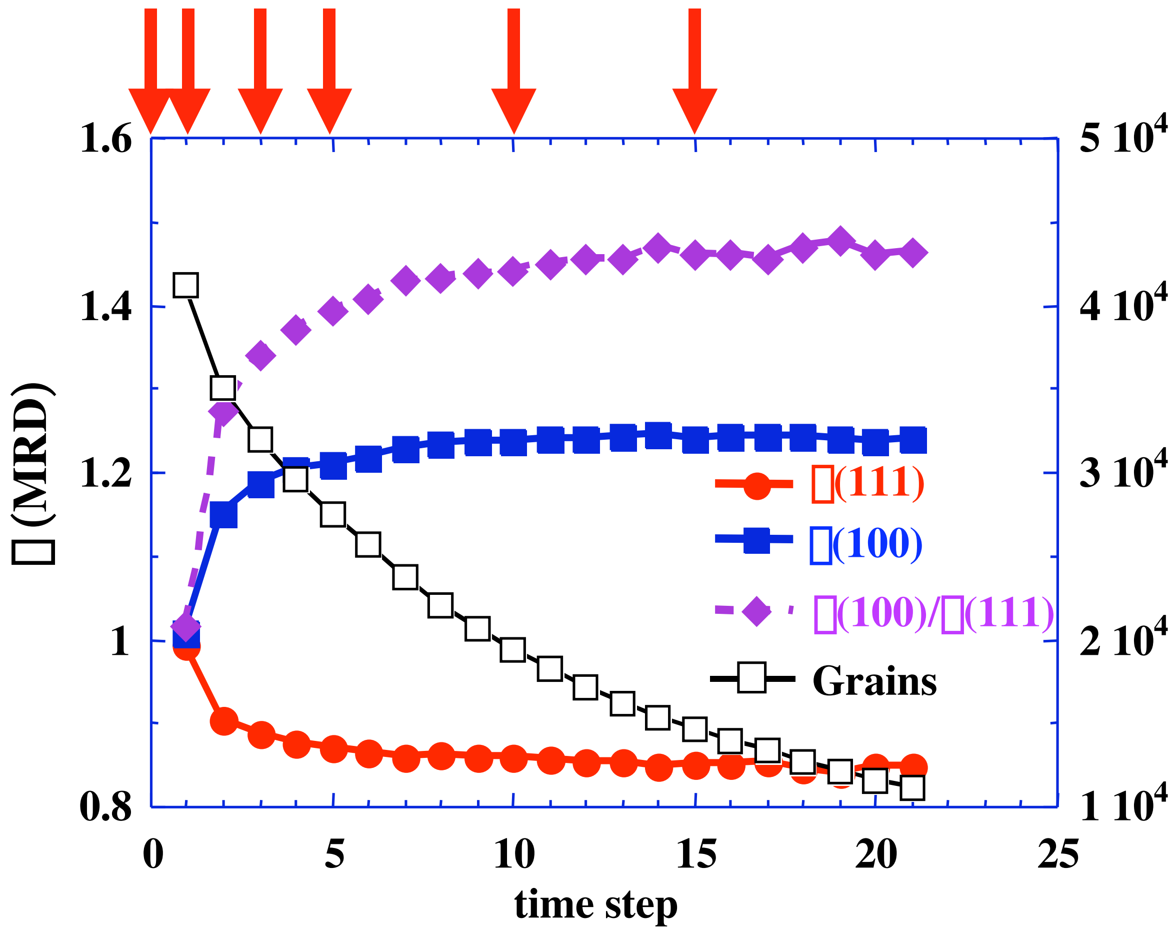
Elements move with a velocity that is proportional to the mean curvature

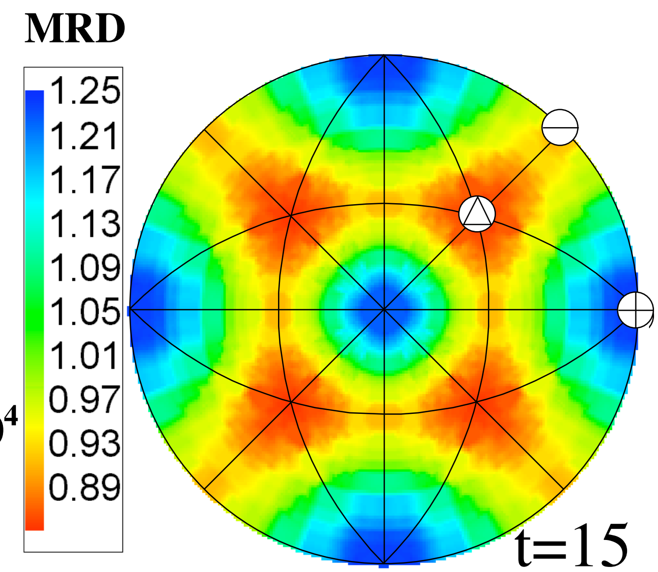
Initial mesh: 2578 grains, random grain orientations (16 x 2578 = 41248)

Use input energy function modeled after that observed for magnesia.

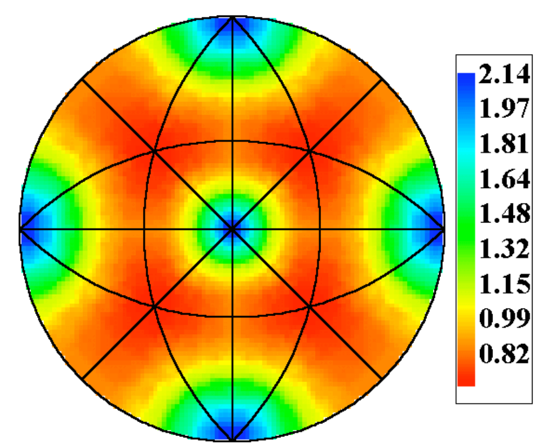
Grain 3D Simulations

- Input energy modeled after MgO
- Steady state arises that correlates with energy.





(n)



Gruber et al., 2nd International Conf. on Recrystallization and Grain Growth, Aug. 2004, in press.

Weaknesses of SEM approach to studying the grain boundary character distribution

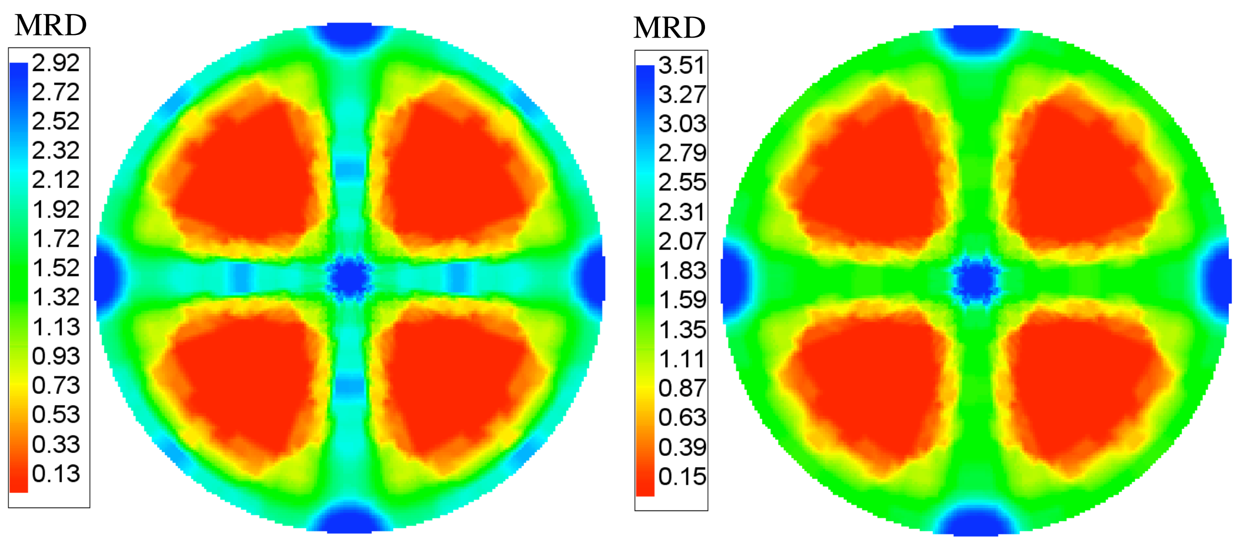
- destructive - does not allow true time lapse studies.
- The resolution in serial sections is limited by the sectioning technique and alignment method.

  -FIB sectioning is very accurate, but only over relatively small areas (few grains)
  -polishing covers very large areas, but has lower vertical resolution

- quantitative accuracy of stereology depends on random sampling of large numbers of bicrystals.
- If lattice parameters are close, fully symmetry is not recognized.

Opportunities for High Energy X-ray Experiments: what we would like to know

- How does □(□g, **n**) vary with time?
- By what mechanism do distributions reach steady state during recrysallization and grain growth?



0 h at 1500°C   24 h at 1500°C

Distribution of planes bounding $SrTiO_3$ in a high temperature liquid

Sano et al., J. Amer. Ceramic Soc., (2004) in press.

Opportunities for High Energy X-ray Experiments: what we would like to know

Other dynamic processes:

- Field driven internal boundary motion
  - grain boundaries driven by electric or magnetic fields
  - ferroelectric domain walls and their interactions
- Relative contributions of grain boundary sliding, plastic flow, and grain rotation to deformation
- Topological data on grain shapes: is there a topological distinction between growing and shrinking grains?
- Nucleation of solid/solid phase transformations

Benchmarks for (static) grain boundary character distribution data

- Angular resolution: 10° (number of distinct boundaries ~ 6500)

- Spatial resolution: 100 nm (within section plane)

- Number of grains: 5,000 (serial sections) or 25,000 (stereology)
  (A complete volume containing 1600 grains should provide a comparable amount of data)

- Time on Microscope: 2 to 10 days (instrumental and materials factors are important) $1000 to $10,000

Summary

Studies of the grain boundary character distribution have advanced in recent years, mainly by taking advantage of digital automation in the collection and analysis of data.

By measuring the the grain boundary character distribution as a function of all five macroscopically observable parameters, we have established a new metric for characterizing microstructures and are beginning to understand its relationship to the physical properties of interfaces and macroscopic materials properties.

High Energy X-ray Diffraction Microscopy techniques have the potential to address important topological and dynamic questions regarding the structure of the interfacial networks within polycrystals.