Upper Tails in Grain Size Distributions

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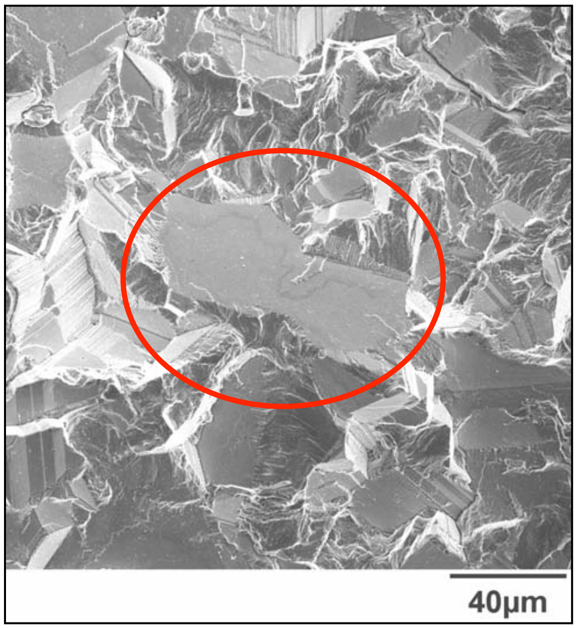
Motivation

Motivation to Incorporate Extreme Values

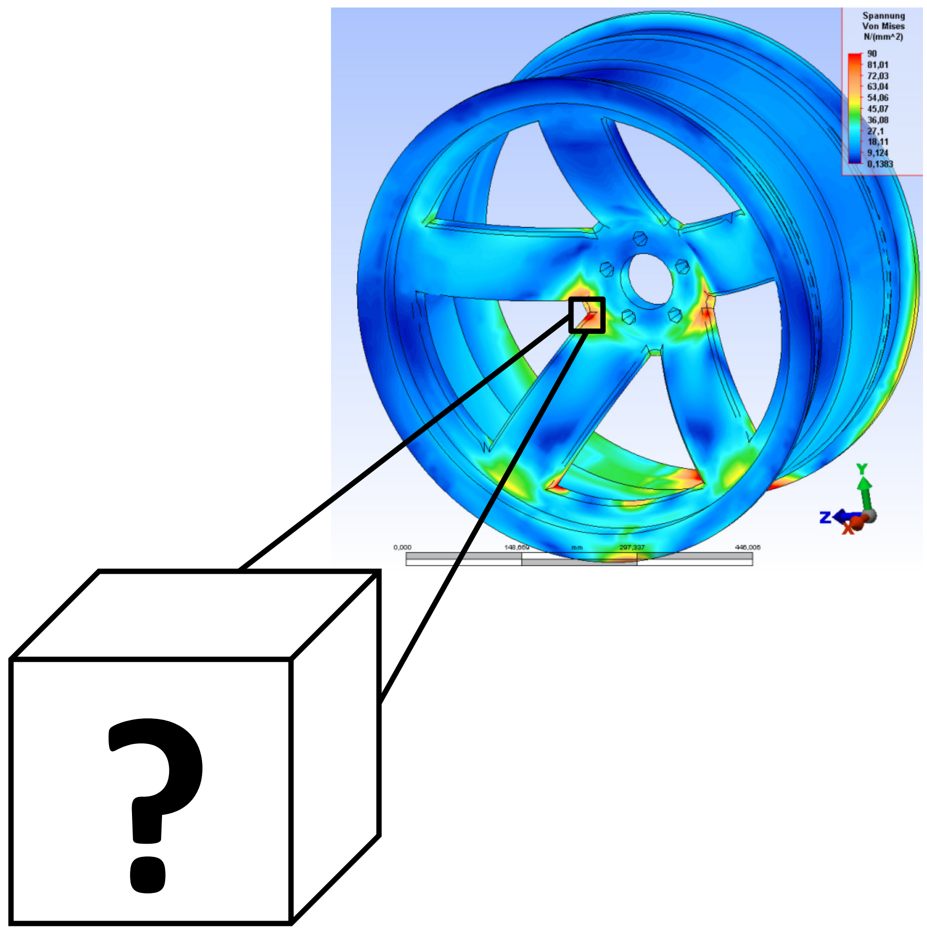
*‘Forget the Representative Volume Element, show me the **Weakest** Volume Element’ – paraphrased from Jim Williams*



Ni-base superalloys

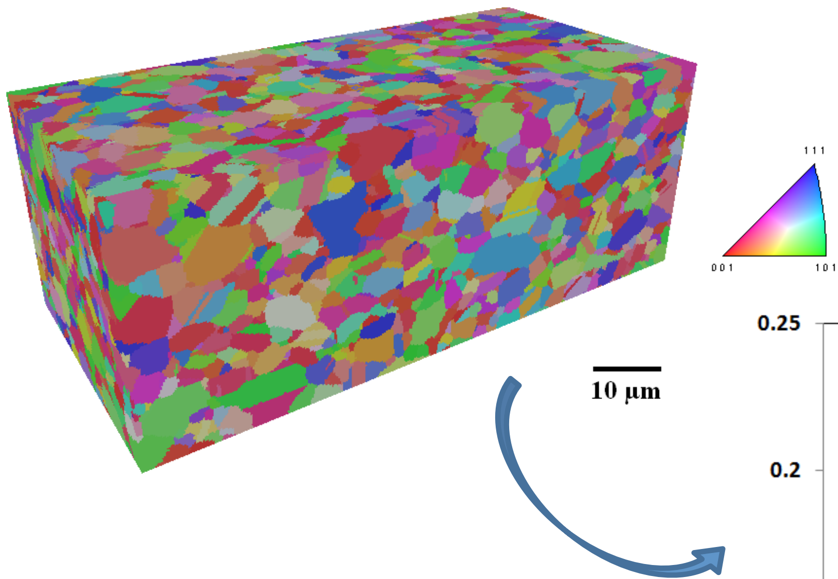
Fatigue crack initiation was observed in large grains oriented for slip

(Jha, et al., 2007)

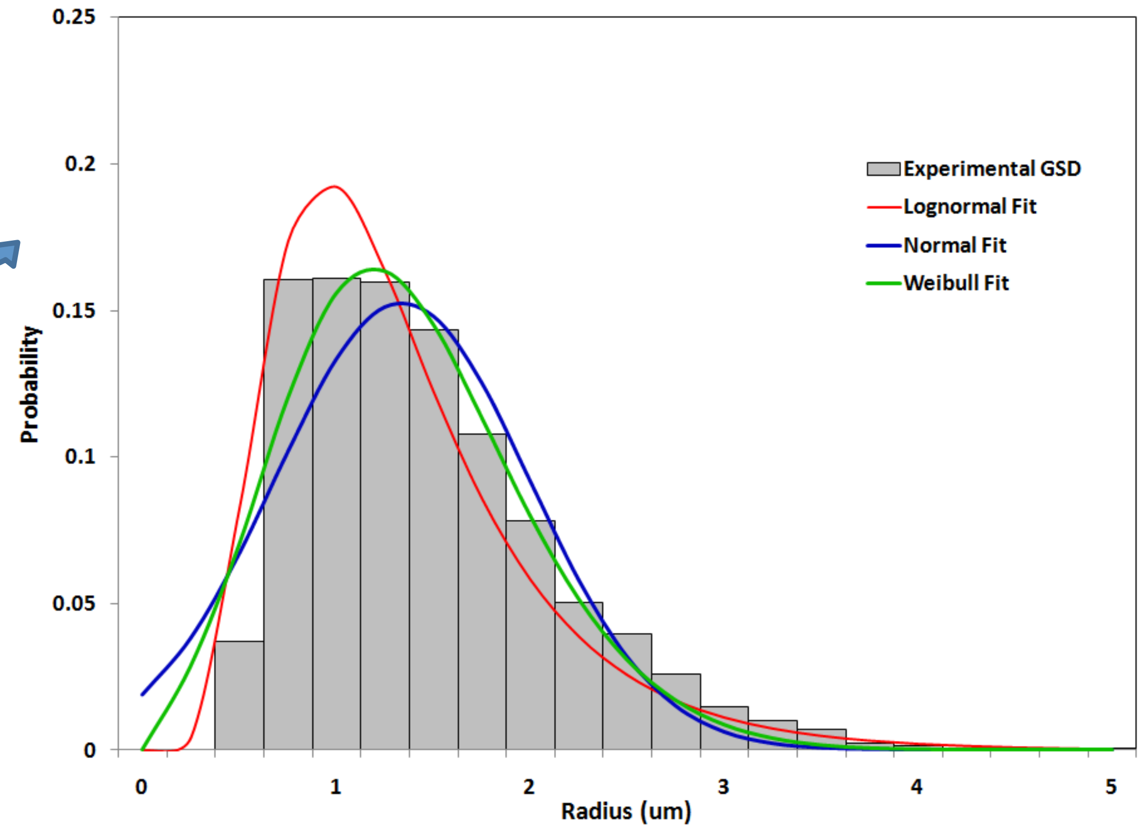


Background

PM IN100 Ni-base Superalloy Example

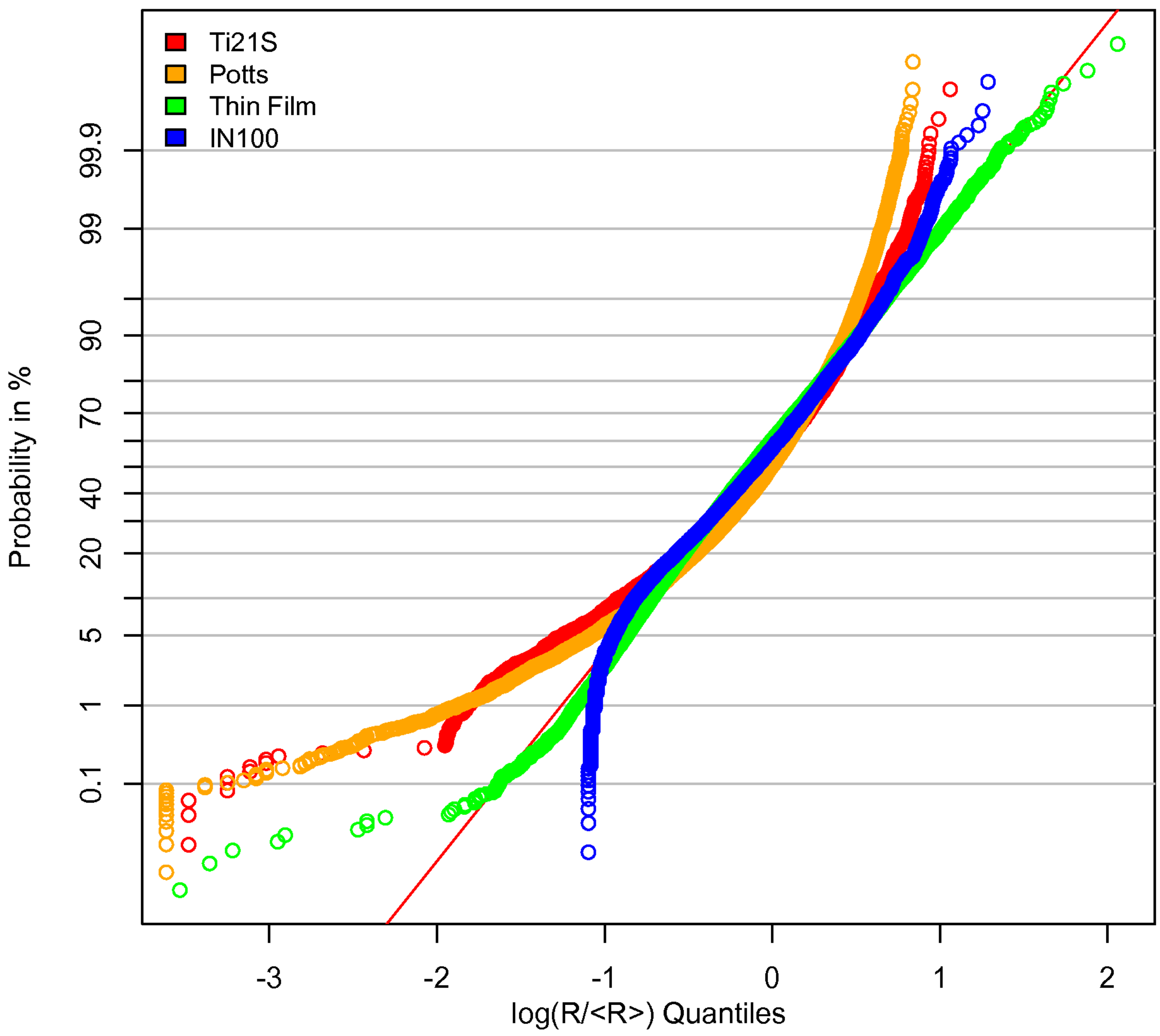


Lognormal Fit Appears 'Adequate'



- 3D EBSD data from FIB-SEM
- 100µm x 35µm x 45µm
- 0.25µm Section thickness
- 0.25µm EBSD point spacing
- > 8500 Grains in total
- > 5800 Grains analyzed
- 24 Voxel minimum size (~ 0.5µm)

Combined Probability Plot



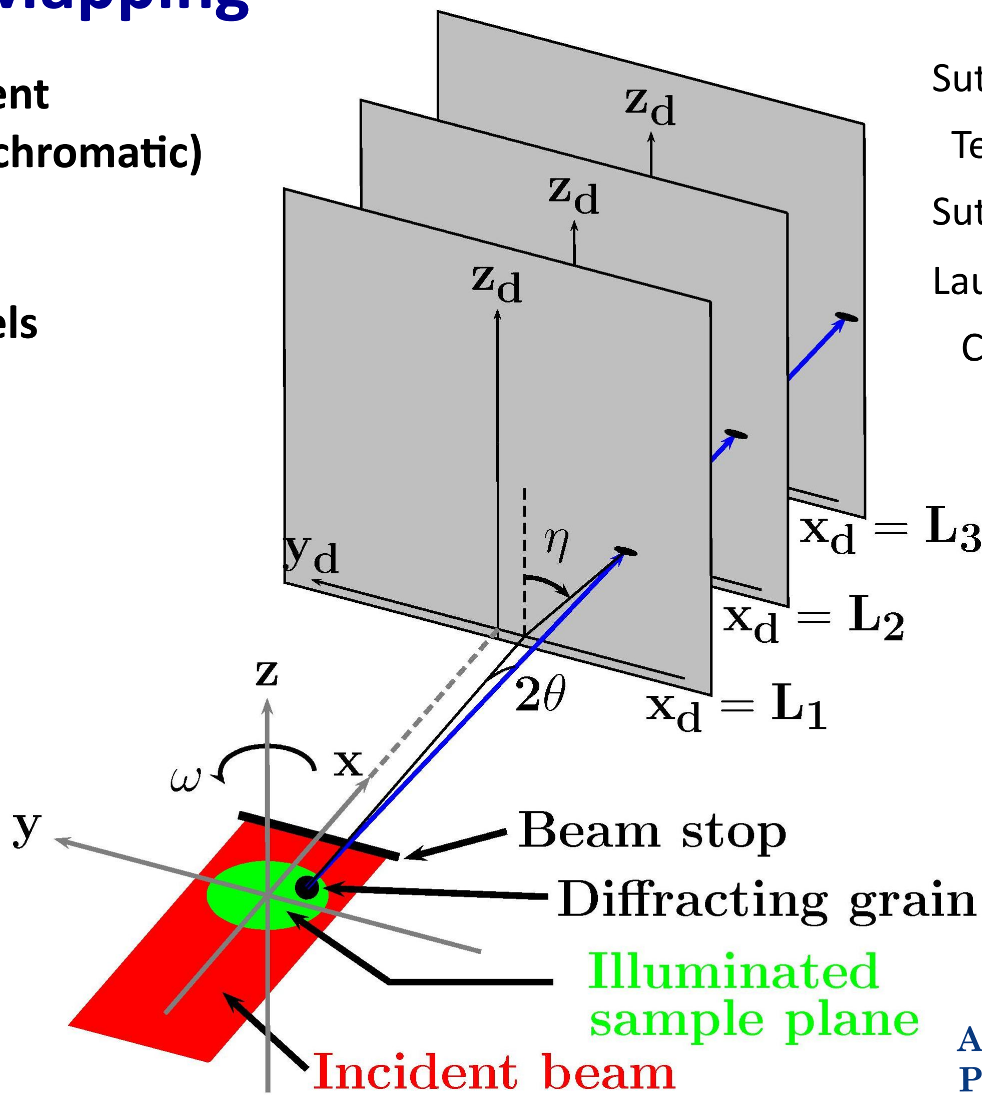
High Energy Diffraction Microscopy (HEDM): Microstructure Mapping

- Near-field measurement
- > 50keV x-rays (monochromatic)
- 1 – 2 μm beam height
- 1.3 mm beam width
- 1 – 4 μm detector pixels
- L = 5 – 10 mm

- Spatial resolution:
  2 – 4 μm
- Orientation resolution:
  < 0.5 degrees
- Analysis:

15 – 50 layers / 12hours

New: 100 layers in less than 1 day



Suter *et al*, Eng Mat & Tech 2007

Suter *et al*, RSI 2006

Lauridsen *et al.*, Appl Cryst 2001

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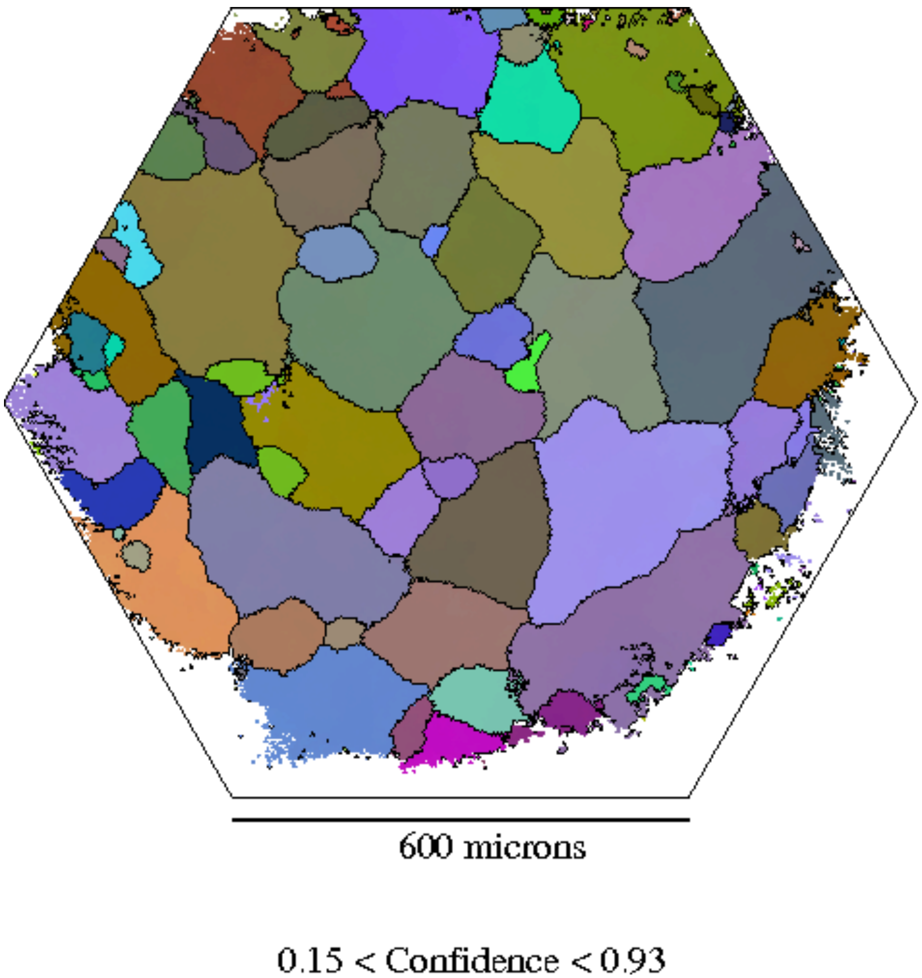
HP Al: Intra-granular Misorientations

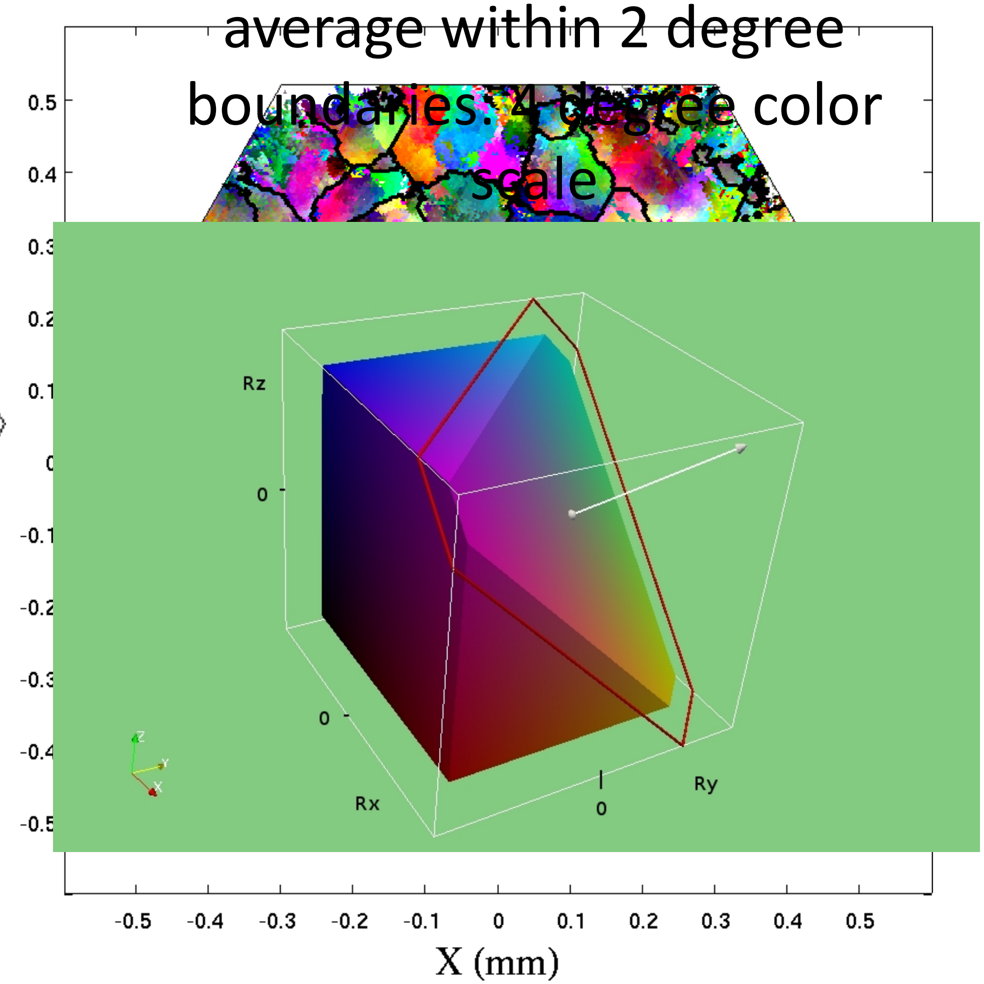
Orientations



2 deg boundaries

Misorientation

Next: Deviations from average within 2 degree boundaries. 4 degree color scale

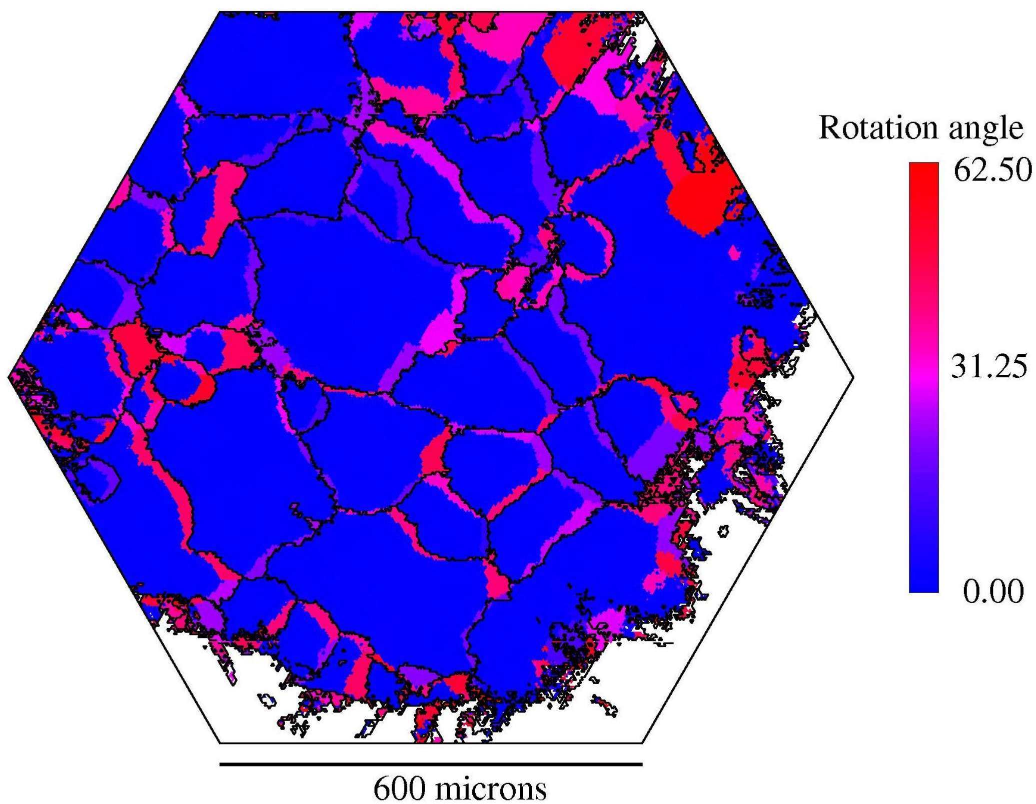


- Expanded color scale
- 2 deg boundaries

Grain Growth Dynamics in High Purity Aluminum

Initial-Final Misorientation

Initial state boundaries



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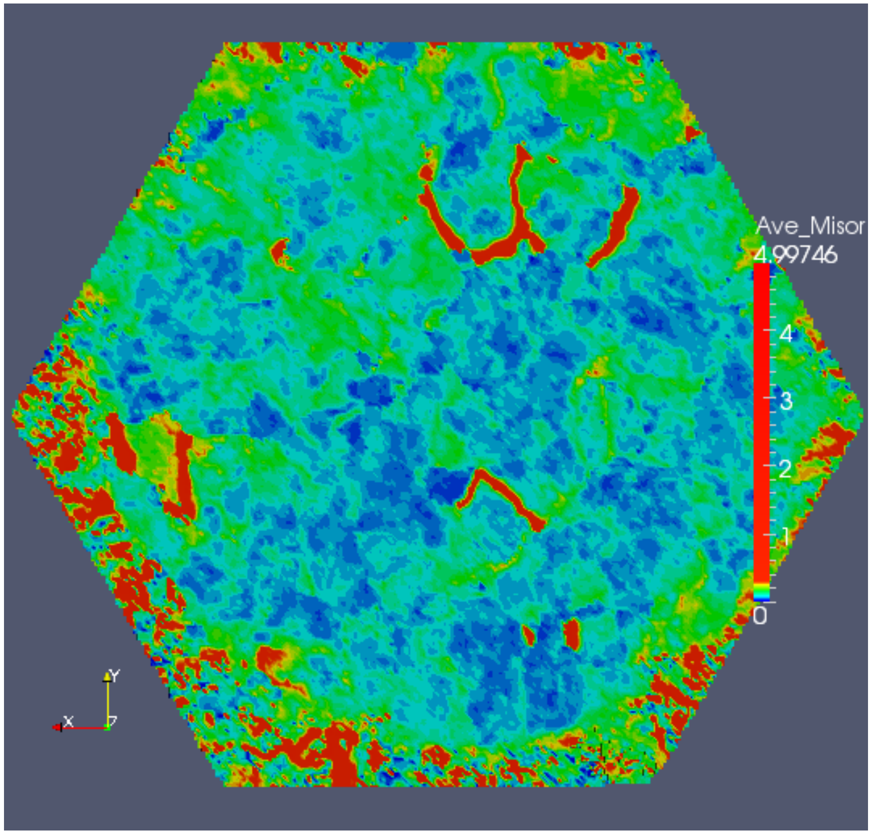
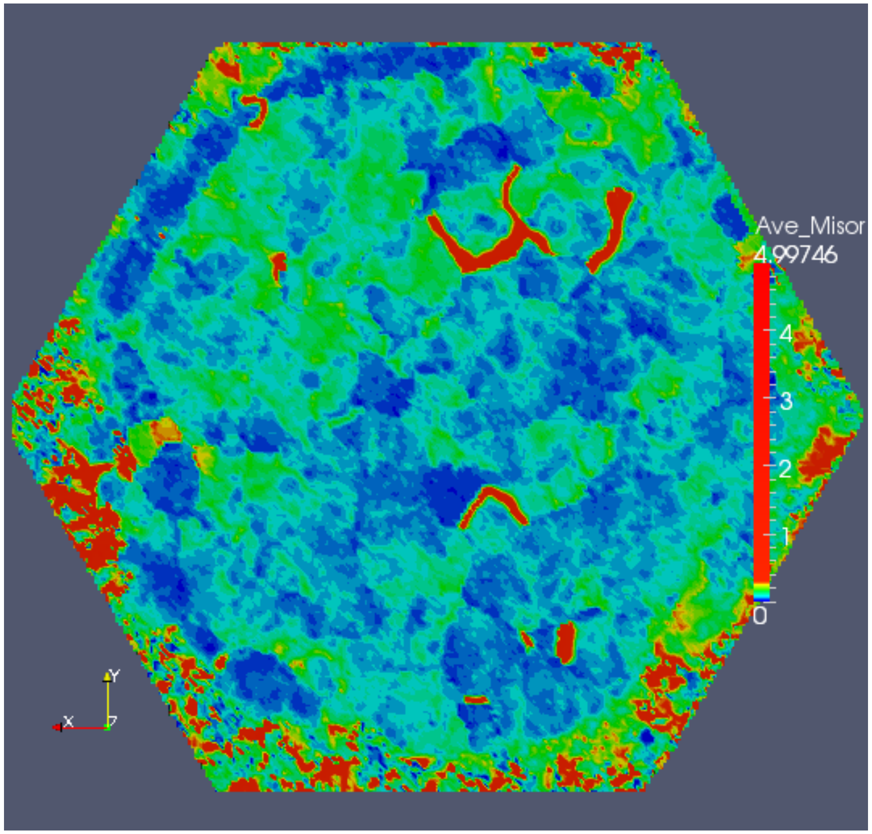
Geometrically Necessary Dislocation Content

Calculated as the Read-Shockley scaled energy associated with each point; max. scaled to 0.3. Trend is for the GND content to decrease.



Initial State



Final State

Analysis by LAGB Content

Matched grains between the initial and final states according to orientation.

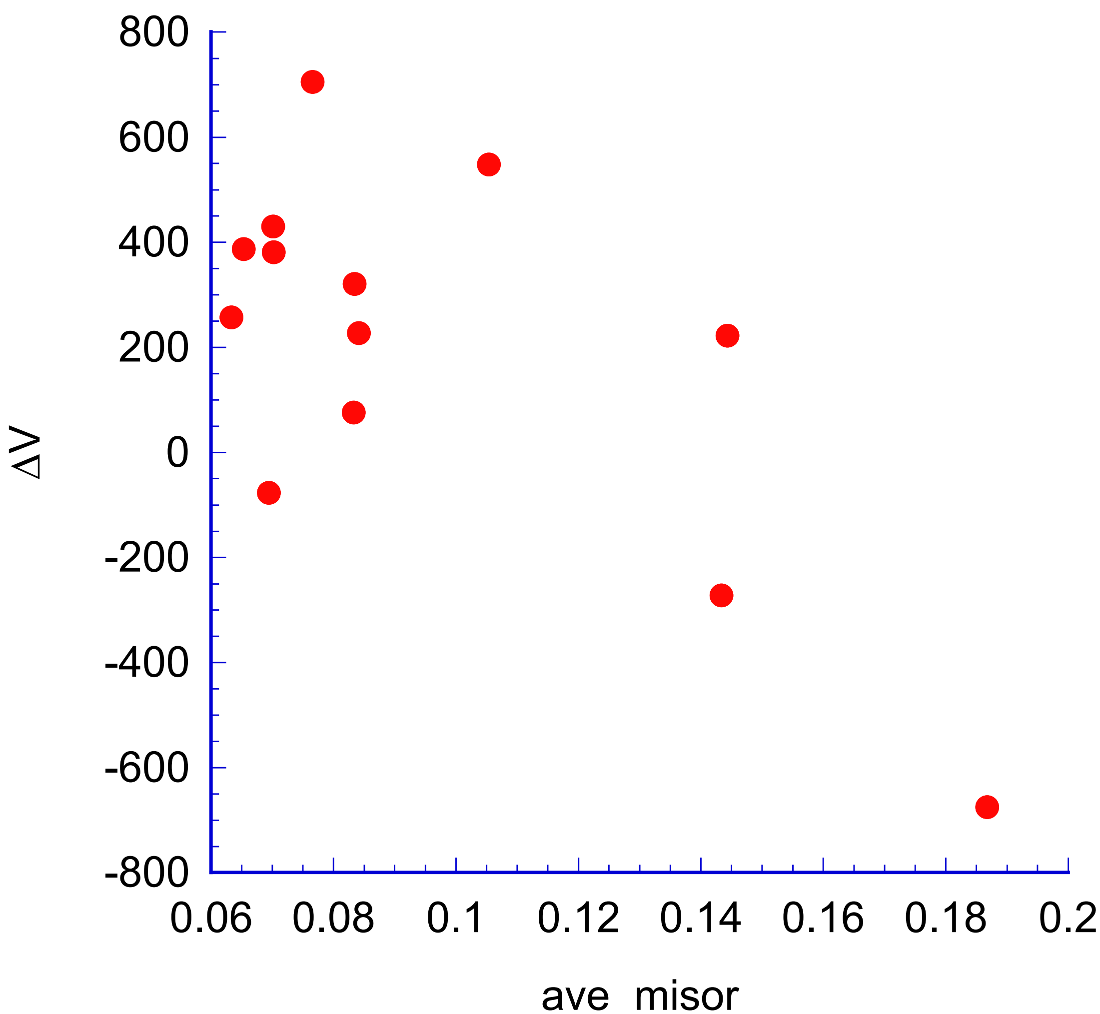
Excluded edge grains.

Computed the change in volume between snapshots.

Computed the Low Angle Boundary Content (as a rough measure of GND) by grain.

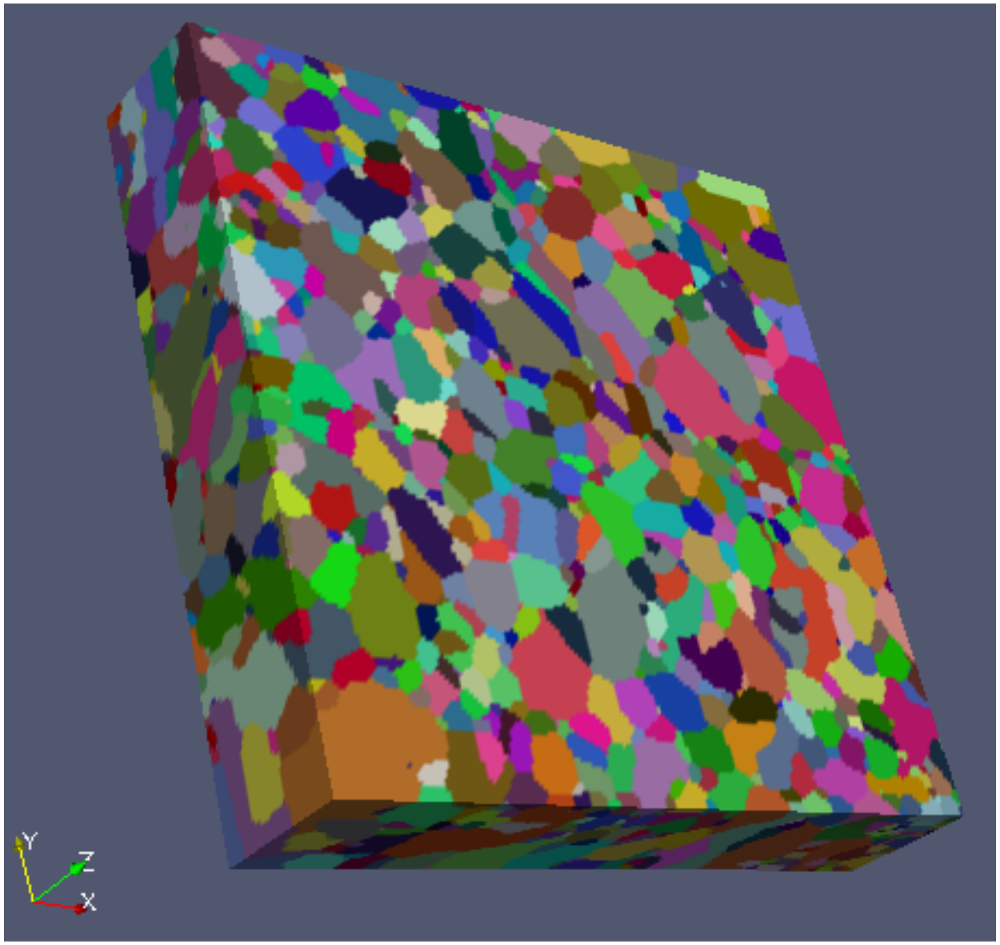
Plotted ΔV vs. GND.

Obtained expected trend of high stored energy leading to decrease in size (shrinks) and *vice versa*.



Three Volumetric Data Sets with Well Ordered Grains

1. Pure Nickel: 42 layers, 4 micron spacing, 0.16 mm^3
2. Bi-doped Nickel: 62 layers, 4 micron spacing, 0.24 mm^3
3. Pure Copper: 177 layers, 4 micron spacing, 0.56 mm^3

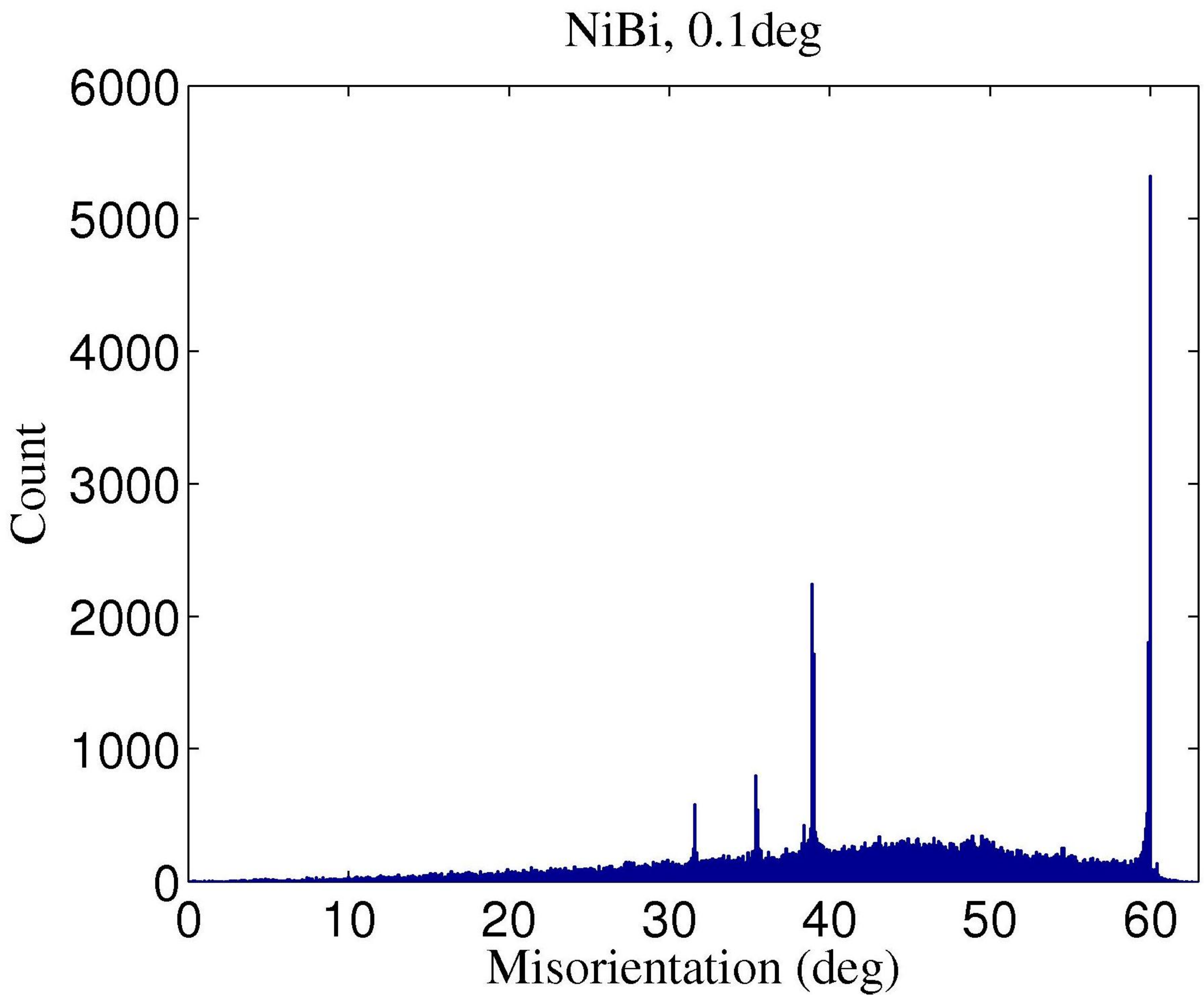


Next slide: movie through volume mesh of Ni-Bi

3,496 grains; ~ 23,598 GBs

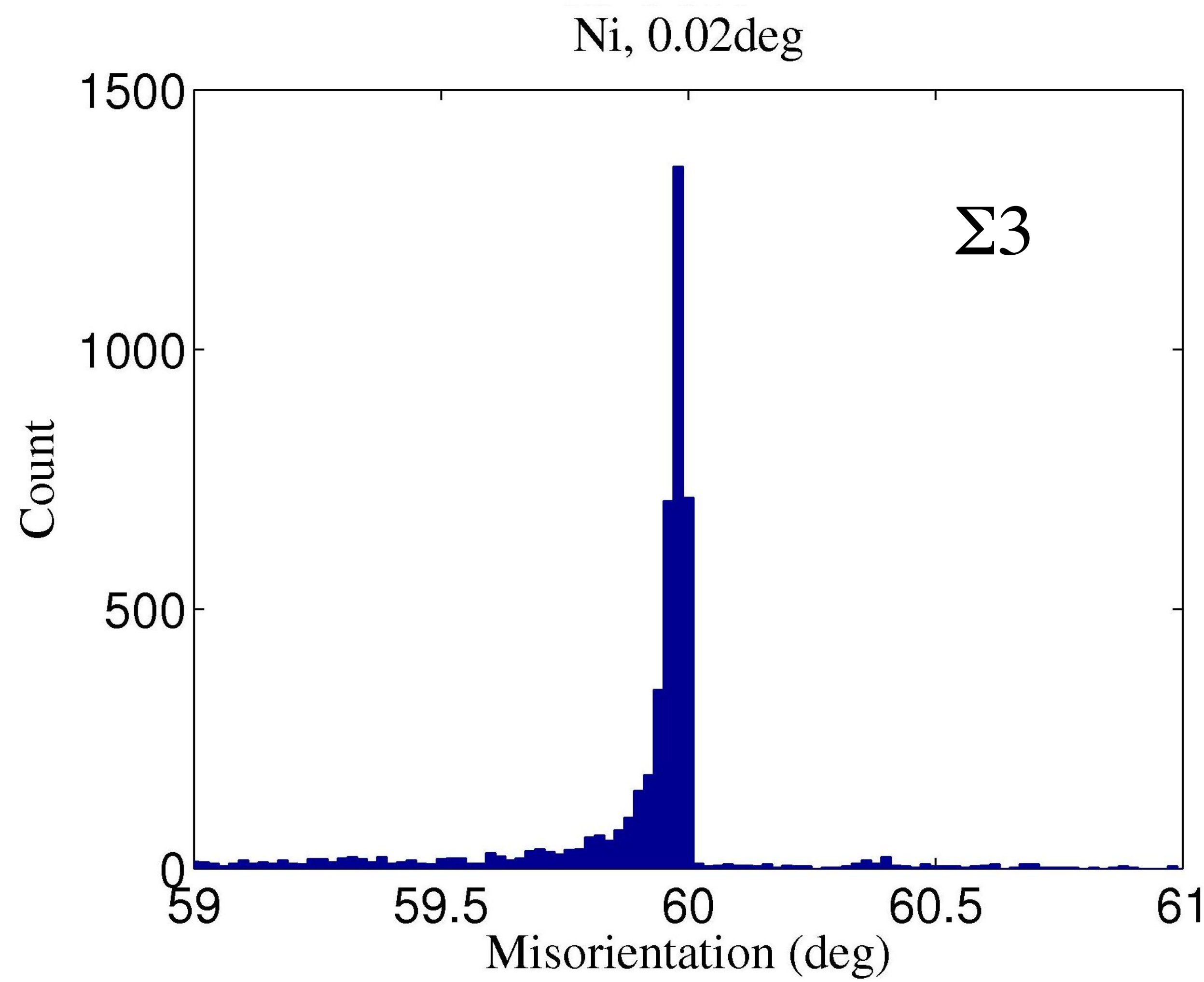
Statistics extraction from large data sets

Neighbor misorientation angle distribution

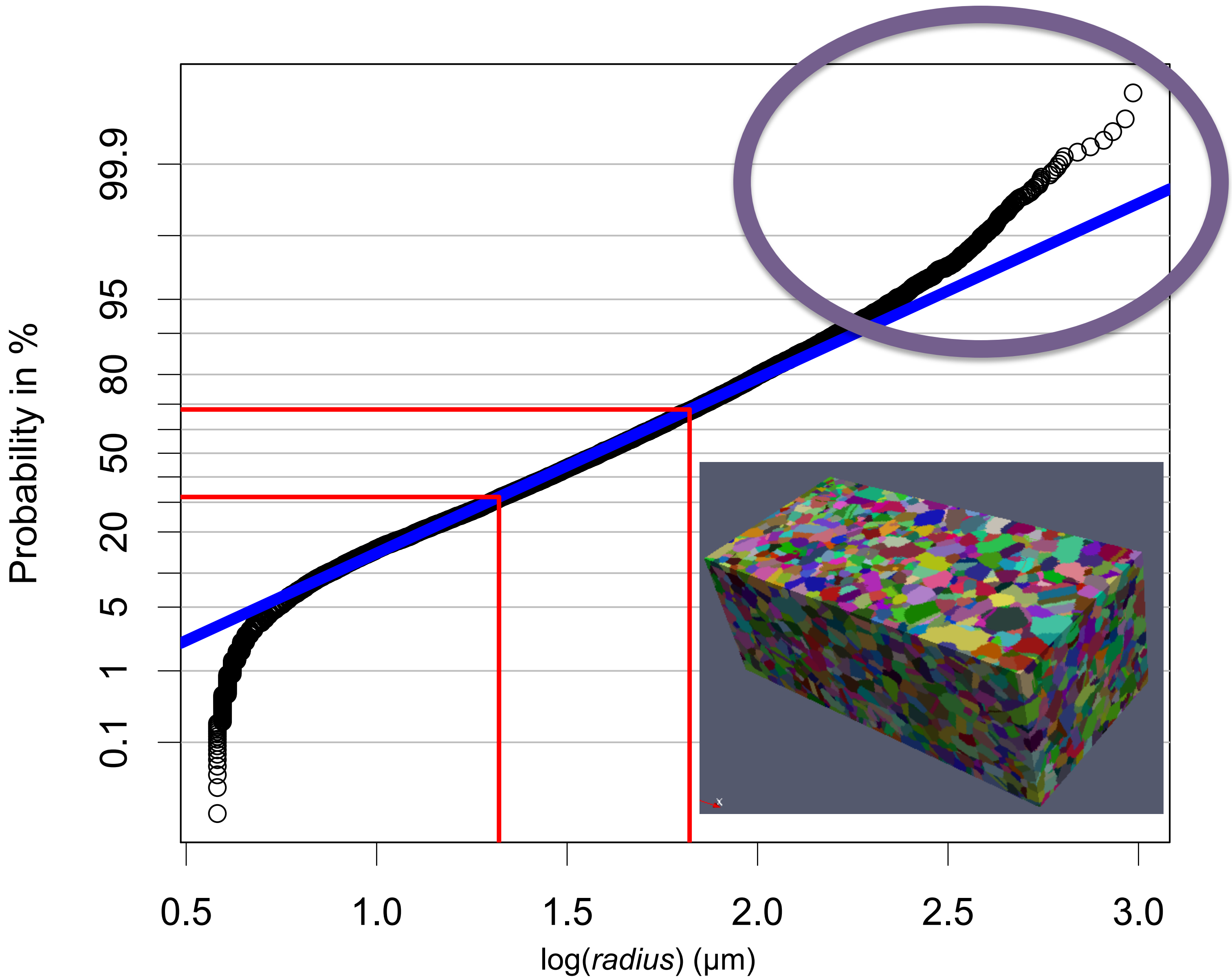


Statistics extraction from large data sets

Neighbor misorientation angle distribution

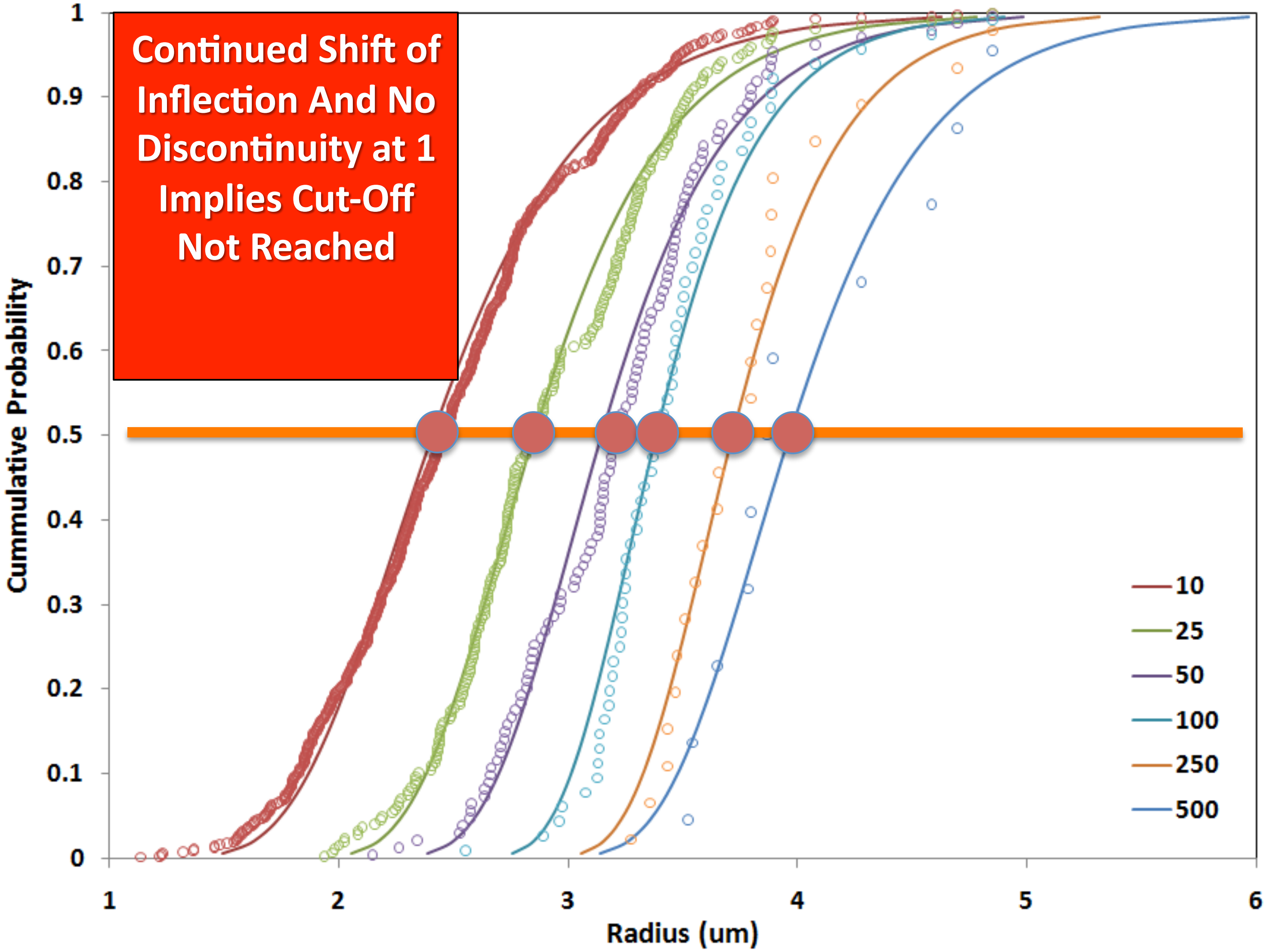


IN100: Probability Plot

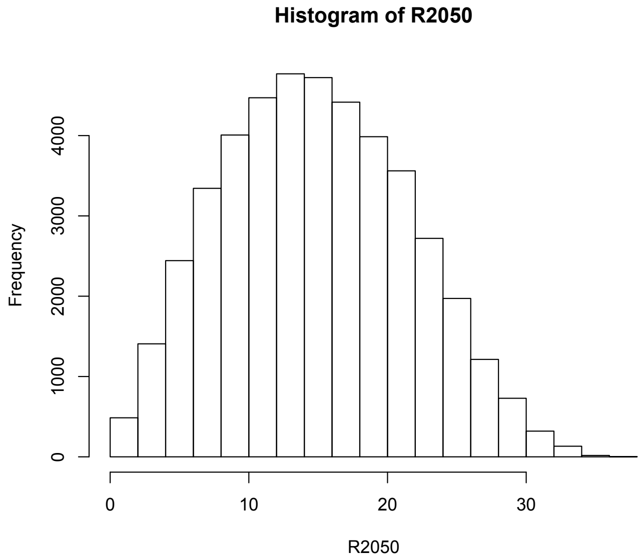


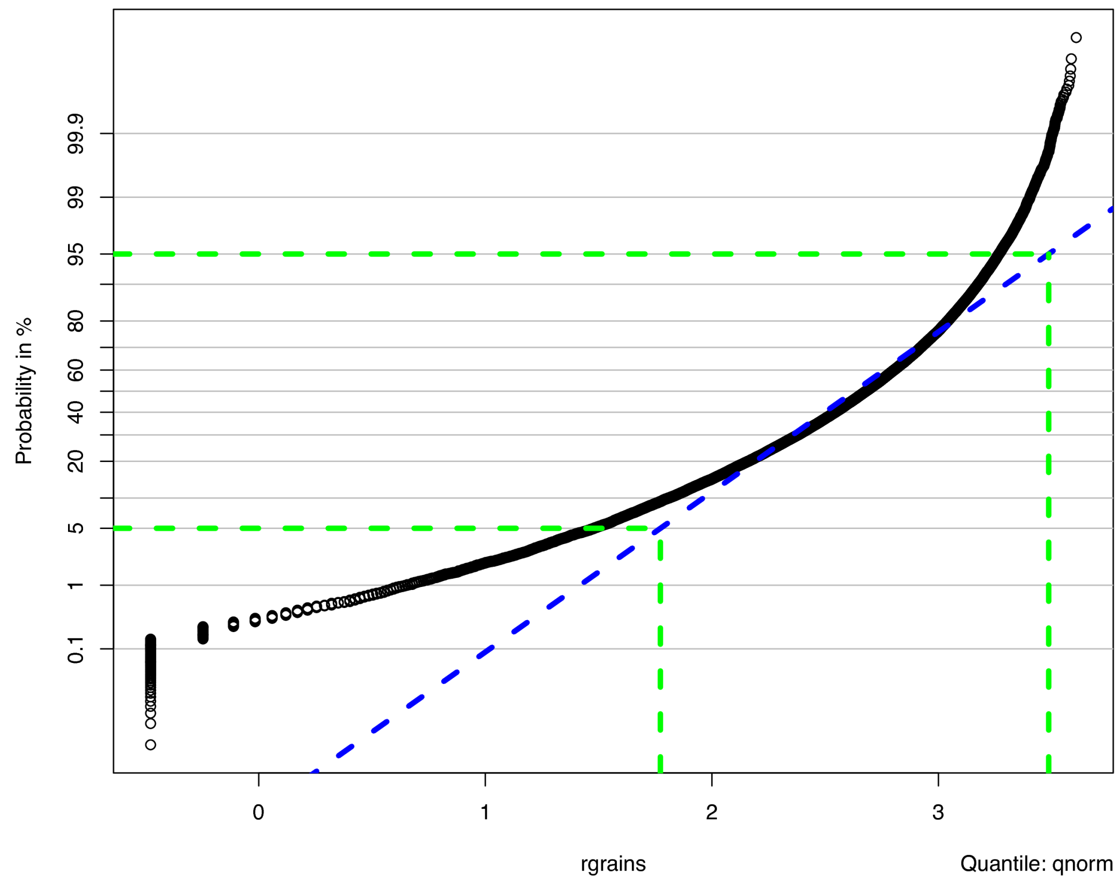
Analysis: IN100

CDFs of Extreme Values as a Function of Sample Size



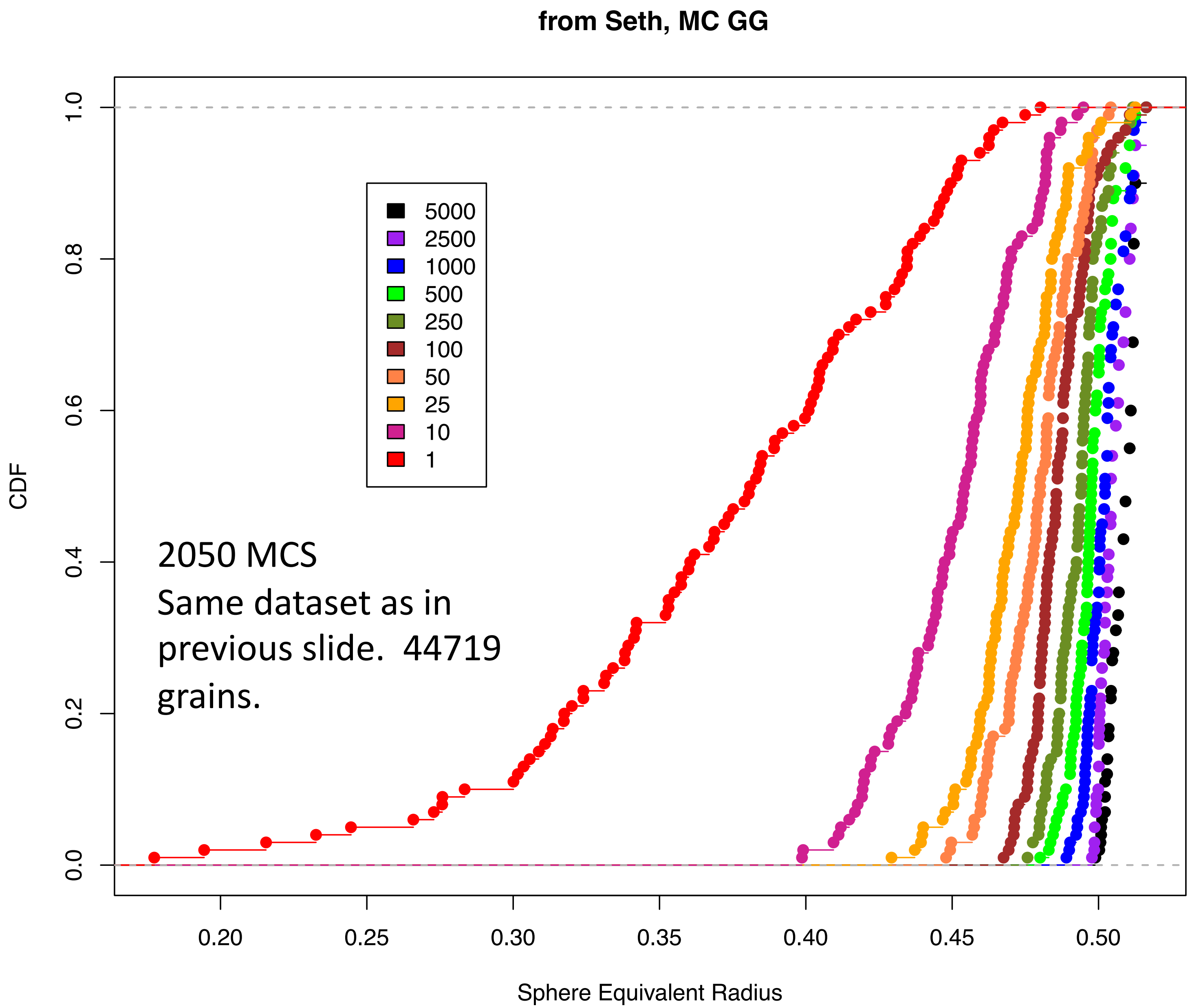
Analysis: Potts model



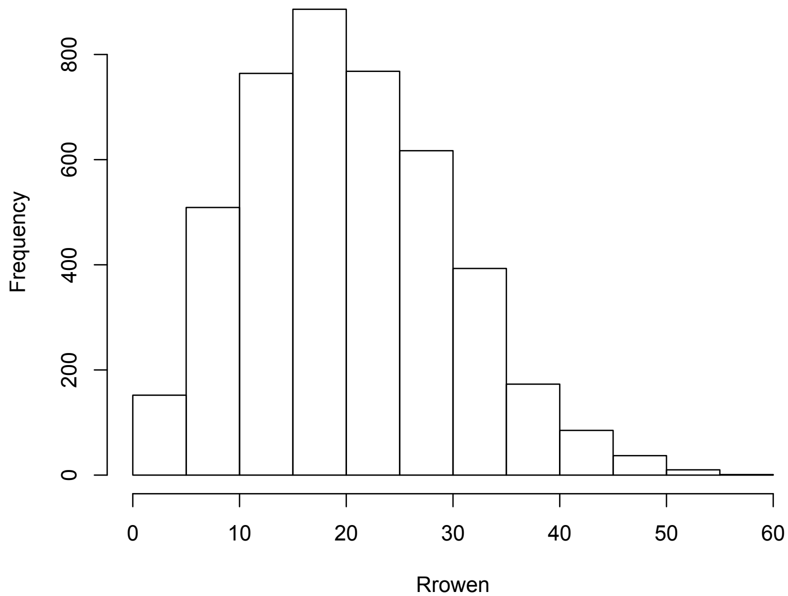


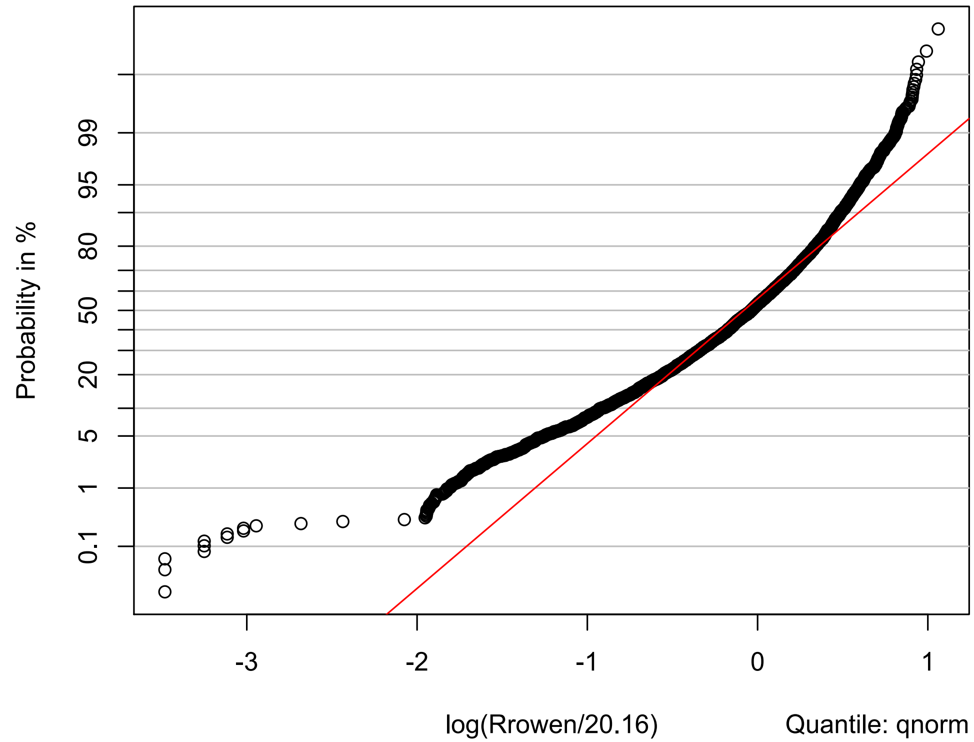
This dataset is taken from a snapshot at 2050 MCS. Clearly, log-normal is a poor description of the distribution of radii, and the strong upward departure of the upper tail fits with the apparent cut-off in size.

Analysis: Potts model



Analysis: Ti beta 21S

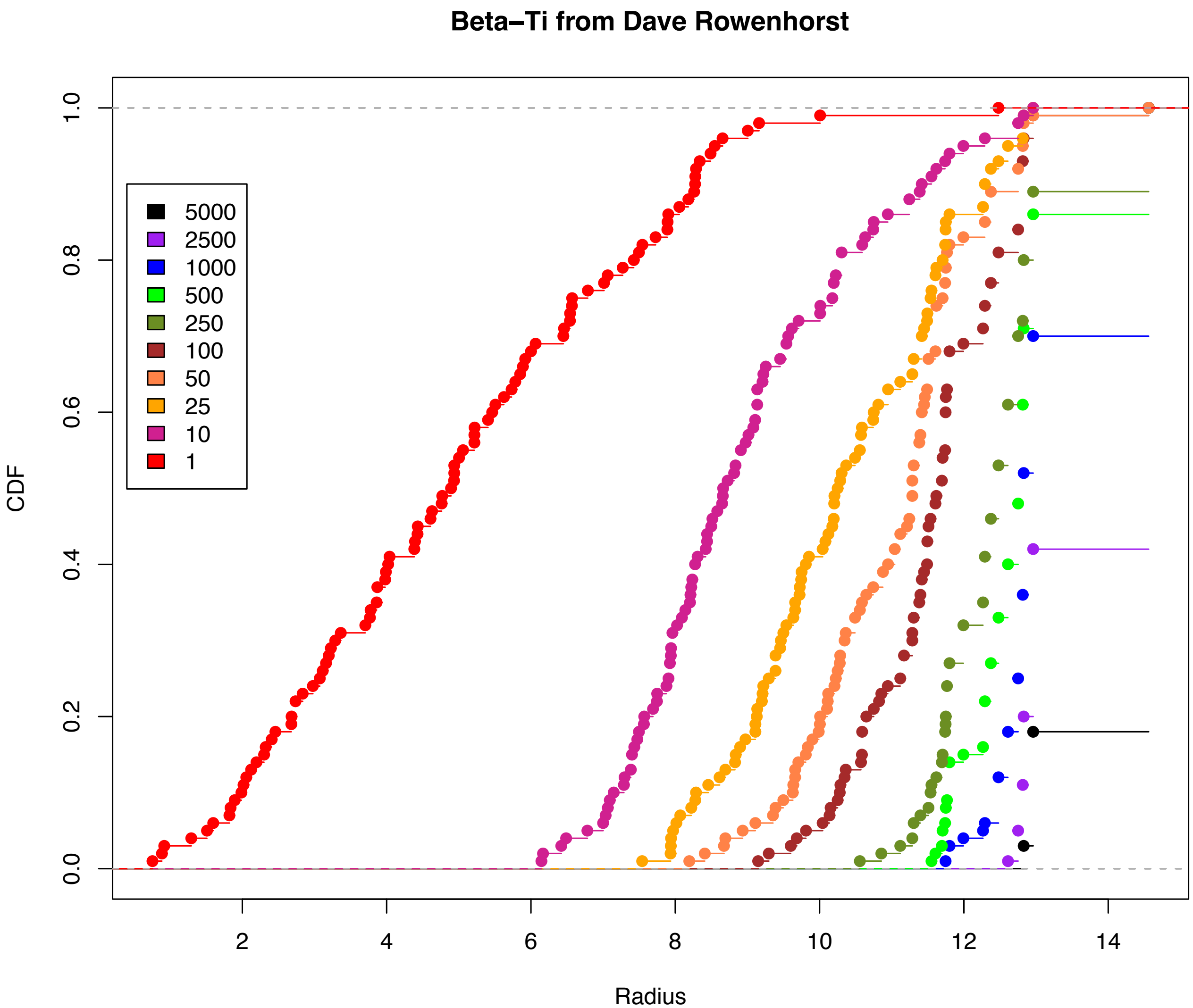




Data analyzed from a dataset with 4396 grains for beta-stabilized Ti alloy (21S). Data courtesy of Dave Rowenhorst, NRL. 3D image generated by serial sectioning, optical + EBSD.

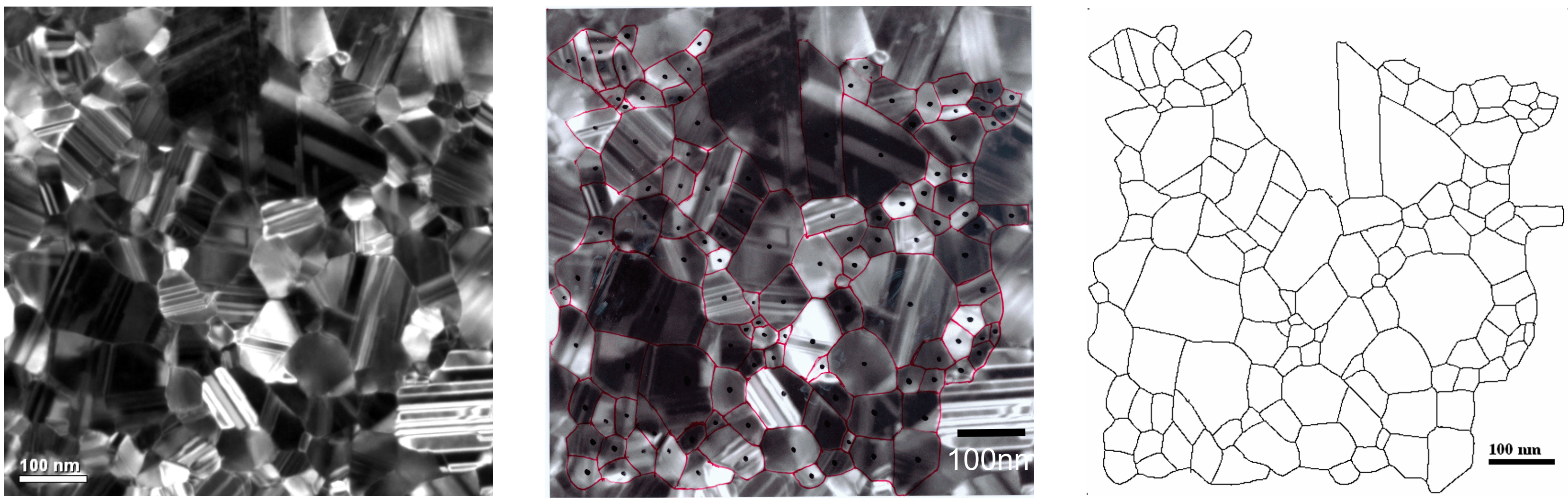
Rowenhorst DJ, Lewis AC, Spanos G. Three-dimensional analysis of grain topology and interface curvature in a beta-titanium alloy. *Acta mater*. 2010; **58**: 5511.

Analysis: Beta-Ti



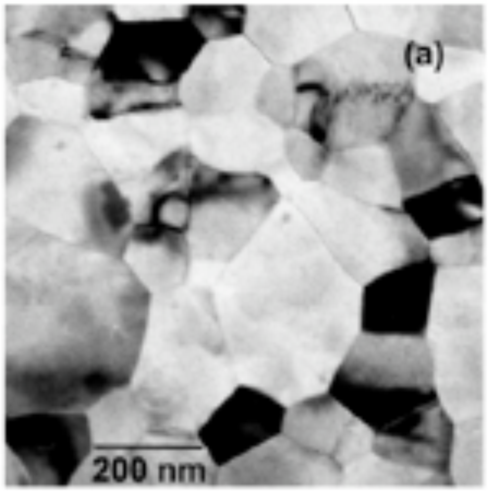
The “bunching up of the CDFs at the larger sizes suggests that there is an upper cutoff in size. This is in contrast to the IN100 result.

Grain Size Measurements Based upon TEM Observations



Result is average grain size and grain size distribution. Reported data includes 21 samples and 17,882 grains

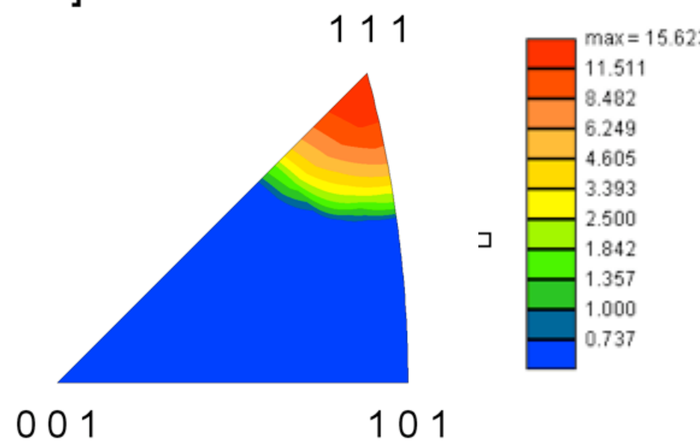
Stagnation of thin film grain growth

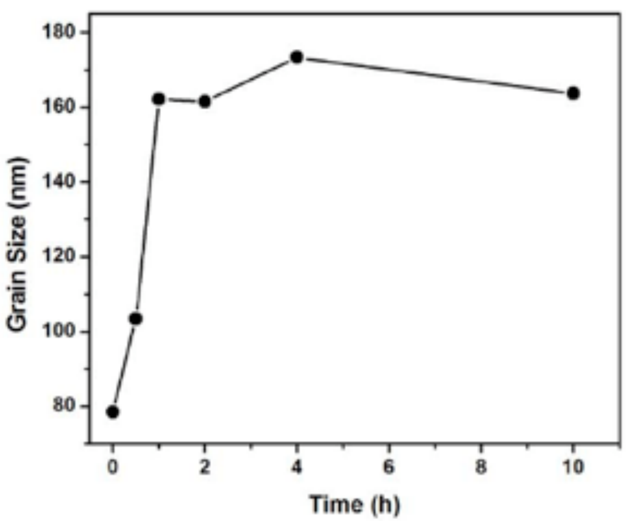


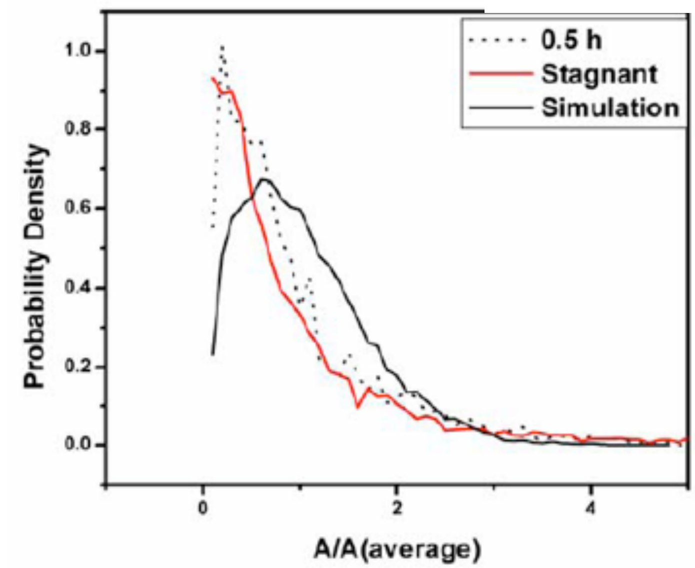
Al-film deposition
Growth experiments
MC&PDE simulations

Strong {111} orientation texture

[001]







Retarding Forces

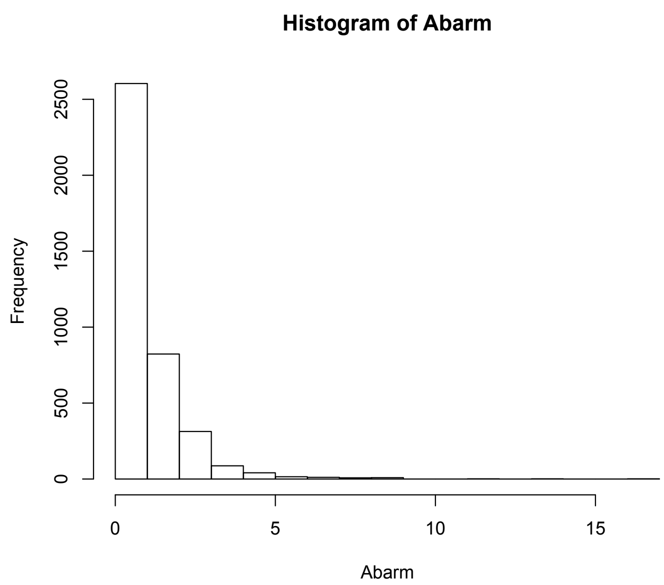
- Grooving
- Impurity drag
- Precipitate pinning
- Triple junction drag
- Stress on boundary

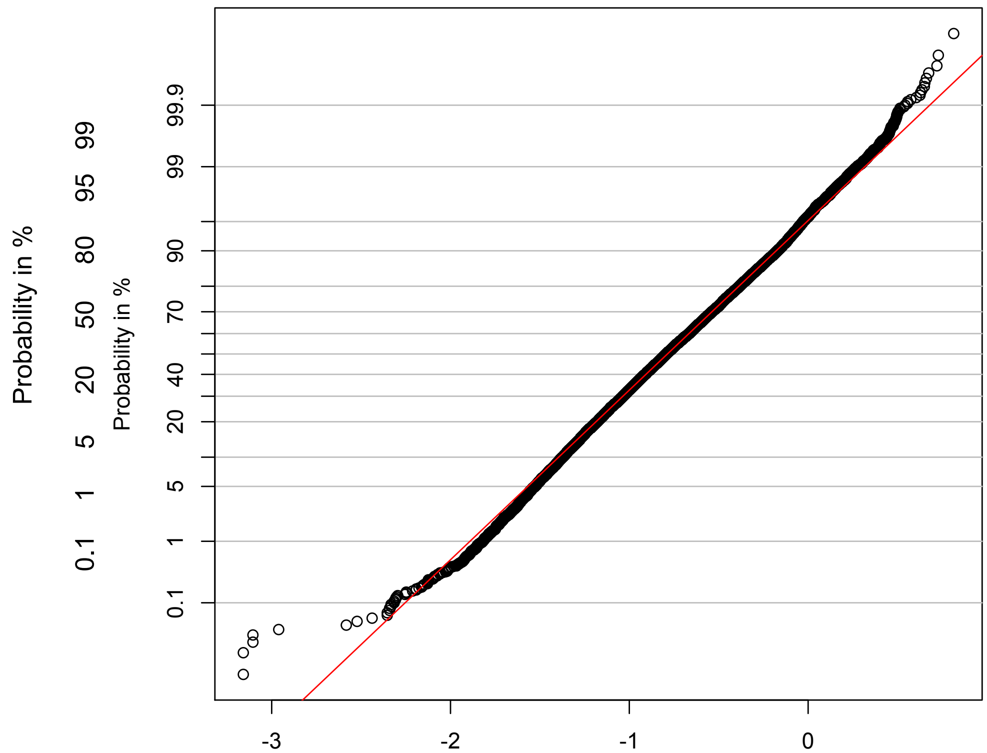
Network, e.g. residual stress

K. Barmak, *et. al. Scripta. Mater.*, 40 (2006)

Analysis: Thin Films

Data provided by Katy Barmak for areas of grains in thin films of Al analyzed (mainly) by Wayne Archibald. Films annealed for 1, 2, 4 and 10 hours; areas normalized by average in each set. Areas converted to circle-equivalent radii. Second plot has additional data from Derrick Carpenter and Jihwan Kim.

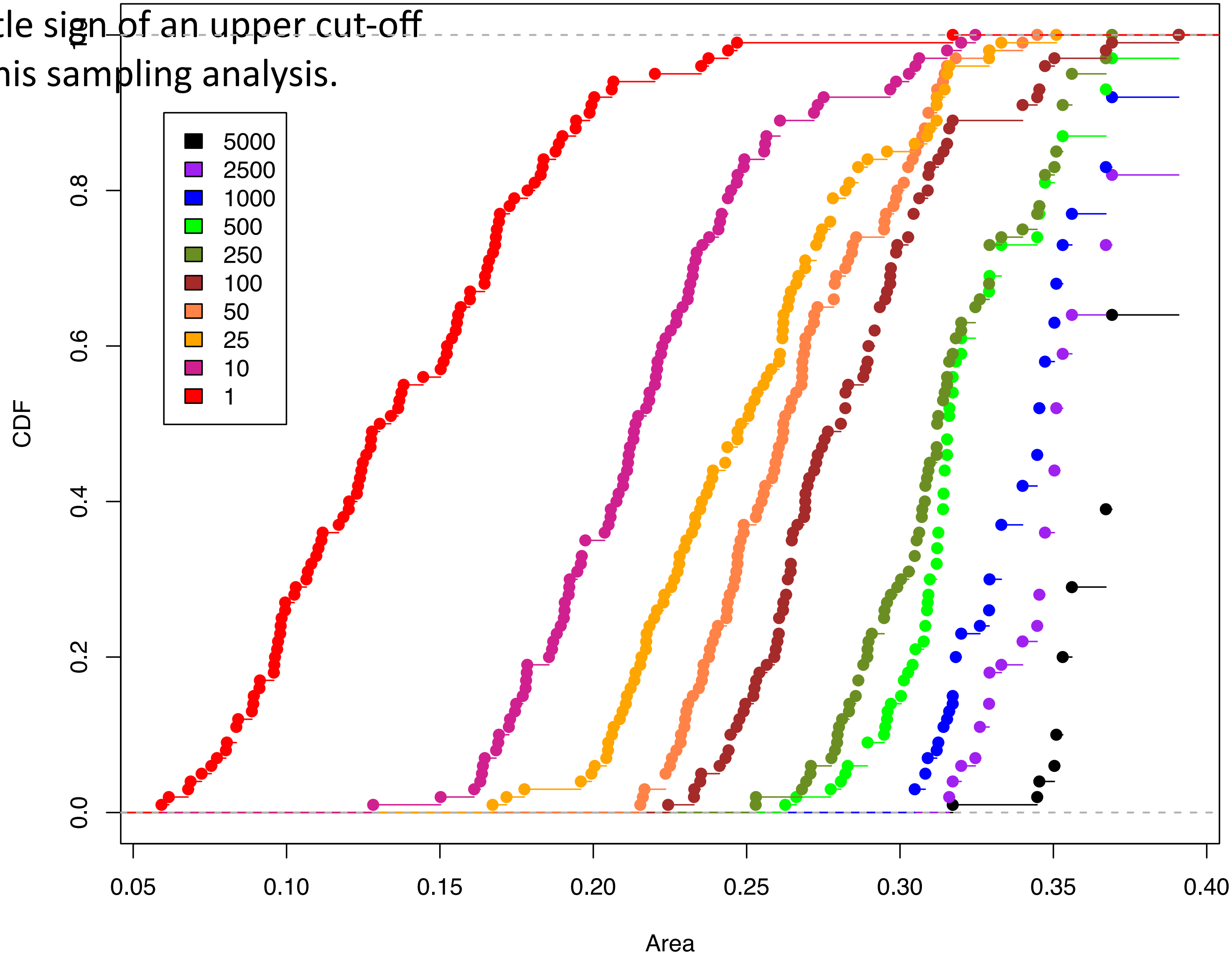




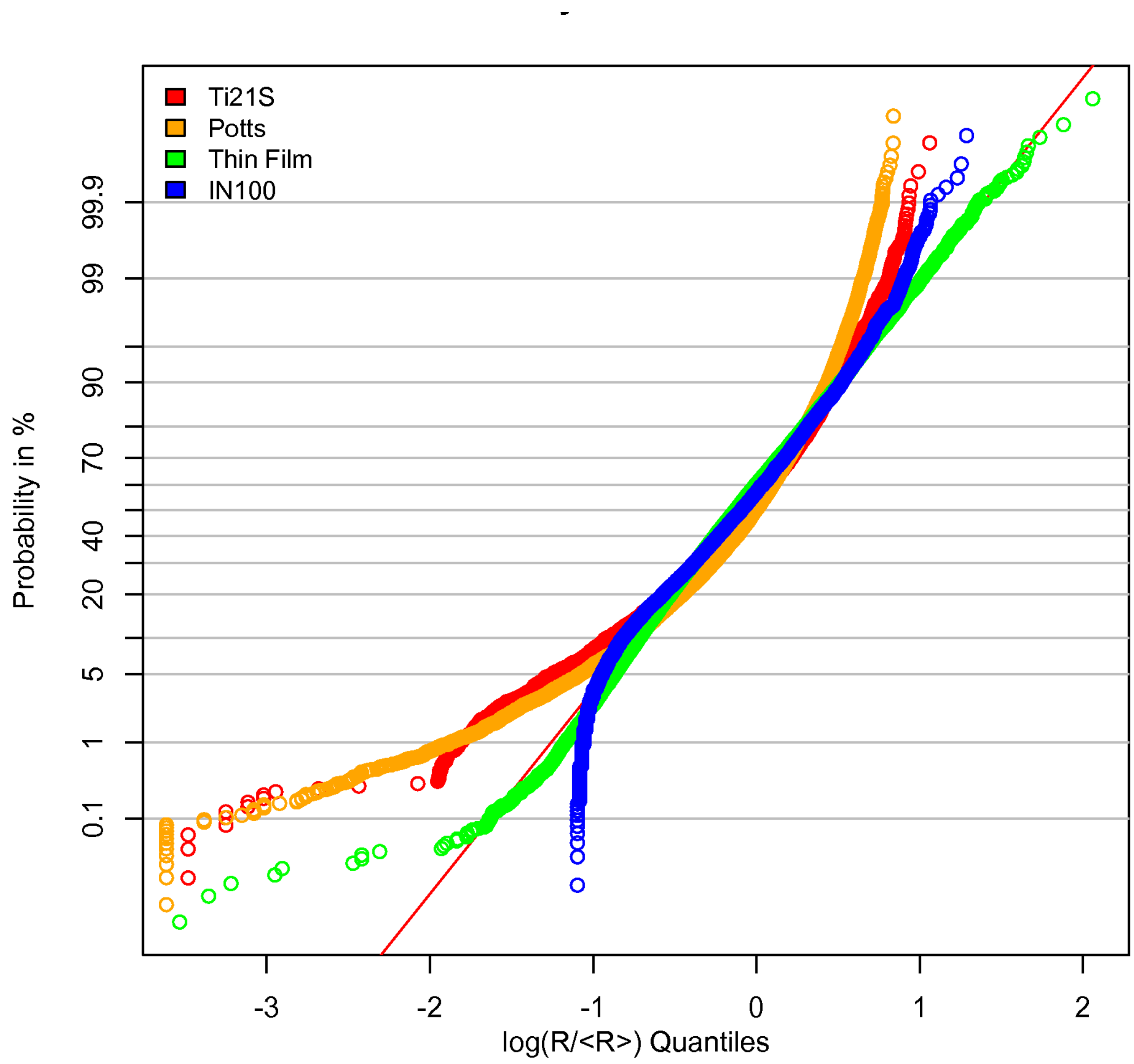
The distribution is remarkably close to log-normal, with rather small departures at the upper and lower ends.

Analysis: Thin Films: Limits

In keeping with the upper tail seen in the nearly log-normal distribution, there is little sign of an upper cut-off in size in this sampling analysis.

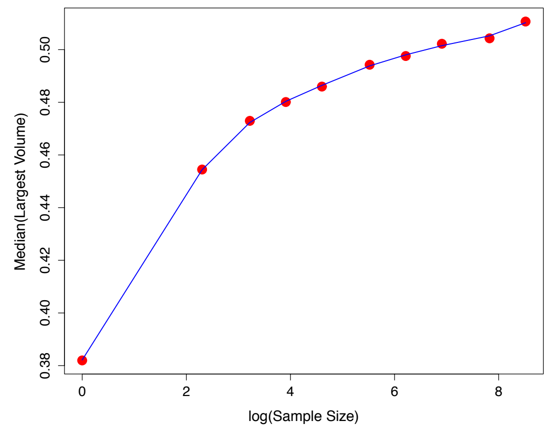


Combined Probability Plot

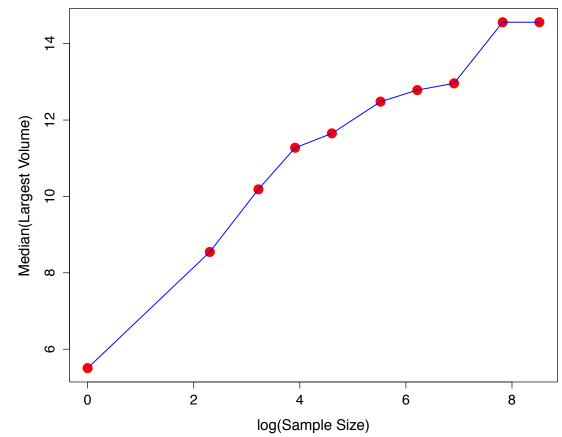


Asymptote for Largest Grain?

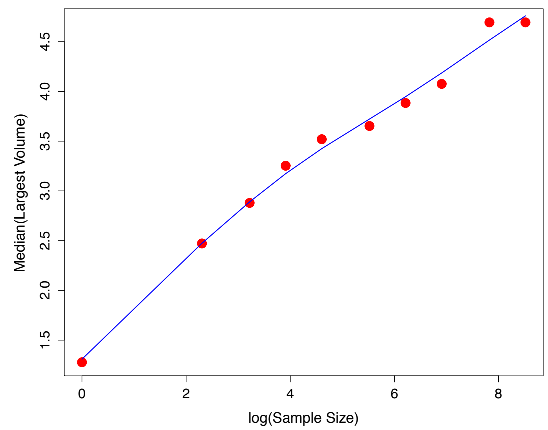
The *median* of each CDF is extracted and plotted versus the logarithm of the sub-sample size



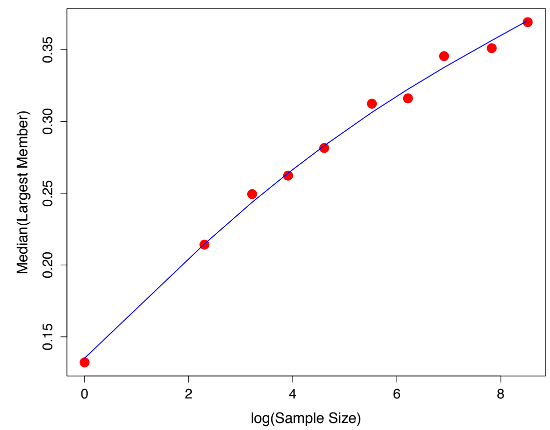
(a) Potts model



(b) Ti (beta-21S)



(c) Ni alloy (IN100)



(d) Al, Cu thin film

Summary

- Damage initiation is, generically, an upper tail problem.
- Large datasets from simulation and 3D characterization enable upper tails to be investigated.
- Log-normal can be applied, as suggested in the literature, but only over a limited range of the data.
- The thin film data most nearly approach log normal.
- The microstructures from the Potts model deviate strongly from lognormal; the subset sampling analysis also suggests the presence of a hard cutoff in maximum grain size, as one expects from coarsening theory
- The IN100 and Ti-21S microstructures also deviate noticeably from lognormal.
- There is an apparent correlation with stagnation or pinning of grain growth: stagnant microstructures approach log-normal.
- Need: more (large) datasets; theoretical distributions; distributions of other microstructural features; apply theorems on extremes of samples.

Critical Issues

- Extrapolation
  - Quantification of Error in Extrapolating CDFs for Large *N*s
- Extreme Values Not Large Deviations
  - Missing Details of Distribution Between Breakdown of Lognormal and Extreme Values (i.e. 2σ to $4^{+}\sigma$)
- Neighborhoods and i.i.d. Assumption
  - How to Build Neighborhoods Around Grains Never Before Seen
  - i.i.d. May not Apply for Abnormal Grain Growth
- Re-Sampling Discrete Samples
  - Quantification of Effect of Sampling Discrete Values Instead of True Distribution