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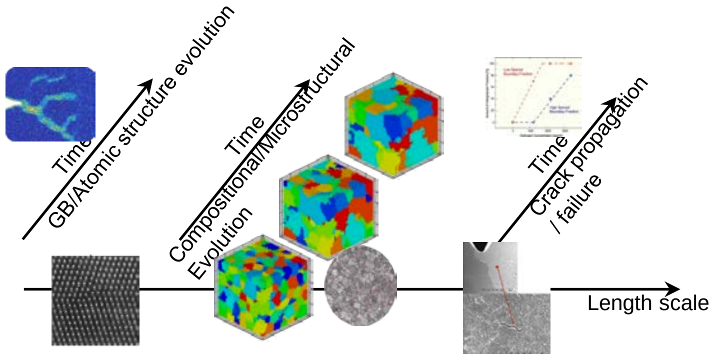

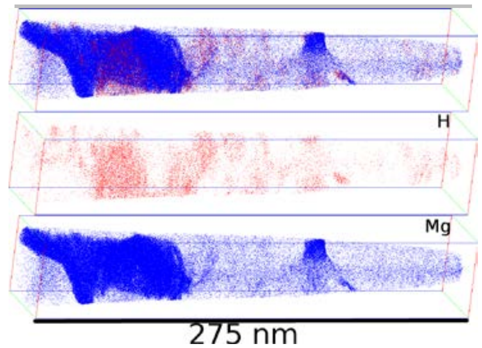

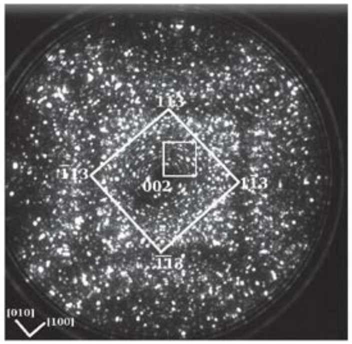

Atom-Probe Tomographic Measurement of Trapped Hydrogen Isotopes

R. Karnesky (Sandia)

N. Teslich, M. Kumar (Lawrence Livermore)

TFG 2014

U.S. DEPARTMENT OF ENERGY NNSA National Nuclear Security Administration

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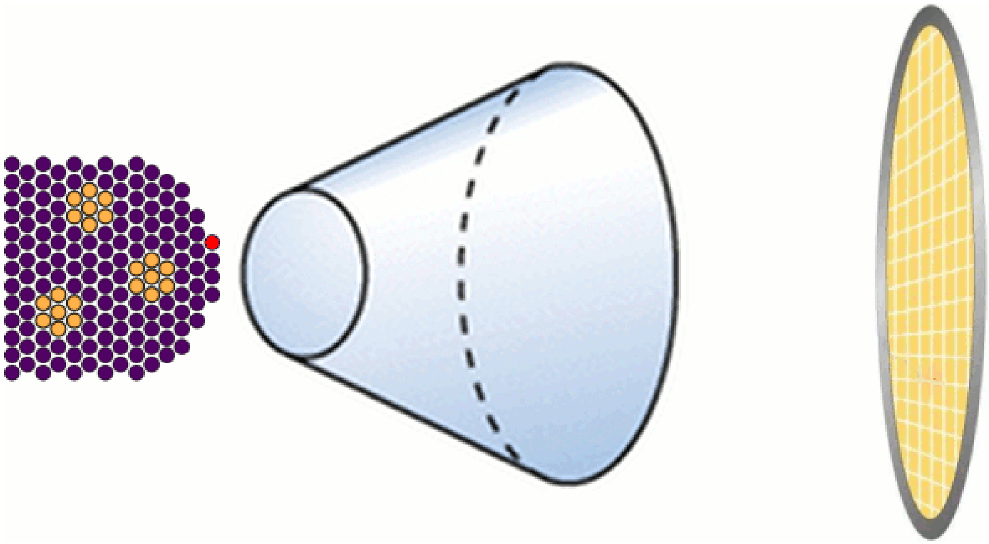
Local-Electrode Atom-Probe (LEAP) Tomography

Compositional and structural analysis at the atomic scale

- Pulse encodes z
- Area detector gives (x,y)
- TOF encodes mass/charge



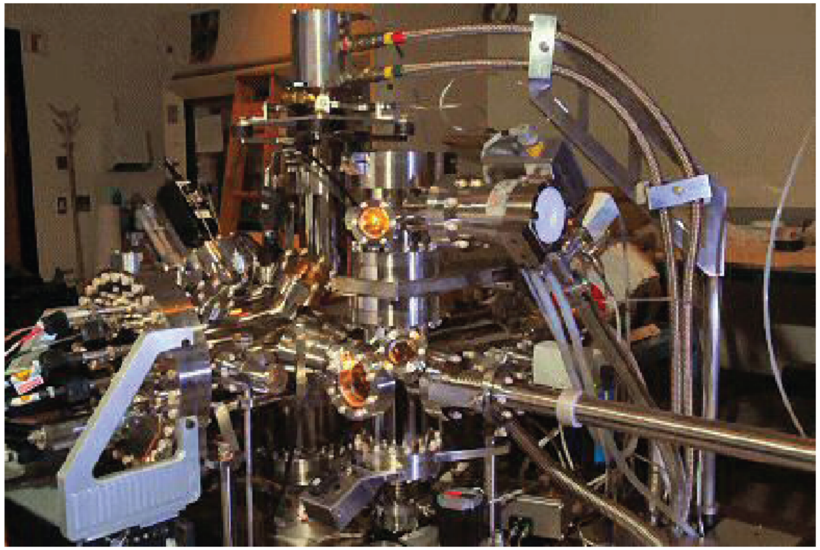
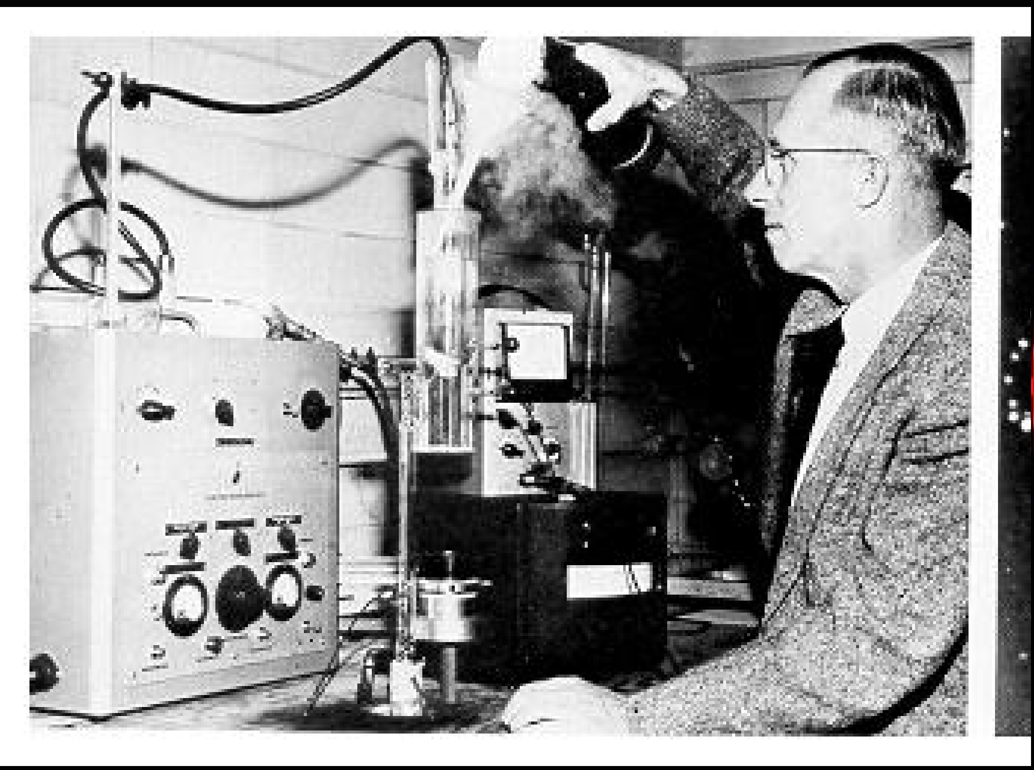


Photo courtesy of R.P. Koll

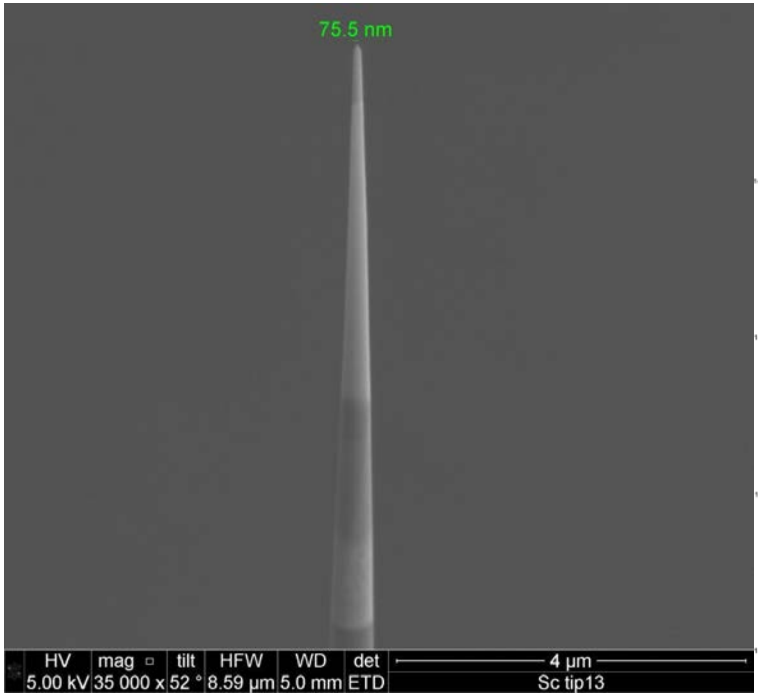
Instrument capability

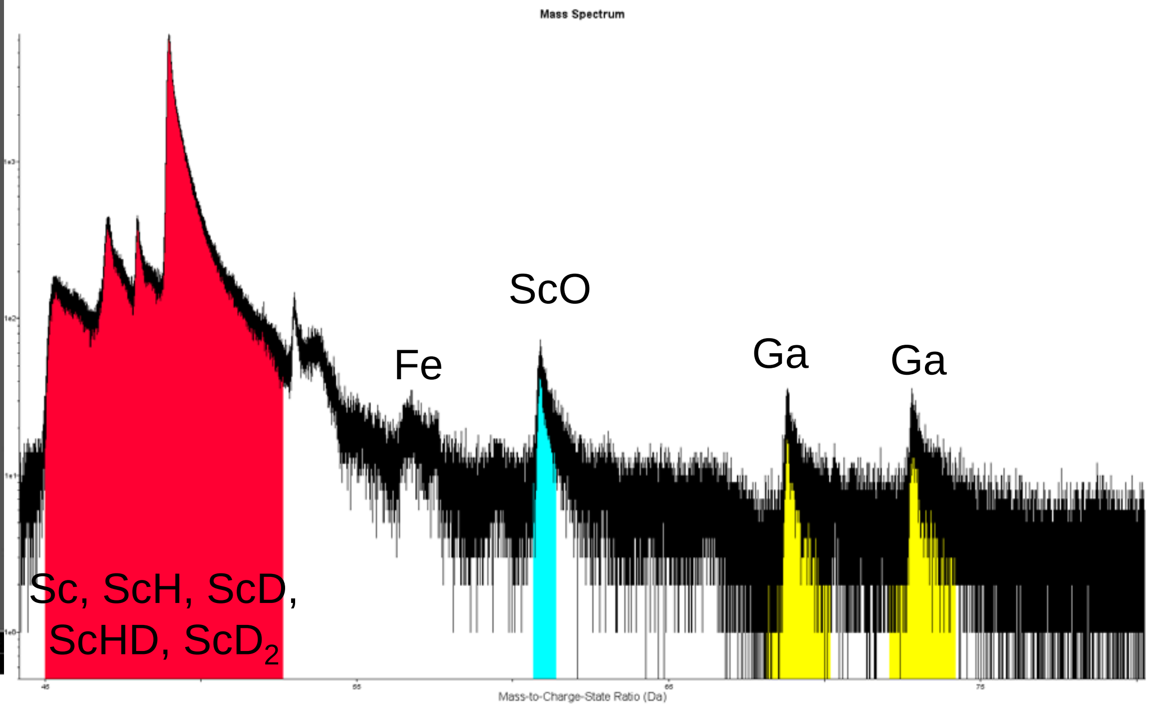
- 10^6–10^7 nm^3 analysis volume
- 3×10^{-11}–10^{-10} torr UHV
- 20–100 K specimen temp.
- 200 kHz electrical pulsing

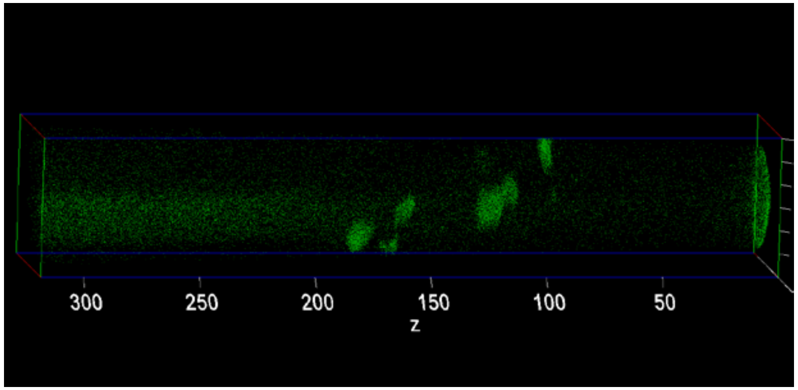
First atomic imaging



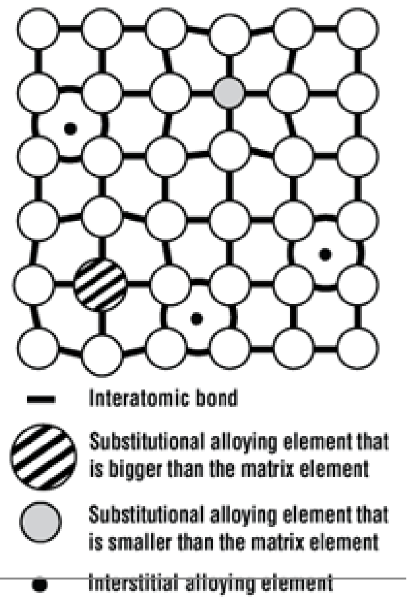
Site-Specific Samples and Hydrogen Isotope Sensitivity

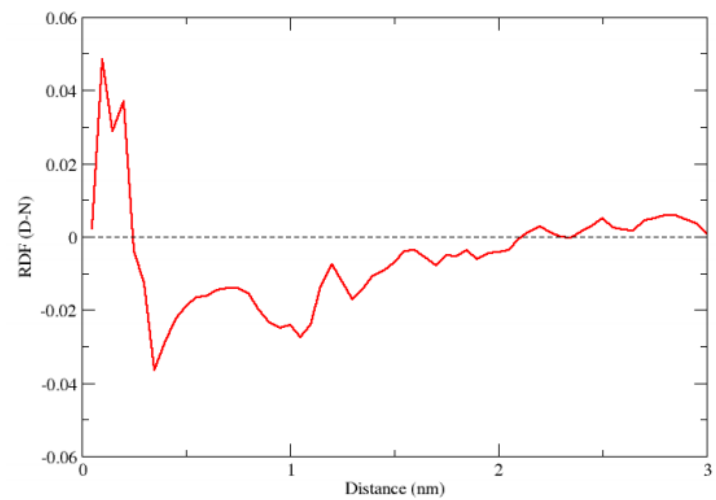


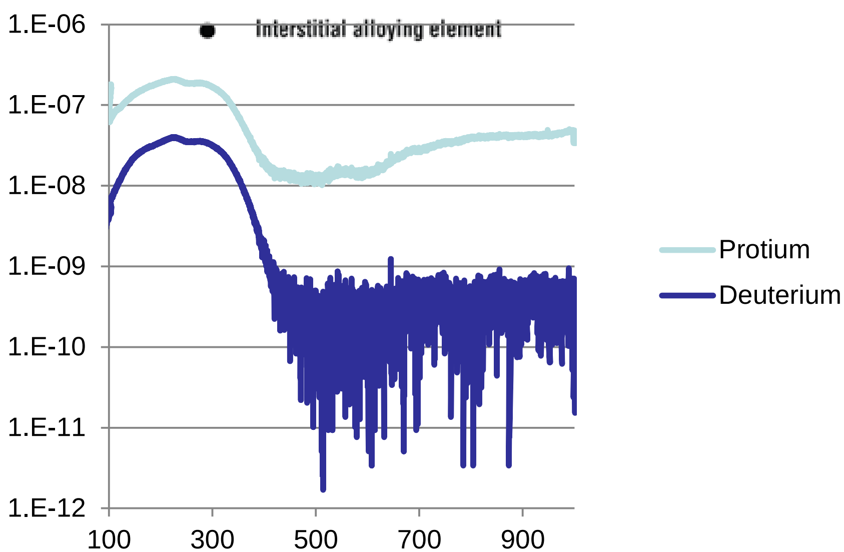


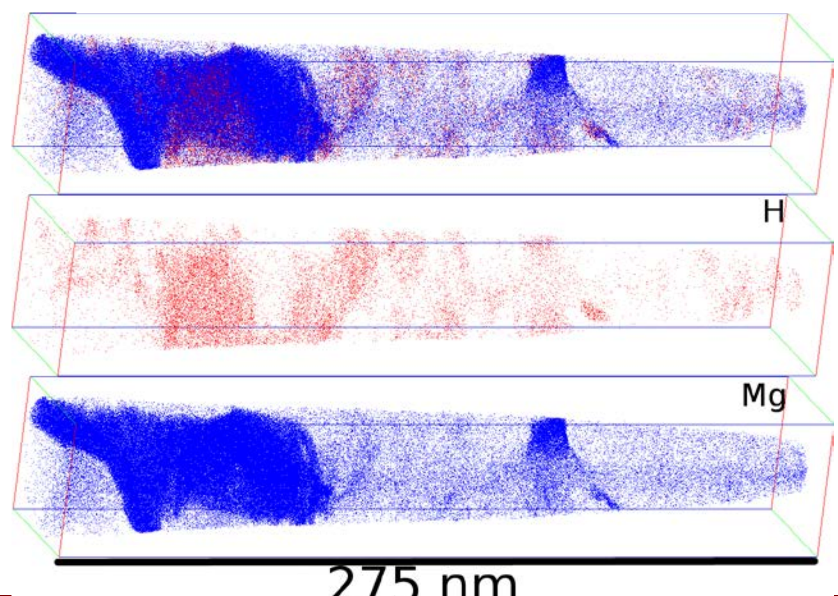


Hydrogen Segregates to Traps and Atom Probe can Image It

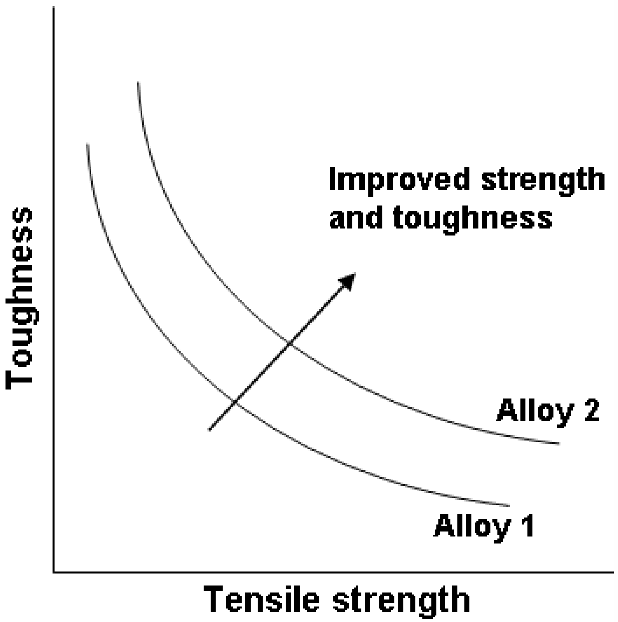


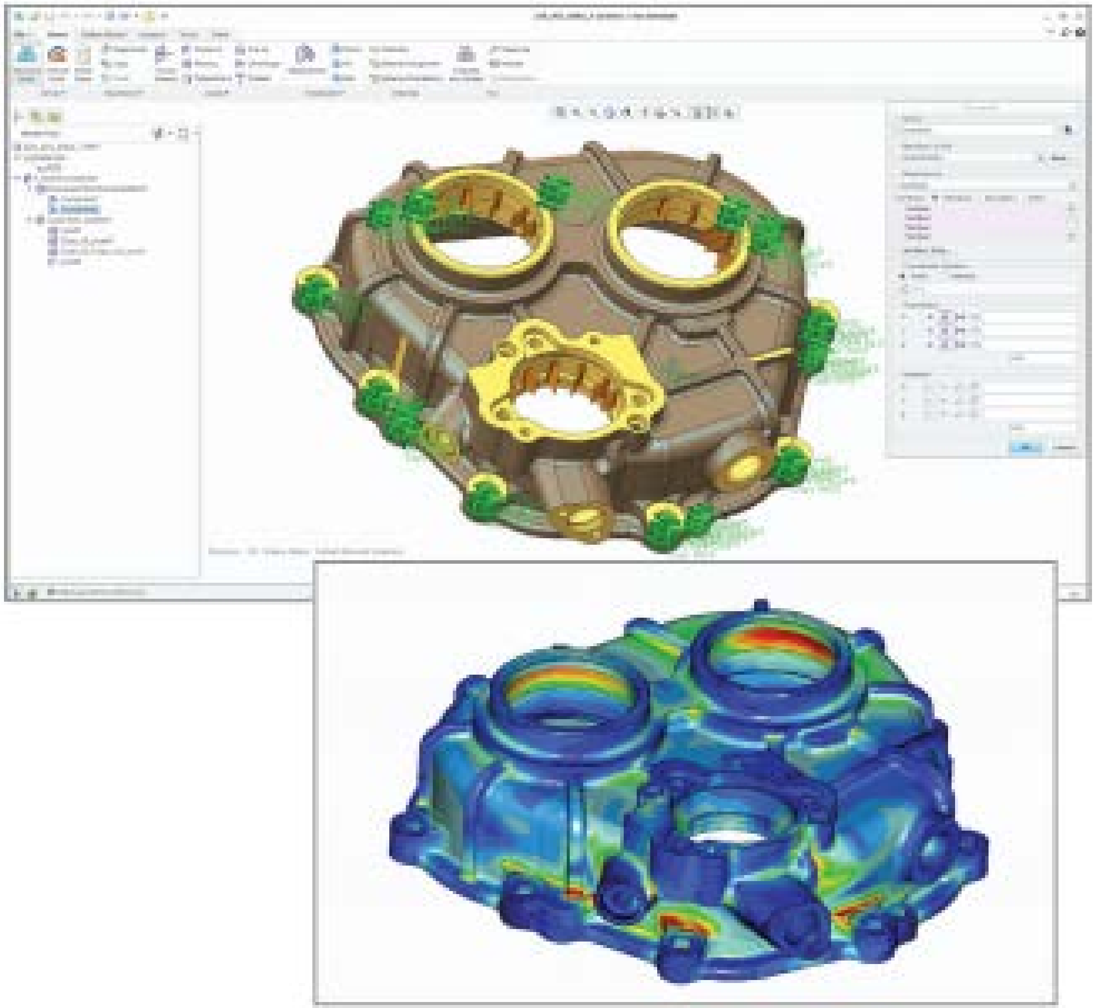






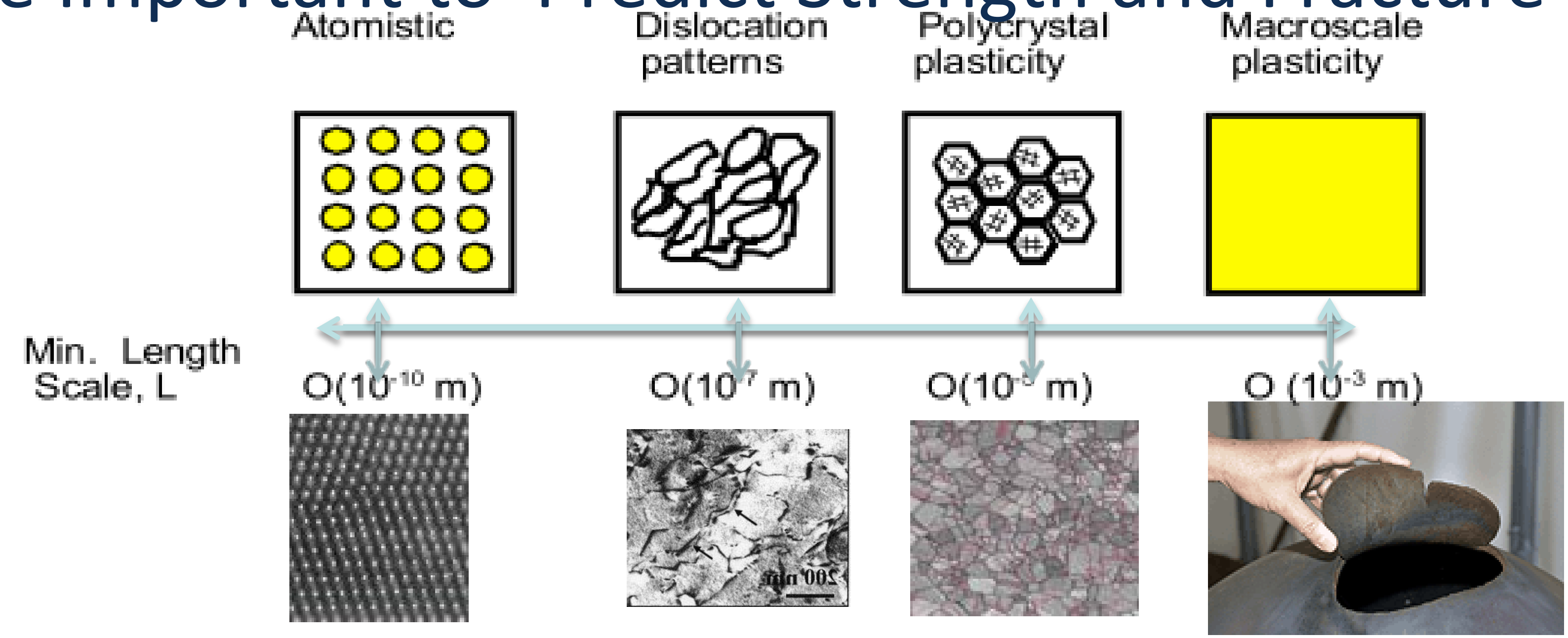
Engineering Needs Require Both High Strength and High Fracture Toughness

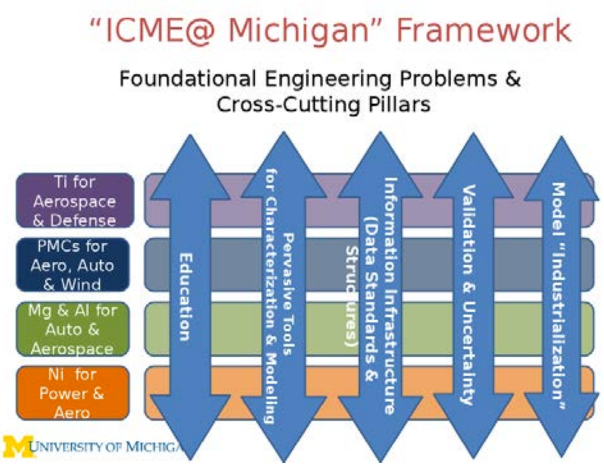


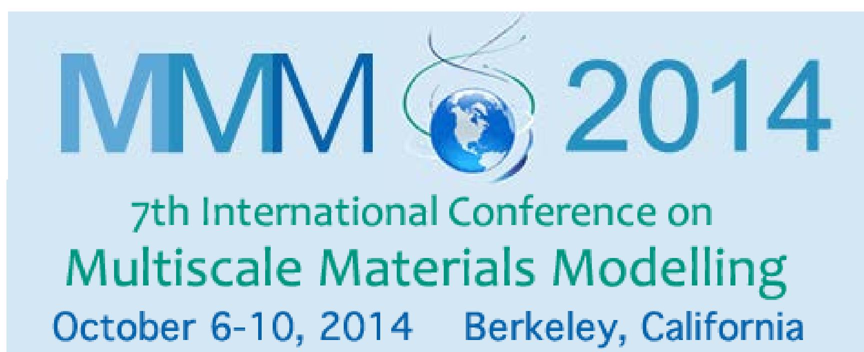


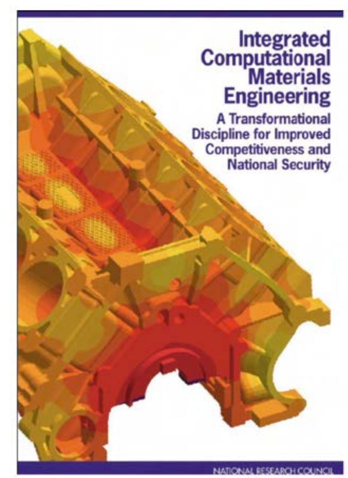
R. Rithie, Nat Mat 2011

Multiscale Experiment/Simulation Couplings are Important to Predict Strength and Fracture

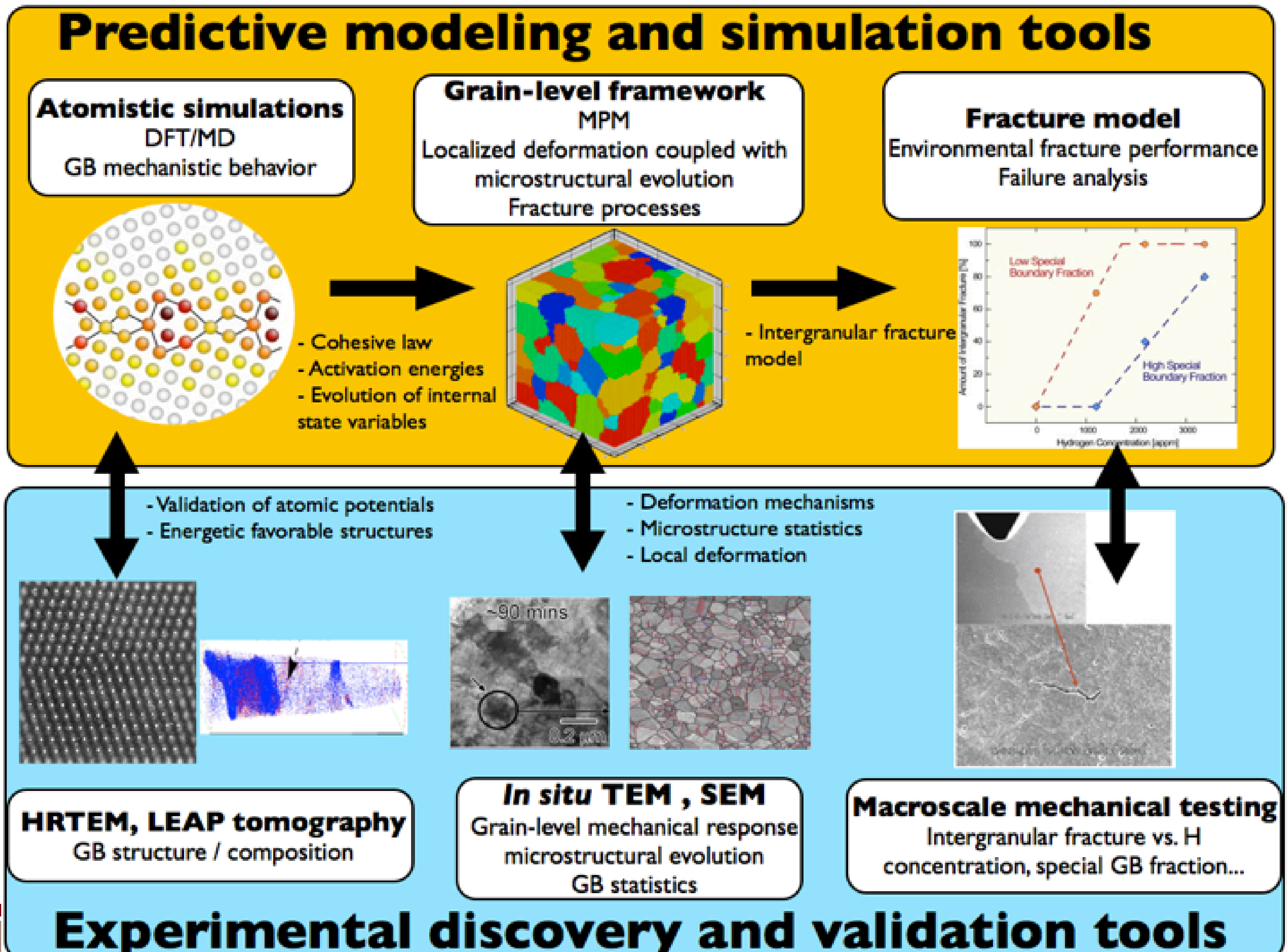




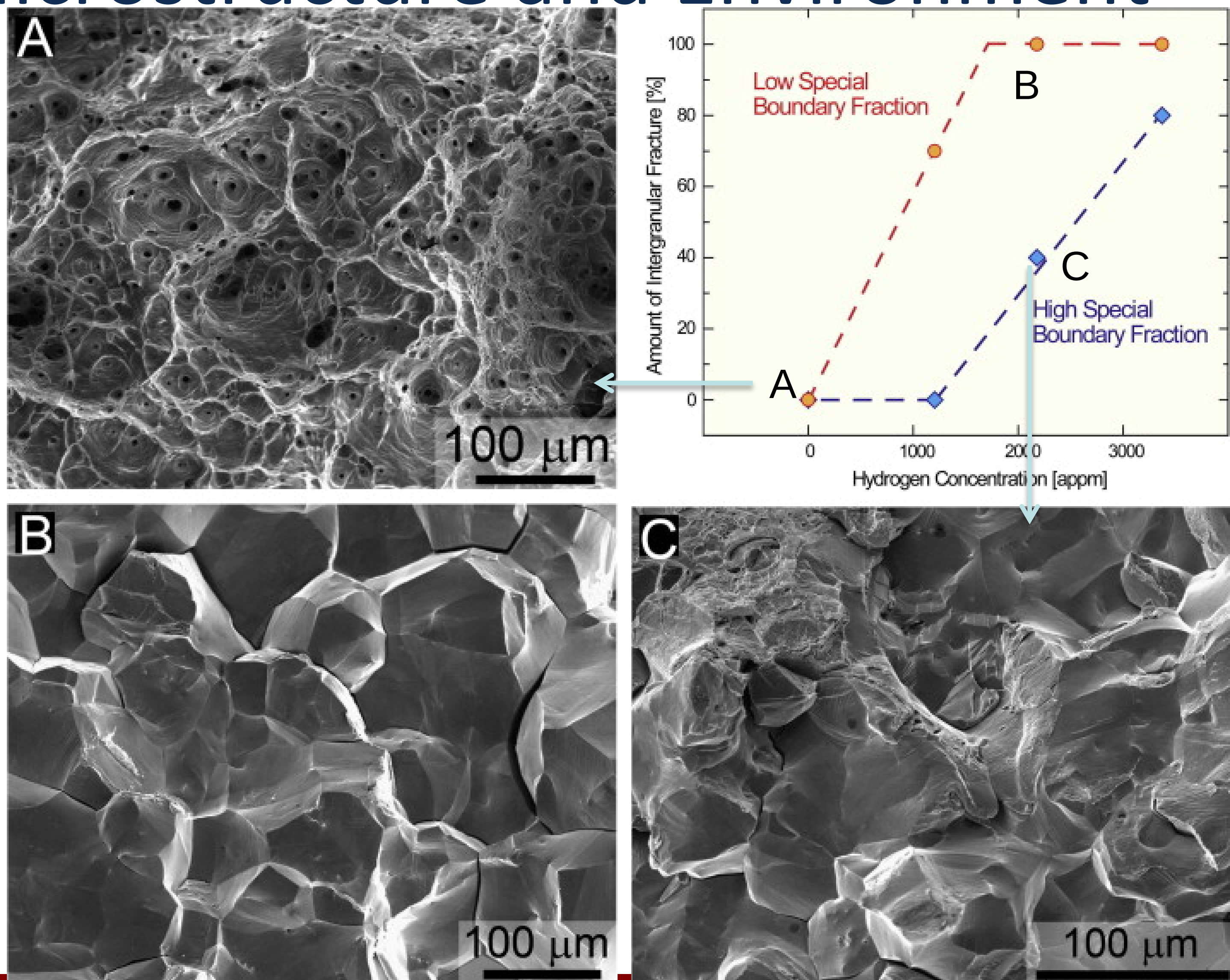




Science-Based Computational and Experimental Capabilities Advance Us Beyond the Empiricism of Current Methods

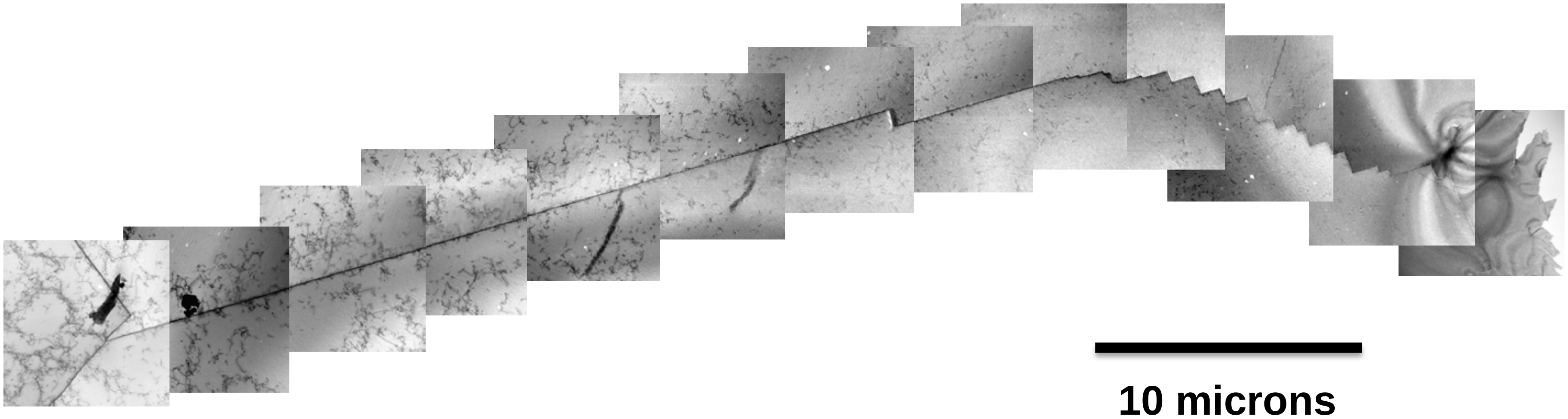


Intergranular Fracture Properties depend on Microstructure and Environment

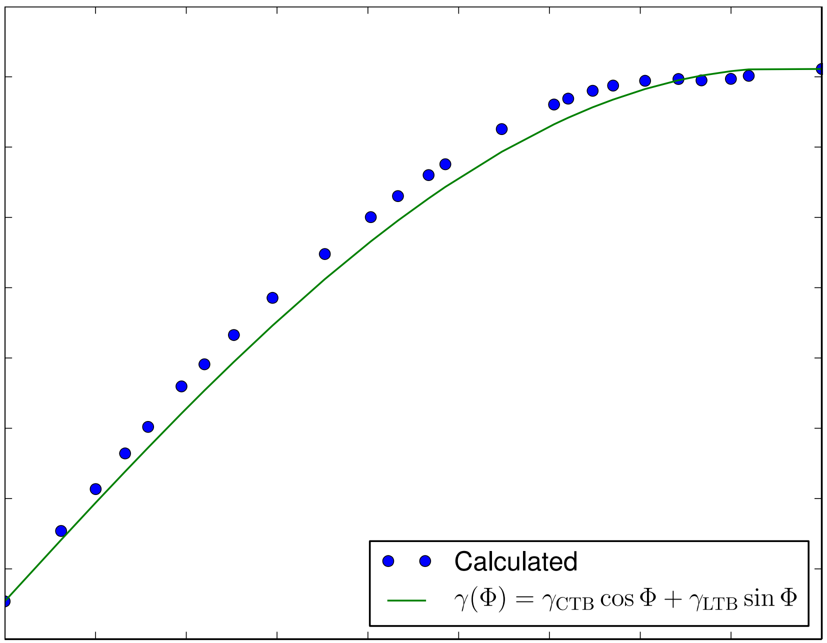


Bechtle et al *Acta Mater.* **57** (2009) 4148-4157

Dislocations and twin boundaries within Ni



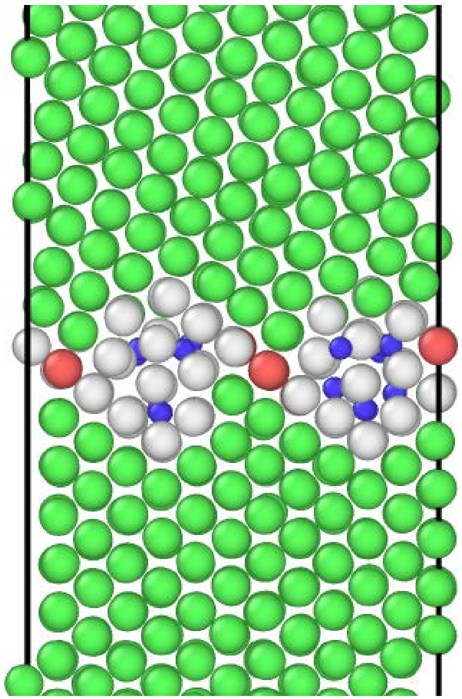
Angular Dependence of Inclined Twin Energy

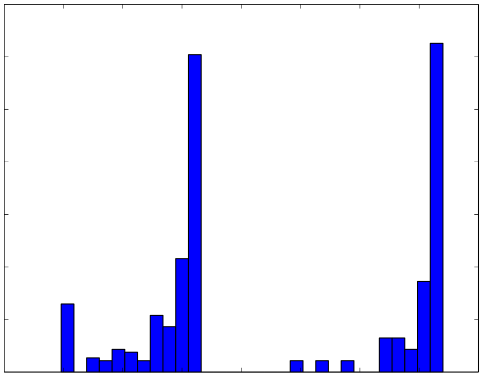


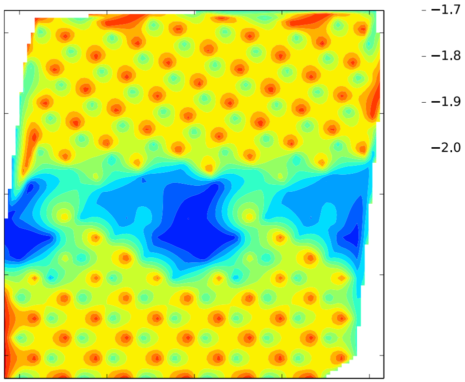
- Calculated values (dots) at 0K versus a model assuming that boundary is made of (111) and (211) twin facets (line)
  - Energetic contributions from junctions between CTB and LTB are neglected

Segregation in 35.26° Boundary

- Image made with $\mu_H = -2.35$eV
  - Corresponds to ~2x10^{-4} bulk concentration at 300K
- H segregates to GB and likely interacts with neighbors
- Adsorption Energy histogram is generated from map at 0K (lower right), it includes large peaks for bulk oct and tet sites.

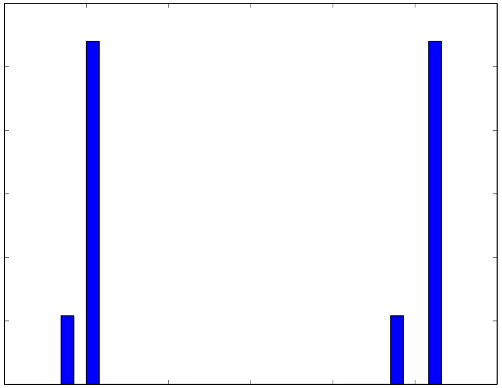




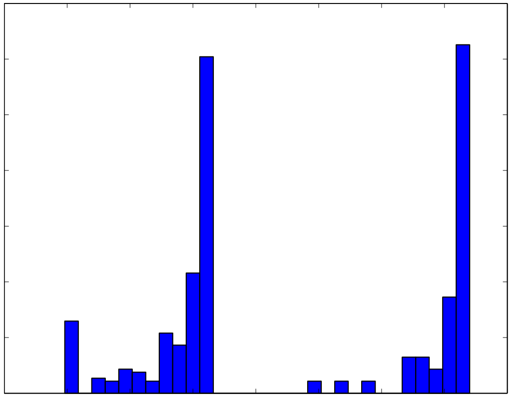


Segregation Energy Histograms (0K)

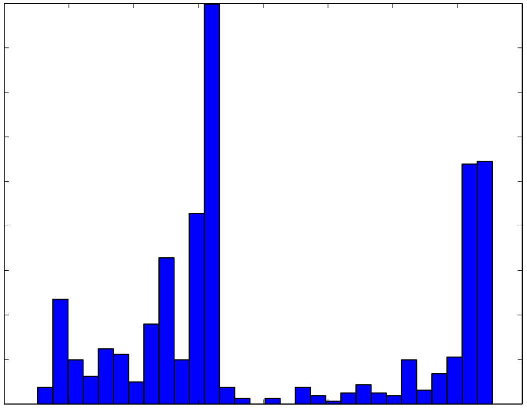
Φ = 0°



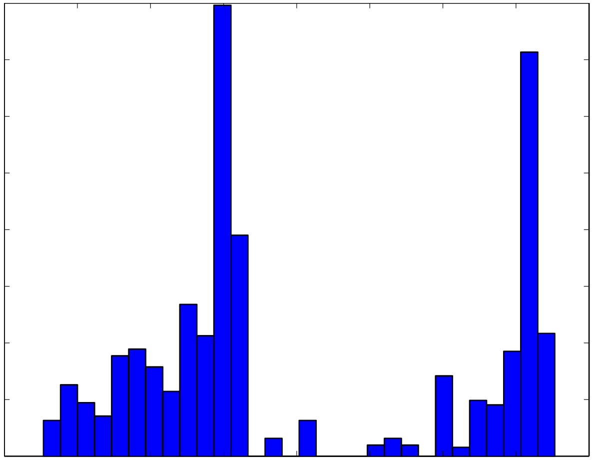
Φ = 35.26°



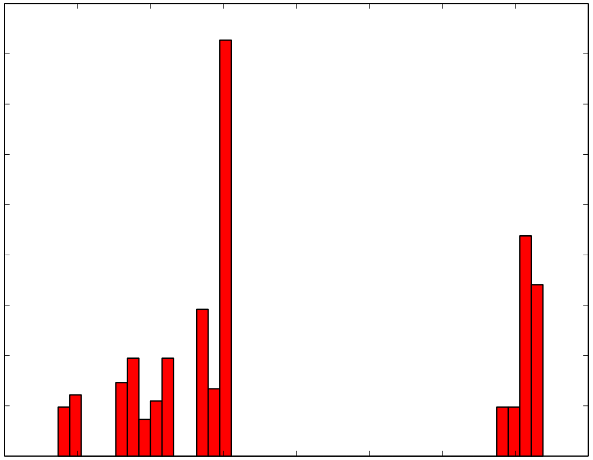
Φ = 74.21°



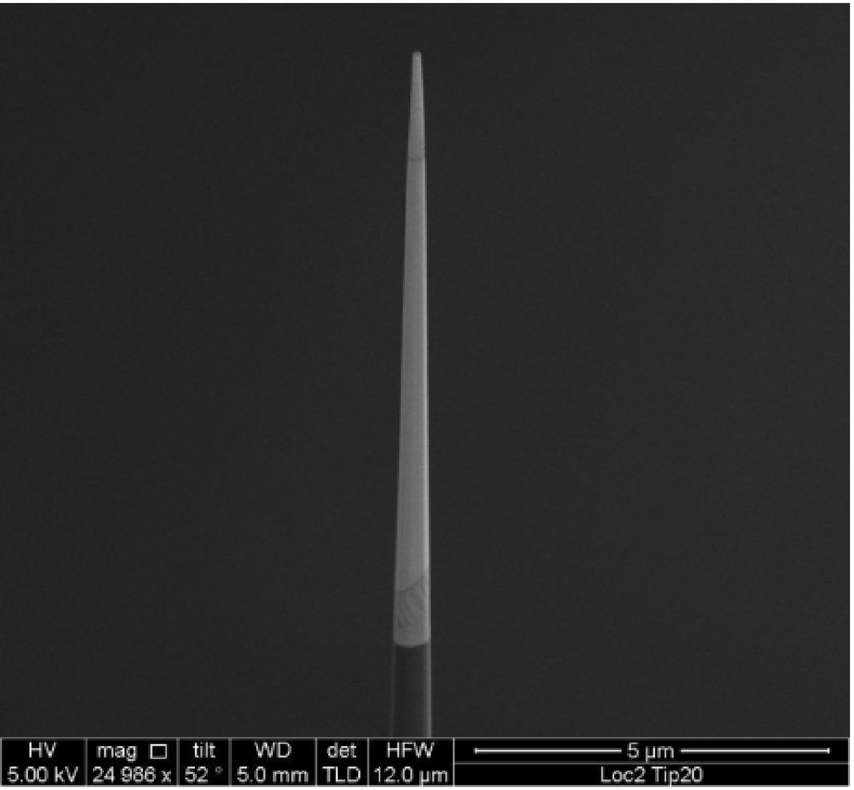
Φ = 79.98°

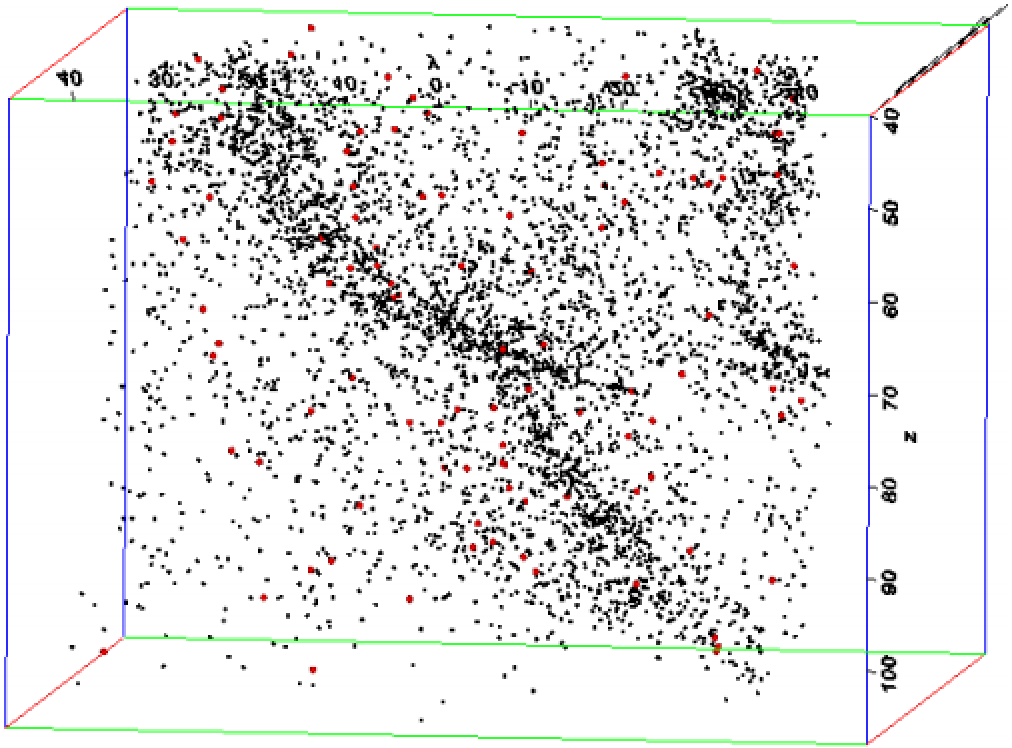


Φ = 90.00°



Higher Angle Boundaries Show Greater S and D Segregation

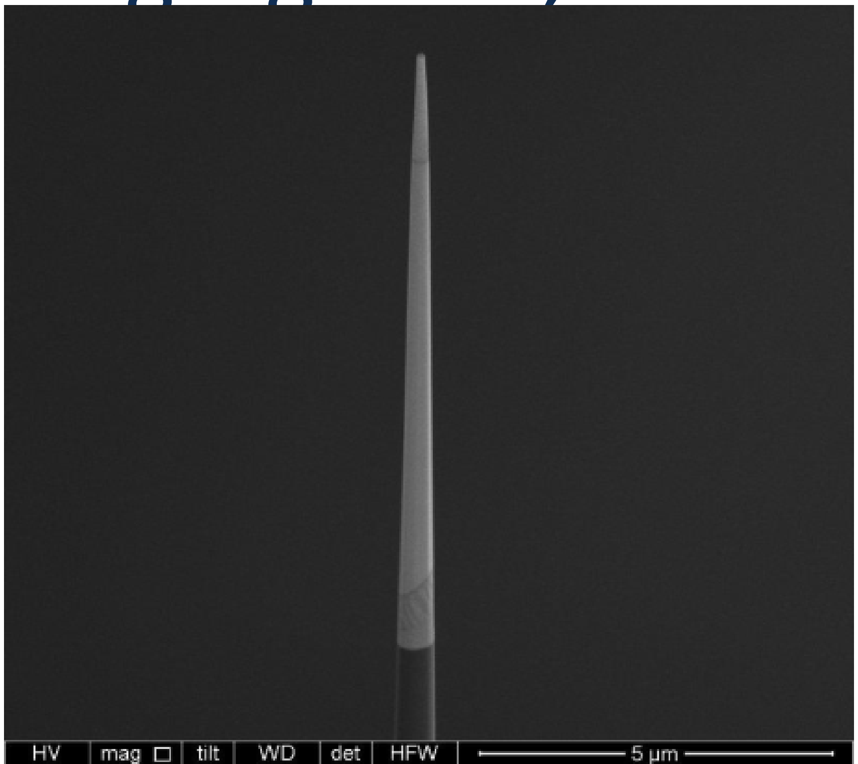


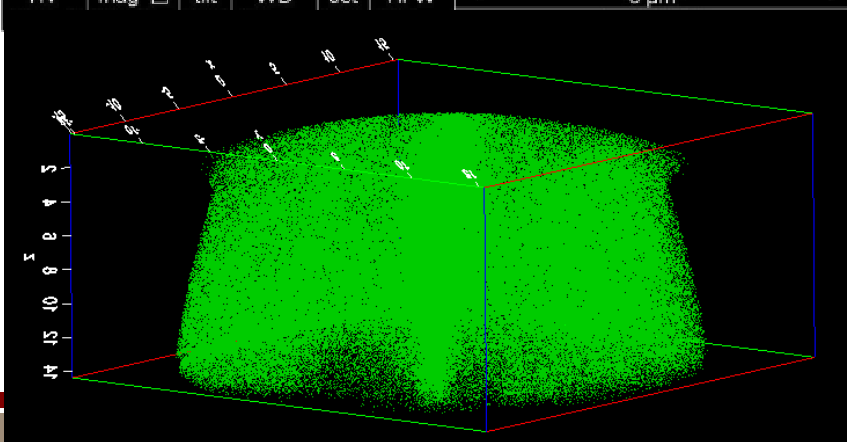


4 at.% D, an enrichment of over ten from the bulk, consistent with Kirchheim, though higher than the results by Birnbaum.

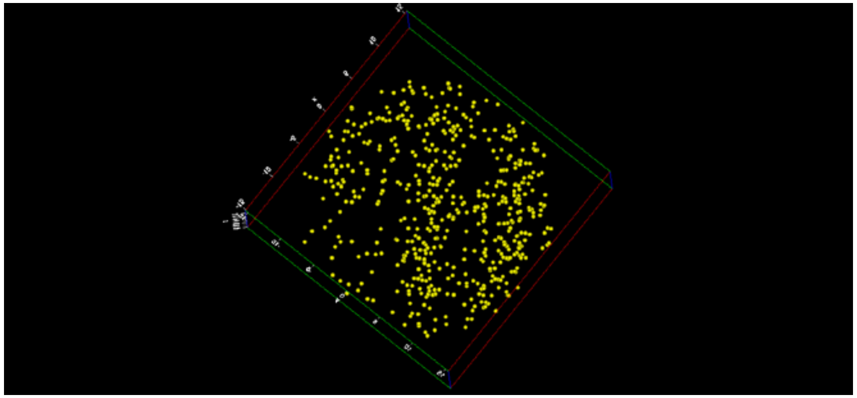
R. Kirchheim et al., *Scripta Metallurgica et Materialia* 28 (1993) 1229-12334.
H. Birnbaum et al., *Journal de Physique* C5 (1988) 397-401.

Twin Boundary Has Some Sulfur Segregation, But No D Segregation

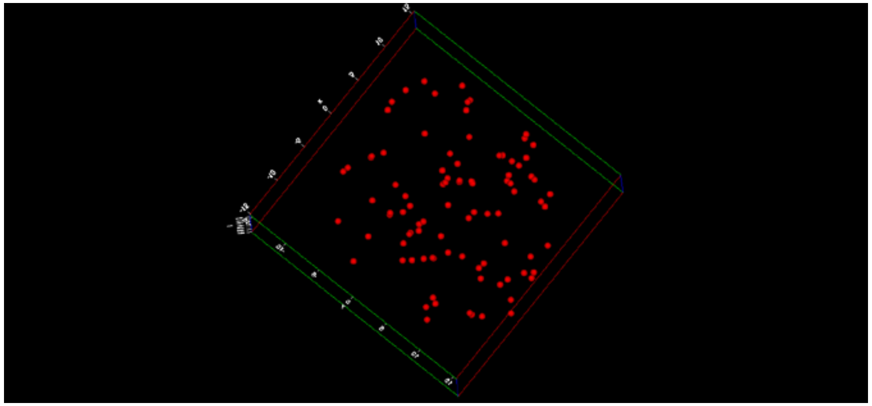




S



D



Summary

- Fracture prediction is impactful and multi-scale
- Current prediction of fracture is poor and will require strong coupling between experiments and simulations
- Atom-Probe Tomography provides a unique capability for 3-D characterization at the atomic scale
- It is sensitive to hydrogen isotopes (H environment greatly impact properties, but H is otherwise difficult to characterize)
- It can serve as input to sub-grain-level models
- It can validate atomic-scale models

Additional Acknowledgements

- ScD2 Project
  - N. Moody
- Fracture Project
  - B. Somerday, D. Medlin, R. Dingreville, B. Somerday, K. Hattar
  - W. Barrow, D. Spearot (University of Arkansas)
- Discussion of H Segregation and Trapping
  - D. Balch, N. Bartelt, C. San Marchi
  - D. Isheim (Northwestern University)

H Mapping Depends On (after T. Kelly)

- Overall H measurements (not localized) may strongly depend on:
  - Local Electrode and chamber cleanliness
  - Transfer time
  - Time in Analysis Chamber
  - Temperature during all of this
  - Pulse rate
  - Ion evaporation rate

- Localized H measurements (of phases) depend on
  - local field-evaporation field
    - causing changes in local tip shape, resulting in local magnification
  - Crystallographic faceting
  - Other defects (dislocations, grain boundaries)