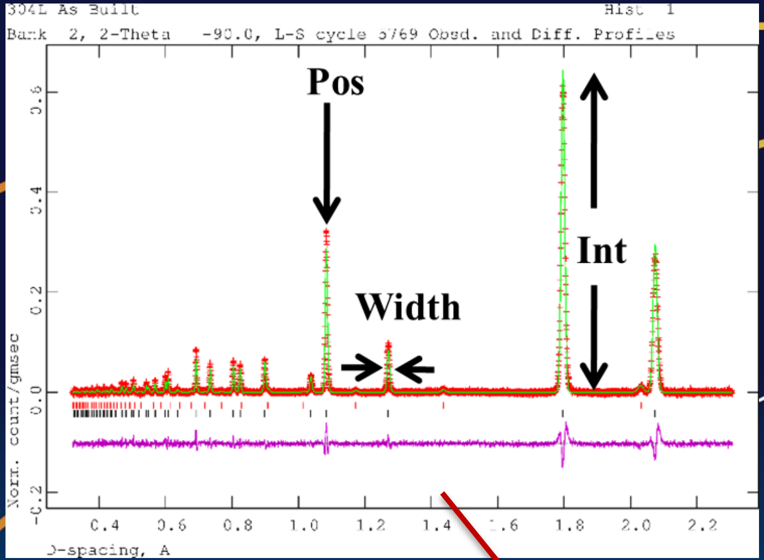

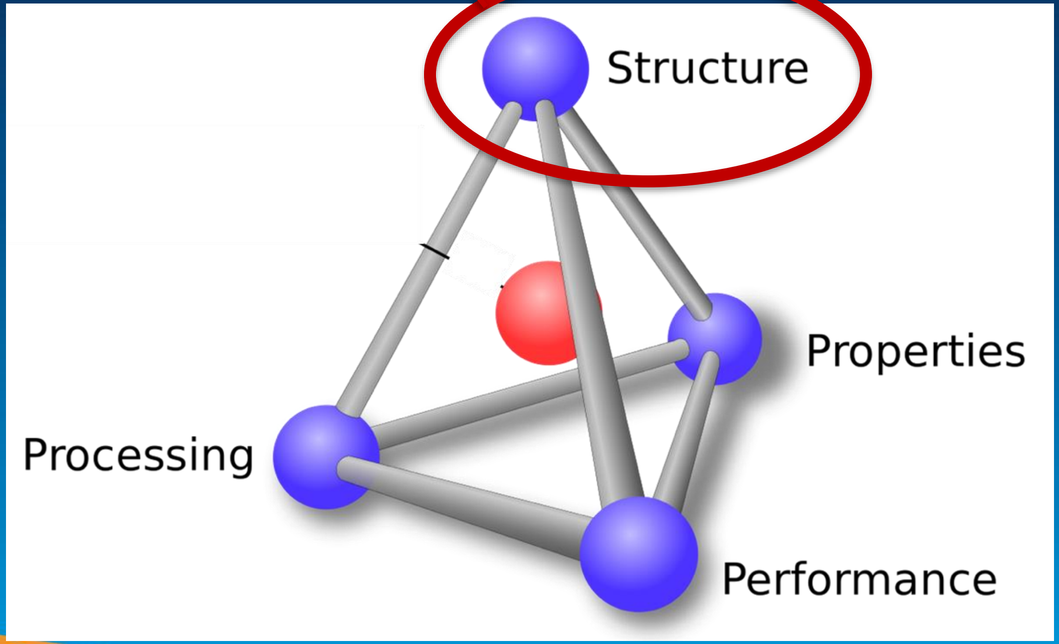

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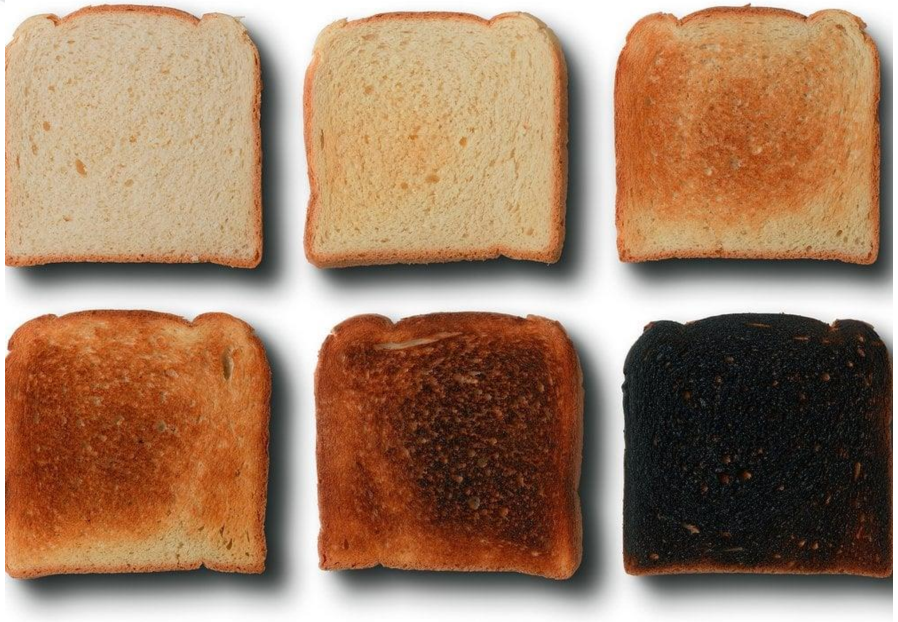
Using Diffraction to Make the Connection Between Materials Processing and Properties

Donald W. Brown

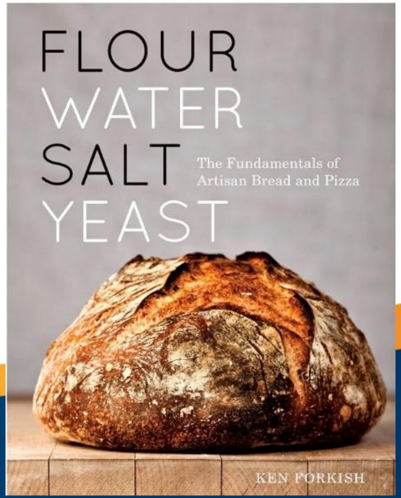
Los Alamos National Laboratory, Los Alamos, NM 87544

Flour, Water, Salt, Yeast…

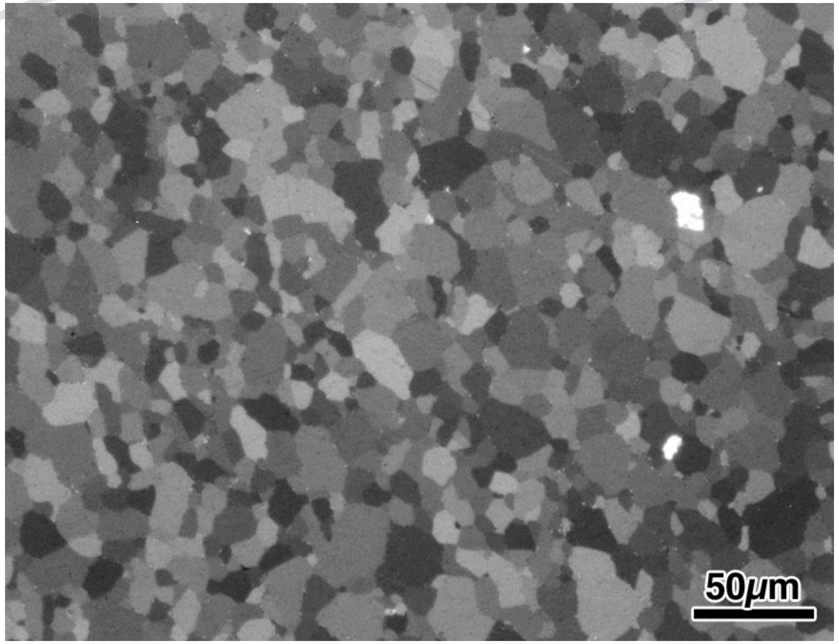


The ingredients are the same, the Properties (taste, texture, density, etc) are different. These are set by the Processing.

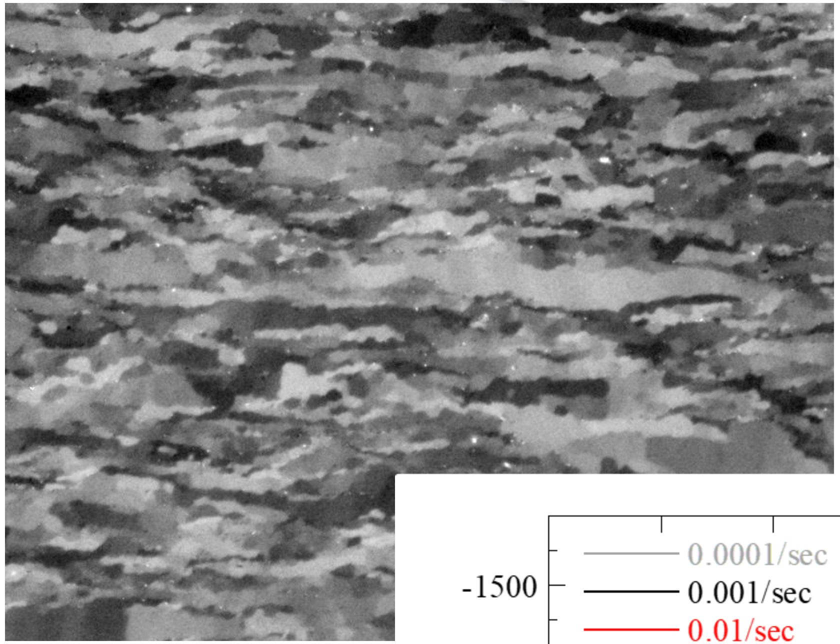
Microstructure Matters!



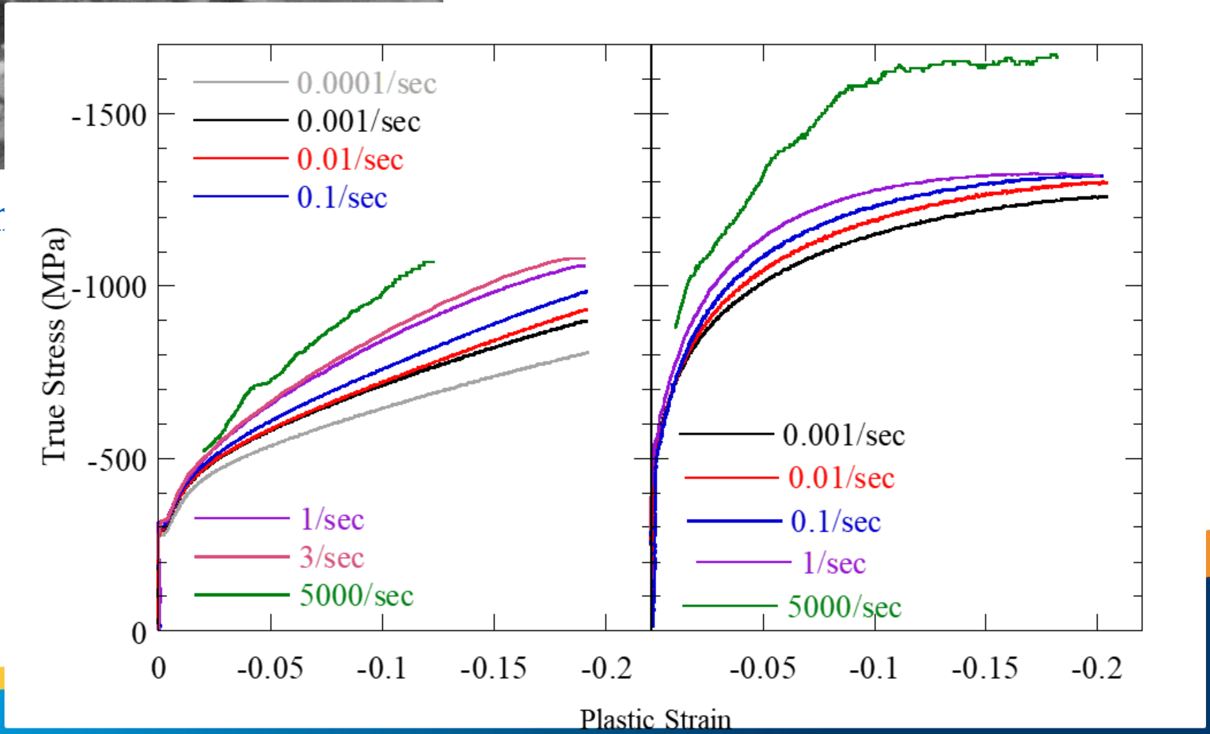
In Materials, We Call This The Process/Structure/Property Relationship.



Hot-Pressed Beryllium



Rolled (wrough



Bottom Line: Scattering Can Be Used to Quantitatively Determine Some Microstructural Features in Engineering Materials

Microstructural Feature	Property Effects	Accessible to Scattering?	Observable
Phase	Strength, Ductility	✓	Unique Diffraction Peaks
Texture	Elastic Modulus, Speed of Sound	✓	Peak Intensity
Defects/ Dislocations	Strength, Phase Stability	✓	Peak Width
Solute Distribution	Phase Stability, Strength	✓	Peak Shift
Residual Stress	Lifetime	✓	Peak Shift
Density Fluctuations (pores, phases, etc)	Mechanical Stability, Shock Propagation	✓	Increase in SAS
Grain Size	Strength	✗	N/A

- So far this is agnostic to Neutrons or HEXRD (>35keV keV).

Los Alamos Neutron Science Center : LANSCE

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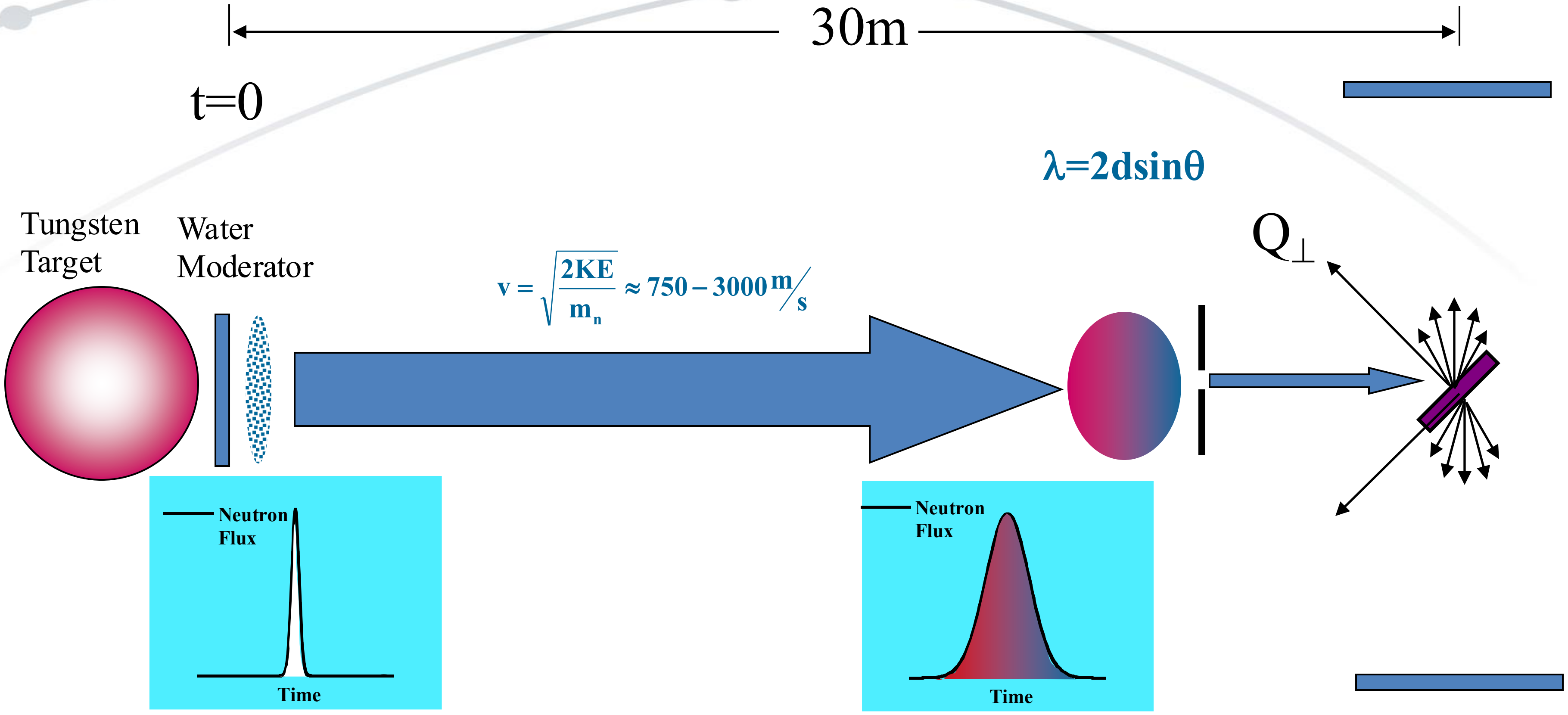
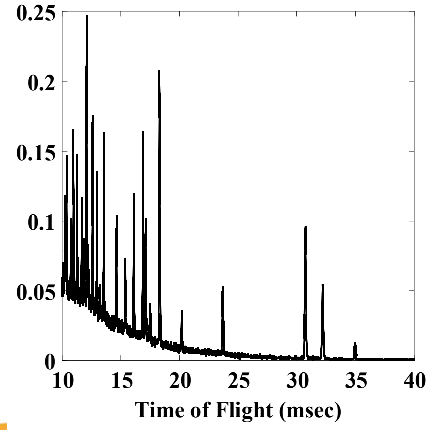
Neutron sources do not sit on desktops

NNSA National Nuclear Security Administration

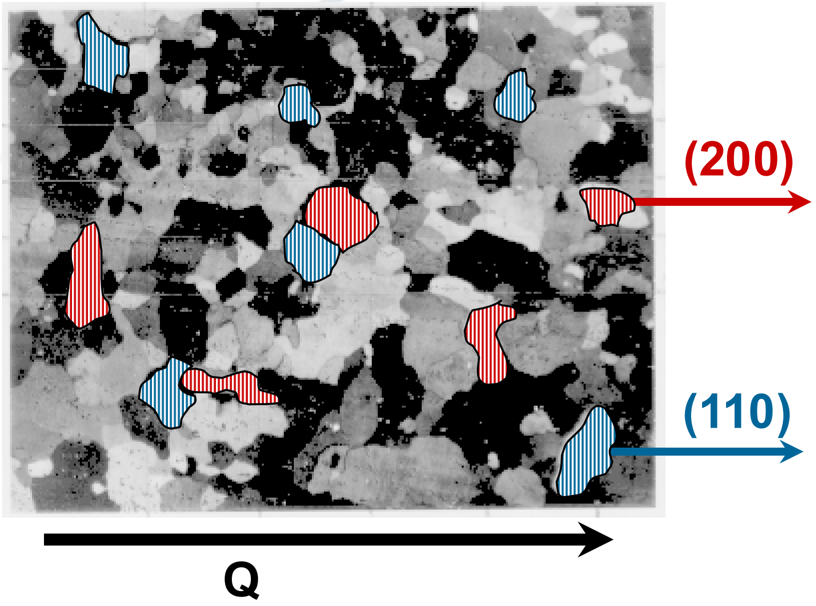
Time of Flight Neutron Diffraction at a Spallation Source

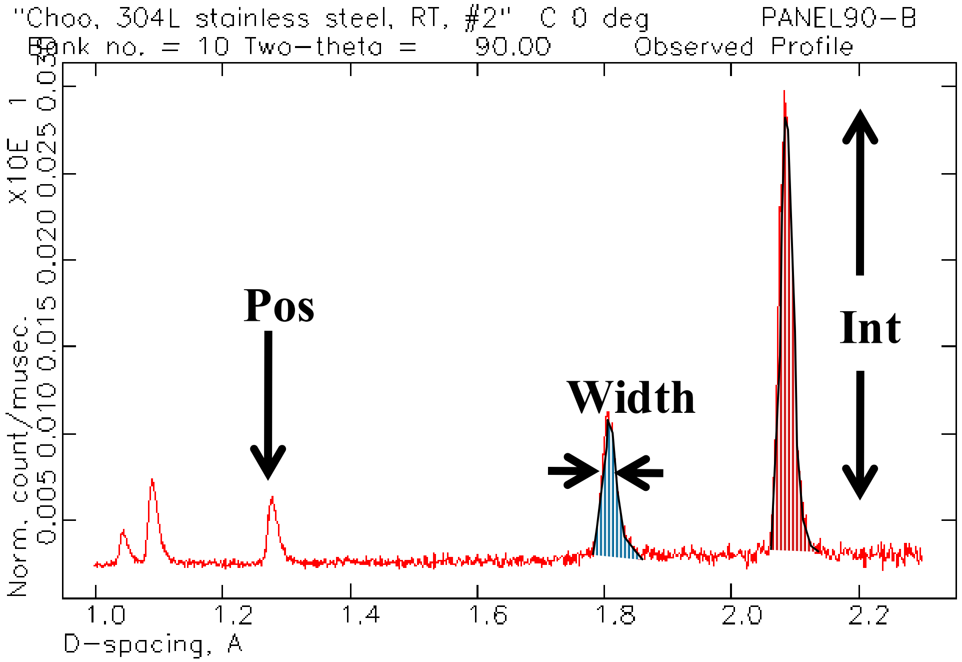




- Velocity (and λ) determined by the neutron time of flight.
- Entire diffraction pattern collected with unique diffraction vector.
    - Advantageous for anisotropic samples.

Diffraction Provides Non-Destructive Access to the Microstructure





- **Diffraction naturally distinguishes between the response of different grain orientations (and phases).**
- **Position : Stress, Temperature, Chemistry**
- **Intensity : Texture, Phase Fraction, Disorder**
- **Width : Size, Dislocation Density, …**
- **Crystal anisotropy means grain response is orientation dependent.**
- **Lattice parameter, a, averages over all orientations and best approximates macroscopic response.**

Stress is Calculated From the Elastic Lattice Strain

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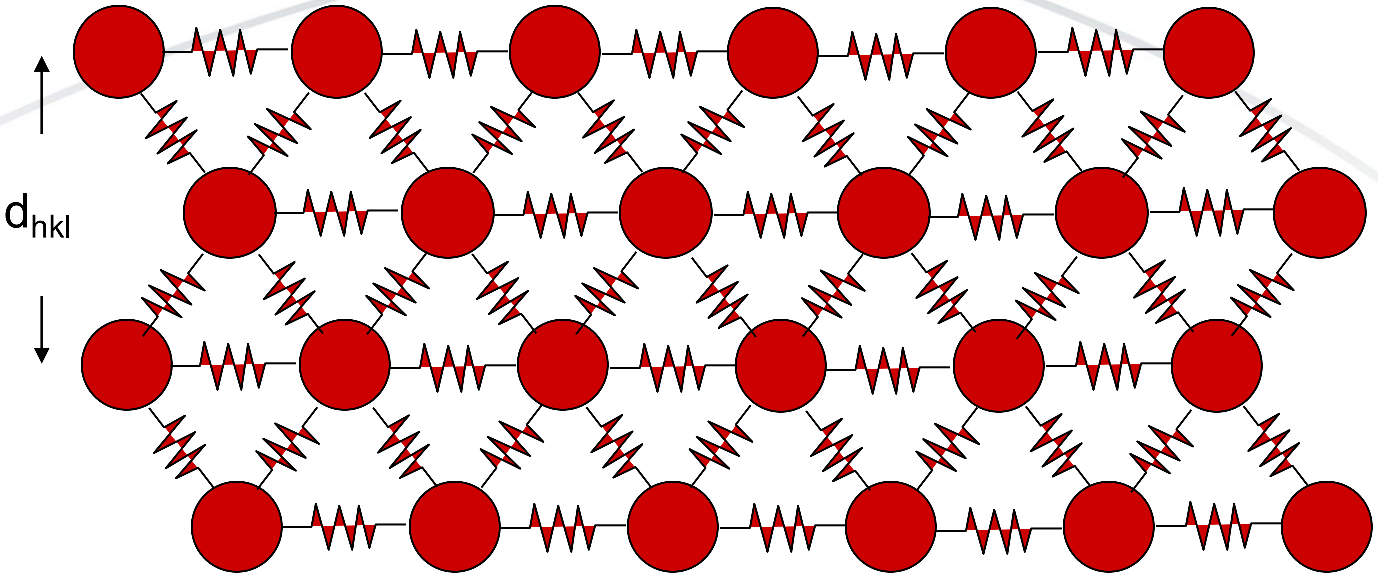

- We measure the spacing between atoms very accurately, ~10 ppm.
- Calculate lattice strains from change in atomic spacing due to stress.
  - Lattice strain : $\varepsilon_{lattice} = \frac{a - a_0}{a}$; $\varepsilon_{hkl} = \frac{d^{hkl} - d_0^{hkl}}{d_0^{hkl}}$
- If we know the spring constants, we can calculate the stresses from the strains.
  - $\sigma_{ij} = C_{ijkl}\varepsilon_{kl}$

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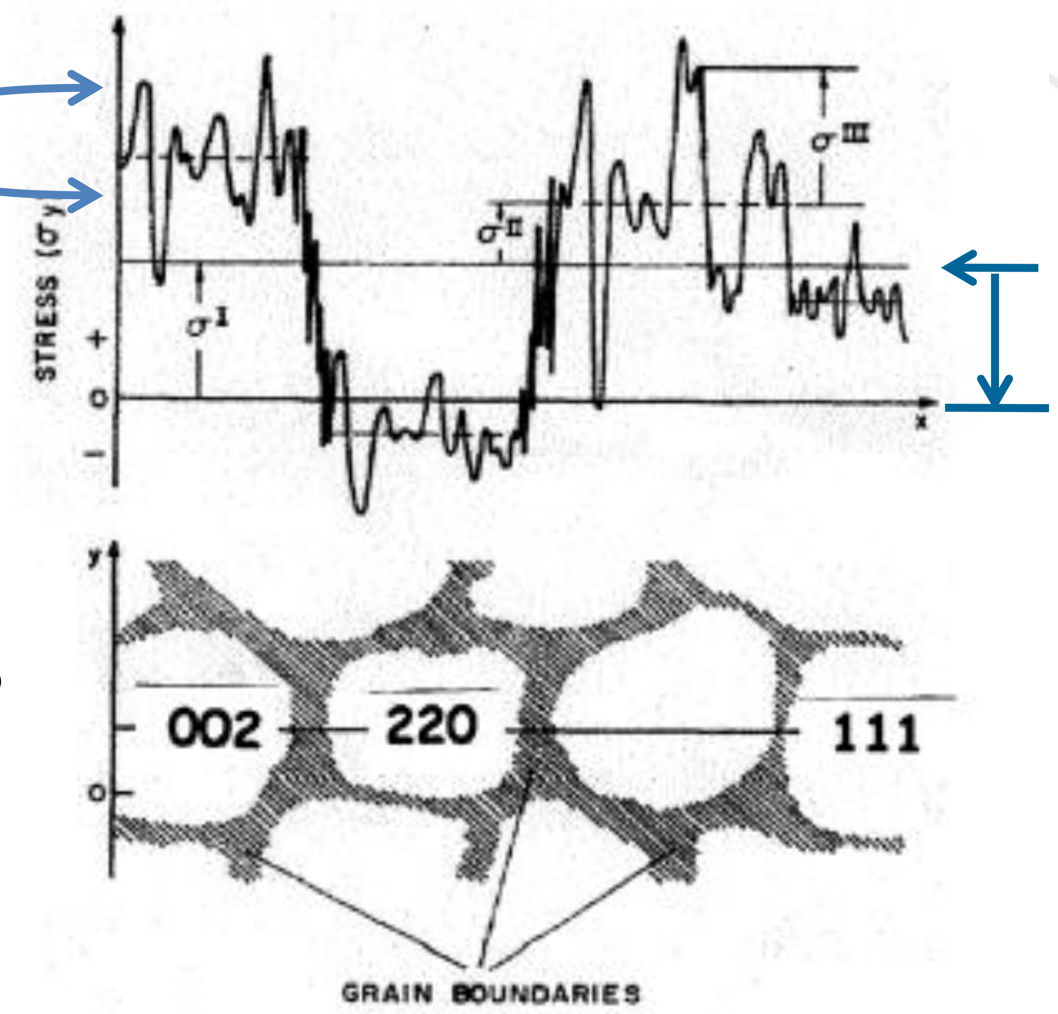
Diffraction Probes Stresses/Strains at Multiple Length Scales

Type III Intergranular Stress: peak width



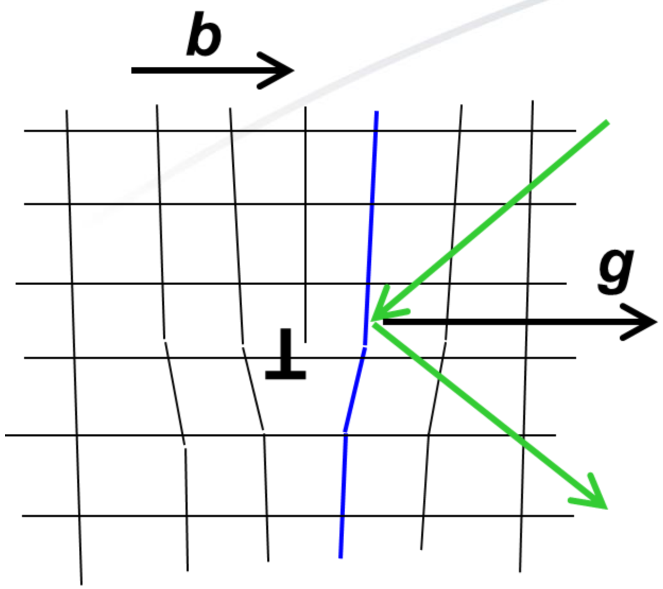
Type I Macroscopic Stress: lattice parameter

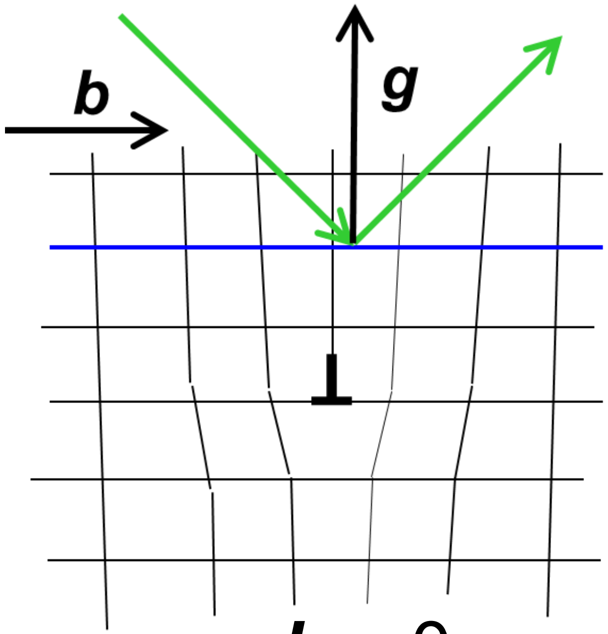
Type II Intergranular Stress: d-spacing (hkl)

- Elastic, plastic, thermal anisotropy, etc contribute to intergranular stress.
- Dislocation density drives intragranular stress.

E. Macherauch: *International Guidebook on Residual Stresses*. (Pergamon Press, Elmsford, New York, 10523, 1987), p.^pp. 1-36.

Stress Field Associated With Dislocations Results in Peak Broadening





gb $\neq 0$

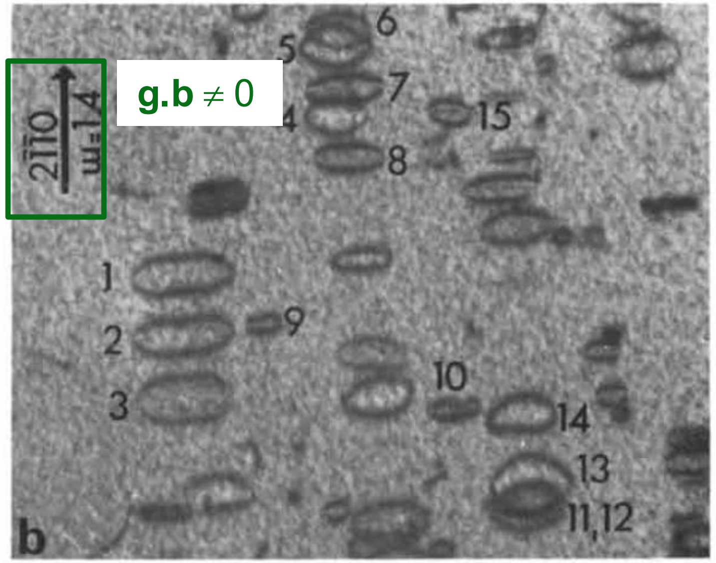
dislocation is visible

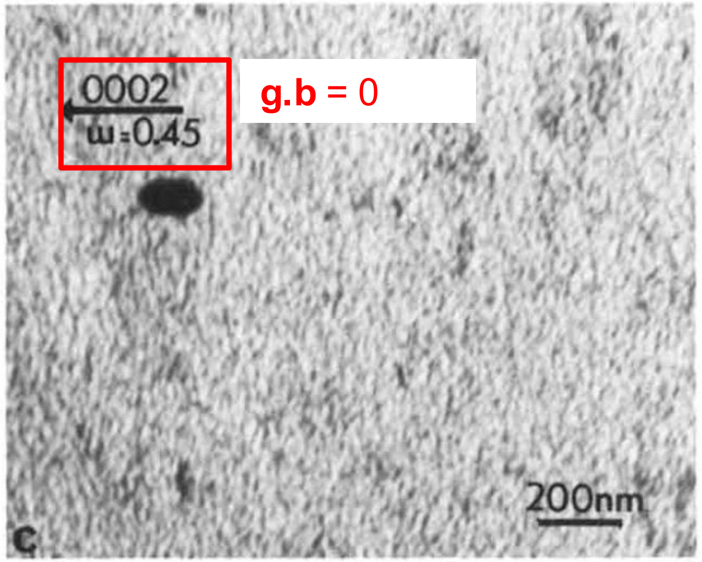
strong contrast

gb $= 0$

dislocation is invisible

weak contrast



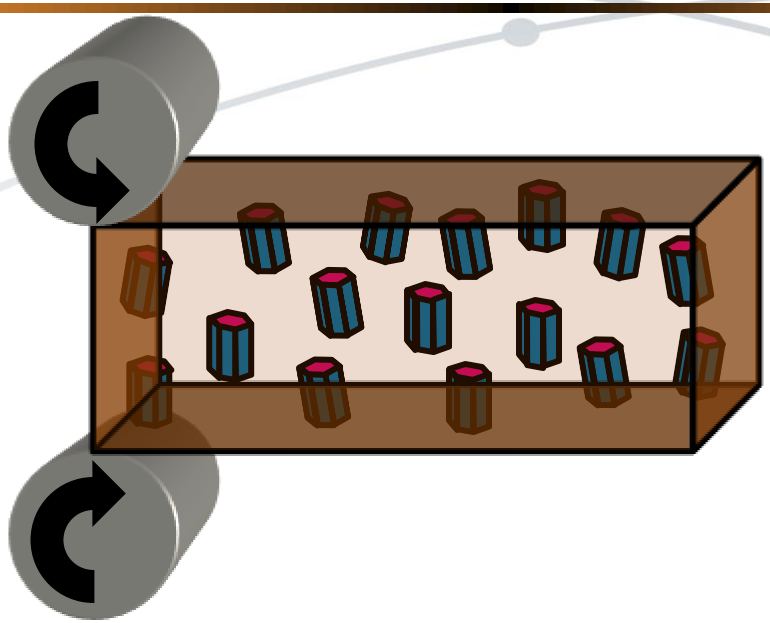


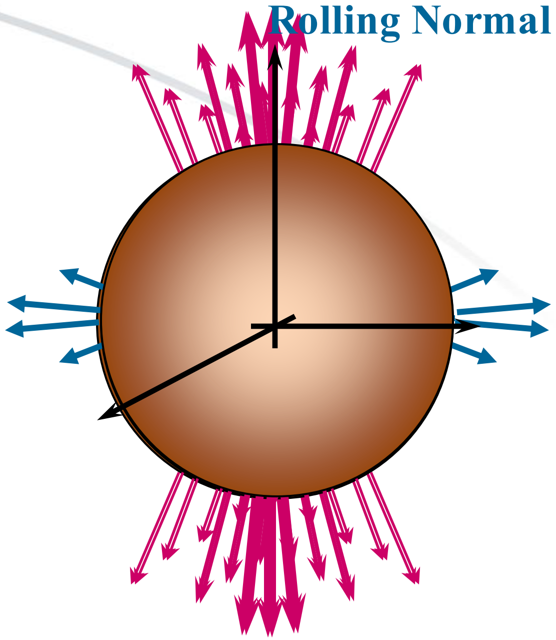
strong line broadening

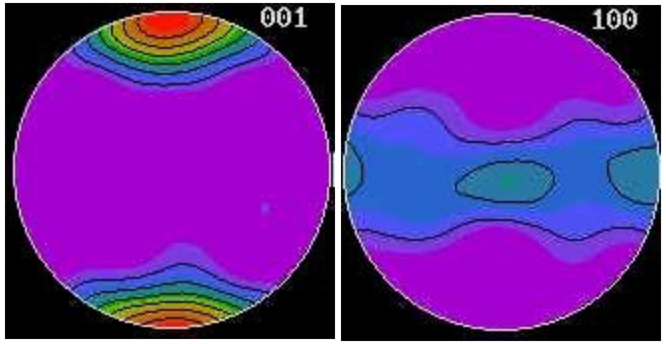
weak line broadening

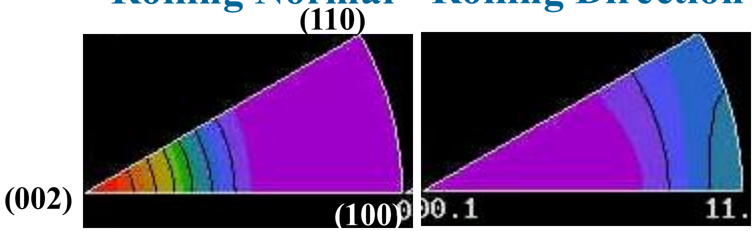
Jostsons, A., Kelly, P. M. and Blake, R. G., *Journal of Nuclear Materials*, **66** (1977) 236-256.

Representation of Crystallographic Texture : Pole Figures and Inverse Pole Figures



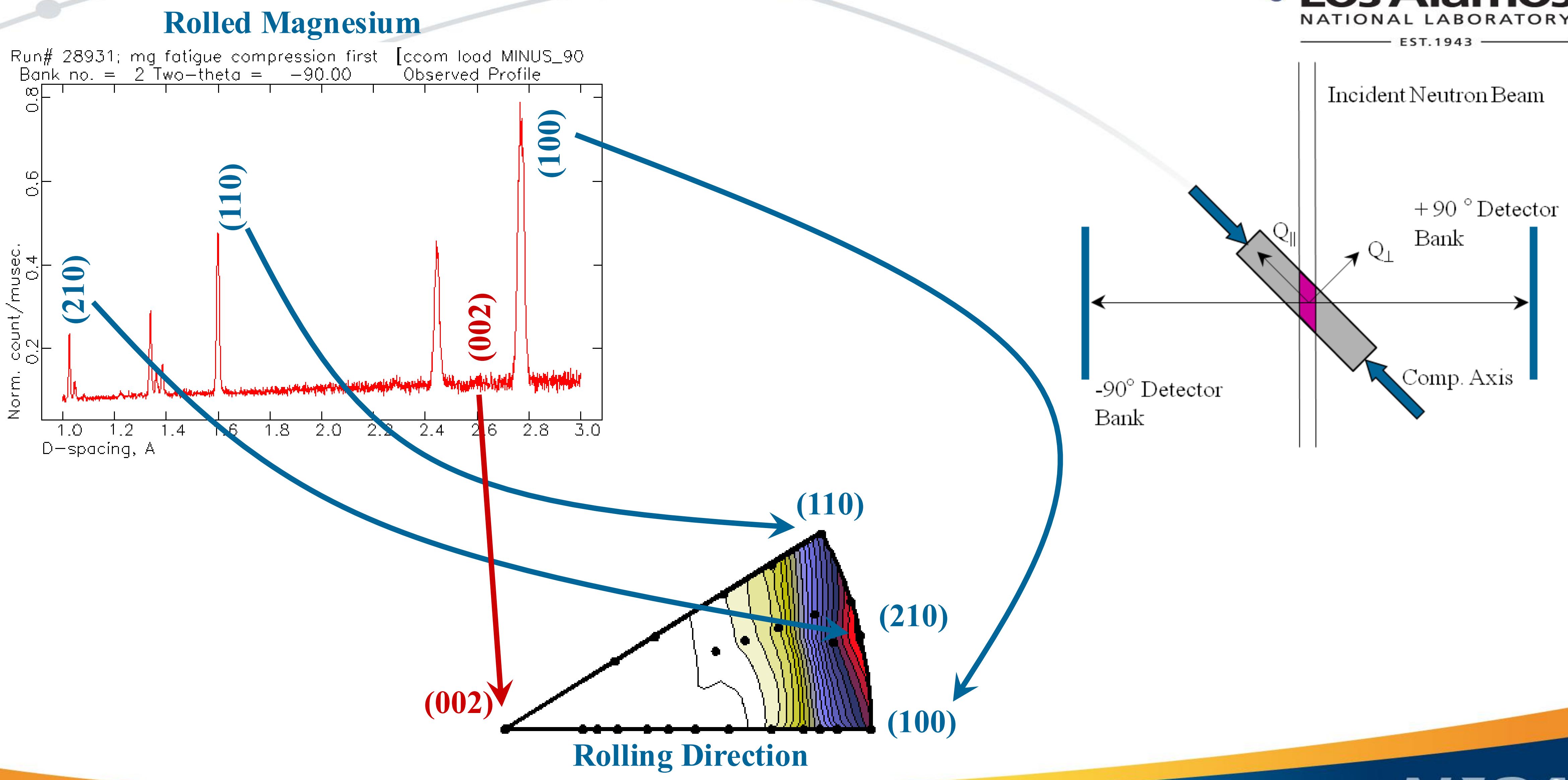




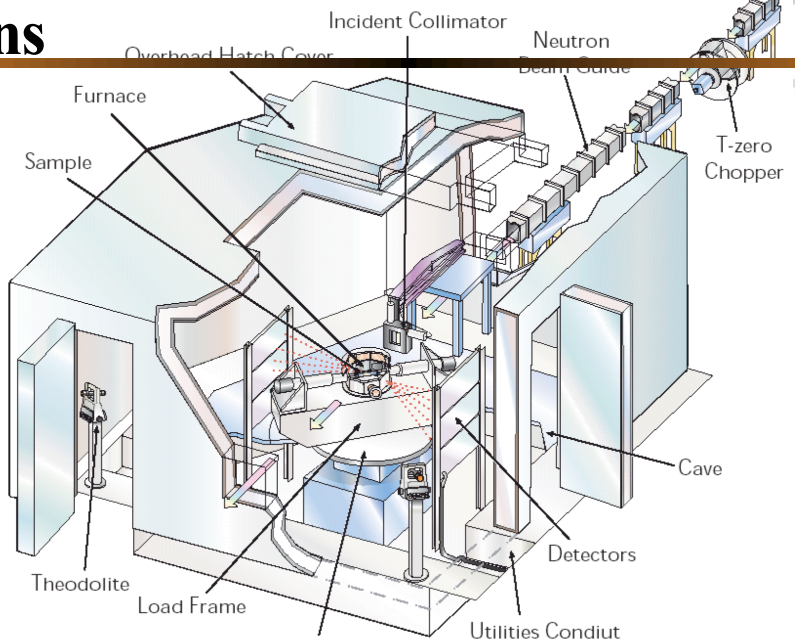


- Pole Figure :Density of a given {hkl} as a function of orientation relative to sample axis.
  - Measured directly with monochromatic x-ray or neutron diffraction.
- Density of all {hkl}'s on stereographic triangle along a given sample axis.
  - Measured directly with TOF neutron diffraction

Peak Intensities Relate Directly to Crystallographic Texture

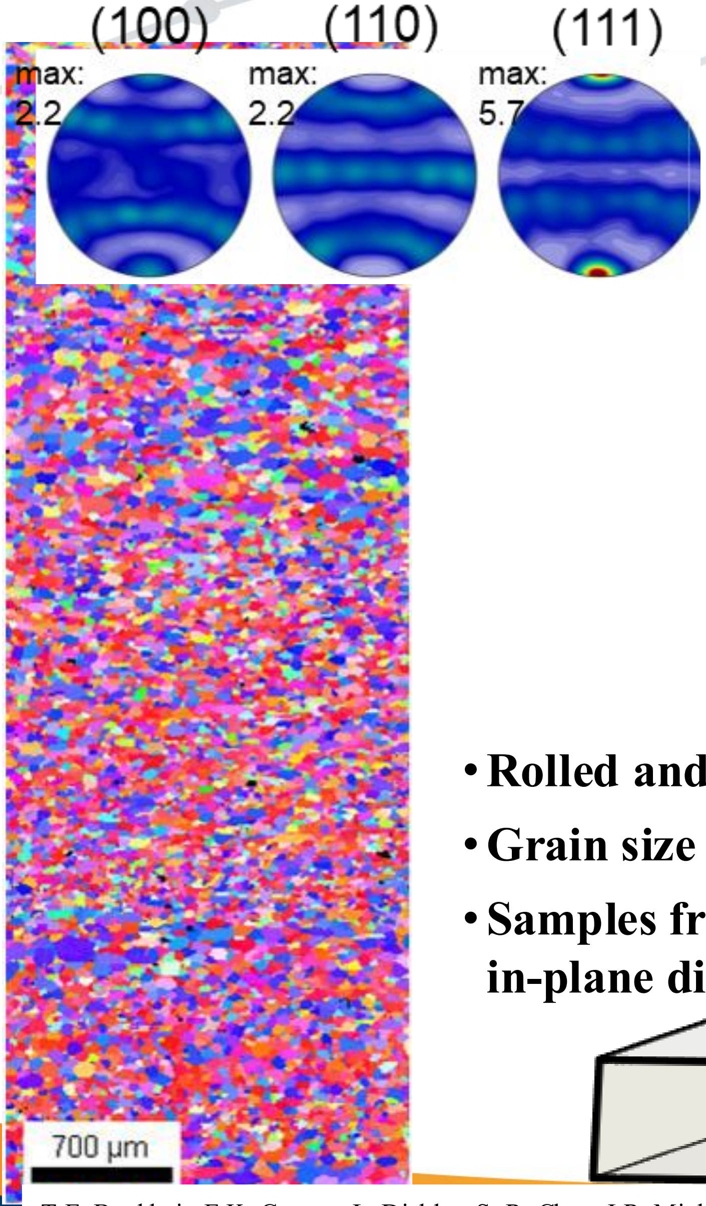


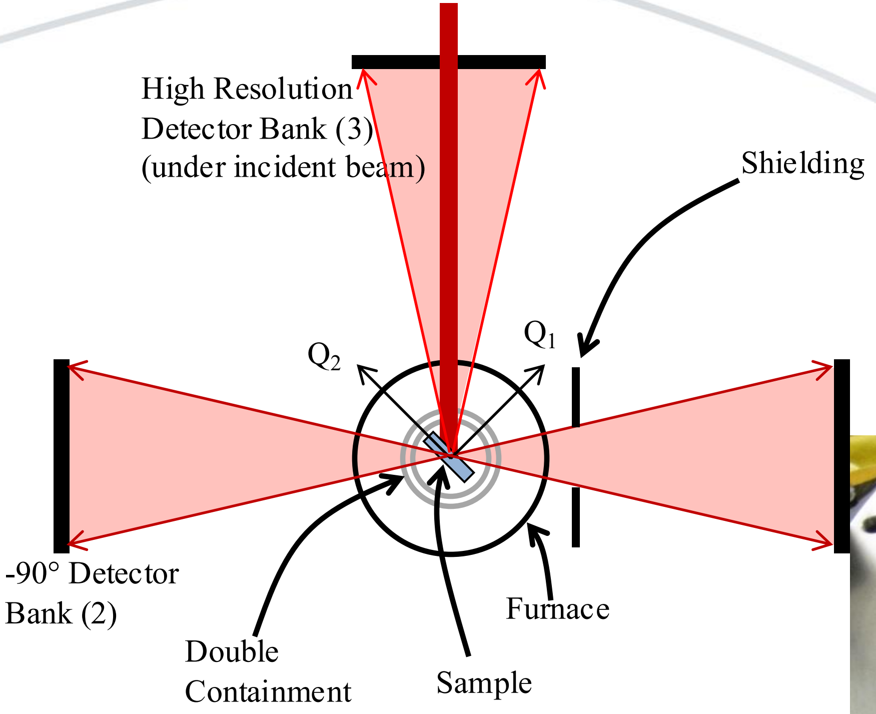
SMARTS Designed to Study Engineering Materials Under Operating Conditions

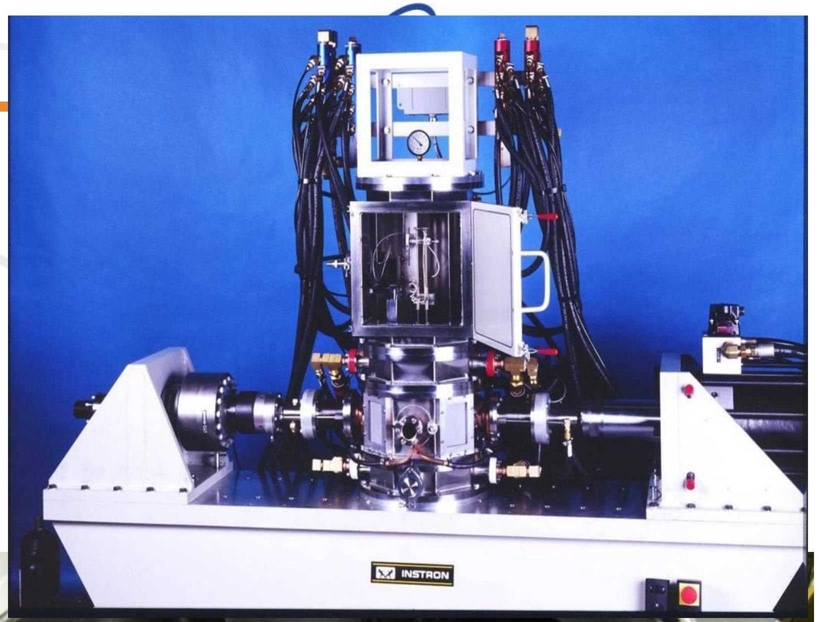


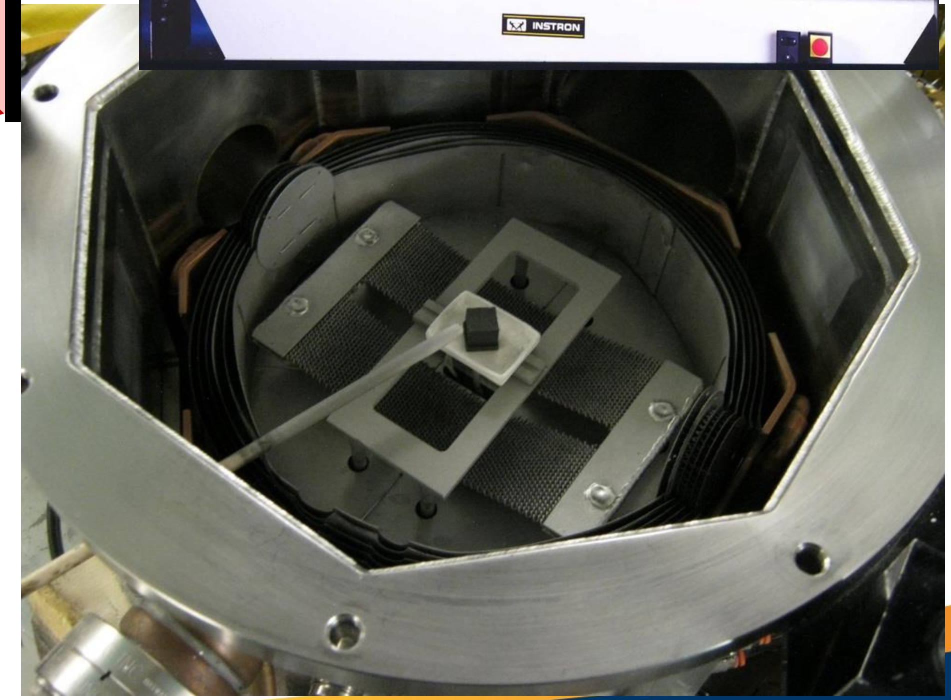
- Moderately high resolution diffractometer.
  - Can determine lattice strain to 1 part in 10^5.
- 250 kN tension or compression, 100 kN T-C cycling.
- 90-2000K (1700K with loading).
- Quenching furnace (>20C/sec).
- Best spatial resolution of 1mm.
- Sample table : horizontal travel ±30 cm, vertical ±60 cm, 370º rotation, 1500 Kg capacity.
- Radioactive samples are easy.
- ~1 minute time scales, 1 mm length scales.
- dbrown@lanl.gov

Sequential Deformation And Recovery Experiments on Tantalum Completed on SMARTS









- **Rolled and annealed plate Tantalum**
- **Grain size of roughly 35 µm**
- **Samples from the through-thickness and in-plane directions**

T.E. Buchheit, E.K. Cerreta, L. Diebler, S.-R. Chen, J.R. Michael (2014). Report SAND2014-17645. Sandia National Laboratory.

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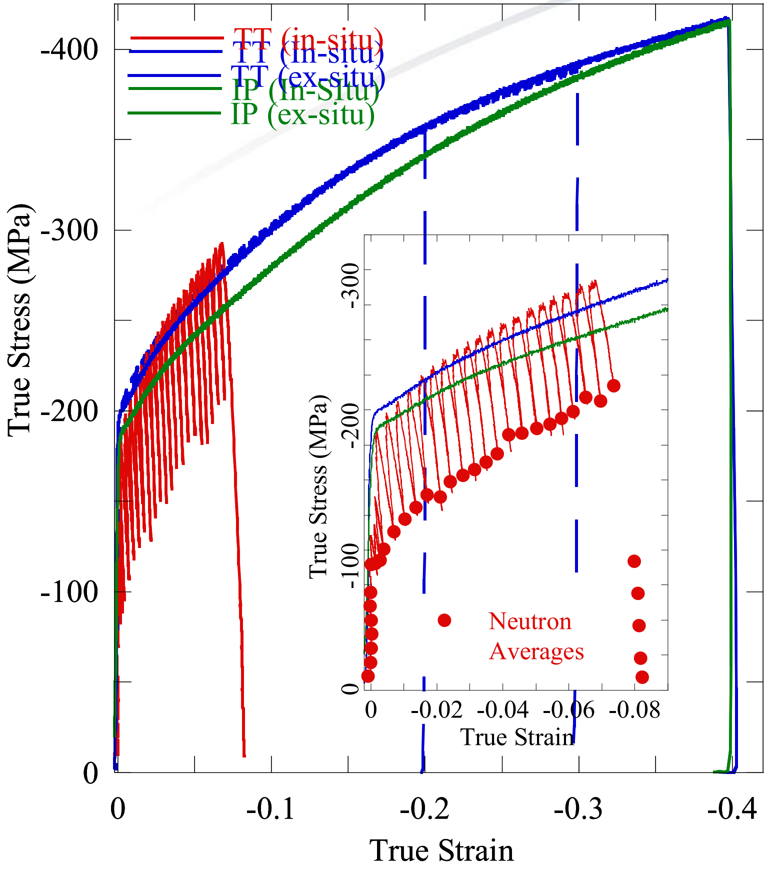
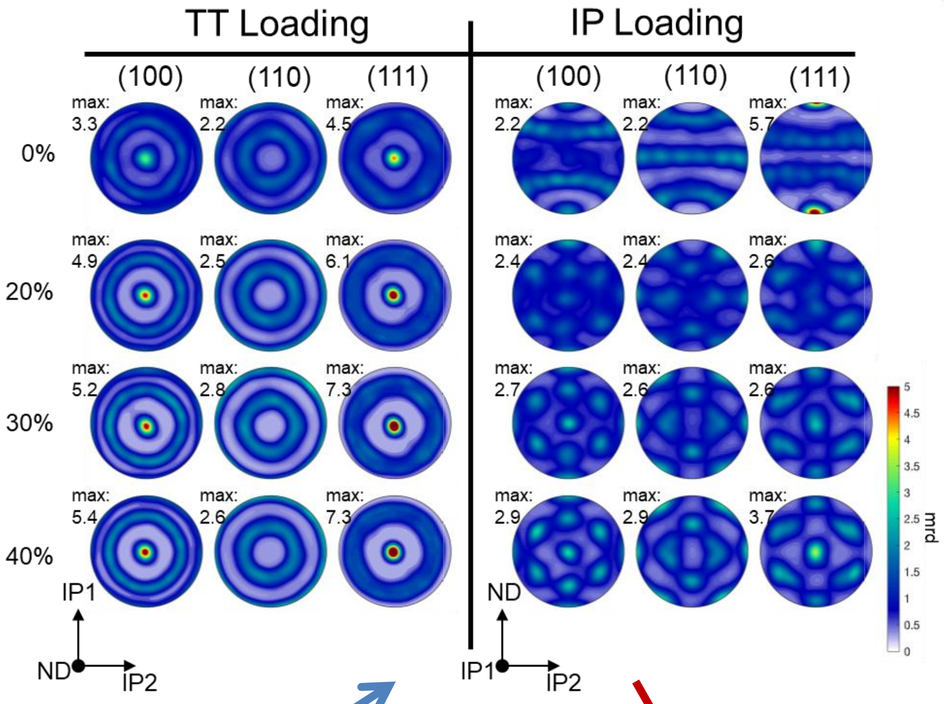
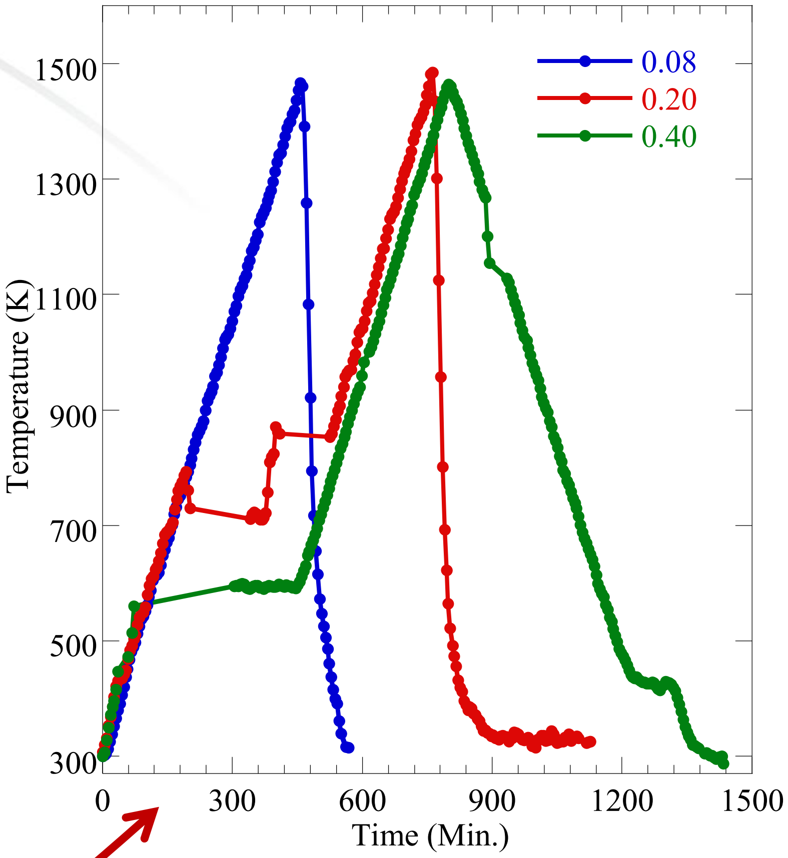
National Nuclear Security Administration

Sequential Deformation And Recovery Experiments on Ta Completed on SMARTS



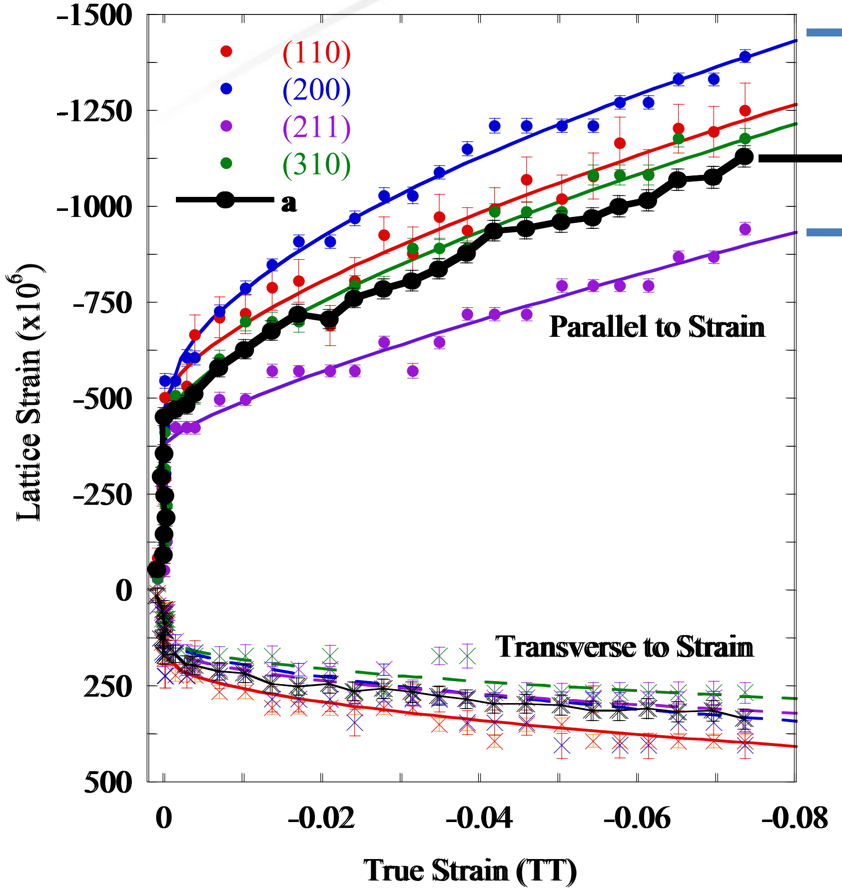




- **In-Situ measurements limited to about 8% strain before buckling typically occurs.**
- **Significant relaxation occurs while neutron data is collected.**

Diffraction Measurements on SMARTS Reveal Stress Information at Multiple Length Scales

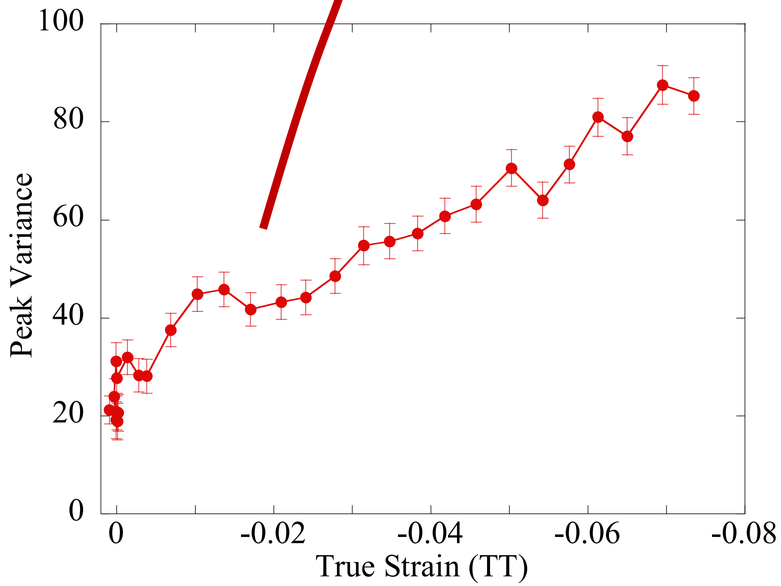
$$\sigma_{ij} = C_{ijkl}\varepsilon_{kl}$$

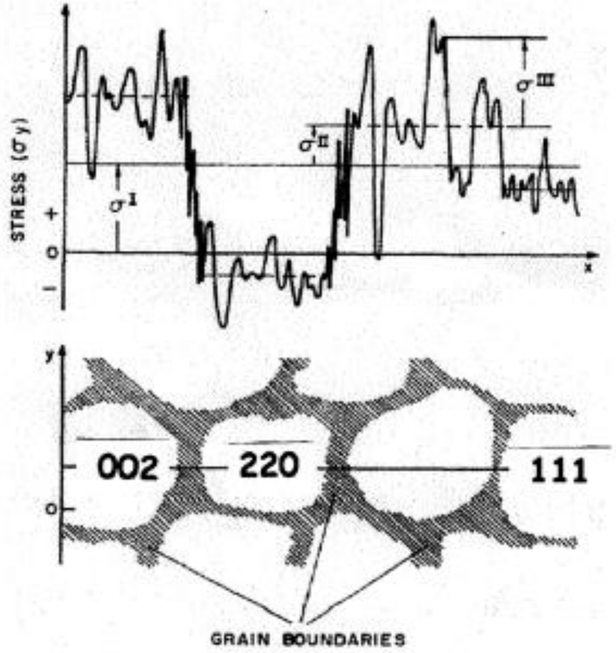


This spread represents the response to intergranular stresses, called anisotropy strain.

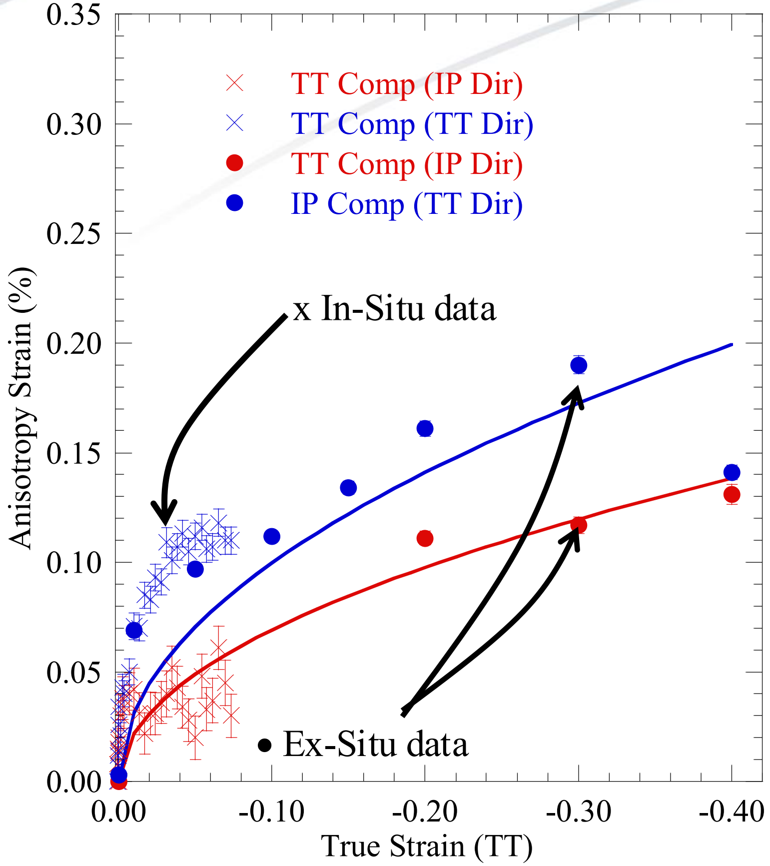
The strain from the lattice parameter (averaged over all grain orientations) is representative of the macroscopic strain.

Increase in peak breadth is indicative of intragranular strains and is linked to dislocation density

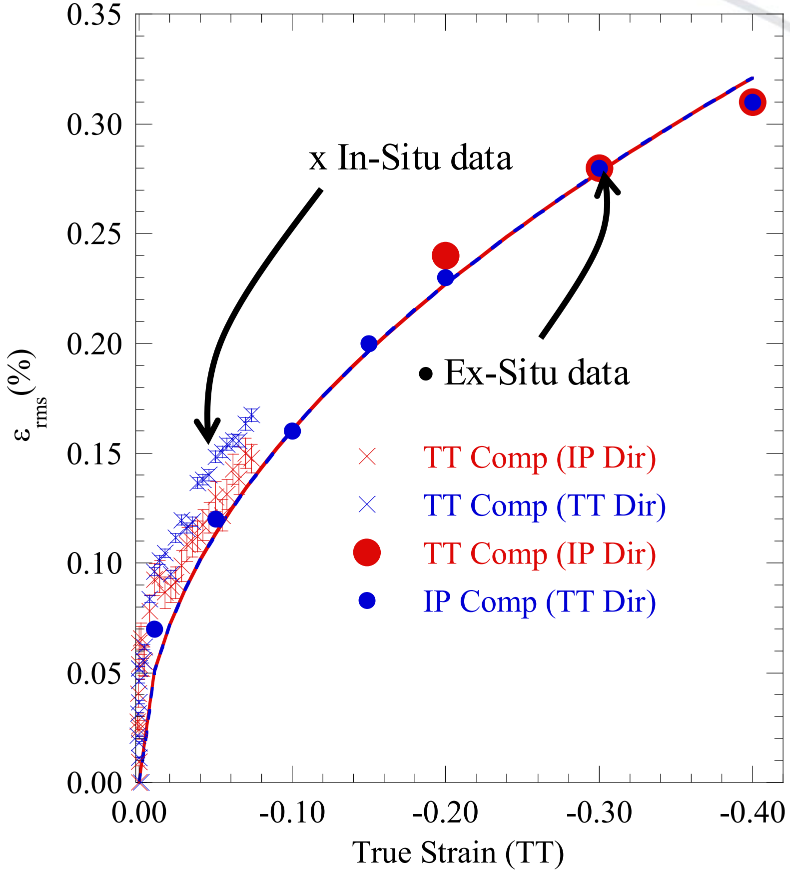




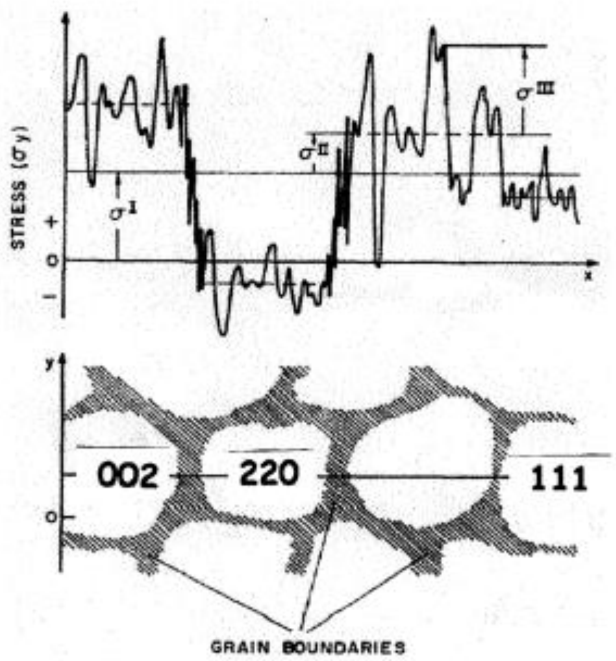
Ex-Situ Measurements Extend to Larger Strains



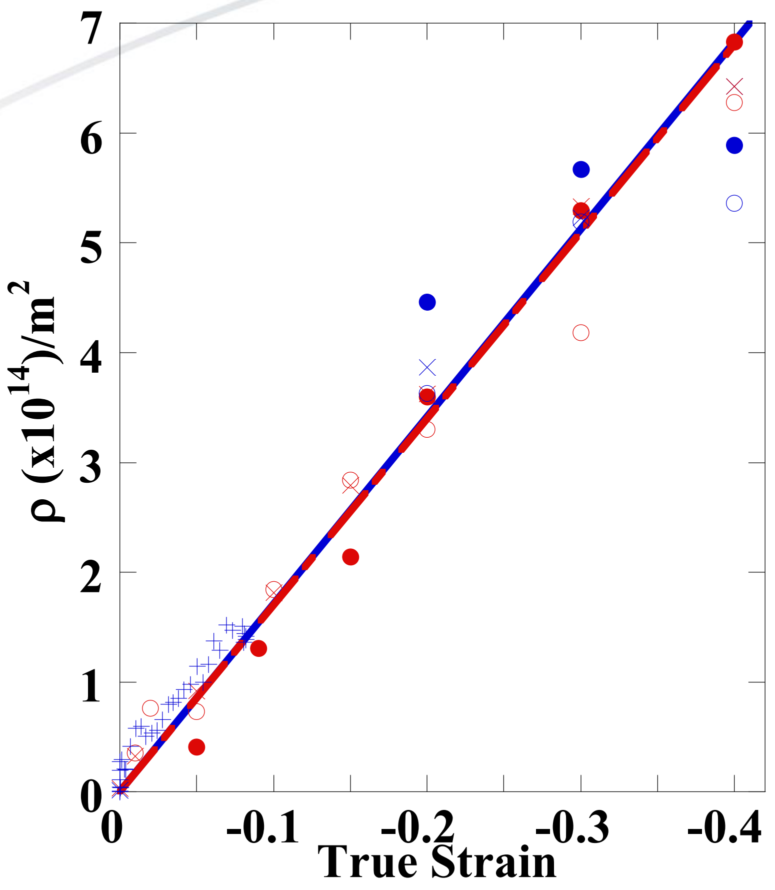
Intergranular Stress

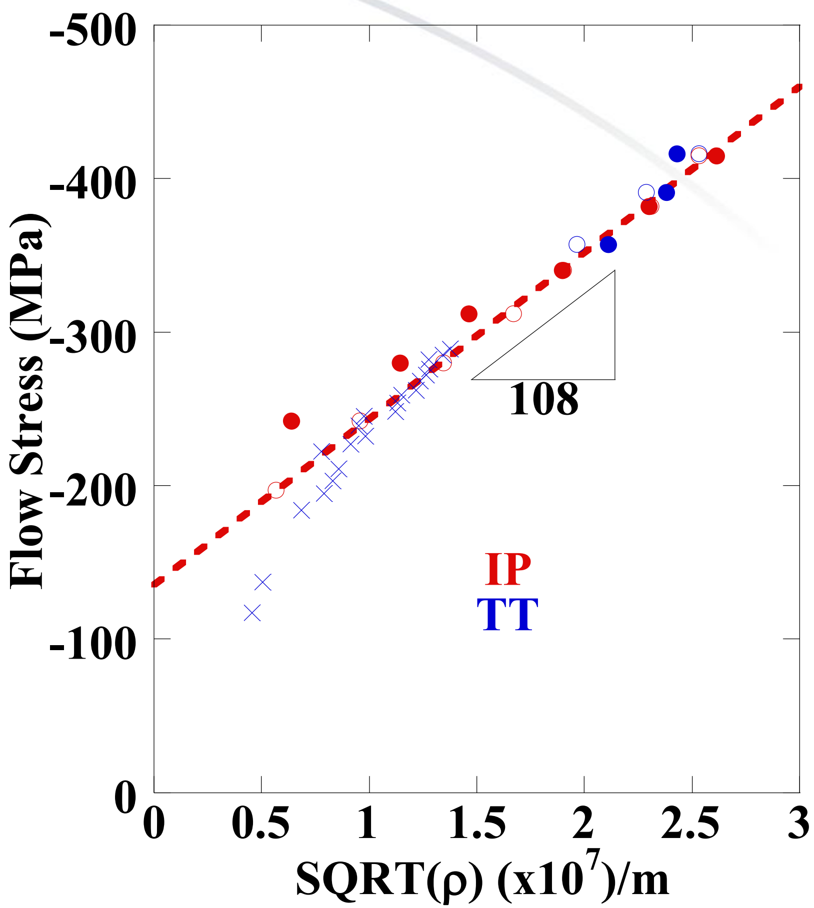


Intragranular Stress
Dislocation Density



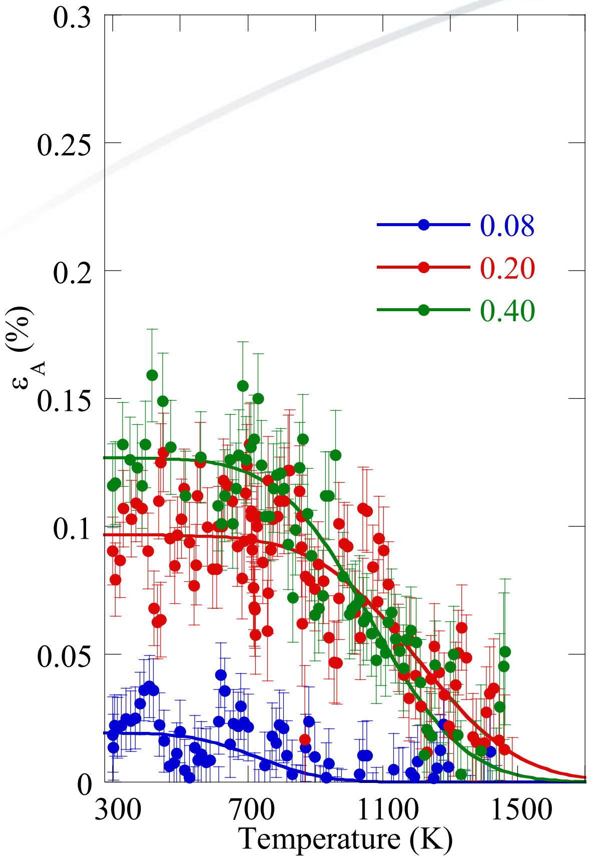
Dislocation Density Determined From the Diffraction Data



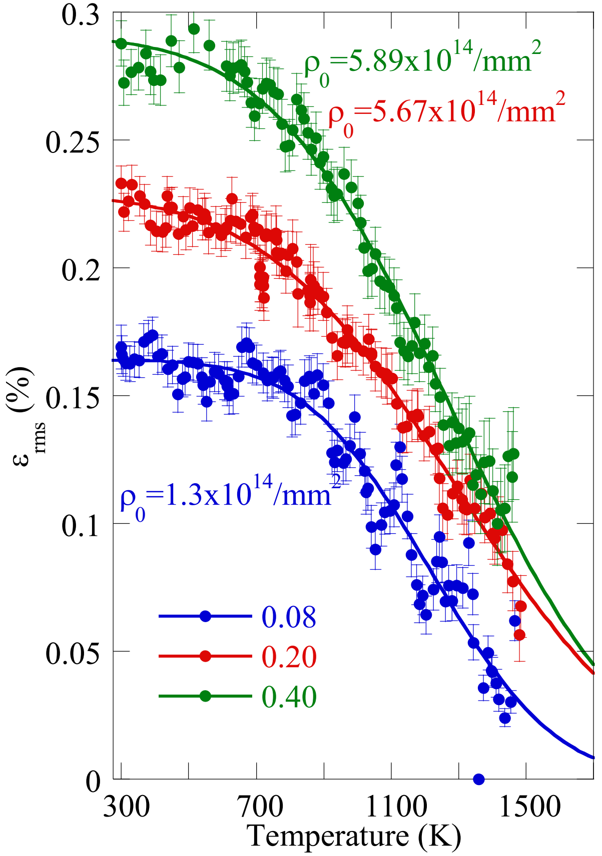


$$\sigma_f = \sigma_c + T\alpha\mu b\sqrt{\rho}$$

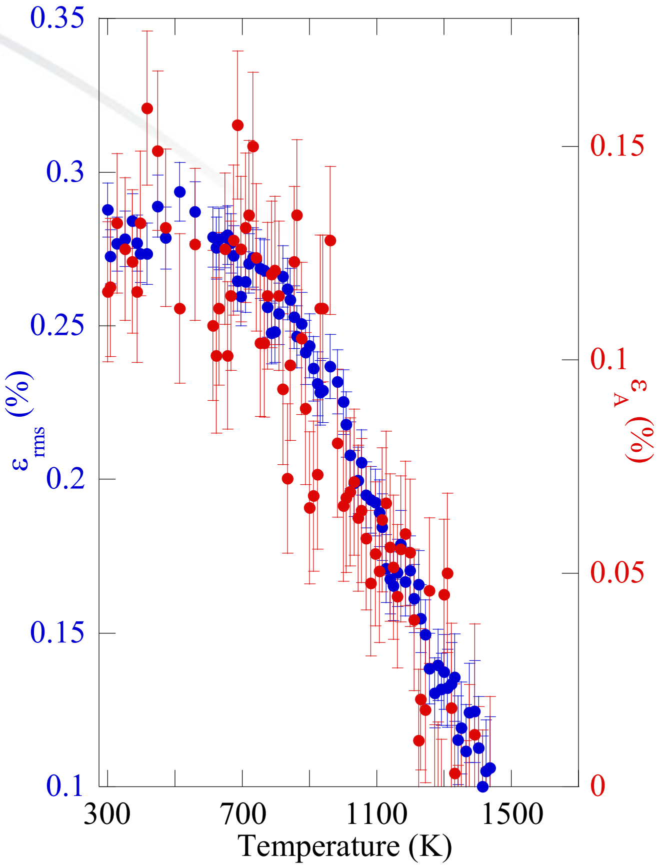
Recovery of Dislocations in Deformed Tantalum Initiates at 700K



Intergranular Stress



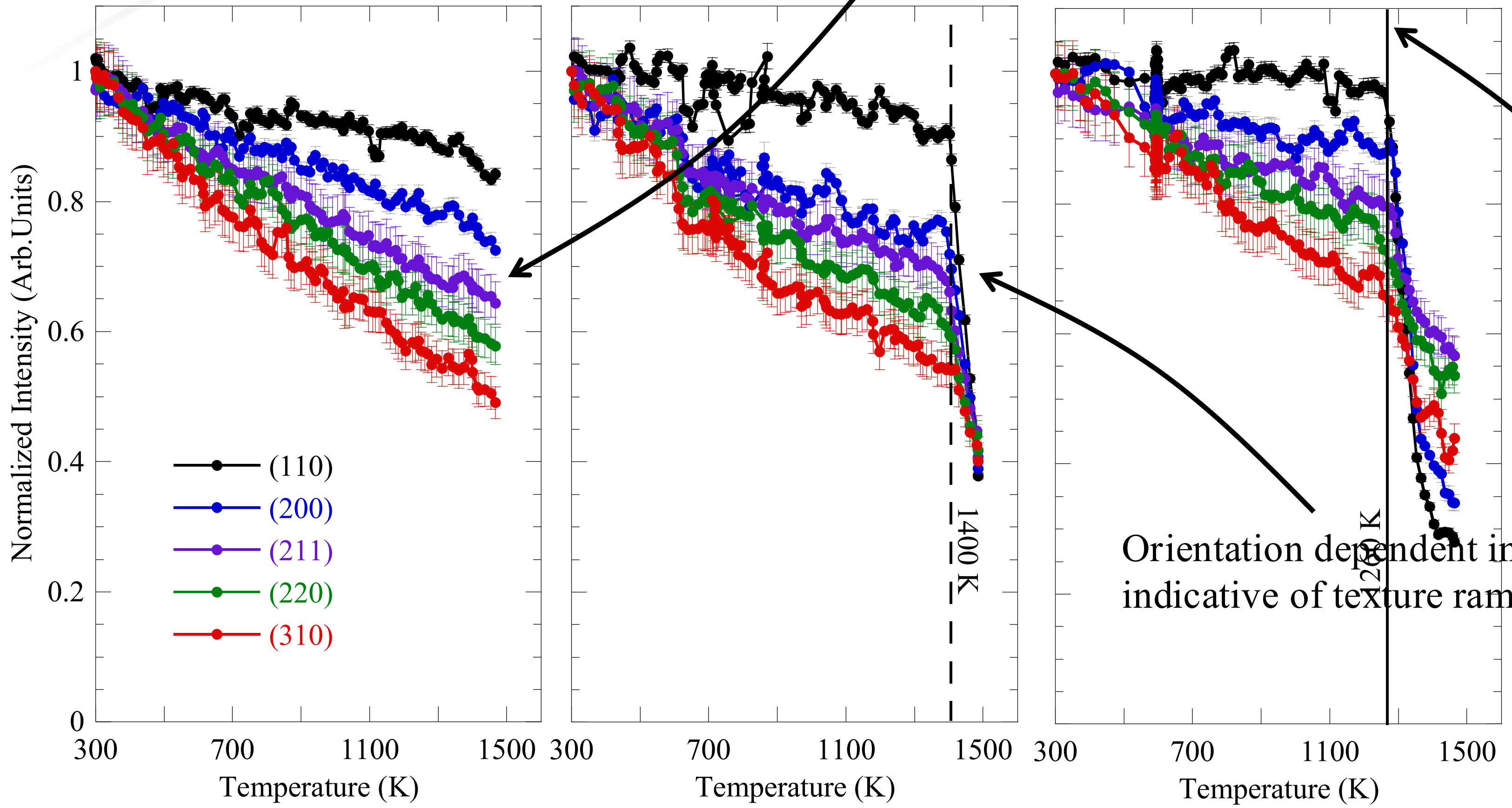
Intragranular Stress



Independent of the initial dislocation density (stored energy).

Recrystallization Manifests as a Texture Change at Higher Temperature

q dependent reduction in intensity is a response to the increase of the Debye-Waller factor



8% Strain

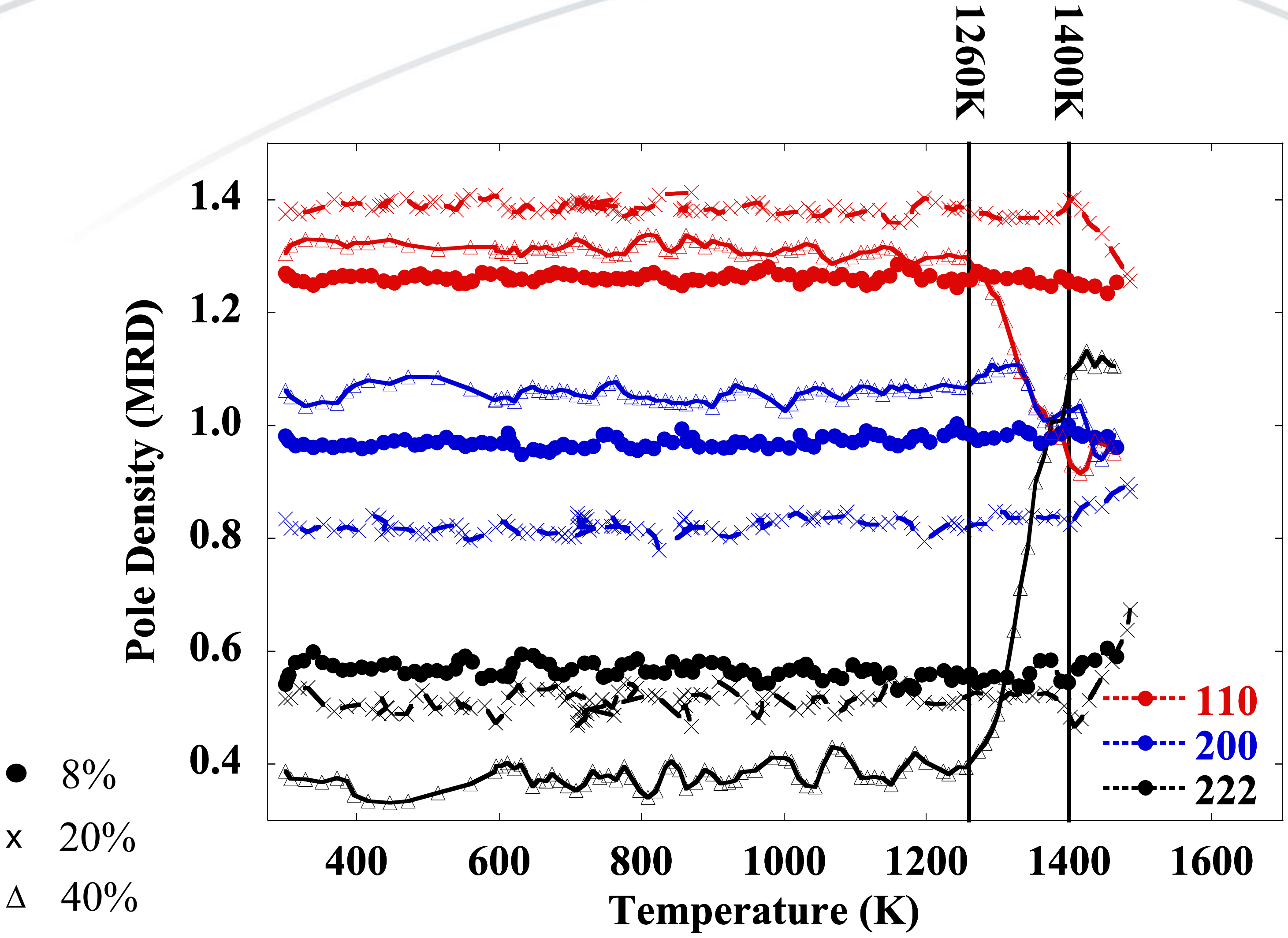
20% Strain

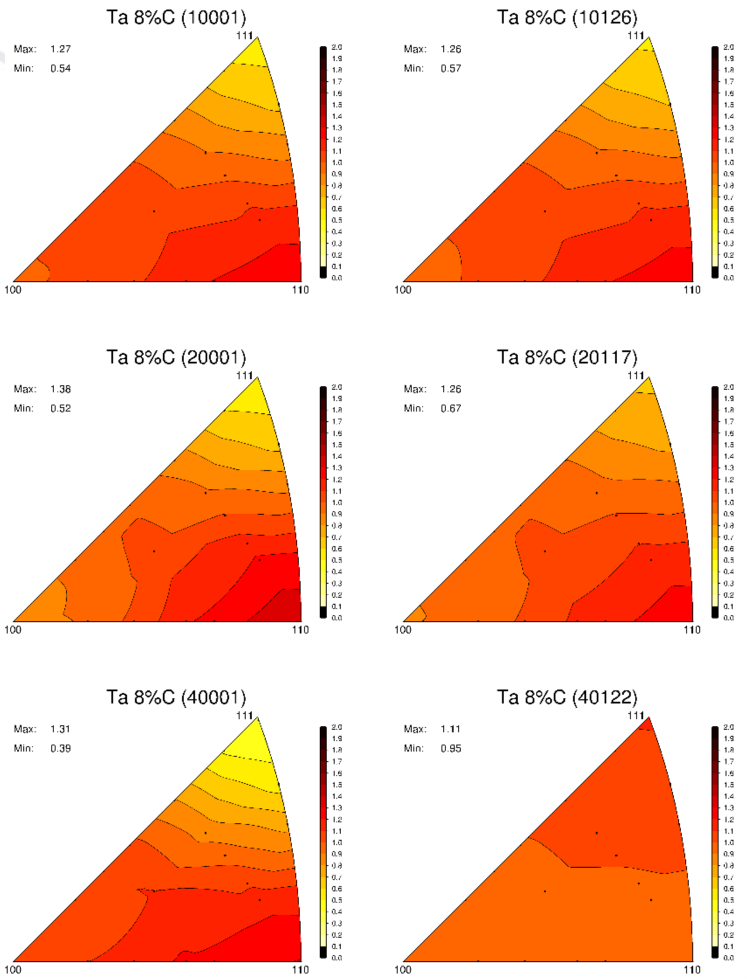
40% Strain

Occurs earlier with increased deformation

Orientation dependent intensity change is indicative of texture ramdomization

Increased Dislocation Density (Stored Energy) Drive RX at Lower Temperatures

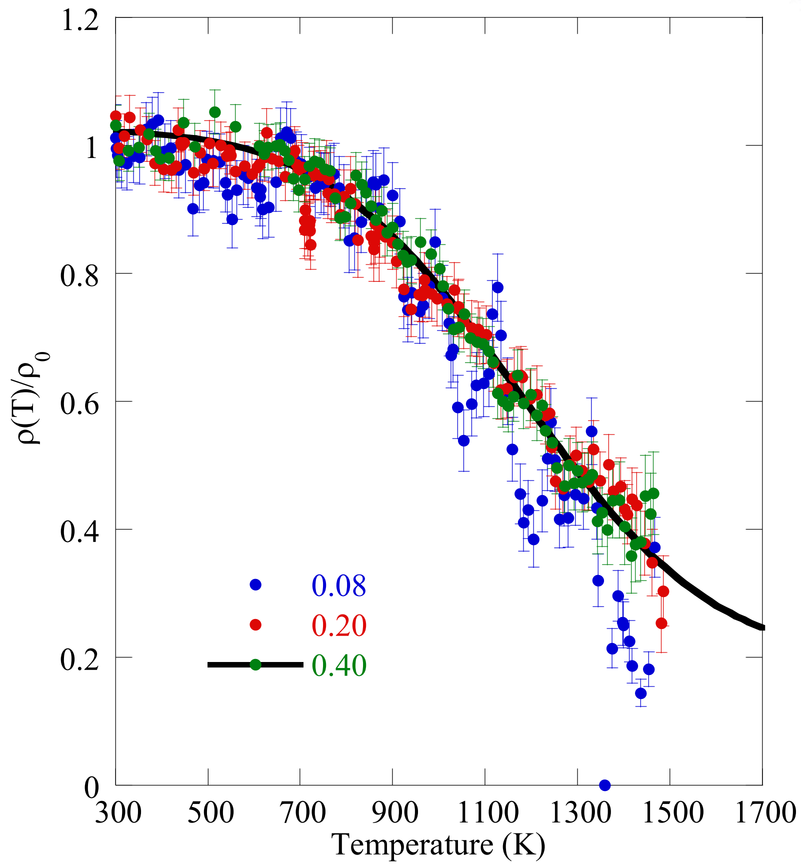




Conclusions

- Neutrons (and X-rays) provide a convenient, quantitative, non-contact probe of the microstructure.
    - Phase, texture, stress, dislocation density, solute chemistry.
- In careful measurements, can separate stresses by length scale.
- The increase in dislocation density during deformation is easy to observe, but difficult to quantify.
- Recovery and RX are observable as distinct events during heat treating
- Study element Ta as a step toward complex BCC alloys.
- This data can be used to develop predictive process/structure/property relationships and enable "materials by design".

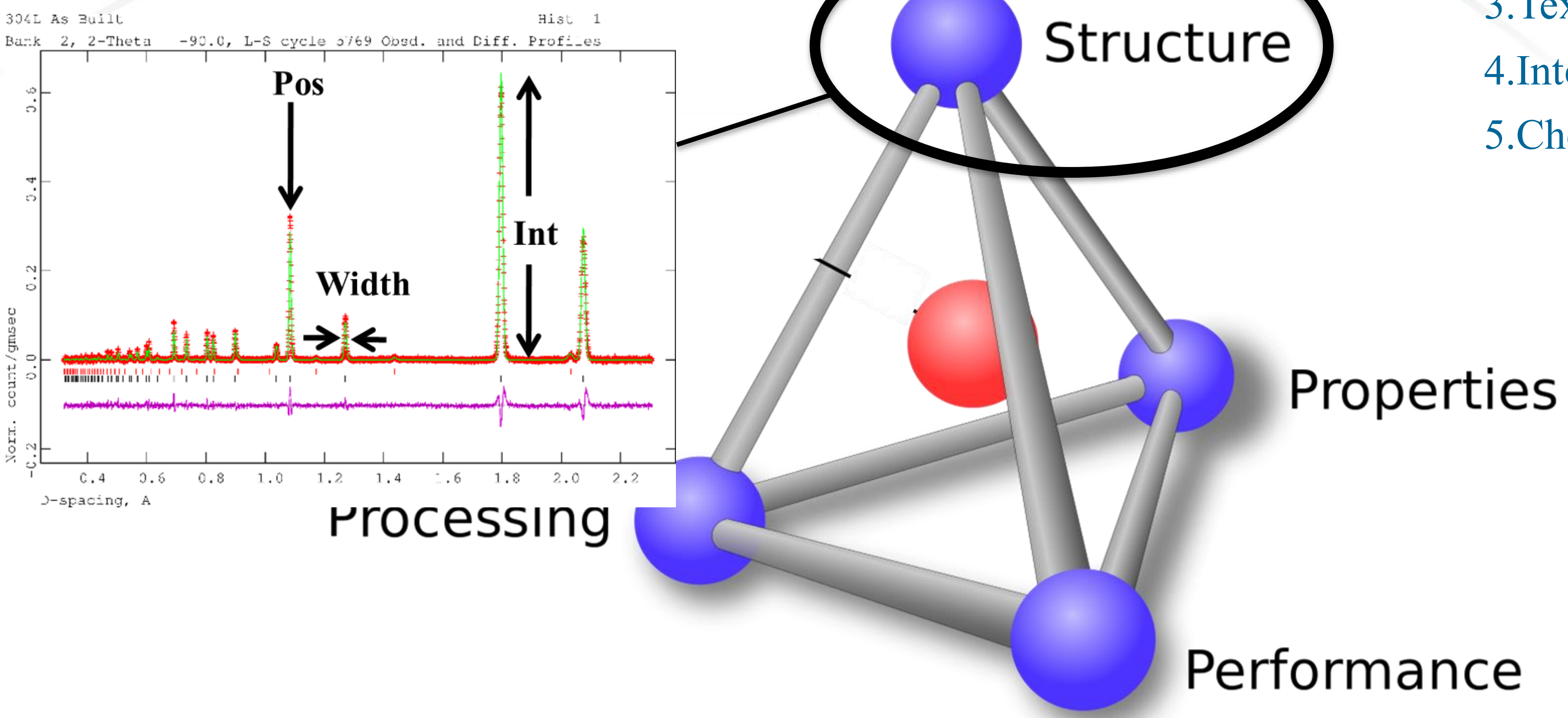
The Rate at Which Dislocations Decrease is Independent of the Initial Dislocation Density



The next steps are to develop the deformation and recovery models, and to get irradiated samples from Idaho National Laboratory

The Goal of the Materials Scattering Team is to Provide Experimental Support of Model Development Across the PSPP Relationship

High Energy X-ray and
Neutron Diffraction/Imaging



1. Phase
2. Defects
3. Texture
4. Internal Stress
5. Chemistry

- **Advanced models are critical for science-based qualification.**
  - **Models and data must bridge length and time scales.**
  - **Quantitative microstructural data is necessary.**