# Single crystal defect properties and tensile behavior of high purity Nb from Nb ingot slice

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DOE ILC, NSCL (Fermi Lab)

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# Overview

- Deformation in multicrystal is less uniform than in a polycrystal.
- Welding deformed crystals leads to recrystallization
- Heterogeneous deformation at grain boundaries → Rx nucleation
- Are starting conditions for cavity forming from all ingots similar/different?
  - Single crystal specimens
    - Tokyo Denkai set from perimeter → Rx study
    - Ningxia set from interior → slip system effects
      - orientation gradients from 0.5-5° / cm exist in this ingot
- Developing material models for crystal plasticity finite element constitutive relationships
  - Challenges and progress in simulating single crystal deformation

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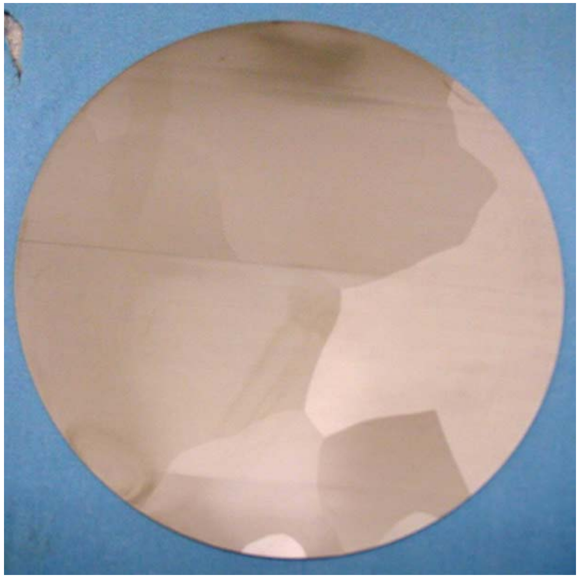
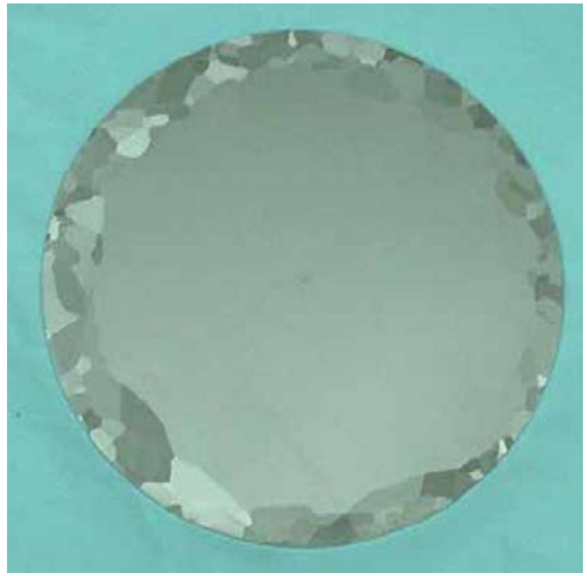
# Single/multi crystal cavities fabricated by welding two half cells – grain boundaries are visible






- Single cell multicrystal cavities made at MSU and J-Lab show effects of grain boundaries, irregular deformation
- MSU single cell cavity grain boundary ridge visible and easily felt with fingers, and cups have ears

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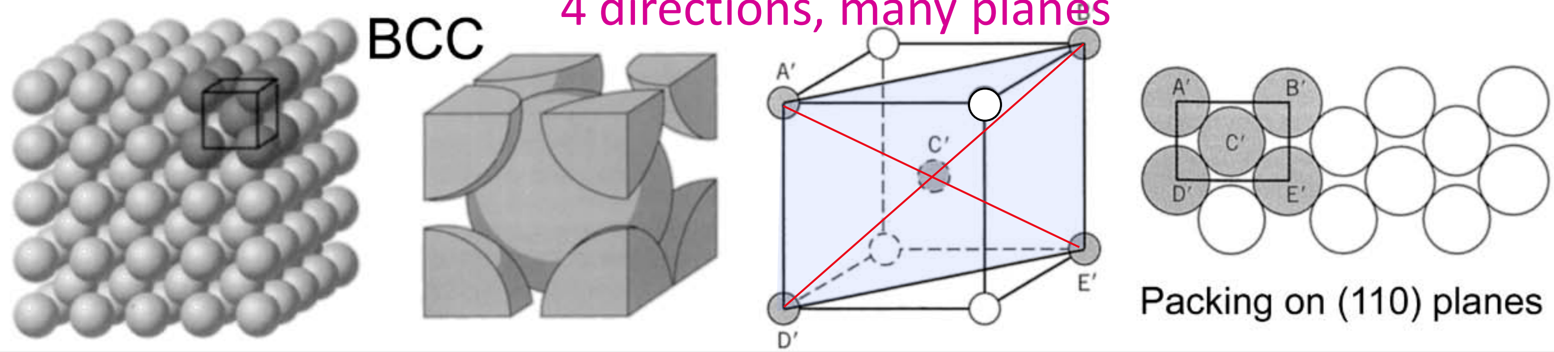
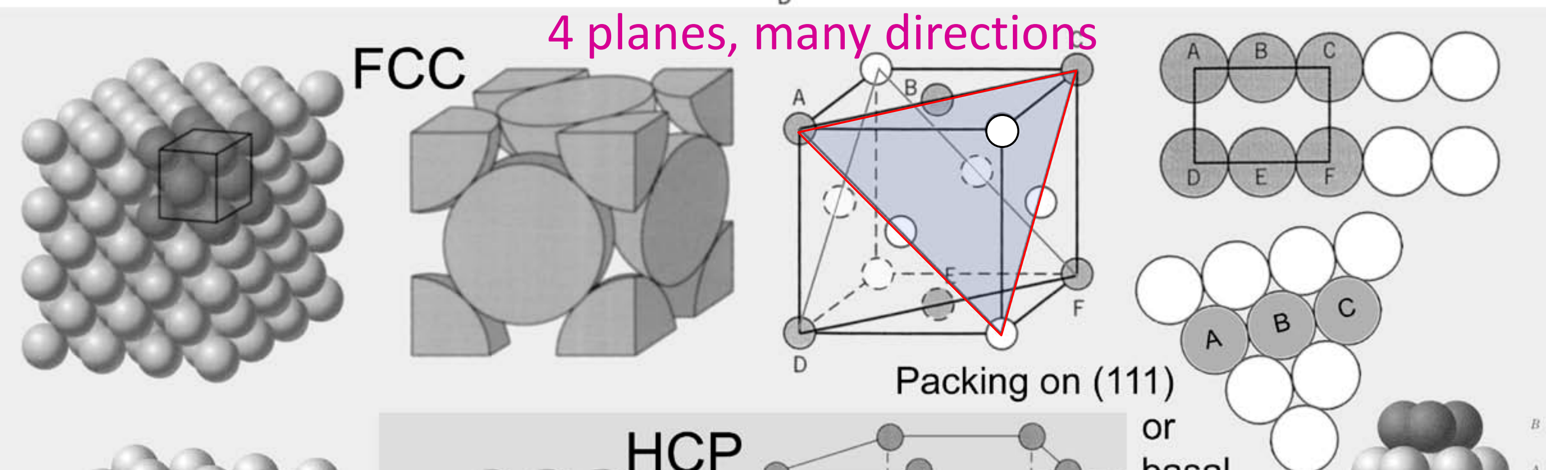
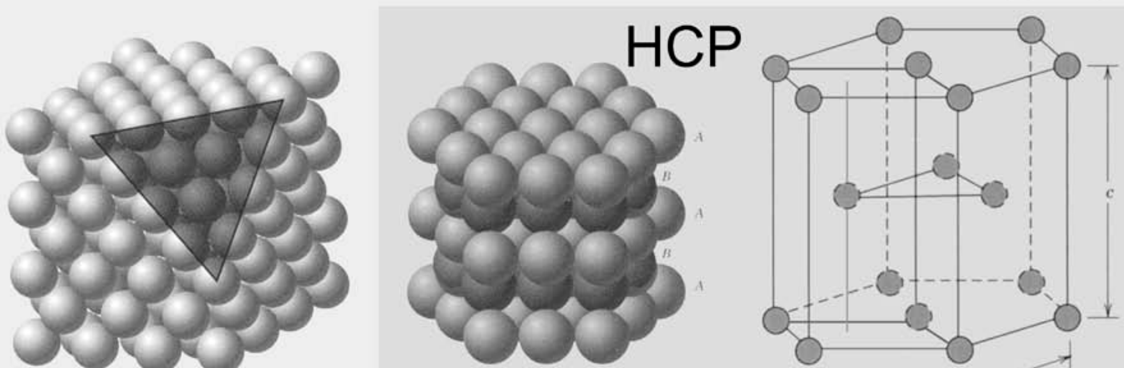
# Metal bending requires dislocation motion on slip systems
# Nb is a BCC metal like Fe; not FCC like Al or Cu, or HCP like Ti;
# Need geometry of crystal orientation, slip planes & directions



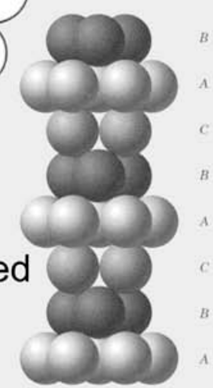

V, Cr, Fe, **Nb,** Mo, Ta, W, are BCC; densest plane is (110)




Al, Ni, Cu, Ag, Au, Pb, are FCC; close packed plane is (111)




or basal planes, HCP and FCC stack close-packed planes in a different order

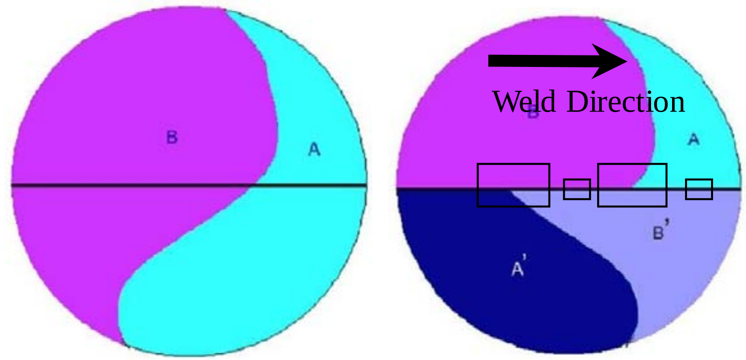


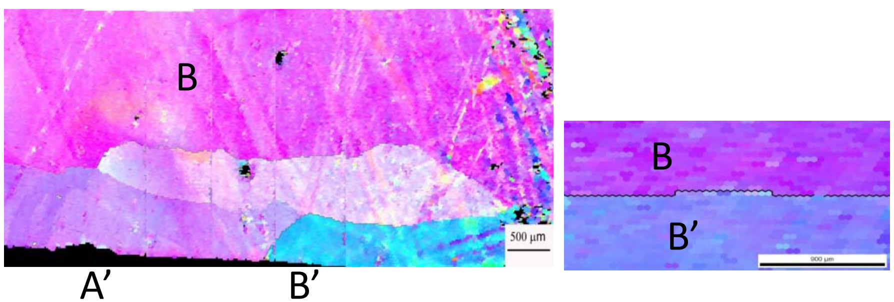
HCP basal plane is also close packed; Mg, Ti, Co, Zn, Zr

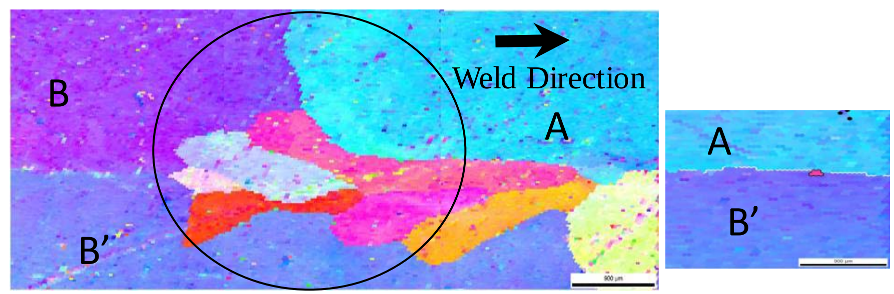
Adapted from Callister, Materials Science and Engineering, An Introduction

# Unstrained single crystals can be welded together gracefully (sometimes)

- Center piece of sliced ingot cut in two, flipped, welded,
- the weld was clean between two crystal orientations, but new orientations developed at triple junctions

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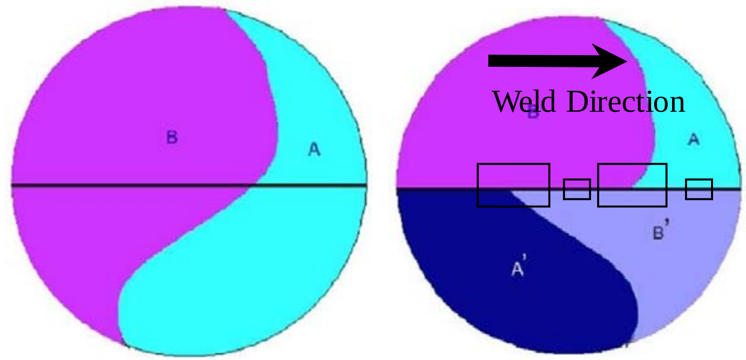


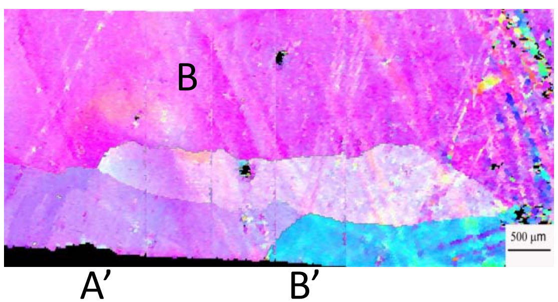


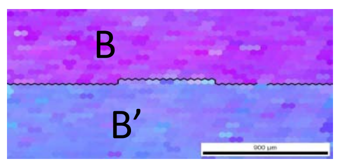


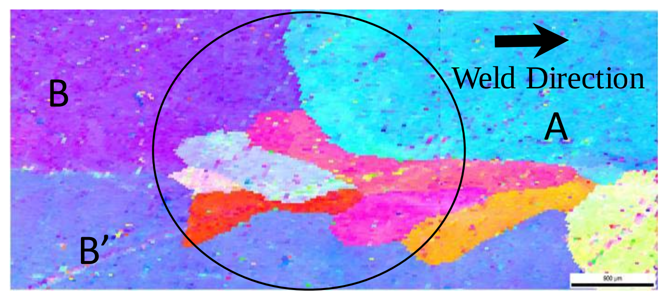


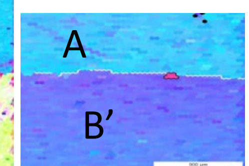


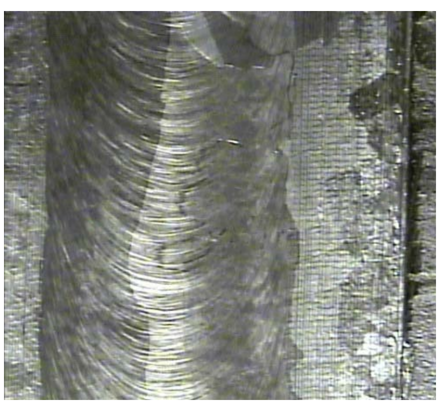

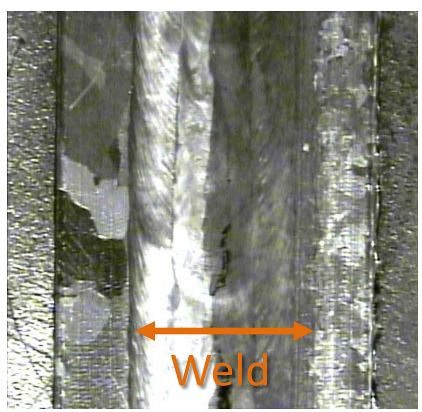


Rx in HAZ

1mm

**But deformation causes different recrystallization features on either side of equator weld of a finished large-grain cavity**

Baars et al. Transactions Applied Superconductivity 17, June 2007

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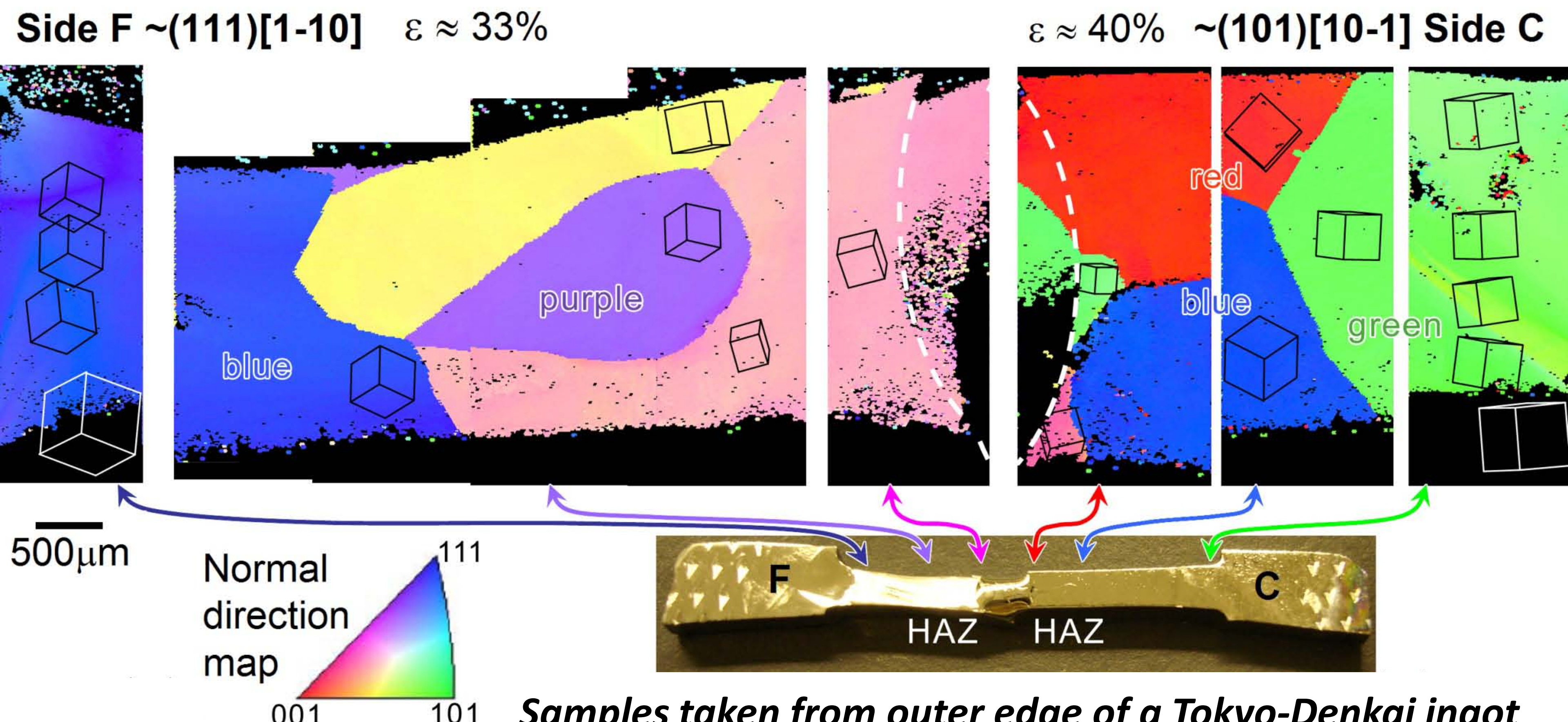
# Not surprisingly, welding plastically strained single crystals leads to recrystallization




***Samples taken from outer edge of a Tokyo-Denkai ingot***

- Two tensile deformed single crystals were welded together
- Parent orientations in grips have white prism orientation
- Black prisms show crystal orientations after weld

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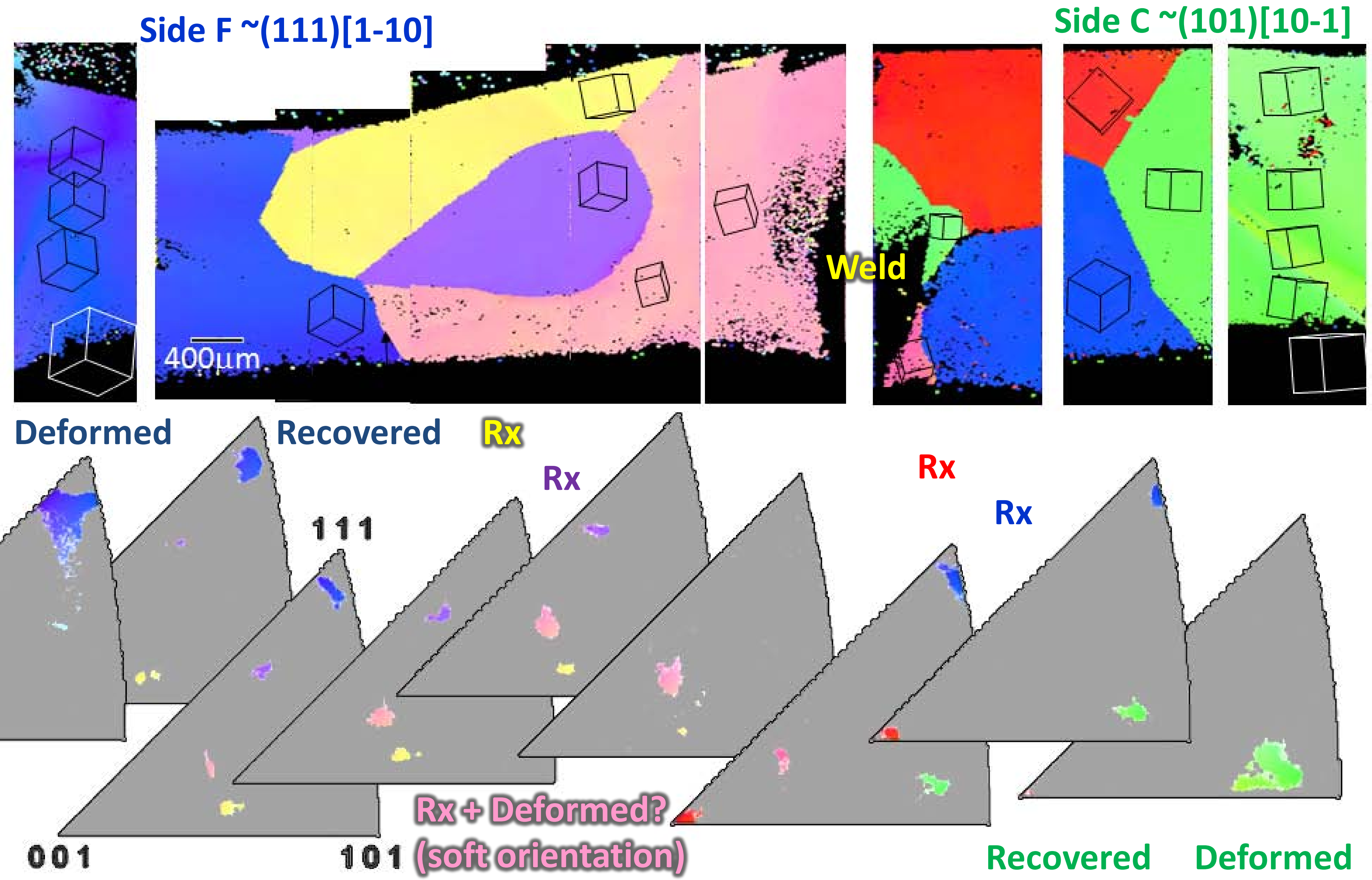
# Orientation gradients are maximized by deformation, minimized by Rx; Rx started in the HAZ and grew into weld



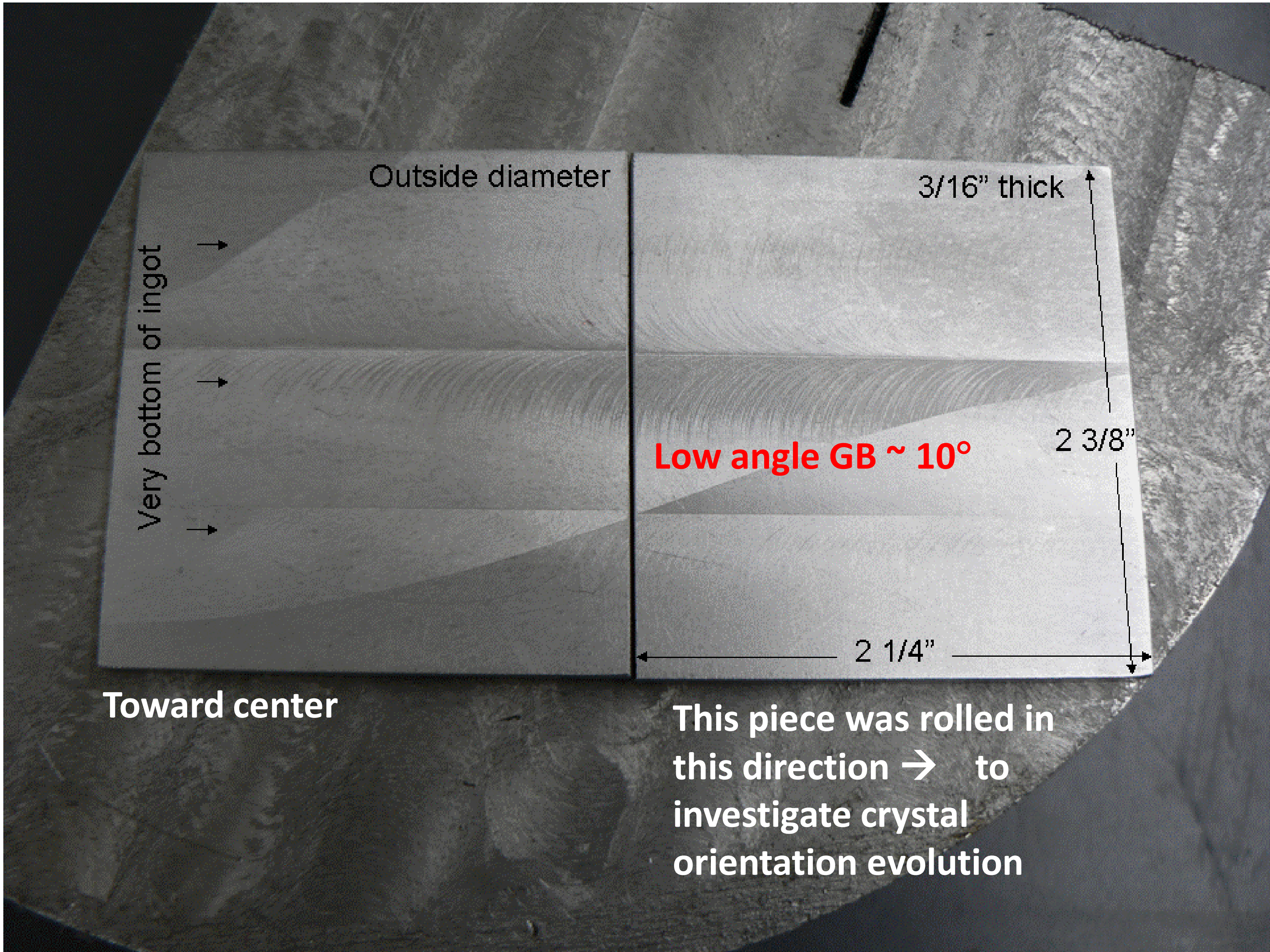
# Characterization of Nb Ingots, and effect of rolling

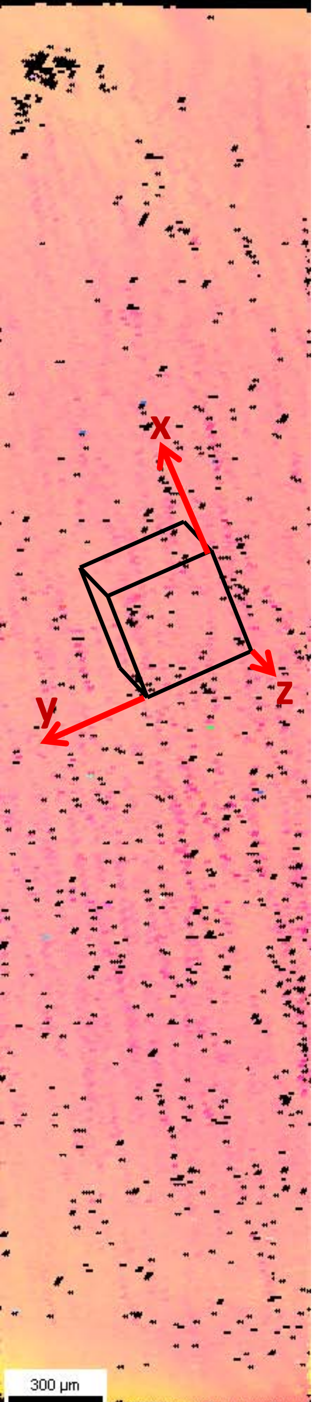
## Can any advantage be gained by rolling pieces of an ingot to maintain an optimal crystal orientation?

Ingot purchased by *Niowave*, sectioned to show longitudinal plane

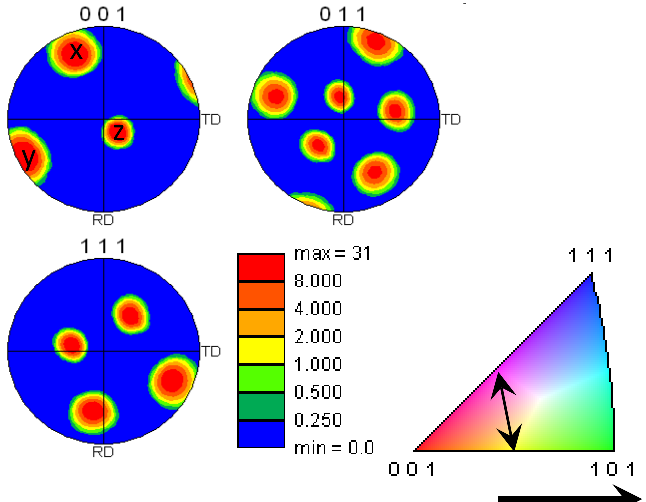
- 3 grains at base, grain at corner overtaken, center grain grows preferentially;

**Is this typical?**

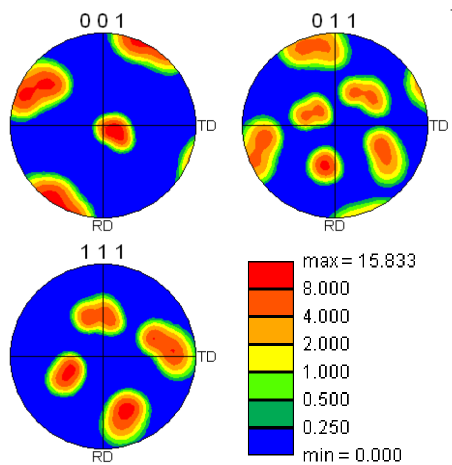




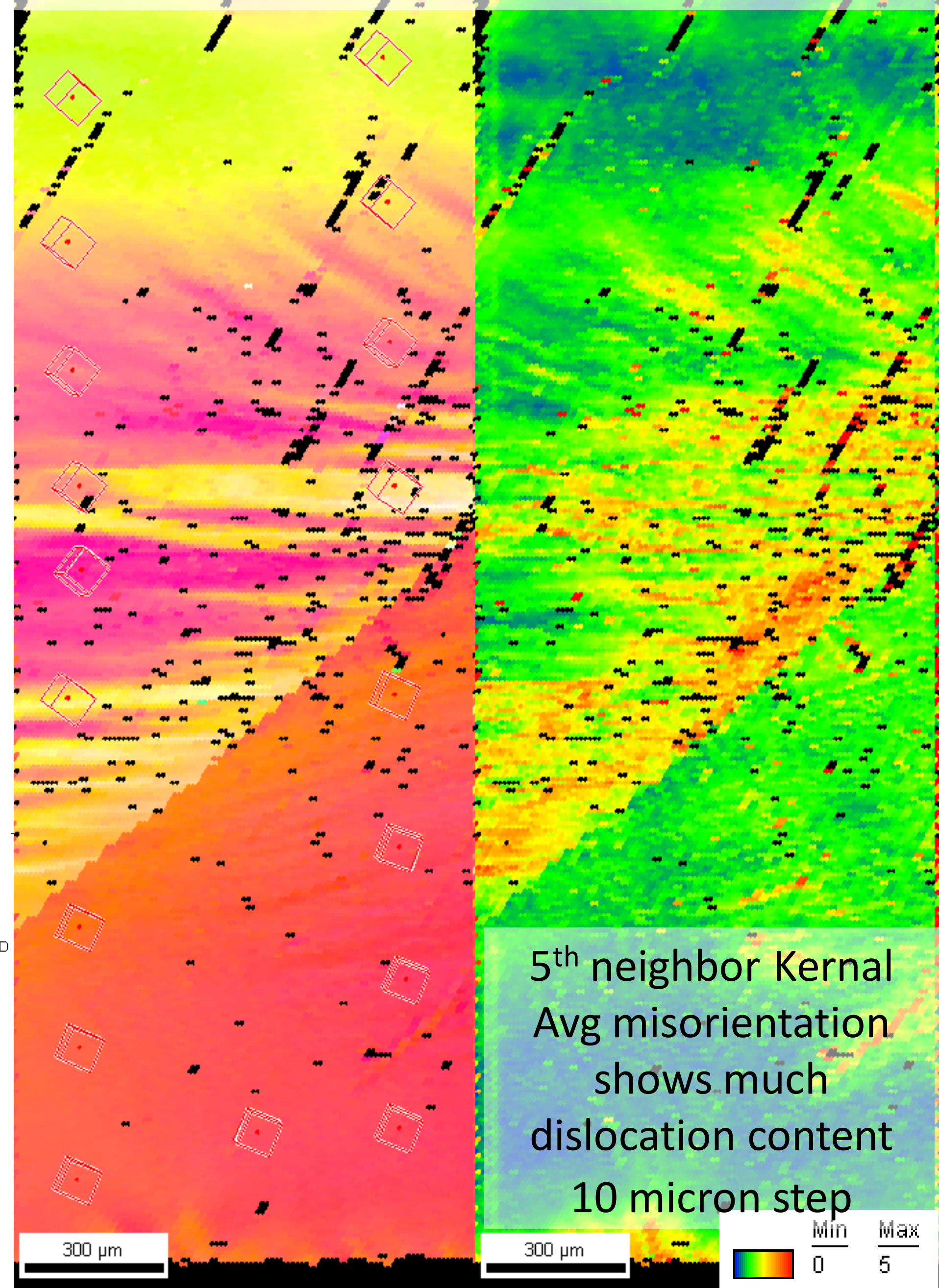

OIM scans show full thickness, with ingot growth (and rolling) direction out of the page.



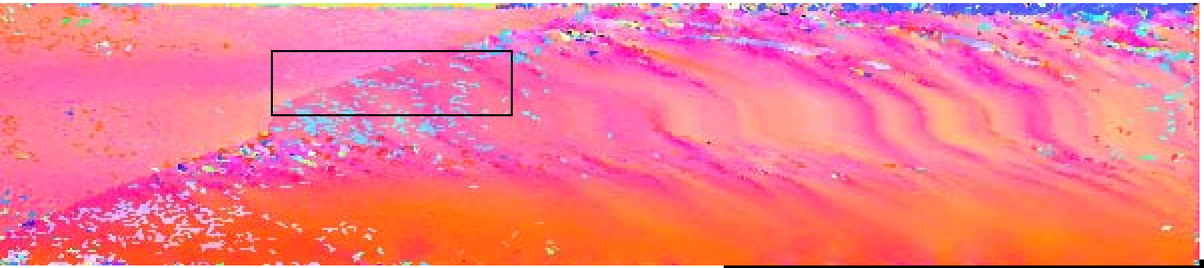

50% rolling, with recovery HT → initial orientation rotated, Orientation oscillates ~ 10°



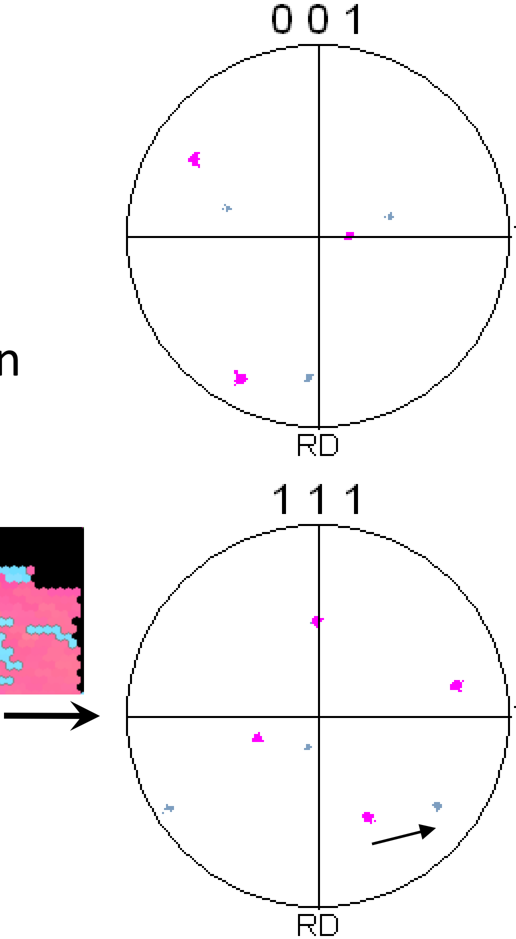

**Rolled 50% reduction**, recovery heat treatment (ramp to 650°C in 120 min and hold 60min)

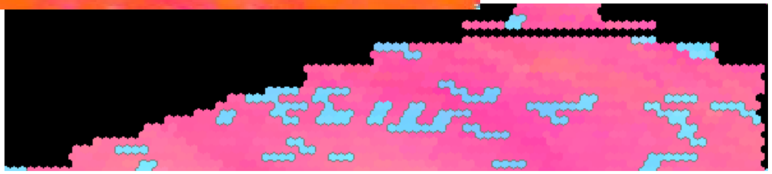


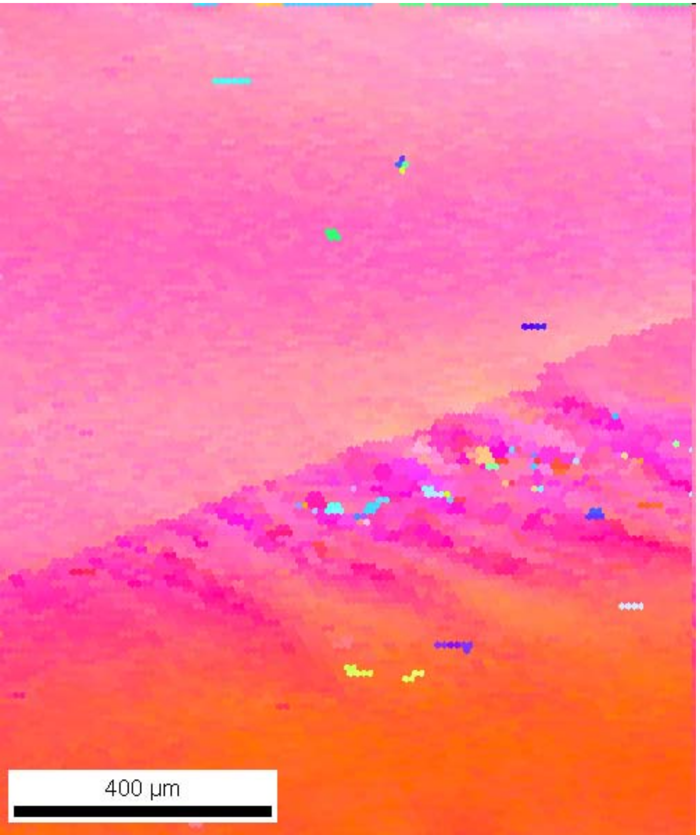
**As-rolled to 71%** near top of sheet shows development of measurable new orientations (blue) in right grain, ***only due to strain***



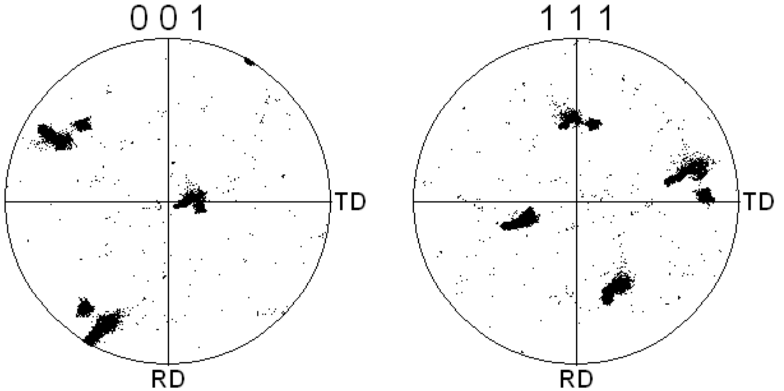
30 deg rotation about <111>






As-rolled to 70% reduction near bottom of sheet – shows lesser development of new orientations in right grain



# So, how do various ingots compare? Will similar deformation from the same starting point be comparable? *

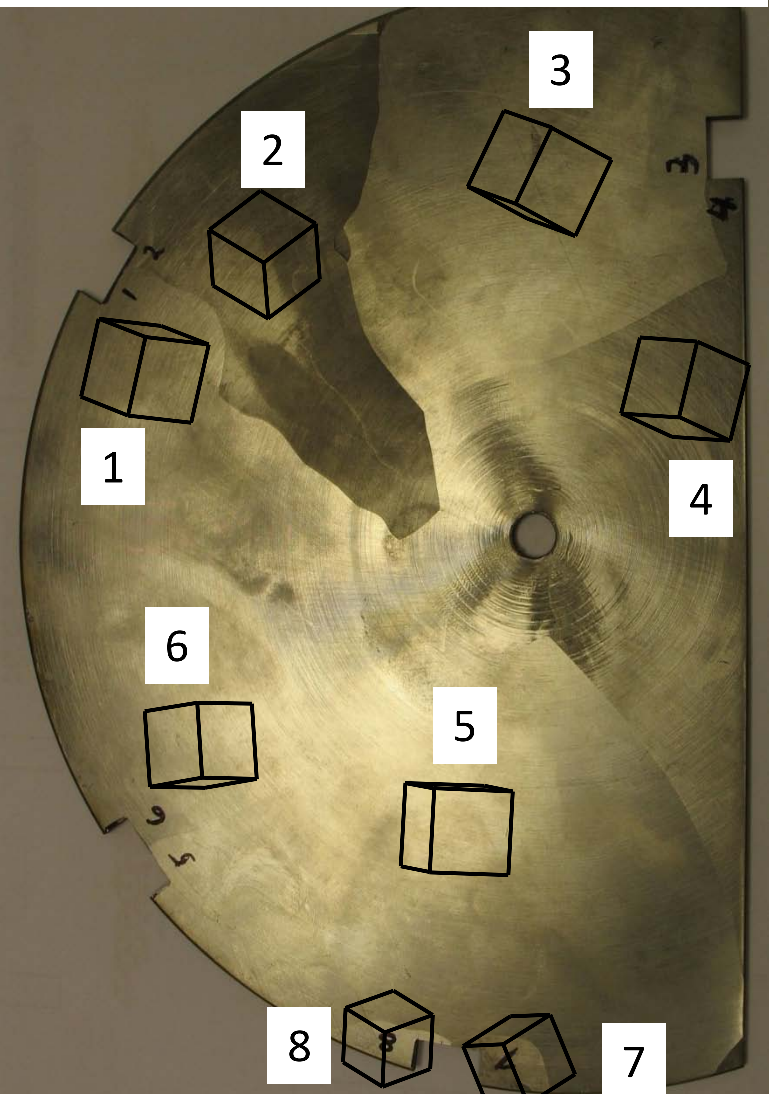
- Three ingots examined to identify similarities and differences,
  - CBMM (via J-Lab)
  - Ningxia (via NSCL)
  - Heraeus (via DESY)
- CBMM done with OIM – taking out small specimens at grain boundaries on perimeter
- Ningxia done with removing center piece, which had most of the grain boundaries, and using OIM
- Heraeus done non-destructively using Laue camera – see poster

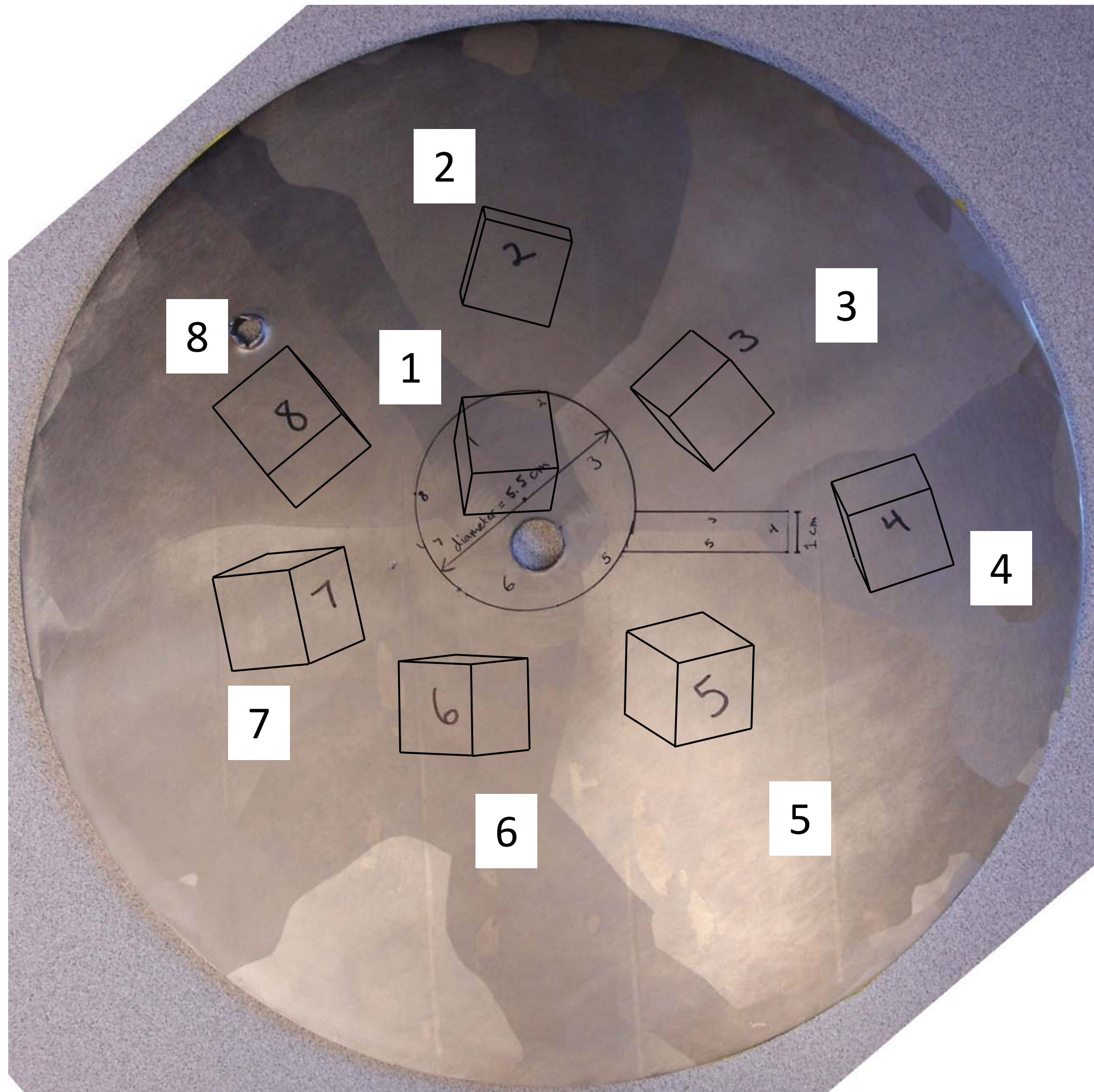
* Perhaps not…

# Slice from CBMM (via J-Lab)

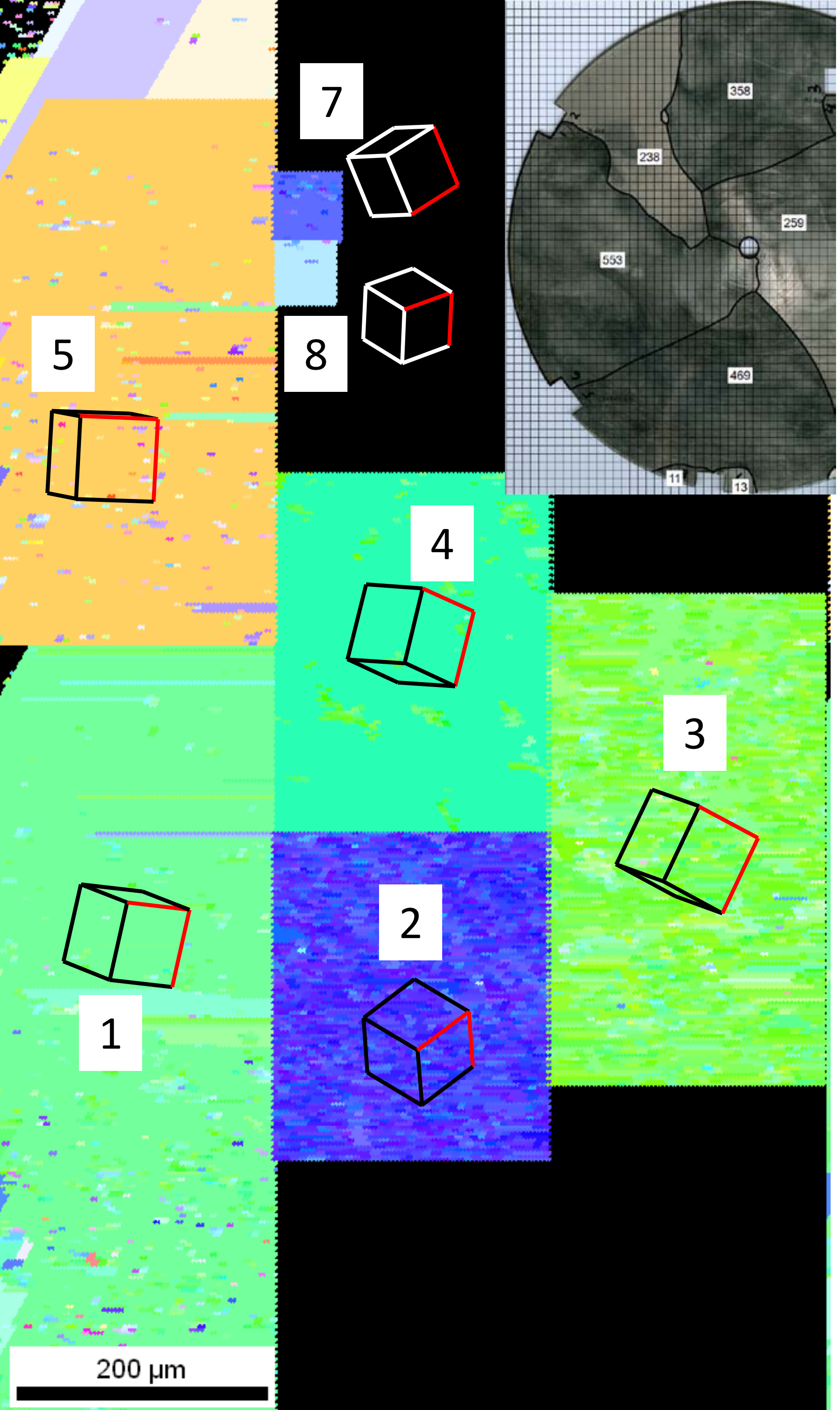
# Slice from Ningxia (via NSCL)

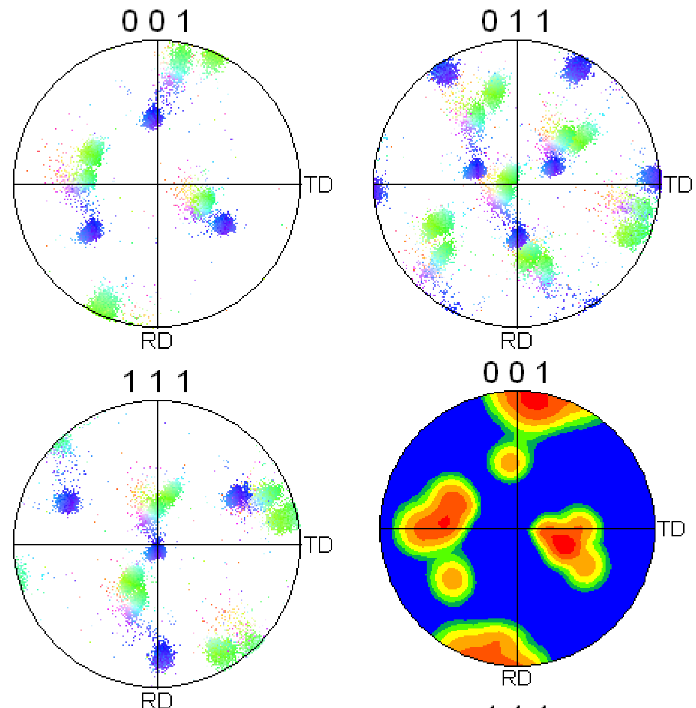
Grains 6 and 1 are the same grain, but there is significant lattice rotation across grain



CBMM ingot plate justified grain orientation data -relative grain areas were calculated, cropped from original files, and merged together in TSL with the proper neighbors (approximately)

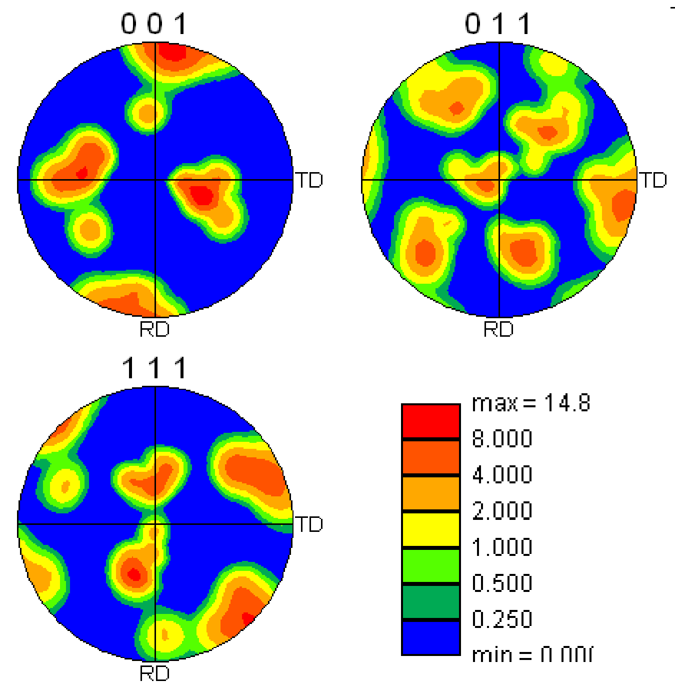



Grain orientations are spread out

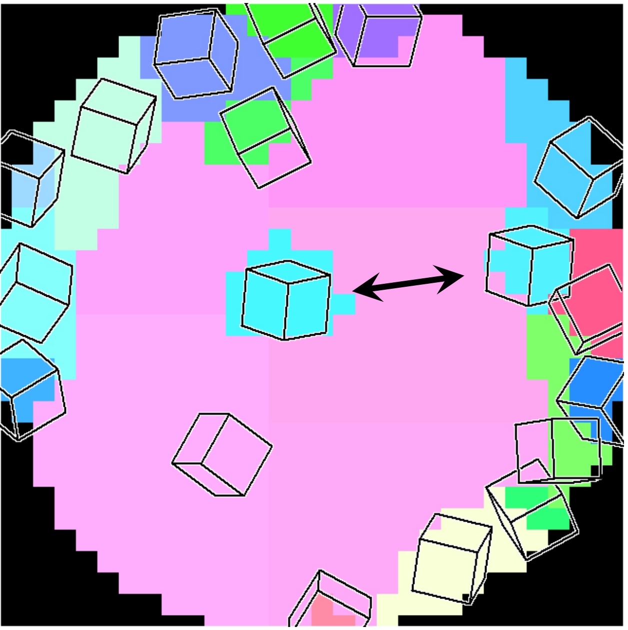
Discrete PFs

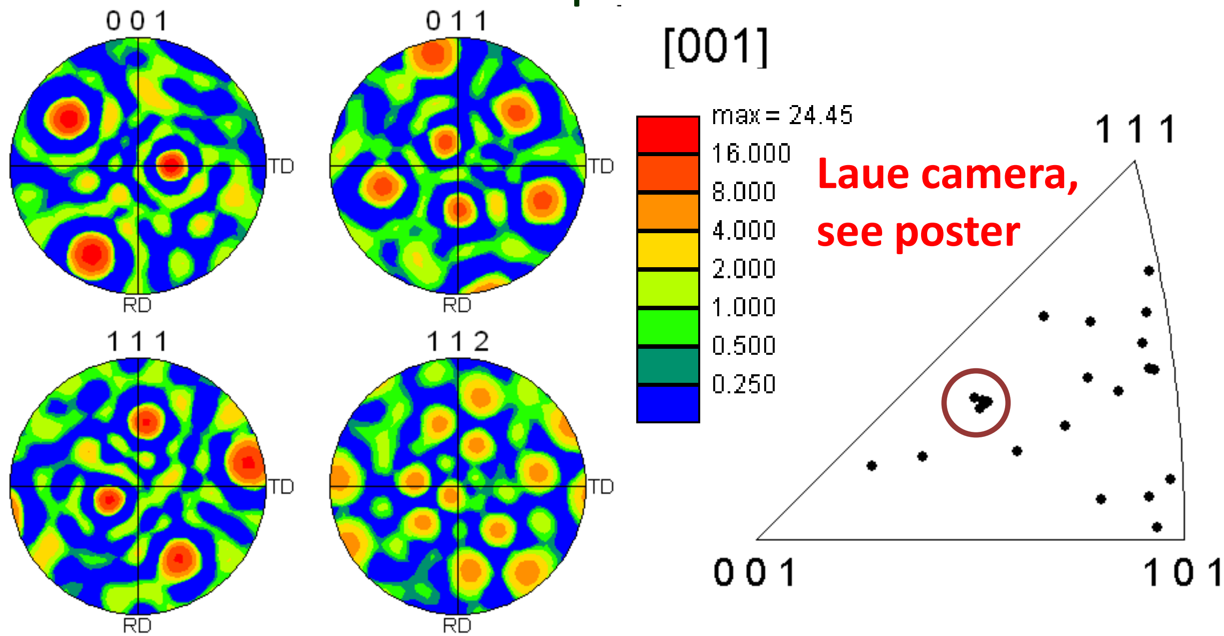
Density PFs

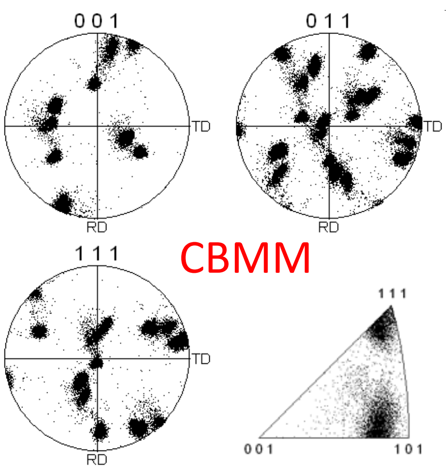


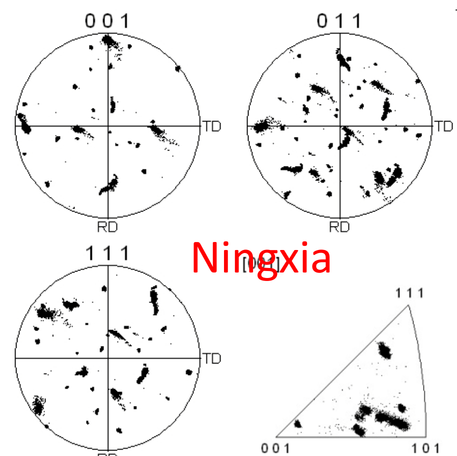
# Heraeus slice has large interior grain with an island (same orientation as a perimeter grain)

- Tendency for random orientations with respect to the dominant one






- Every ingot is different… Some have more spread than others, but may be an artifact of surface preparation.
- Are orientation gradients real?

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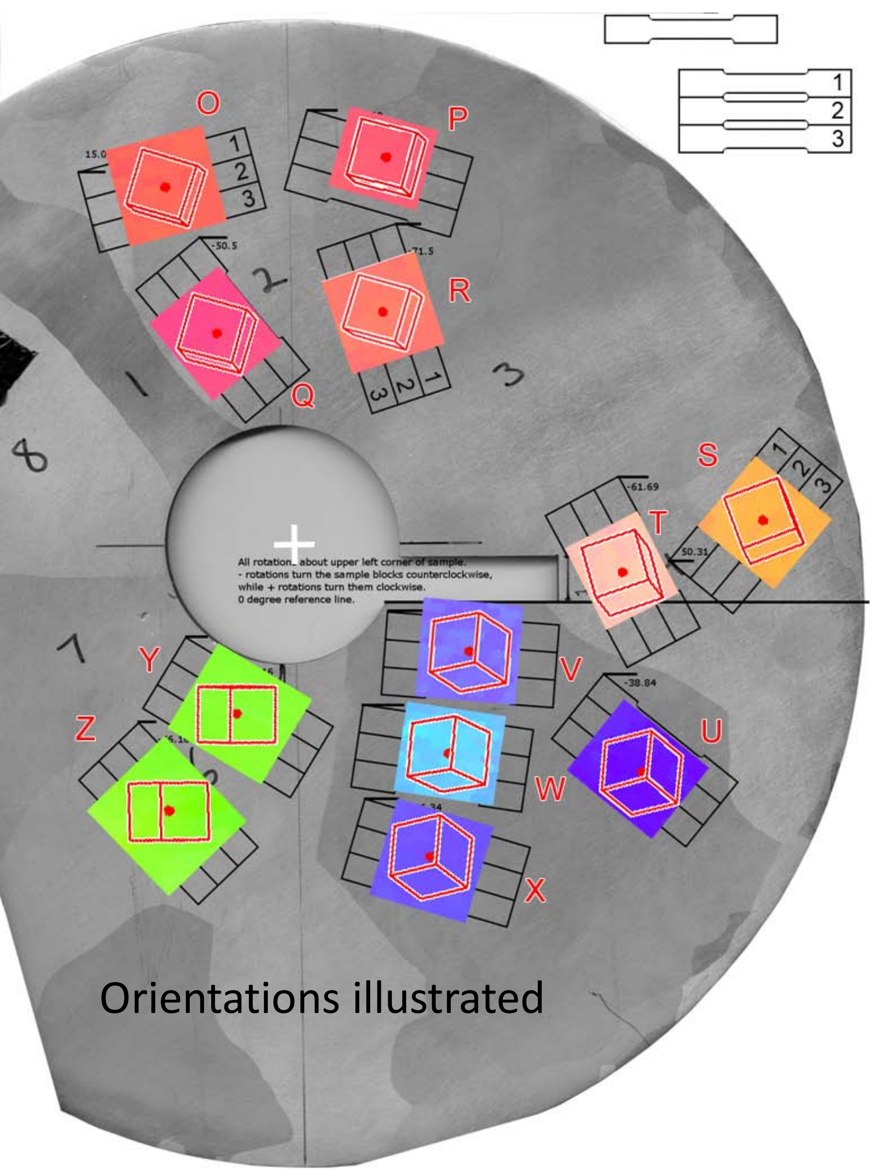
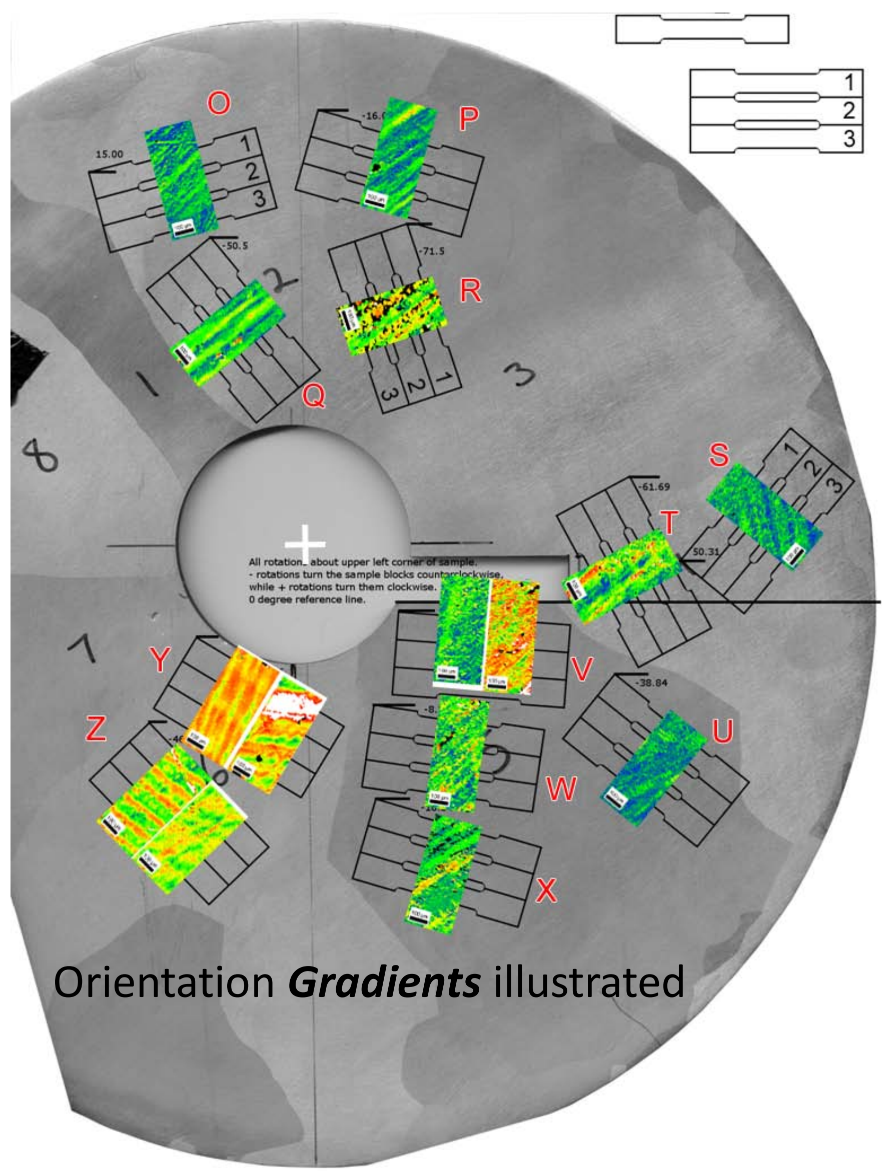
# How does initial orientation affect deformation?

Orientations strategically chosen to examine slip system activation, interactions

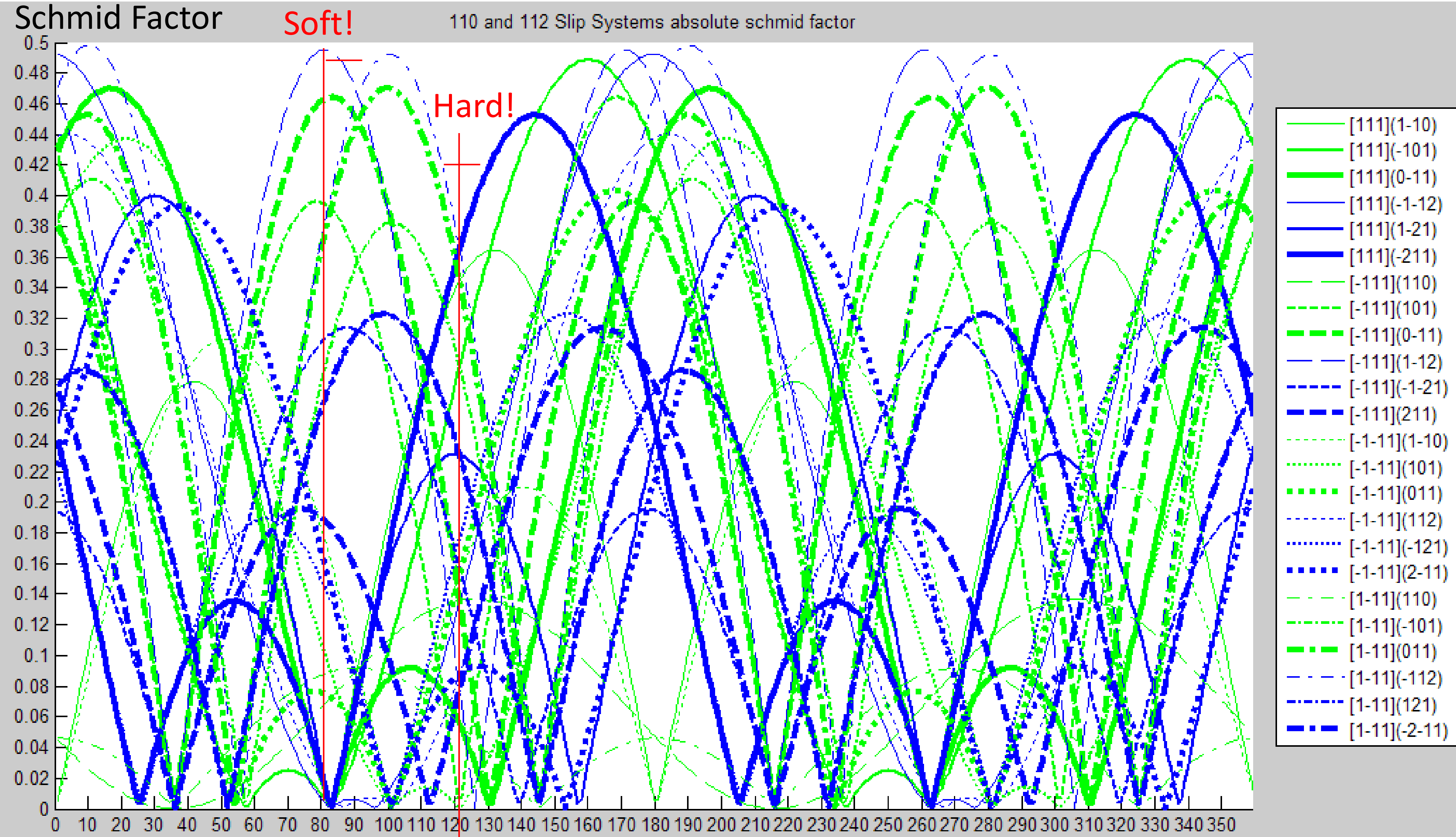


Orientations illustrated



Orientation ***Gradients*** illustrated

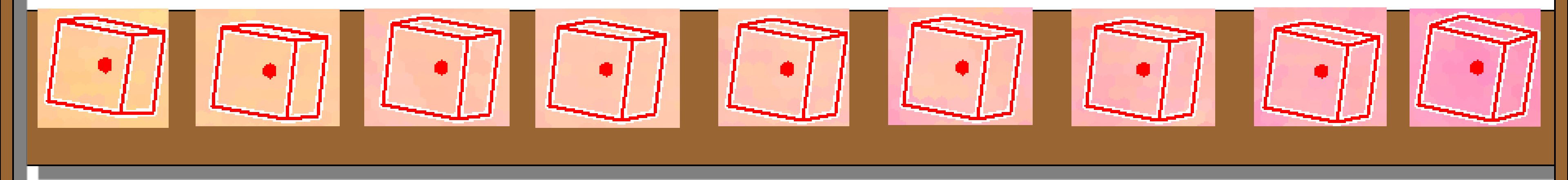
Grain 3 with Euler angles 319° 39° 83°, then consider what slip systems are highly stressed (Schmid factor) with every orientation in the plane of the slice, and choose... orientation gradients of 10° can lead to differences in flow stress of 20%, or alter the operating slip system(s)



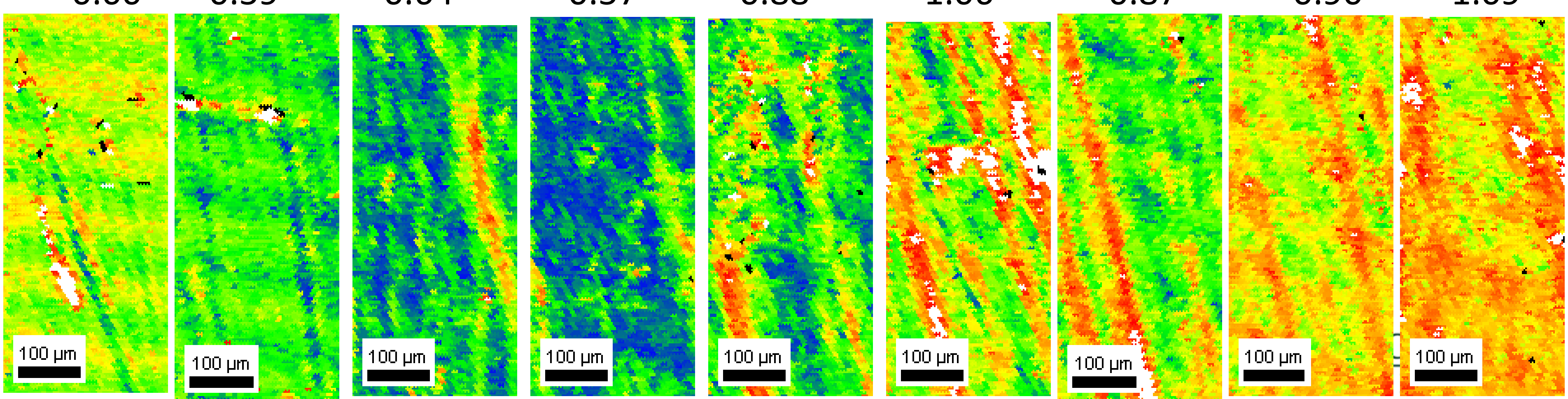
# Ningxia slice used for single crystal tensile experiments, (will be used for Rx too)

- Actual deformation may vary from prediction because orientation gradients vary from 1-10° / cm… does not appear to be systematic (via various methods of searching for patterns)

**T1**



| | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| $\varphi_1$ | **6.6** | 8.7 | 10.7 | 10.6 | 12.4 | 14.1 | 13.3 | 14.1 | **14.6** |
| $\Phi$ | 23.0 | 23.4 | 24.0 | 24.3 | 24.3 | 24.4 | 23.9 | 23.8 | 23.6 |
| $\varphi_2$ | **343.7** | 341.8 | 339.7 | 339.6 | 337.8 | 335.5 | 336.4 | 335.1 | **334.4** |
| | 0.94 | 0.83 | 1.06 | 0.81 | 1.31 | 1.74 | 1.33 | 1.12 | 1.36 |
| | 0.00 | 0.59 | 0.64 | 0.57 | 0.88 | 1.06 | 0.87 | 0.90 | 1.09 |



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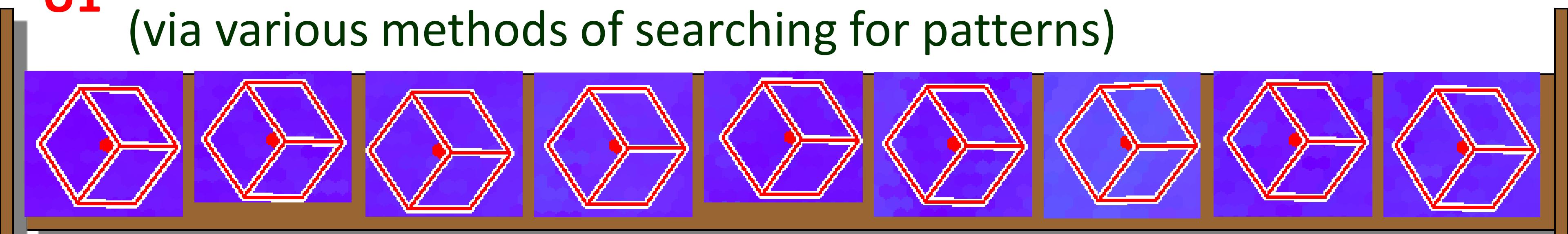
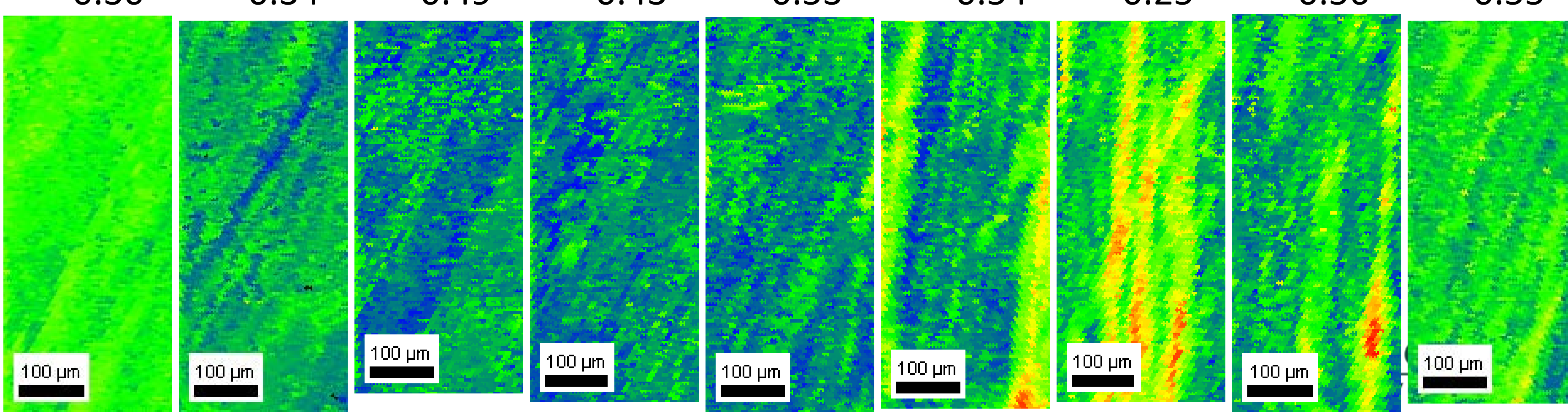
**U1**

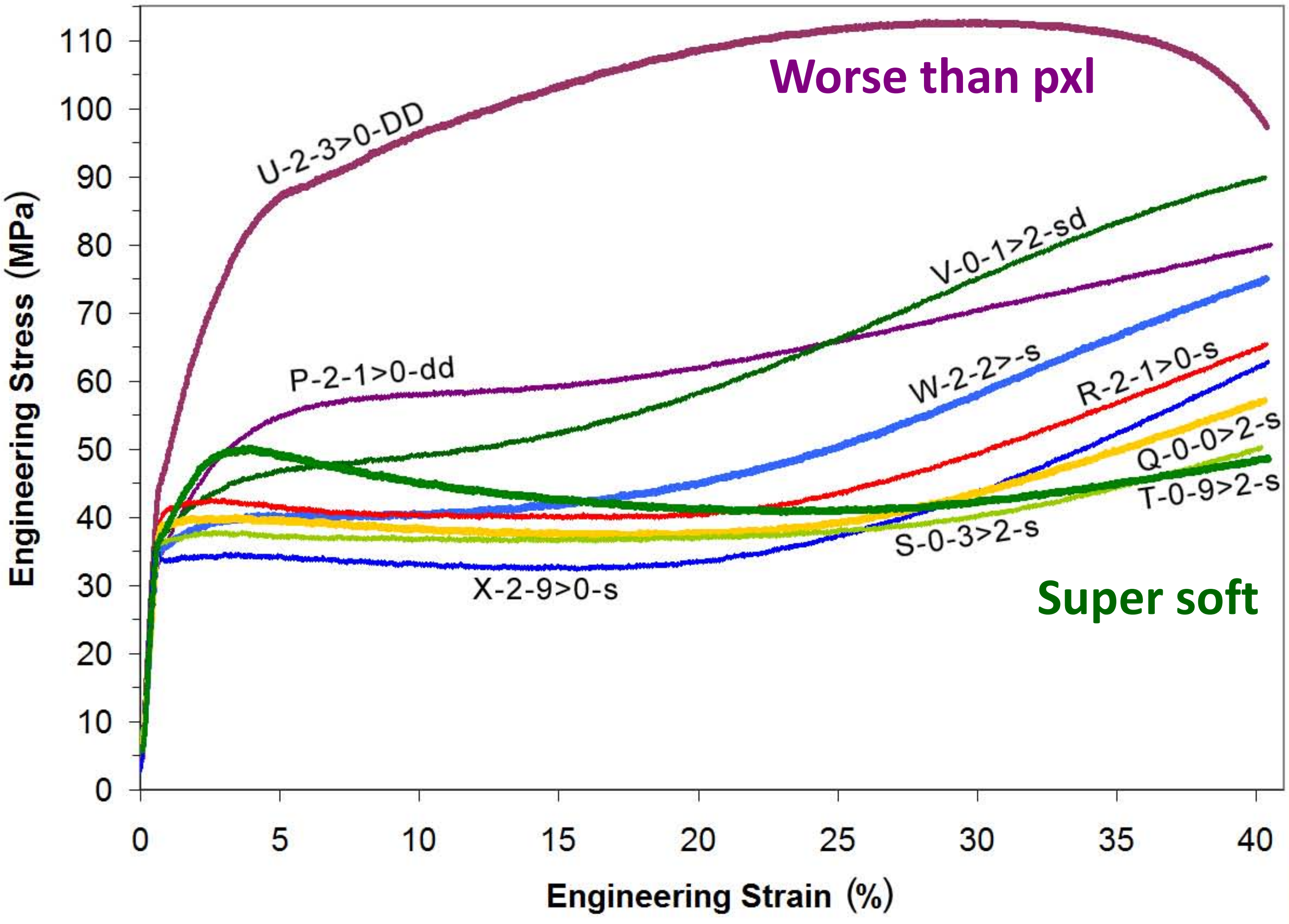


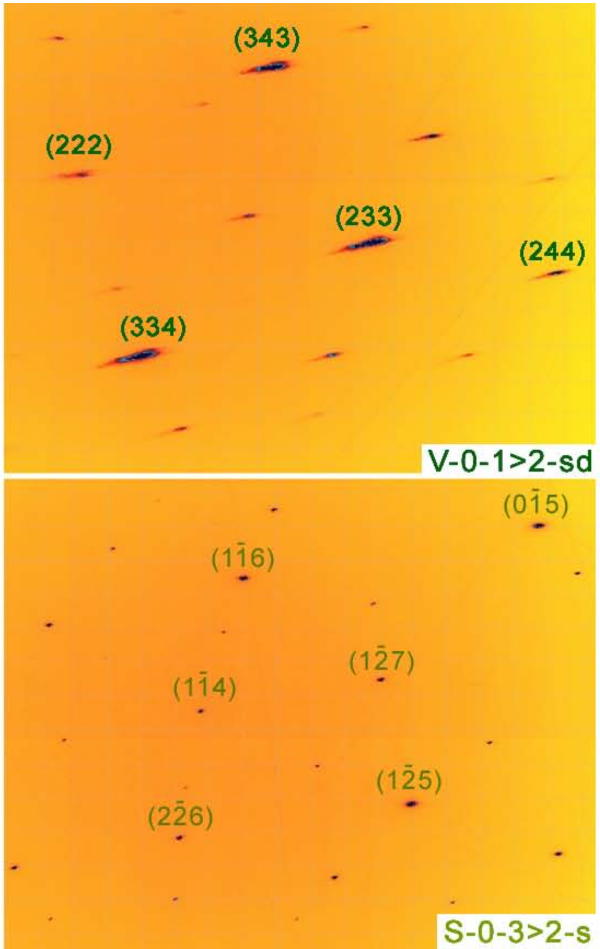
| | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| $\varphi_1$ | **232.9** | 233.8 | 233.8 | 233.6 | 233.8 | 233.5 | 233.4 | 233.4 | **233.3** |
| $\Phi$ | 59.4 | 59.3 | 58.8 | 58.7 | 58.6 | 58.2 | 57.6 | 58.3 | 58.5 |
| $\varphi_2$ | **144. 7** | 144.5 | 144.6 | 144.5 | 144.2 | 144.7 | 144.8 | 144.4 | **144.0** |
| | 0.44 | 0.44 | 0.60 | 0.55 | 0.67 | 0.95 | 1.13 | 0.91 | 0.74 |
| | 0.30 | 0.34 | 0.49 | 0.43 | 0.53 | 0.54 | 0.25 | 0.56 | 0.55 |



# As deformation depends on initial orientation how does initial orientation affect deformation? **A lot**

- Diffraction patterns are typically streaked, indicating large deformation and orientation gradients that affect nucleation of recrystallization → need to change direction of working of ingot

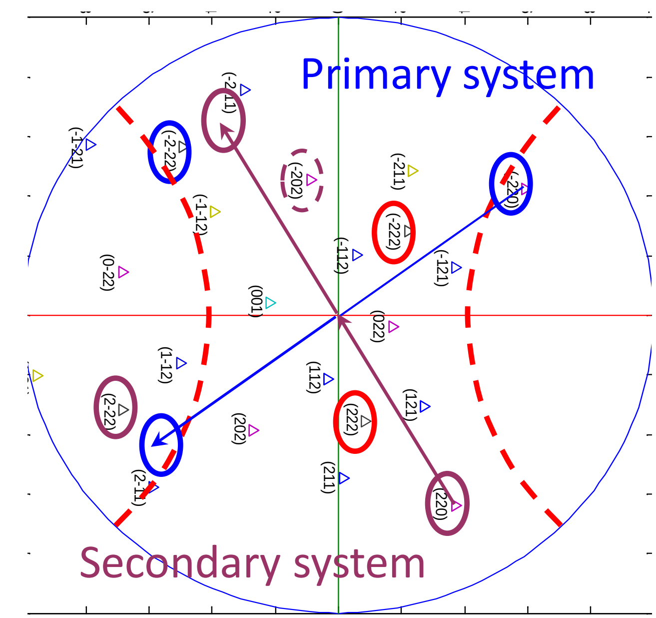


# A crystal oriented for single slip rotates until a different slip system becomes more favorable

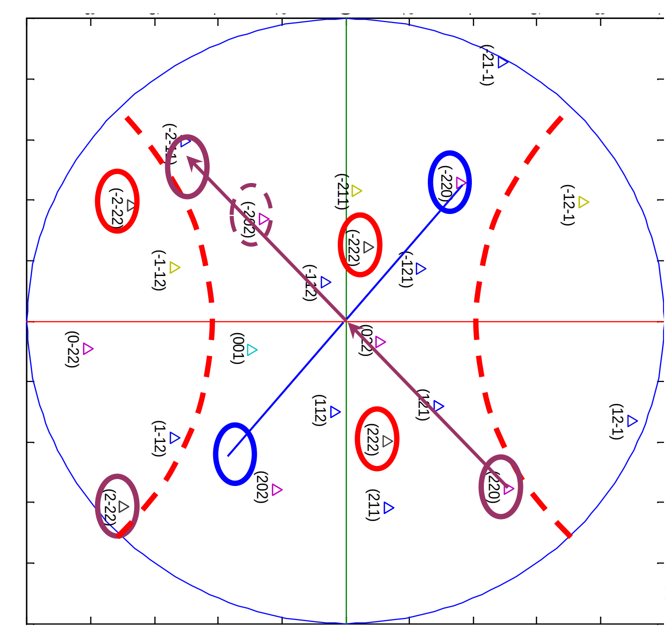
- Slip directions (circled) on 45° small circle (dashed red arc) are favored
- When rotation makes secondary slip system favorable, leftover dislocations on primary slip system block secondary dislocations, cause hardening

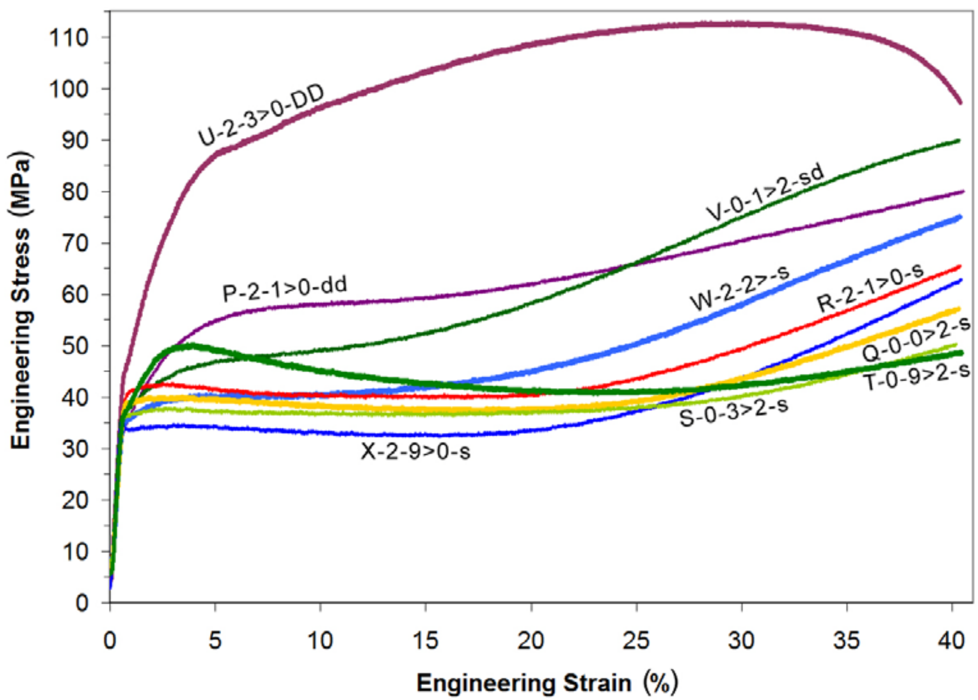
Initial T2 346.1 25.5 2.1



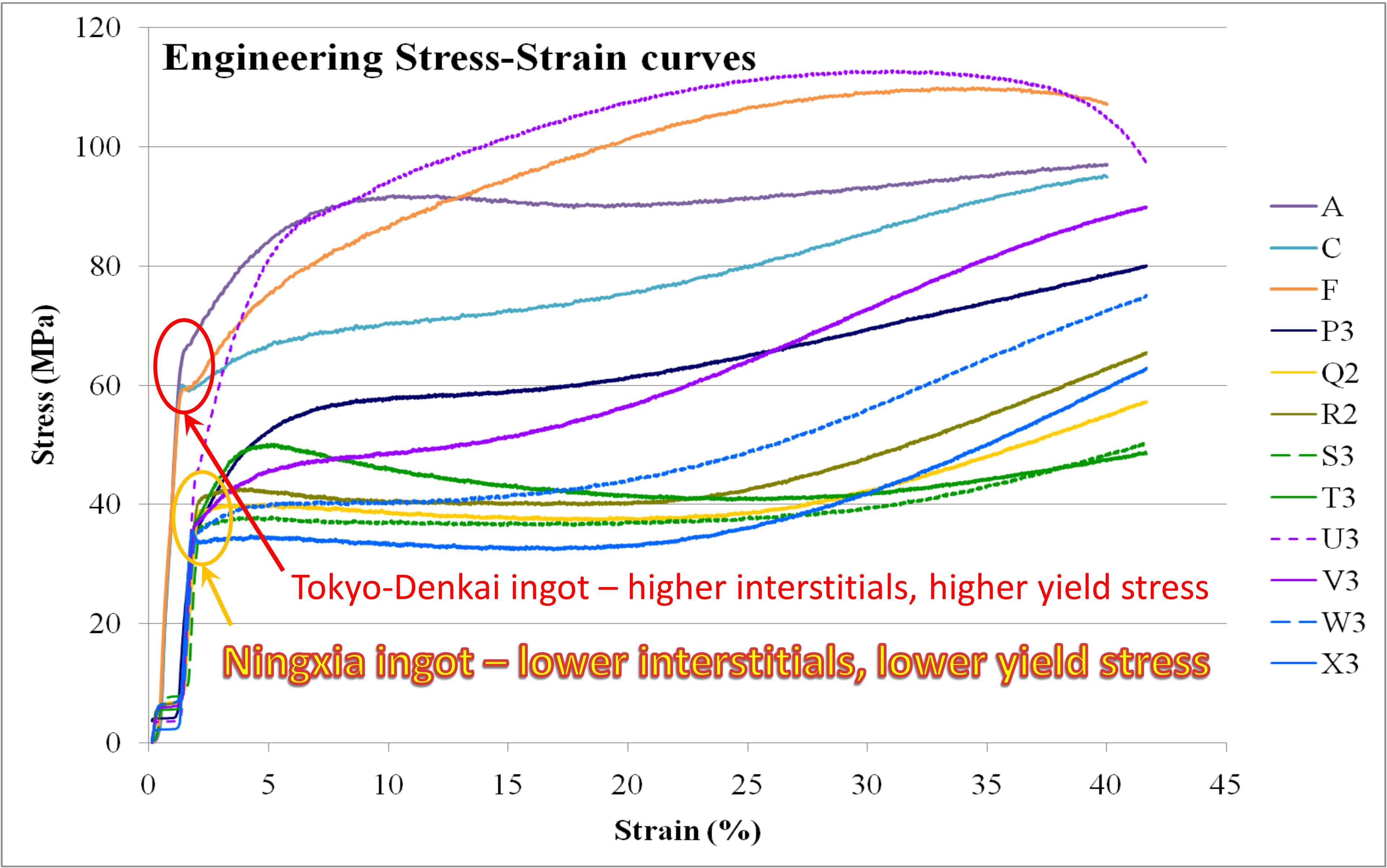

Tensile Axis

Deformed T3 17.9 34.5 344.1





# Compare/contrast single crystal tensile specimens from two different ingots (Tokyo Denkai, Ningxia)

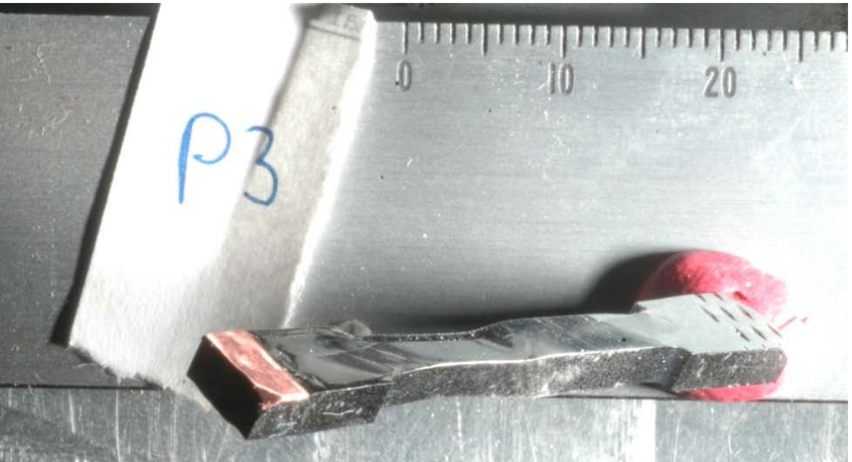


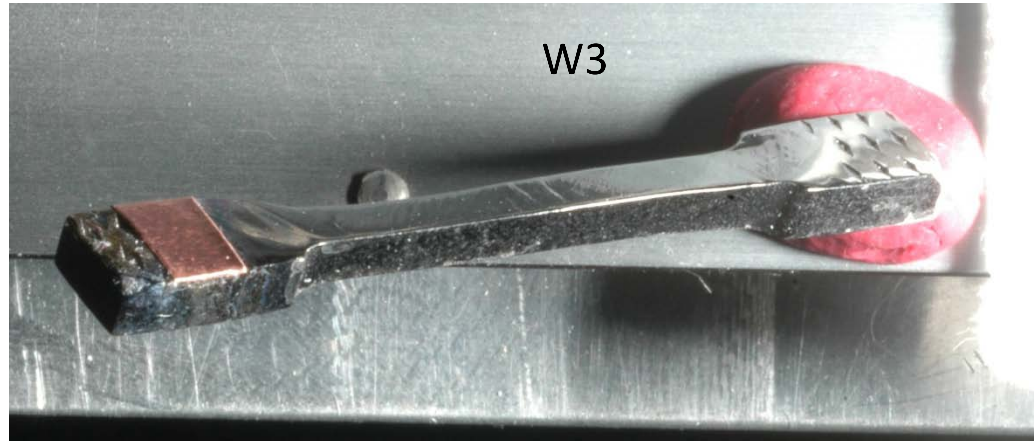
# Future activities:

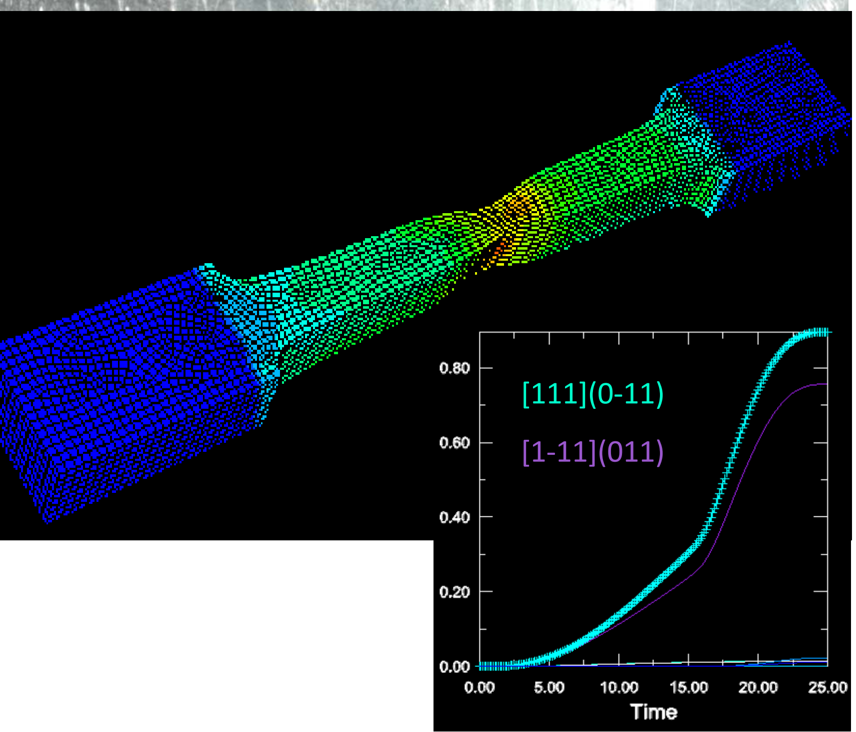
- **Analysis** of tensile test specimens is ongoing…
  - Seeking relationships between deformation, Rx, and Recovery
  - Slip is apparently slightly easier on {112} than {110} planes, and this differs from ultra high purity or RG Nb
- **Modeling**… using crystal plasticity finite element models to force deformation to follow slip systems that operate in metals, which can account for the obesrved non-uniform deformation (see following examples)
  - Shape changes are reasonably effectively predicted, stress-strain history is more difficult to model
  - Leaning toward implementing a dislocation velocity based approach, which may help account for the different modes of hardening with crystal orientation
  - Effect of orientation gradients is not yet explored

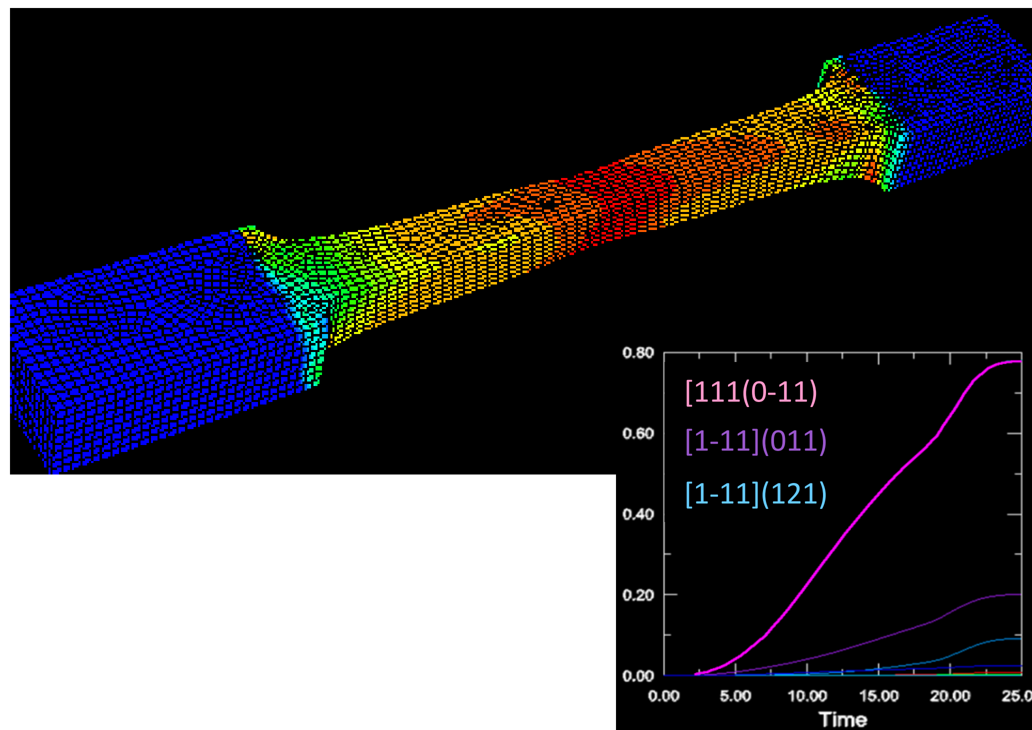
This slide is intentionally blank, because I removed the equations for different hardening models and solving strategies

# Simulations of shape changes are decent…

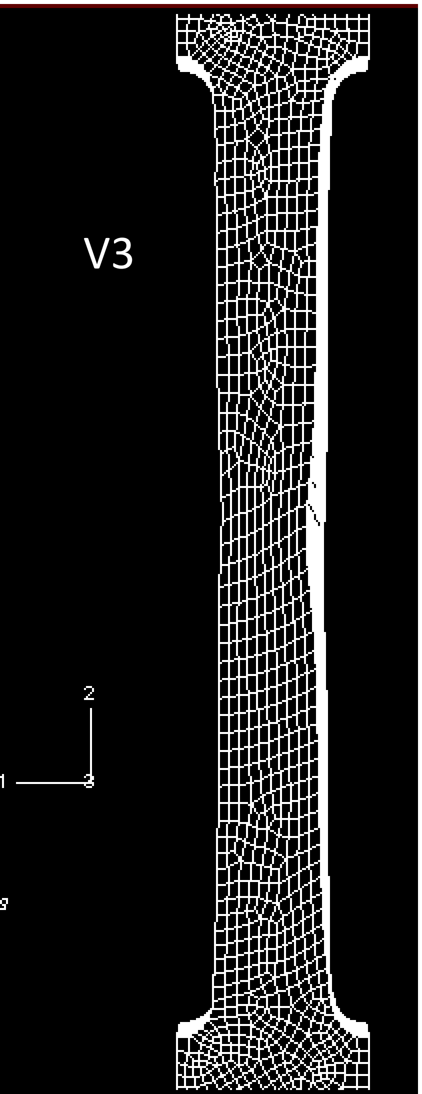


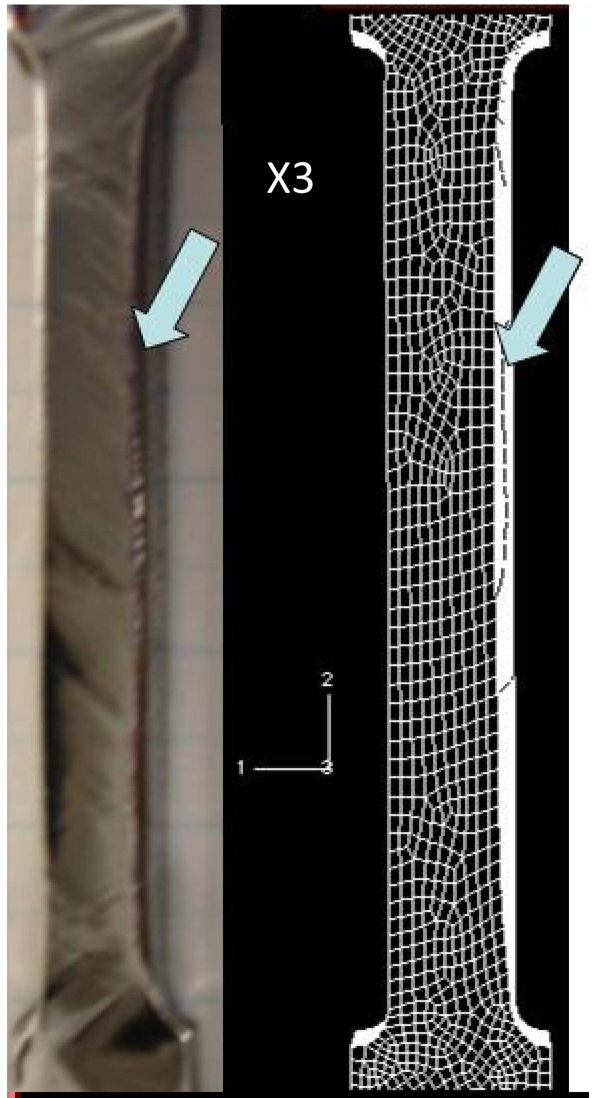



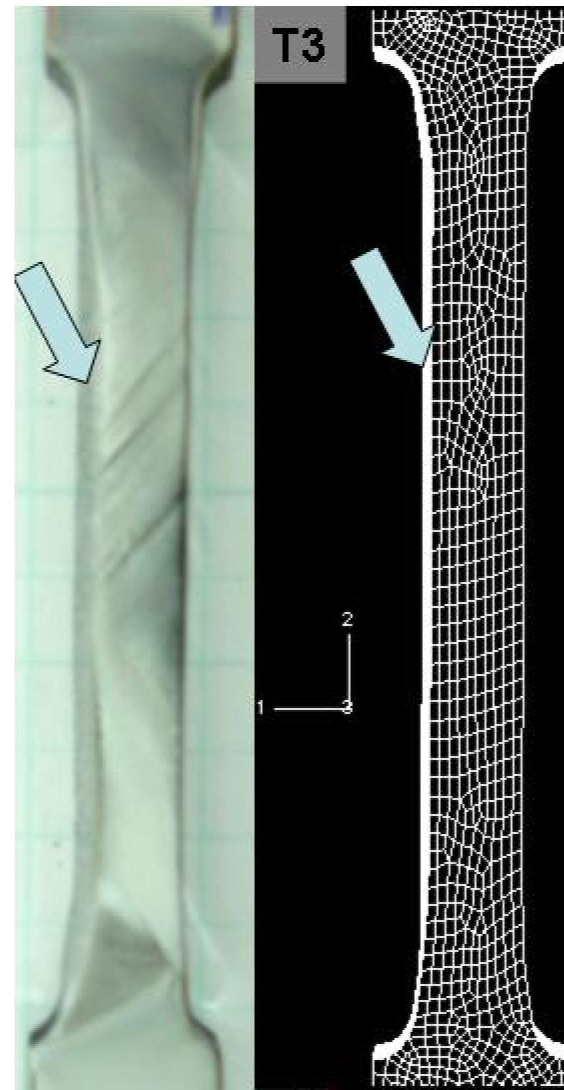



# Asymmetric shape changes come out right too...



# Summary

- Deformation of single crystals is complicated
  - (112) vs. (110) slip, rotations and hardening
  - Multiple slip in the same or different <111> directions
  - Deformation of single crystals with orientation gradients is more complicated.
- Deformation of bi-crystals is ***even more*** complicated, as grain boundaries introduce hardening sites leading to more heterogeneous deformation that affects formability and recrystallization
- Recrystallization rules seem elusive, tend to get random orientations
- Modeling in a predictive way, to give guidance on preferable crystal orientations (and misorientations) is possible, but non-trivial
- Dislocation content is potentially significant for flux pinning and thermal conductivity, so managing dislocation defect structure may be worth the effort to minimize unwanted dislocations and cryo plant costs
  - (watch for Saravan Chandrasekaran's talk)