

New studies of niobium material

(Update on Physical-Mechanical Metallurgy of Nb for Physicists)

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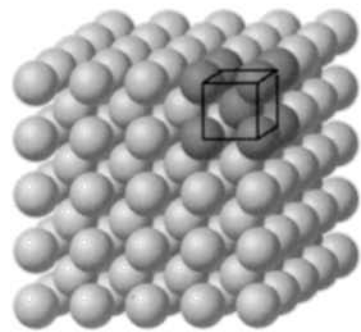
Work funded by DOE (ILC, OHEP), Fermi National Lab

Outline – Metallurgical issues along the processing path

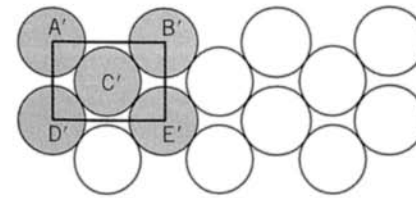
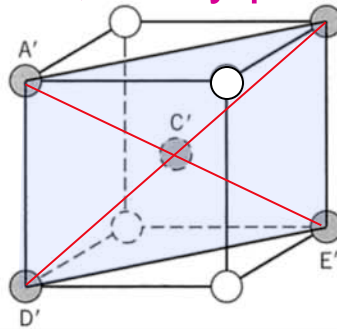
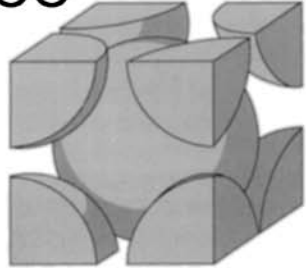
- PRST–AB overview paper (v.13, 031002, 2010)
- Some basics on Nb metallurgy and dislocations
- Conventional cavity fabrication process path concerns
 - Based upon sheet metal production/reproducibility
 - Requires welding at (the most?) critical locations
 - implications
- Alternative paths worth considering
 - Large grain slices + welding (lower cost, few GBs)
 - Hydroforming (removes welds but requires careful planning of plastic deformation history)
- New research thrusts reflecting our group and collaborations

Nb is a BCC metal like Fe;
not FCC like Al or Cu, or HCP like Mg or Ti

4 directions, many planes



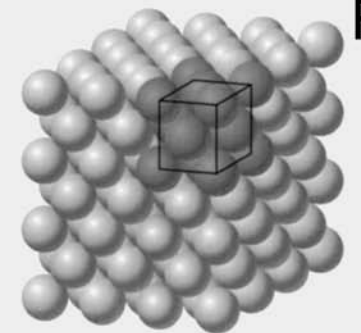
BCC



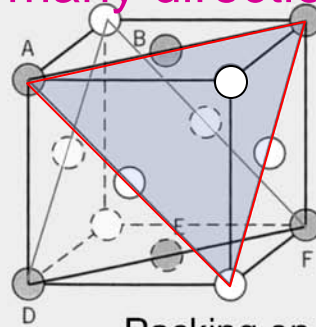
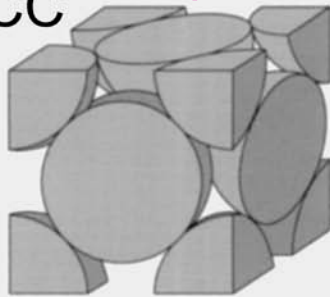
Packing on (110) planes

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

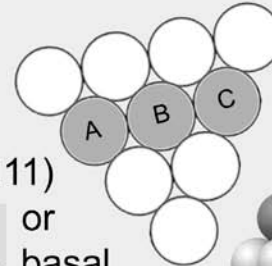
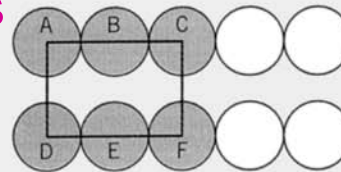
4 planes, many directions



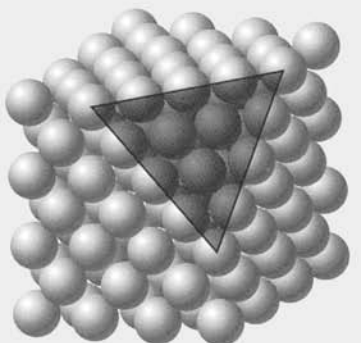
FCC



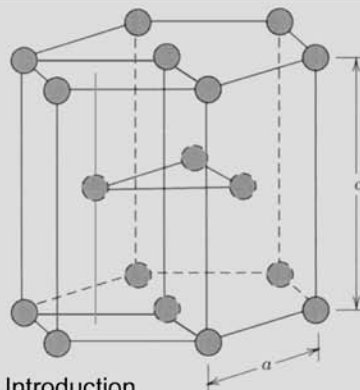
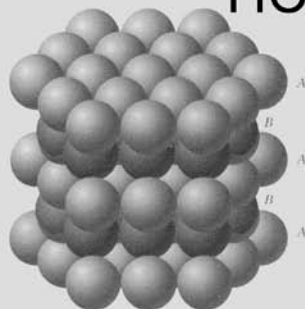
Packing on (111)



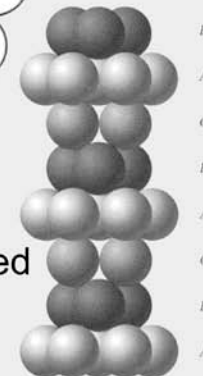
Al, Ni, Cu, Ag,
Au, Pb, are
FCC; close
packed plane
is (111)



HCP



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



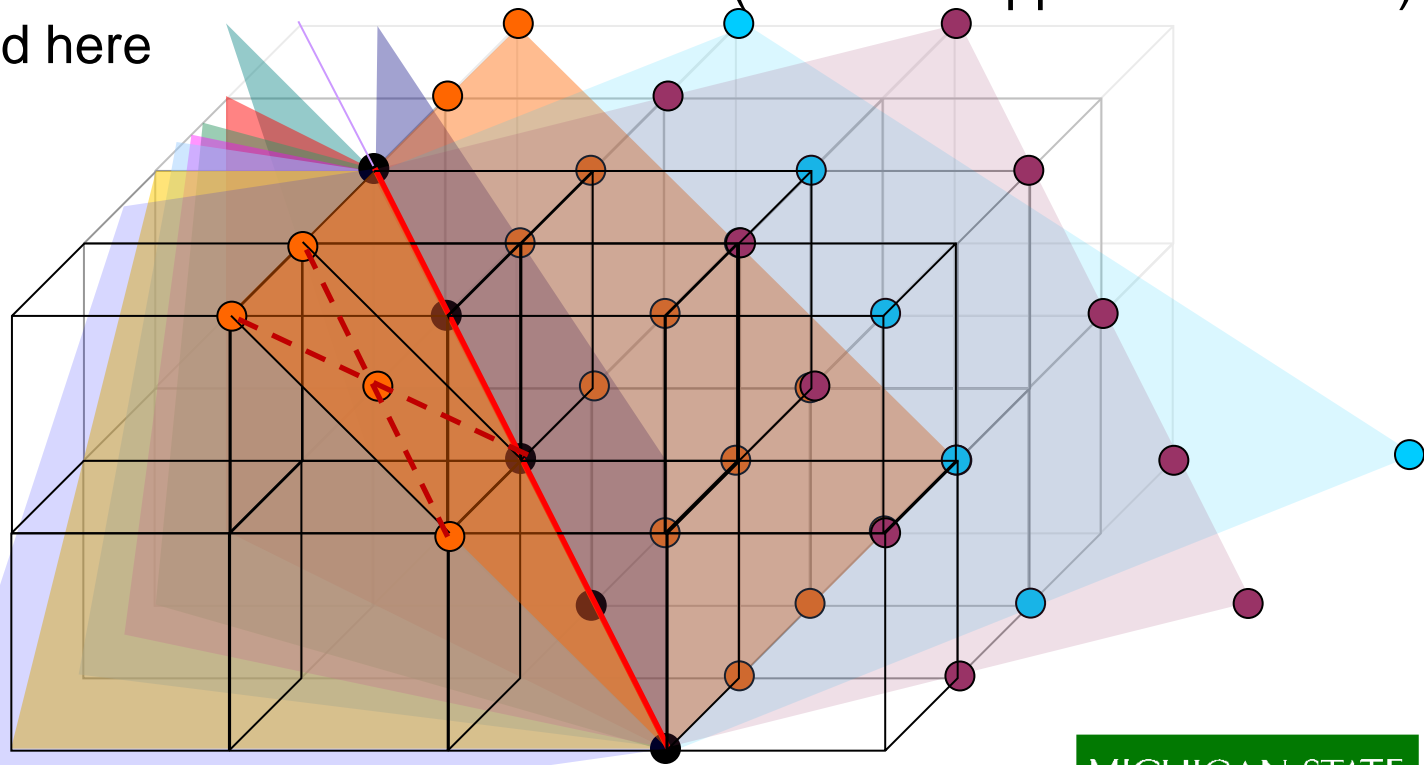
HCP basal
plane is also
close packed;
Mg, Ti, Co,
Zn, Zr

BCC dislocations can move in 4 directions in as many as 48 slip systems*

- Dislocations enable large plastic strains by facilitating shear on slip systems – there are 48 ways to do this, but most think that 24 of them are responsible for most of the deformation
- 4 groups, 12 associated with each $\langle 111 \rangle$ direction (connects opposite corners)
One group illustrated here

$[-1\ 1\ 1]$ $(1\ 1\ 0)$
 $[-1\ 1\ 1]$ $(1\ 0\ 1)$
 $[-1\ 1\ 1]$ $(0\ -1\ 1)$
 $[-1\ 1\ 1]$ $(1\ -1\ 2)$
 $[-1\ 1\ 1]$ $(-1\ -2\ 1)$
 $[-1\ 1\ 1]$ $(2\ 1\ 1)$

$[-1\ 1\ 1]$ $(1\ -2\ 3)$
 $[-1\ 1\ 1]$ $(2\ -1\ 3)$
 $[-1\ 1\ 1]$ $(-2\ -3\ 1)$
 $[-1\ 1\ 1]$ $(-1\ -3\ 2)$
 $[-1\ 1\ 1]$ $(3\ 1\ 2)$
 $[-1\ 1\ 1]$ $(3\ 2\ 1)$

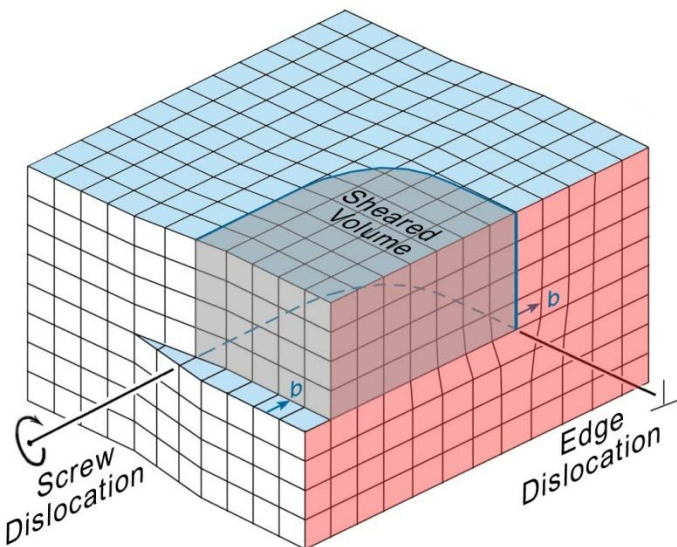


* FCC: 6 directions in 12 systems

BCC dislocations

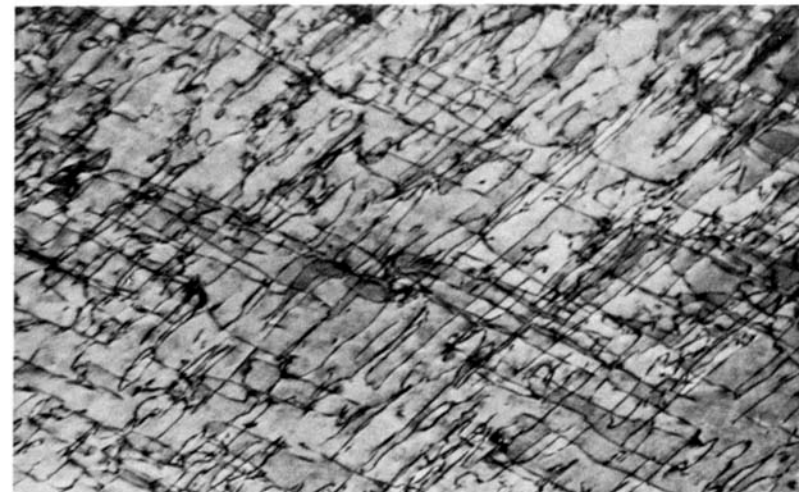
nomenclature, geometry, messy details

- $[-1\ 1\ 1]$ $(0\ -1\ 1)$ nomenclature defines the **slip direction** direction in $[\]$ and **slip plane normal** in $(\)$; integers \rightarrow vectors
 - or generalized as $\langle 111 \rangle$ on $\{110\}$, $\{112\}$, $\{123\}$ planes
- Slip systems with highly resolved shear stress operate first
 - only 5 are needed to achieve a general shape change – which 5?
 - Coordinate transformations needed to sort this out - **messy**
- **Dislocation loops** generated by ledges or other defects in grain boundaries, and/or entanglements inside grains



Edge and Screw geometry – edge dislocations are more mobile, leave extended loops in material

Krauss, 1980, HT of Steel



Rolling to make sheet metal - hard to control **microstructure** and **texture** (grain orientations)

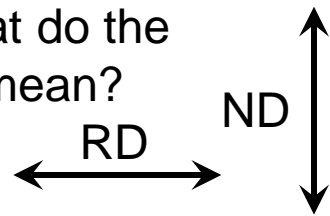
- Consistency is a challenge... Producers start with a hunk of ingot, and then use combination of breakdown forging, annealing, rolling to hopefully get a uniform microstructure
 - does initial ingot orientation haunt the microstructure to the end ?

- We have never seen the same texture/microstructure twice...

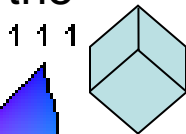
Jiang et al. IEEE Transactions Applied Superconductivity 17, June 2007

– Can be fixed

So, what do the colors mean?

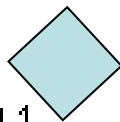


The crystal direction pointing out of the sheet

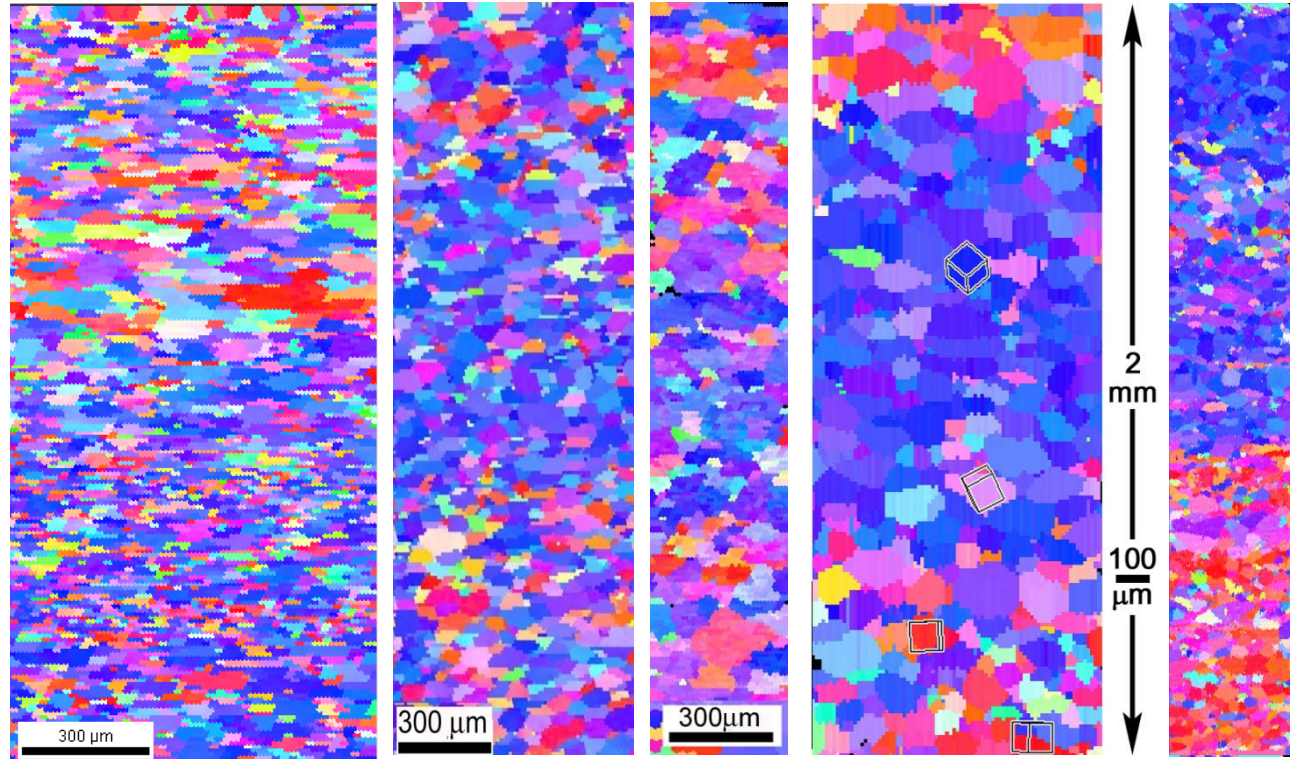


1 1 1

1 0 1



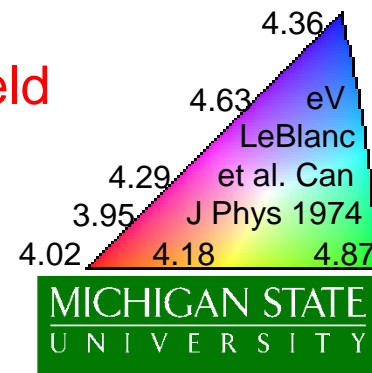
0 0 1



What kind of microstructure/texture is best?

Most have mix of red \leftrightarrow blue

- For formability, the blue orientation 'γ fiber' $[111] \parallel$ ND resists thinning and is most likely to yield a uniform surface
- Why? The **activated** $[111]$ slip vectors are as close as possible to the plane of the sheet \rightarrow much strain causes **least amount of change in thickness** (less roughness).
 - **Hard orientation** - a large stress is needed to activate slip
 - *Has a moderate work function for electron emission (4.36 eV)*
- The red orientation 'cube', or 'rotated cube' has the activated $[111]$ slip directions as highly inclined to the sheet as possible causing **most change in thickness** \rightarrow roughness
 - Also, it is **very soft**, and will be the first orientations to yield
 - *Has low work function for electron emission (4.02 eV)*
- The green orientation is a minority orientation
 - *Has highest work function for electron emission (4.87 eV)*

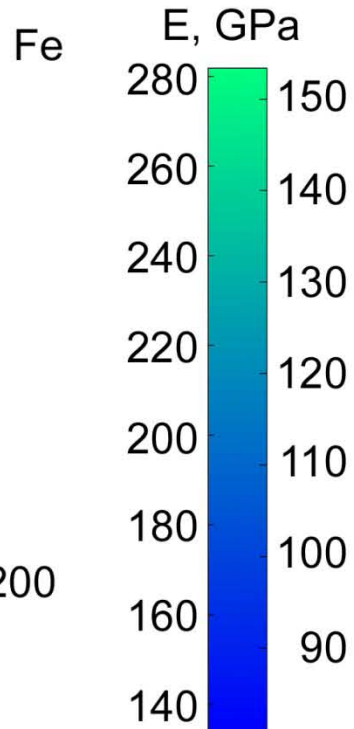
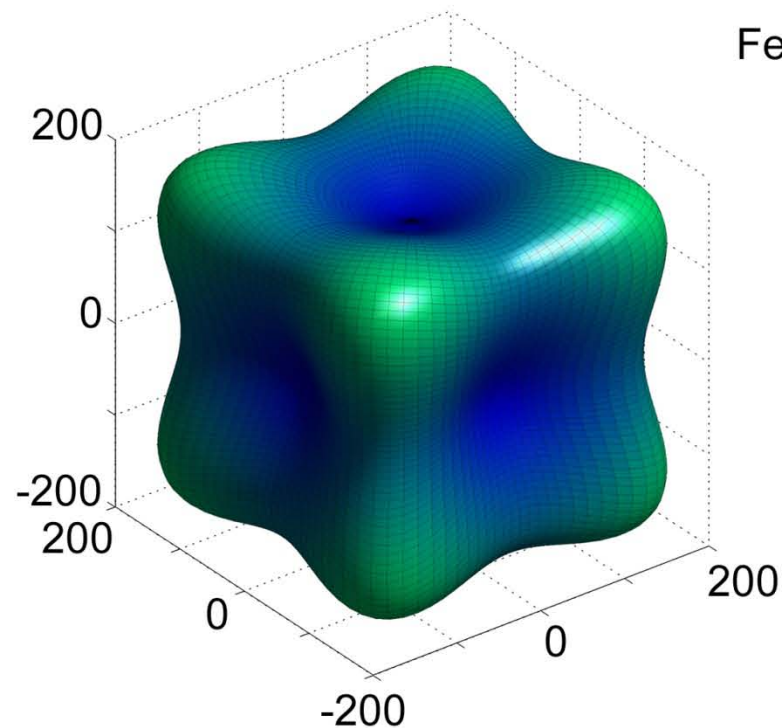


What should the SRF community be requesting from commercial producers?

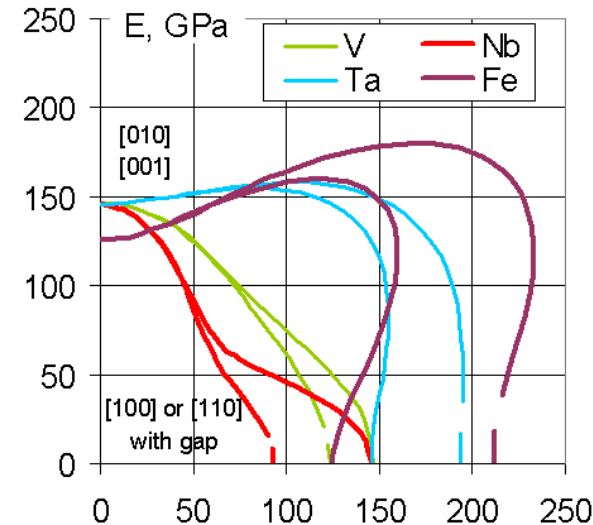
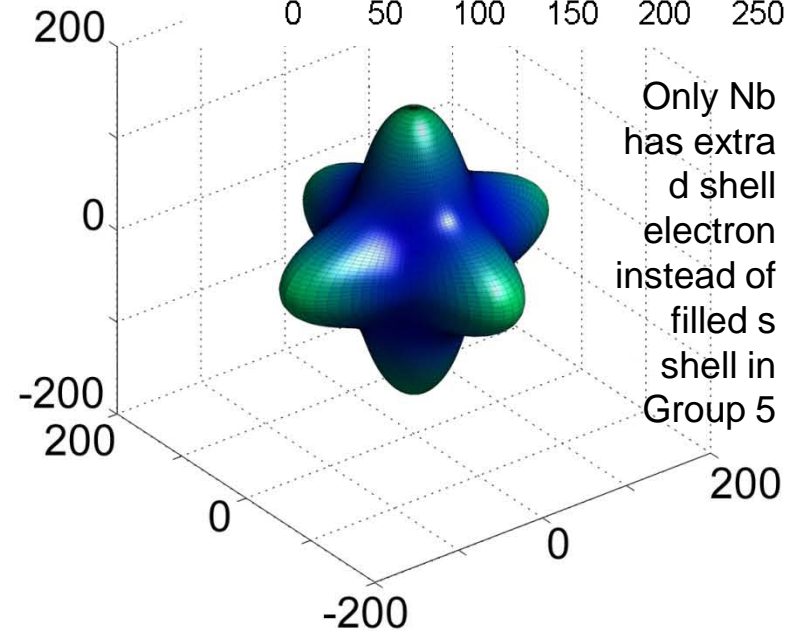
- Is purity important? → higher purity makes is softer!
- Is yield strength important? (dimensional stability)
 - Only possibility is a small grain size – tough to get!
 - Nothing to pin dislocations or grain boundaries if high purity
- Is texture/microstructure on surface important?
 - Etching and grain boundary ledges (roughness)
- Is overall texture important for half-cell formability?
 - It is very ductile anyway
 - How much anisotropic behavior is tolerable?
- Sheet vs. tubes?
 - Hydroforming tube → cavity is an important possibility
- Is **dislocation substructure** important (annealing)?
 - Affects recrystallization, grain size, ductility, much like steel... How similar is Nb to steel?

Elastic anisotropy of Nb is opposite of steel, and every other BCC metal except V

- There is about a factor of ~ 2 between modulus in 111 and 100 directions for Fe and Nb, **but in opposite directions**
- **Nb is most compliant** in the direction where Fe is most **stiff**; affects dislocations



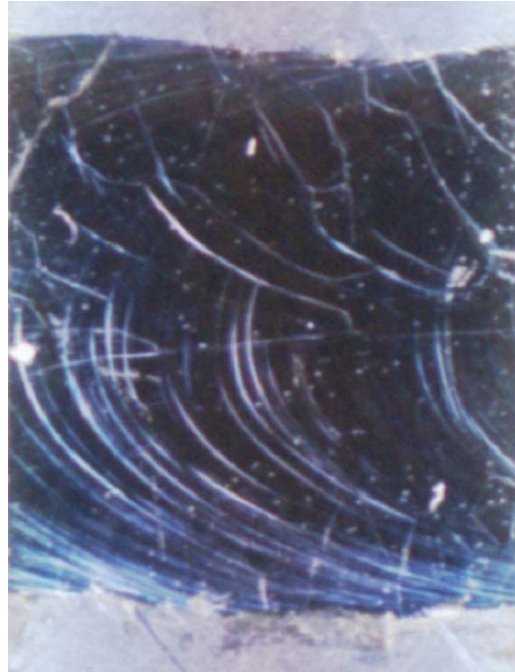
Nb



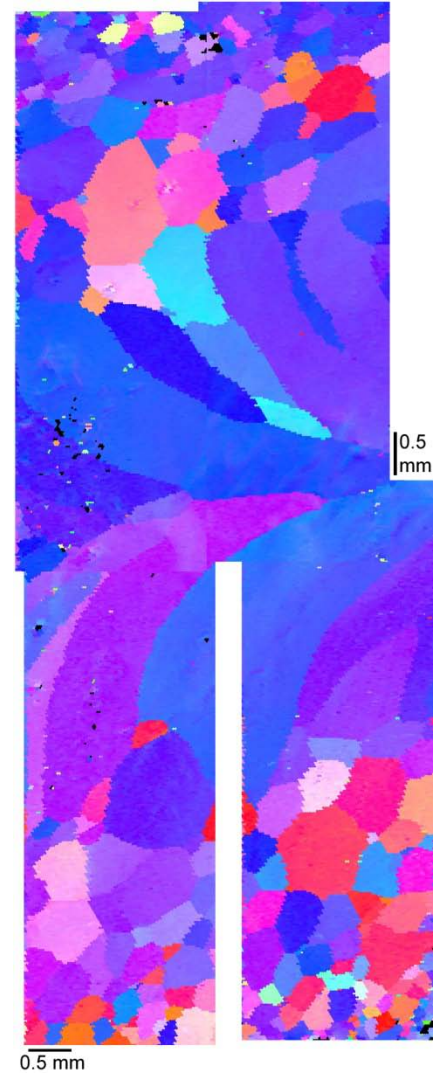
Whether single crystal or multi-crystal, Nb has to be welded → huge grains



Differential oxidation after exposure to air before fully cooled delineates grains

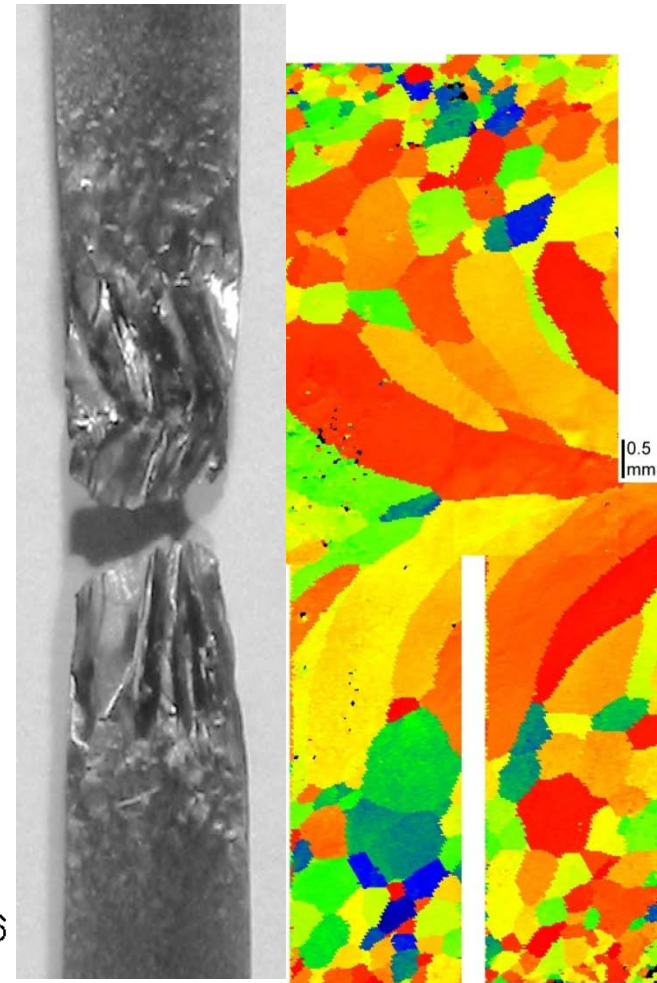
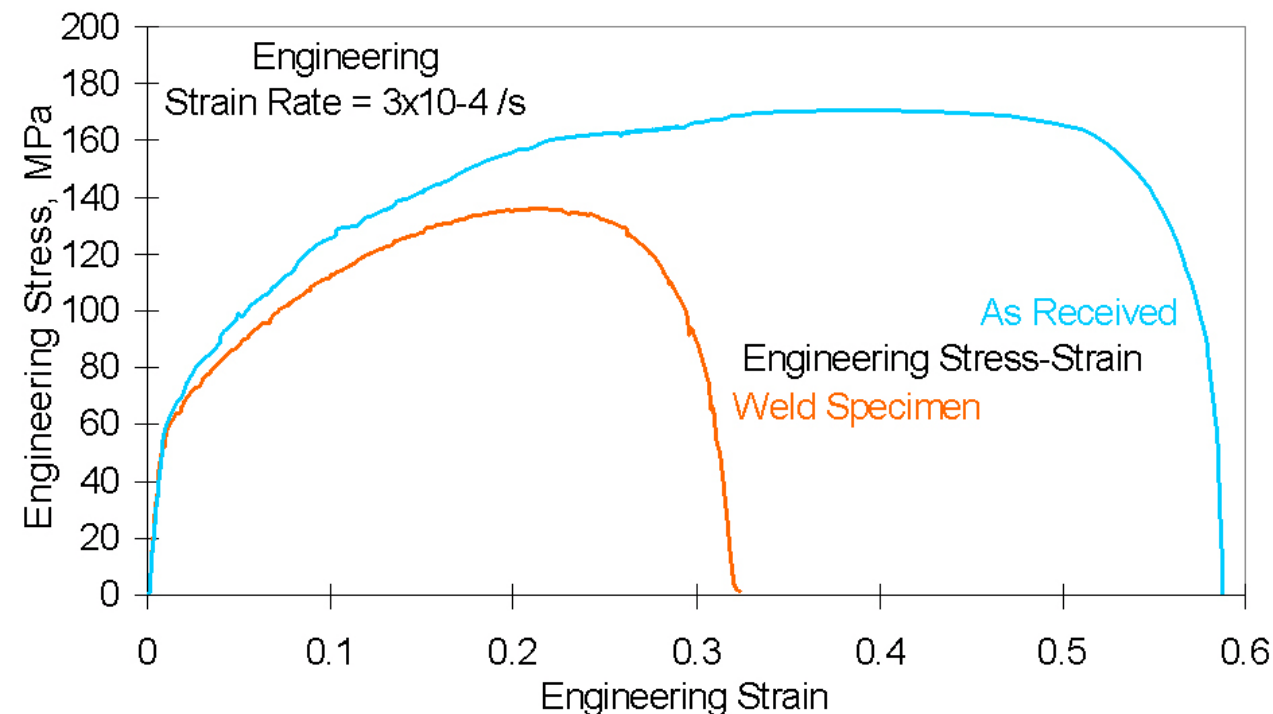


Thermal etching makes
Grain boundaries visible



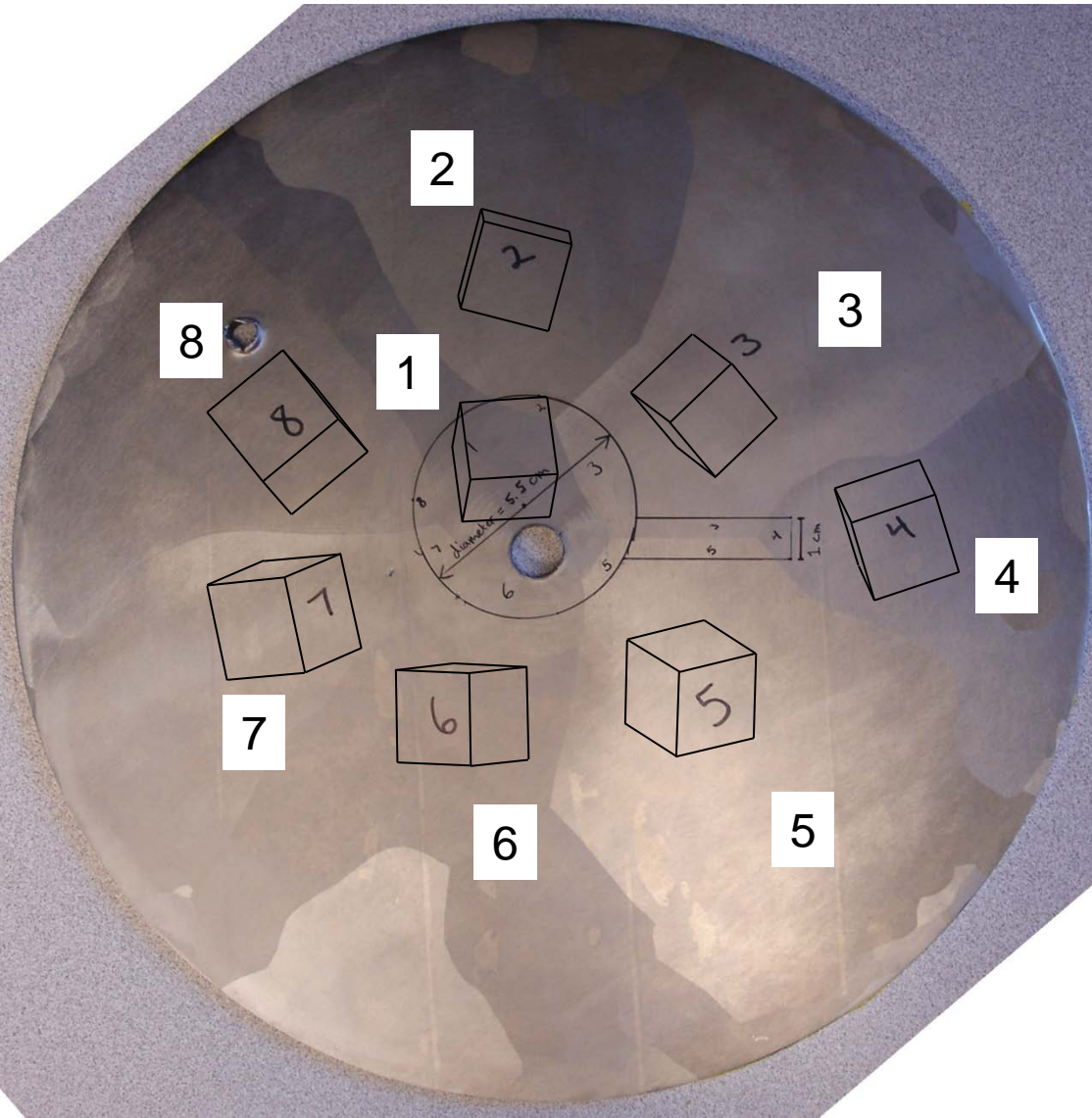
Tensile properties are lower in weld, due to huge grain size \rightarrow exaggerated 'orange peel'

- Variation in grain size and orientation will lead to differential strains in different grains.
- Schmid factor map - Red orientations will yield before yellow-green-blue grains



0.5 mm Schmid Factor Min 0.25 0.5 Max

Making cavities from ingot slices?



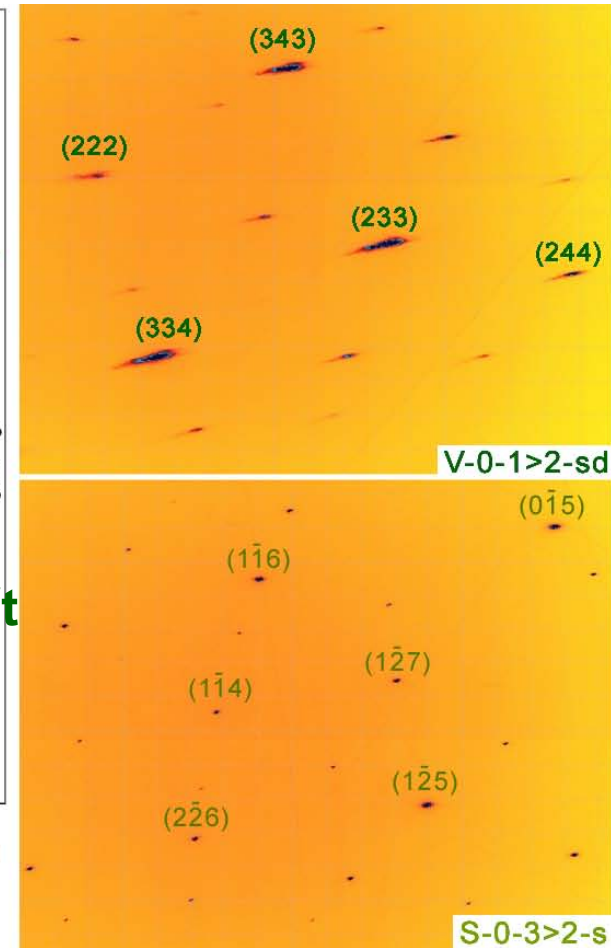
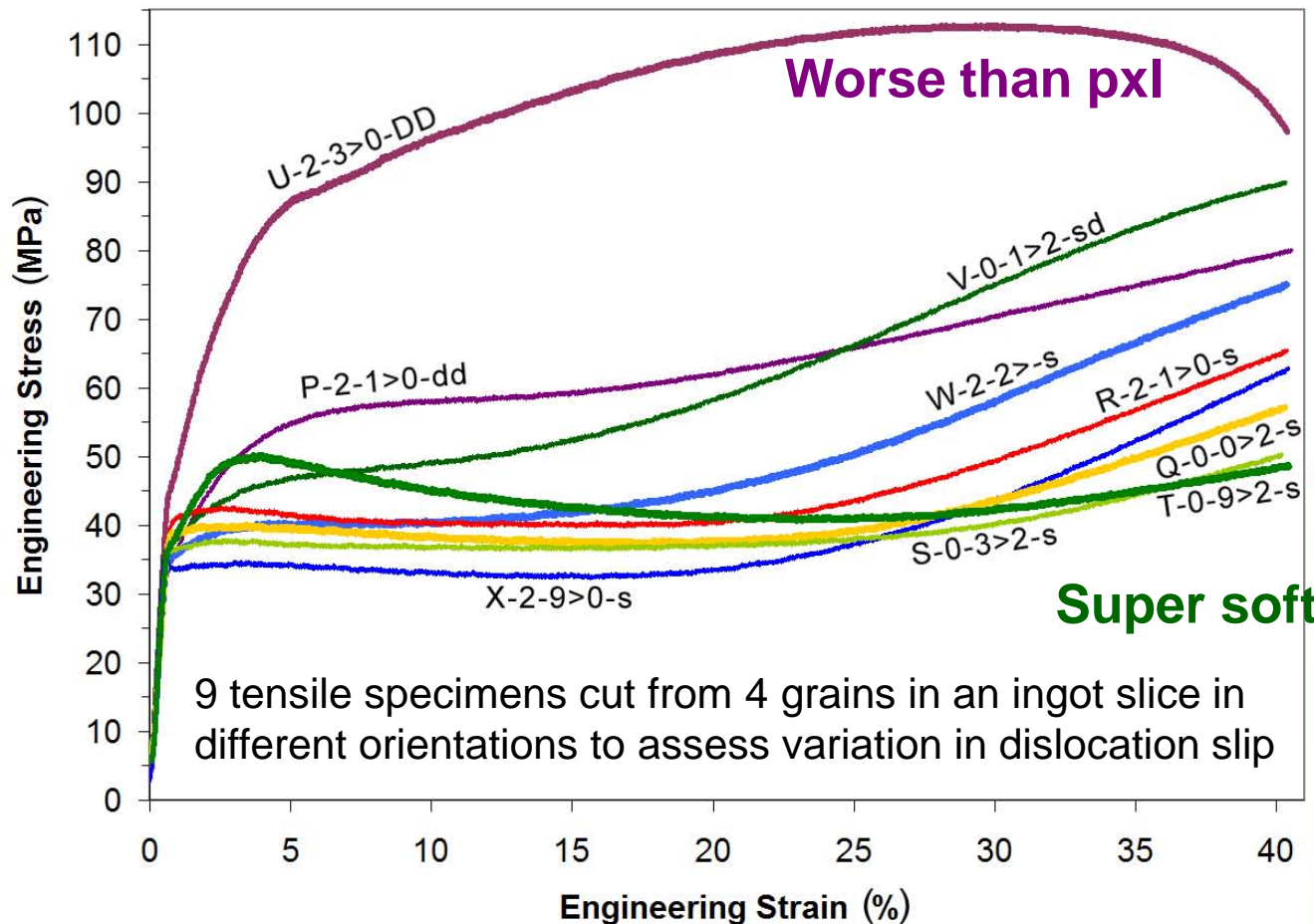
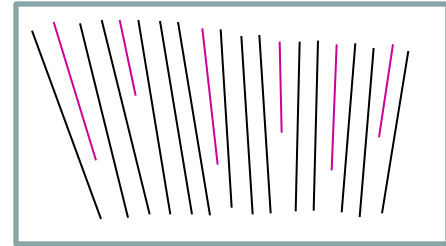
- Large grains deform heterogeneously, due to different crystal orientations
- Ridges form near grain boundaries due to restricted deformation



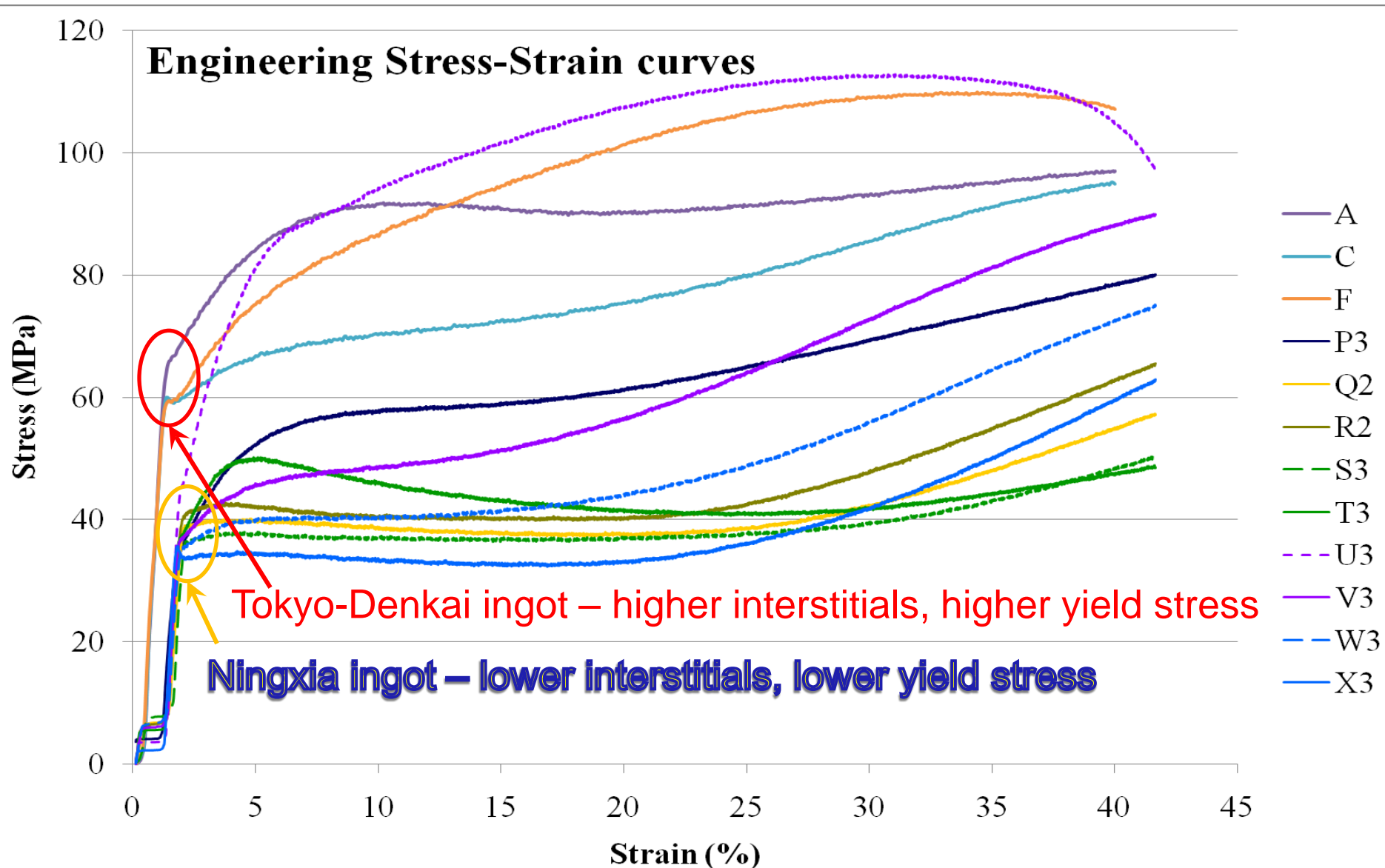
As deformation depends on initial orientation, how does orientation affect deformation? **A lot**

Diffraction patterns typically streaked due to dislocations

Deformation and orientation gradient details affect nucleation of recrystallization and defect annihilation

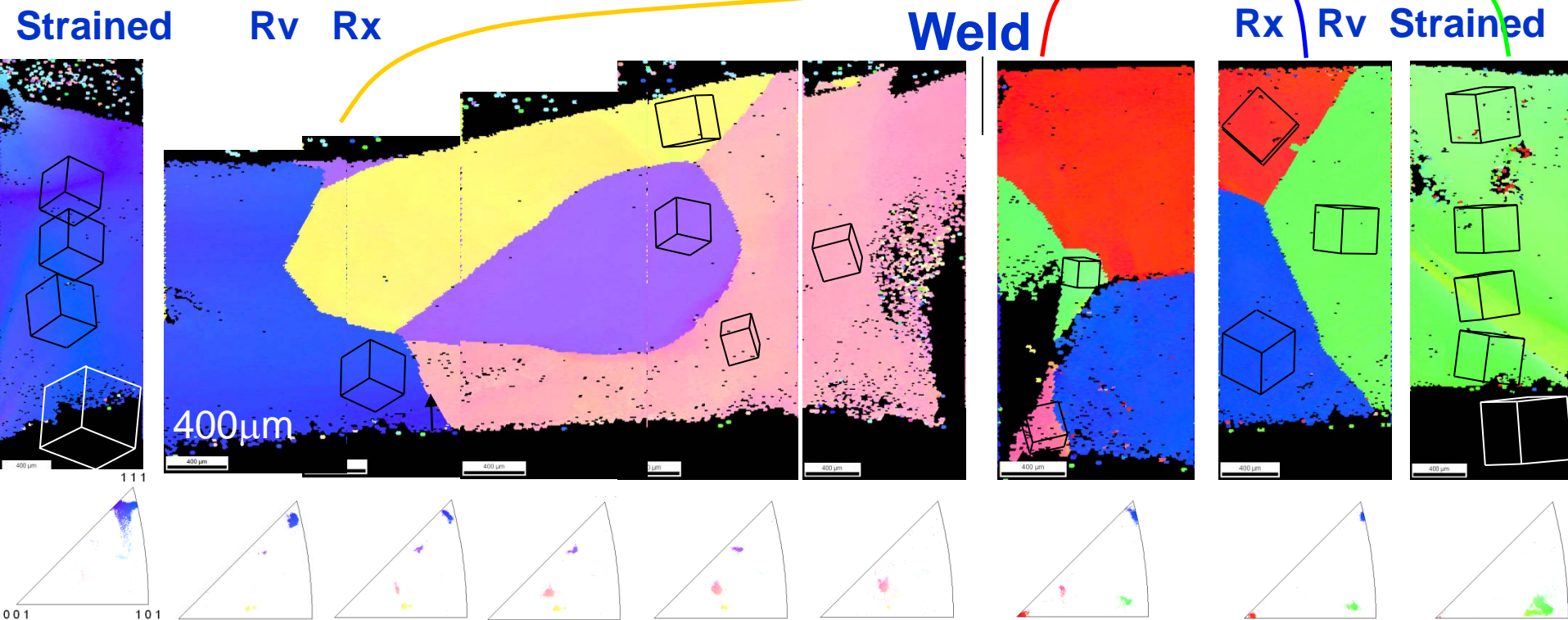
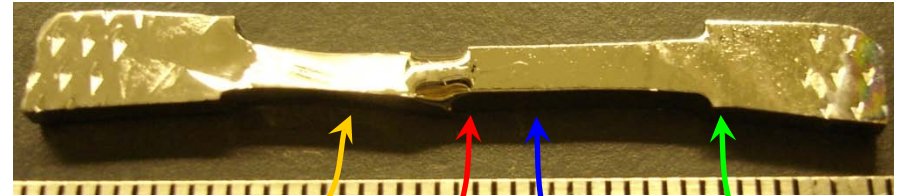


Compare/contrast single crystal tensile specimens from two different ingots (Tokyo Denkai, Ningxia)

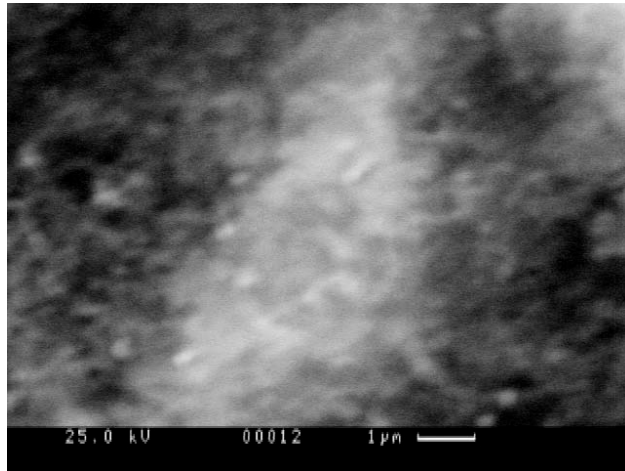


Not surprisingly, welding plastically formed single crystals leads to recrystallization

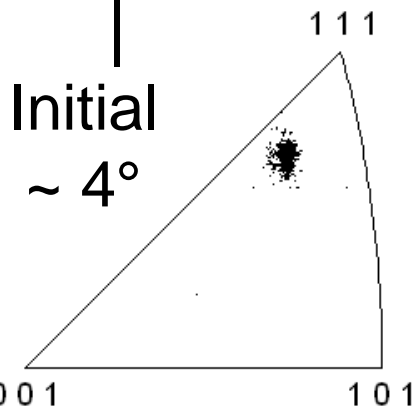
- Two tensile deformed single crystals deformed to ~40% strain welded together (iris); orientations after weld
- Parent orientations in grips have white prism orientation
- Black prisms show crystal orientations after weld



So, what impact does processing history have on dislocation substructure?

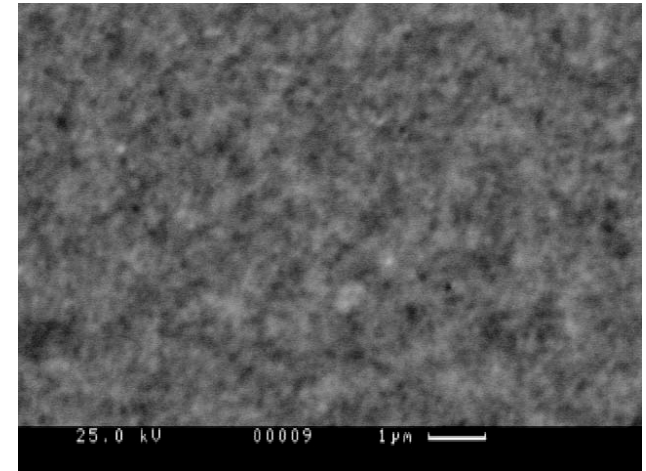


As-received \rightarrow many entangled dislocations in cell walls

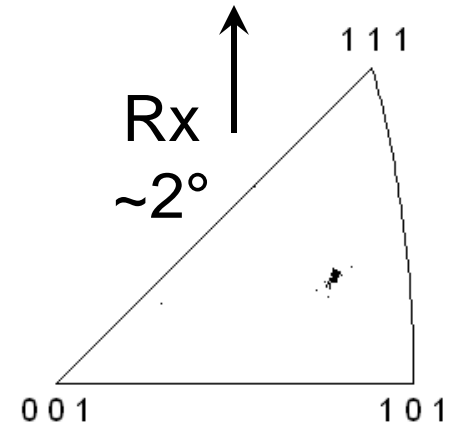
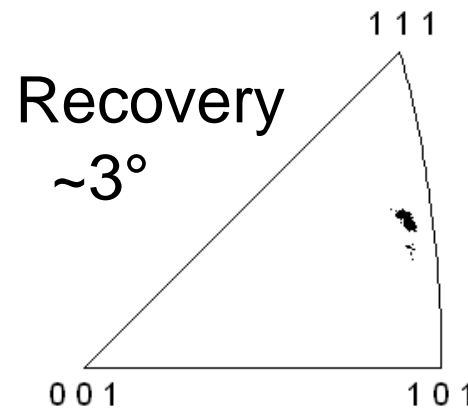
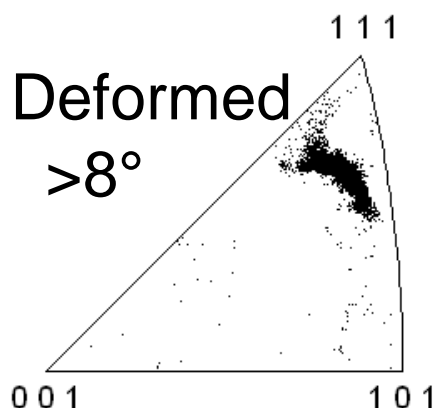


$\sim(111)[-110]$ specimen:
Peak spread is minimum in Rx,
nearly as sharp in recovered

Normal Direction
Inverse
Pole Figures

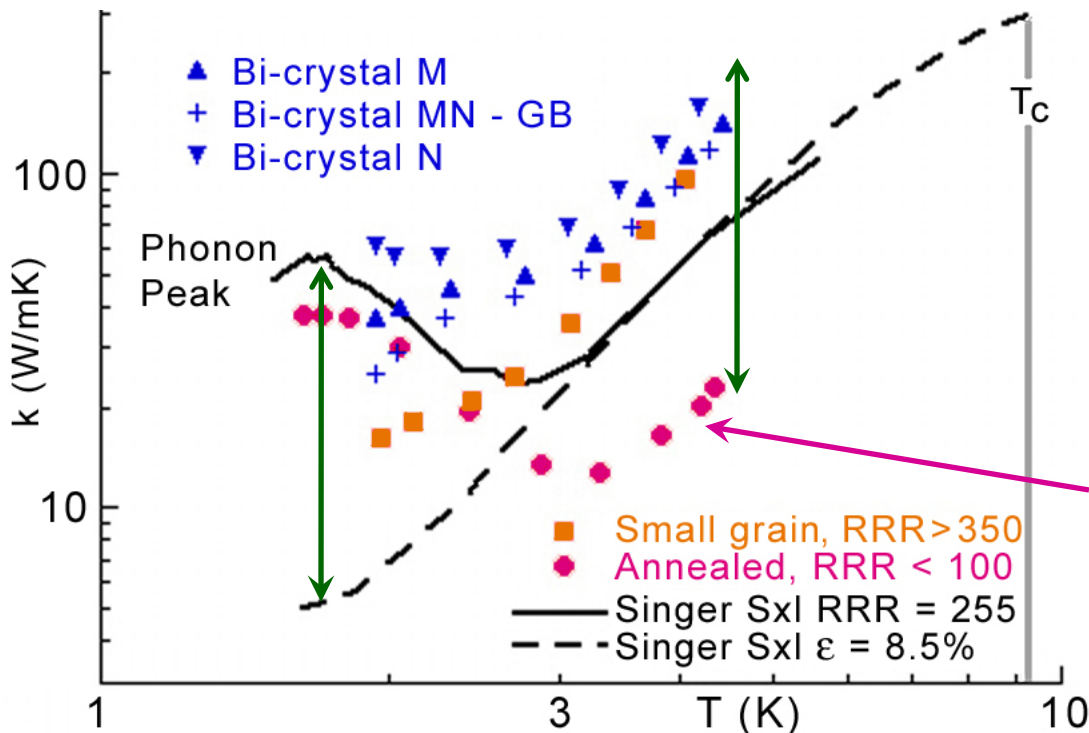


Rx grain \rightarrow fewer, but still many dislocations, evenly distributed



Dislocation content and purity affect thermal conductivity and presence of 'hot spots' by 10x

- Phonon peak appears with annealing, so dislocation content seems to be a key factor for thermal conductivity, → Cryo €\$¥
- How does dislocation substructure and purity affect RRR?



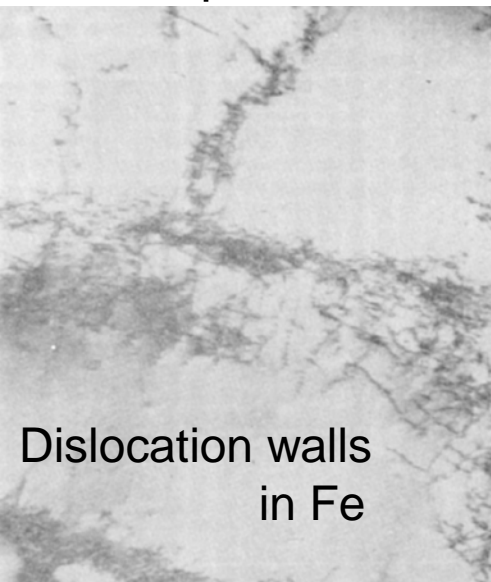
Different phenomena above and below ~ 3K

Recent sxl measurements show higher phonon peak after 850 Rx than 600°C recovery anneal

This specimen was annealed at ~1400°C, but a lower *purity* environment decreased RRR

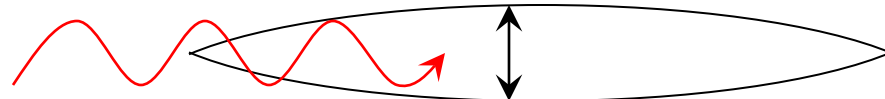
Dislocation substructure & thermal properties

- Prior work (Cotts, Northrup, Anderson 1981-83) examined phonon transport and dissipation in LiF single crystals with known dislocation substructures
 - **When phonon transport direction is parallel to a mobile dislocation segment, phonon was dissipated (phonon converted to friction)**
- Phonons travel according to (anisotropic) elastic properties
 - What about transport through grain boundaries?
- Is the low T phonon-peak killed when there are dislocations that can couple with phonon and disperse its energy?

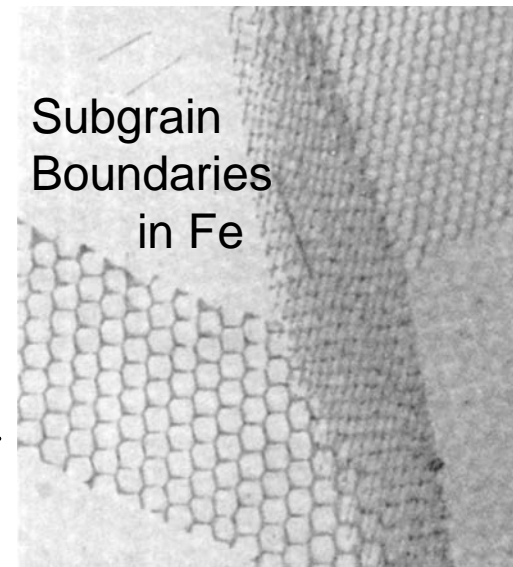


If so, then dislocation substructure may need to be managed with respect to crystal orientation to maximize phonon transport

So, **must** we Recrystallize?



Hull & Bacon Introduction to Dislocations, 1984



Most desirable Nb material state?

- Dislocations, grain boundaries, interstitials, are not desirable
 - Dislocations are necessary to form the complex shape
 - Etching and heat treating introduce interstitials, exposure to air oxidizes surface, heat treatment dissolves oxide
 - Grain boundaries result from recrystallization but grain growth sweeps out dislocations
 - Grain boundaries in some orientations permit magnetic field flux penetration (Giovati, Gurevich, others at FSU)
- Does Nb love its dislocations so much that it will not let us sweep them all away? (some are required to satisfy entropy)
- ? Perfect cavity a recrystallized single crystal with remaining dislocation segments **not** lined up in a radial direction ?
 - Would tuning erase this benefit?

Large grain slices to make cavities

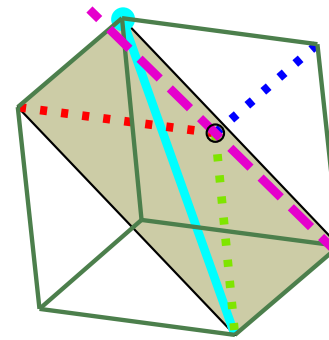
- **Benefits:** Reduced cost due to not wasting corners, lack of grain boundaries that may disperse phonons, can improve Q (and reduce cryo cost)
- **Issues:** Still has welds, which are a major source of uncertainty and often implicated with respect to 'hot spots'
 - Not the welds themselves, but the heat affected zones, where higher impurity absorption may occur, or other unidentified phenomena related to recovery/Rx
 - Doesn't have many grain boundaries that move and sweep out dislocations (depends on Rx...)
 - Anisotropic properties may contribute to variability

What we are doing to support large grain cavity fabrication research

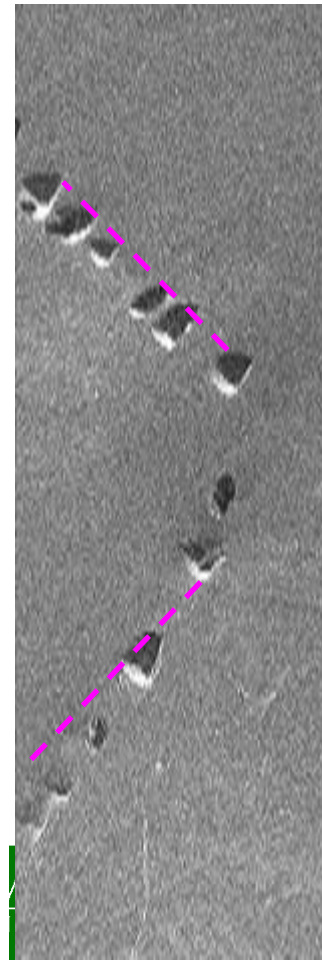
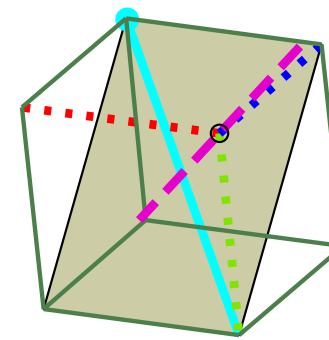
- Etch pits are a potential source of hotspots there are documented correlations (Fermi, J-Lab); OIM (collaboration with J-lab) on etch pit characterization
- Laue camera to nondestructively automate measurement of crystal orientations
- Identification of desirable / undesirable crystal orientations near iris and equator to minimize defects (reject slices that would lead to lower quality cavities) → material specification

Assuming biaxial stretching

$$\text{Or-1 m \# 6} = 0.209$$
$$\text{ss1 n} = [1 \ 1 \ 0][-1 \ 1 \ -1] = b$$



$$\text{Or-1 m \# 1} = 0.291$$
$$\text{ss10 n} = [0 \ 1 \ 1][-1 \ 1 \ -1] = b$$



Hydroforming requires complex forming path, but can eliminate welds at critical locations

- Cons: Requires development of optimal processing path from ingot to form tube that is optimal for forming cavity
 - There are many potential hydroforming paths once you have a decent tube, unexplored territory
 - Requires computational modeling to assist interpretation of results and identification of optimized process
 - Cost of deformation processing (and annealing)
- **Benefits: No welds, that are probably a major contributor to variability in performance**
- Once process path is established, reproducibility is likely to be high (e.g. path is well defined for beverage can industry)
- While upstream material processing cost may be higher, high yield and fewer parts to handle could lower cost

What we are doing to support seamless cavity fabrication research

- Develop constitutive model for computational simulation of tube and cavity formation, that will support prediction of optimal processing paths
- Optimize tube fabrication path
 - Supporting / analyzing flow formed tubes and texture/microstructure evolution with Roy Crooks, Black Labs, VA
 - Equal Channel Angle Extrusion of tubes, with K.T. Hartwig at Shear Form (TX)
- OIM characterization and analysis of hydroformed cavities, Mike Sumption (OSU)
- Hot tube large / single grain conditioning and forming, Jim Murphy (UNR)
 - May be possible to make preforms or maybe even cavities at high temperature by bulging tubes in single / large crystal condition

