ENGINEERING

Microstructure Studies of Niobium

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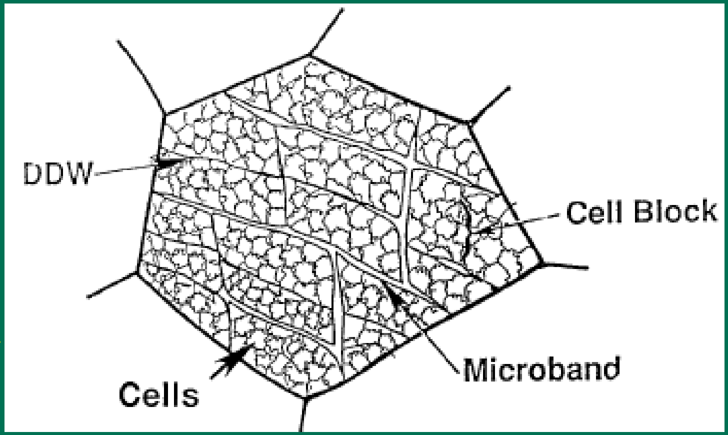
Dislocation substructure, recrystallization, and texture are important for SRF community

- Polycrystalline Nb
  - As-received texture of rolled sheet varies even between batches from the same manufacturer
  - Recrystallization and/or grain coarsening at e-beam welds leads to soft region along weld
- Large grain/Single crystal Nb
  - Tubes (welded or seamless) for hydroforming
  - Will deform differently in different crystal directions
  - Relating dislocation substructures to recrystallized grain orientations would aid design of desired textures
- Electric/Superconducting properties
  - Romanenko's thesis on high-field Q-slope correlates hot spots to higher local misorientation of lattice (higher dislocation density)
  - Phonon scattering due to dislocation lines parallel to phonon travel

Dislocation structures

- Grains divide into
  - Cell Blocks (CBs) bounded by long flat boundaries
  - Microbands (MBs)- double walled, small strains
  - Dense Dislocation Walls (DDWs)- single walled, small strains
  - Lamellar boundaries (LBs)- appear at large strains, form very flat CBs, replace MBs and DDWs
  - All the above are considered geometrically necessary boundaries (GNBs)
    - Separate regions that deformed differently
    - Arranged in parallel families
    - Special macroscopic orientation relative to deformation axis
    - Increasing strain- increase in misorientation angle and decrease in spacing, much more rapidly than IDBs
  - Cells within CBs (SSDs)
    - Equiaxed, bounded by incidental dislocation boundaries (IDBs)
    - Low misorientation angles



Doherty et al, Mat Sci Eng A238 (1997) 219-274

Cold-worked state must be understood since recrystallization depends on it

- Commercial Nb sheet is made by rolling ingots containing very large grains
- Deformation banding from rolling is mechanism of grain refinement in Nb bicrystal
  - Dislocations moving on different planes tangle with each other
  - In compression, greater amount of deformation banding occurred within grains having ‘unstable’ <110> || to the compression axis
- Different grain orientations will develop different deformation features ►affects recrystallization

Grain boundary

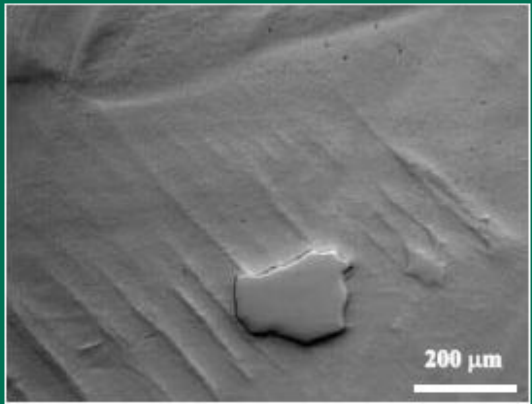
Grain 1

33% reduction

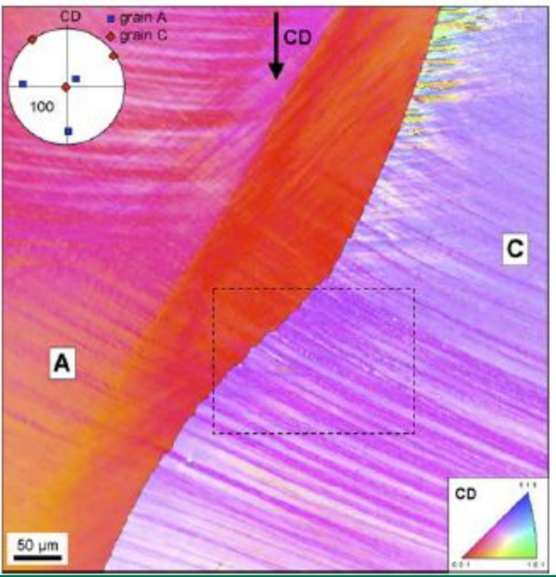
Grain 2

Grain 1: Nucleation in shear band after 70% reduction, heated 800°C 1hr

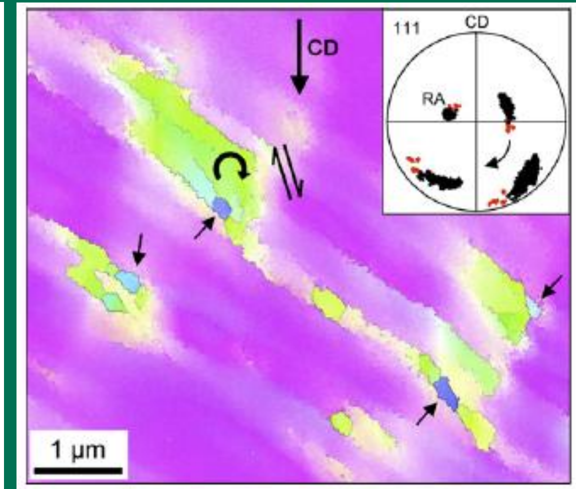
Sandim et al *Mat. Sci. & Eng.*, A354, 2003



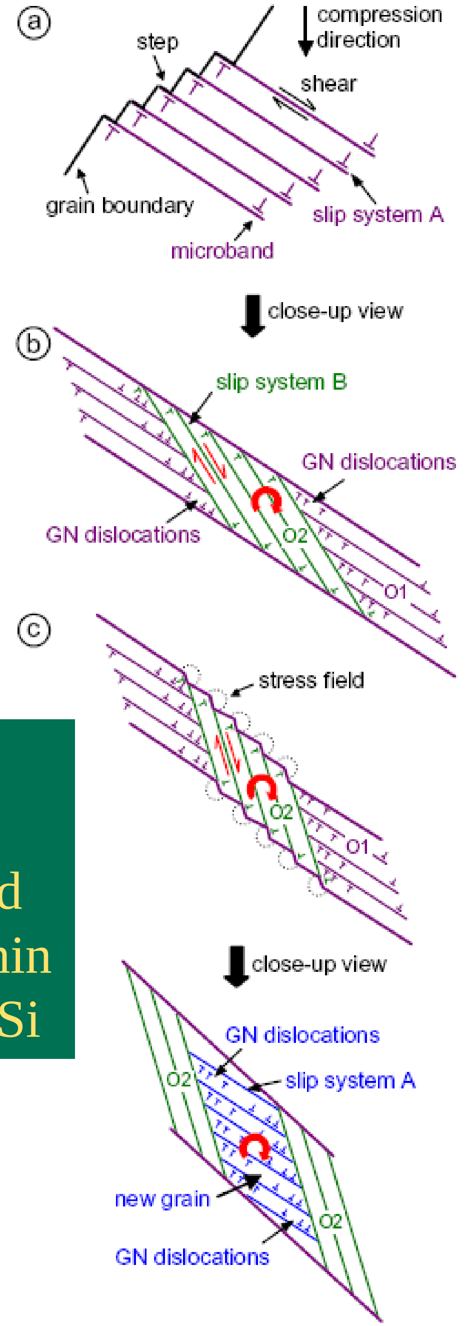
Example of possible embryos/nuclei



Overview of deformation microstructure of two grains at their boundary. Crystal orientation map EBSD step size 1.4 µm. CD=compression direction. Pole figure of initial orientations



Lattice rotations about ~{111} of possible embryos, black lines >15° misorientation



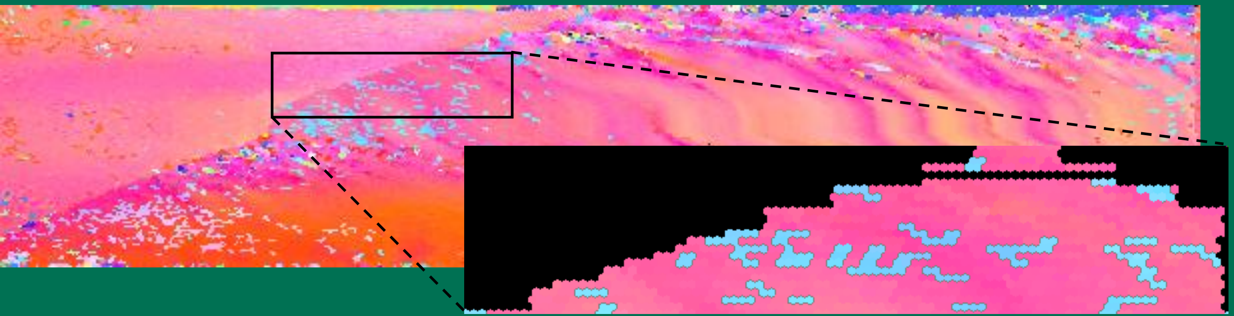
Posited action of intersecting slip systems leading to local rotation about a {111} axis and making possible embryos within microshear bands in Fe-3wt%Si

‘New grains’ ~200-300nm diameter would probably not be visible in deformation texture. No anneal yet…

Dorner et al. *Scripta Mat.*, 57, 2007

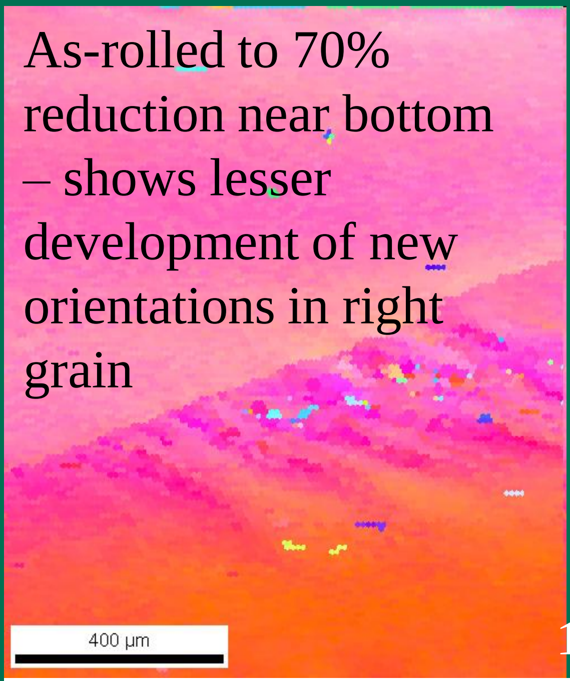
Rolled to 71%, **no heat treatment**

Near top shows development of measurable new orientations (blue) in right grain, ***only due to strain***



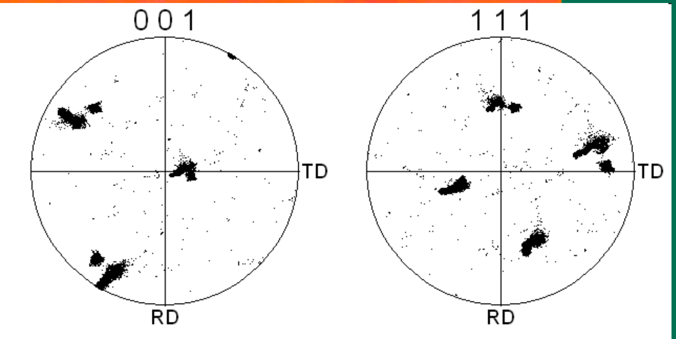
2micron step

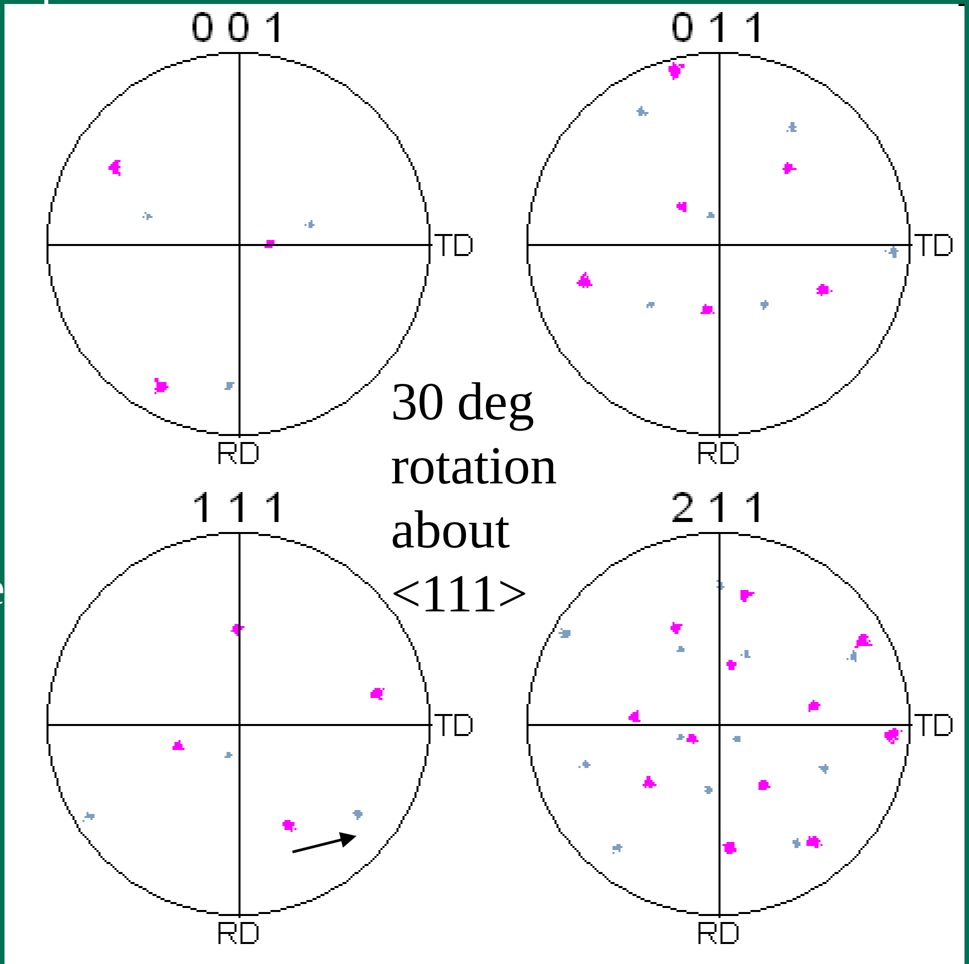
Sample 6



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

10micron step

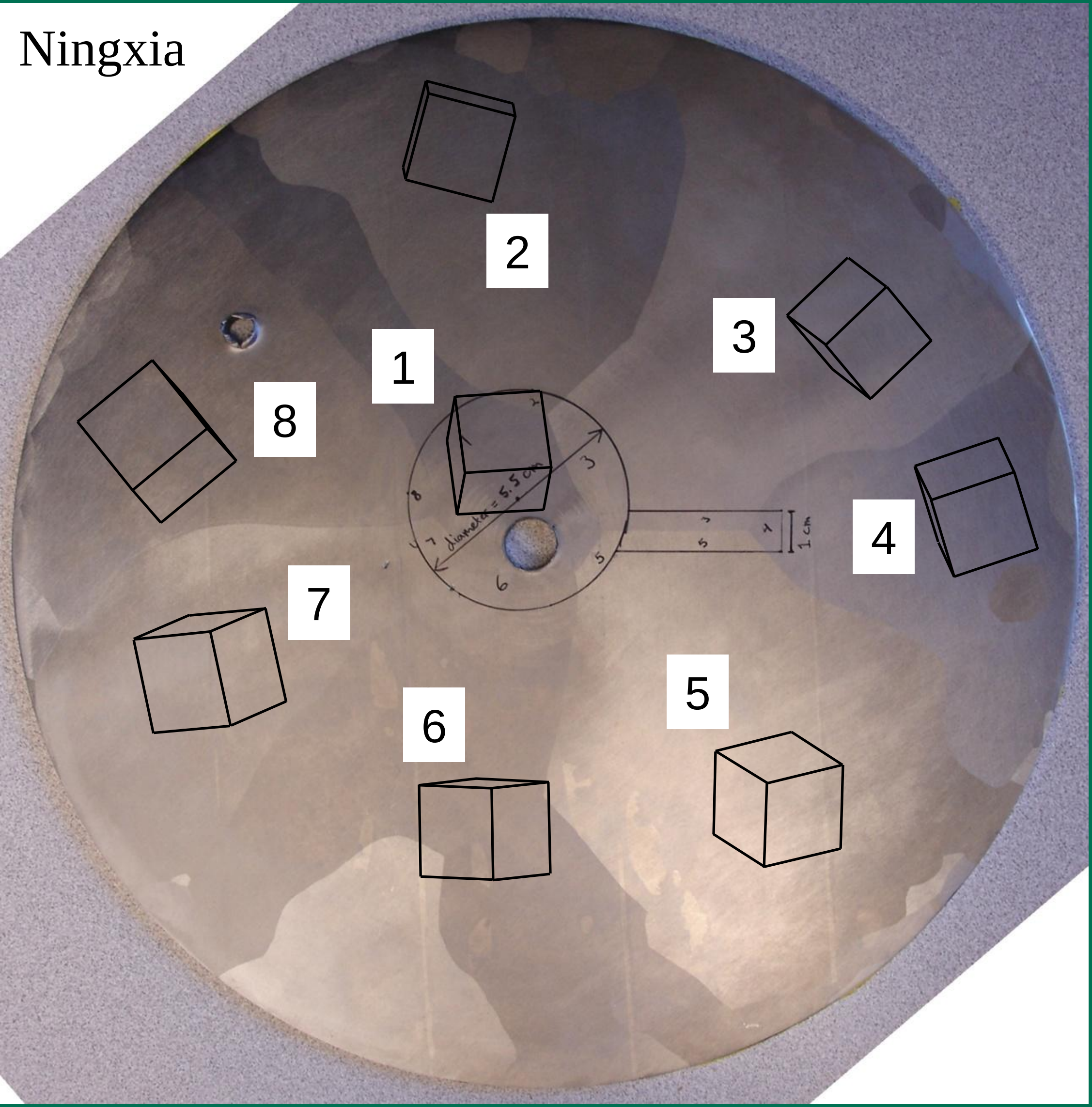


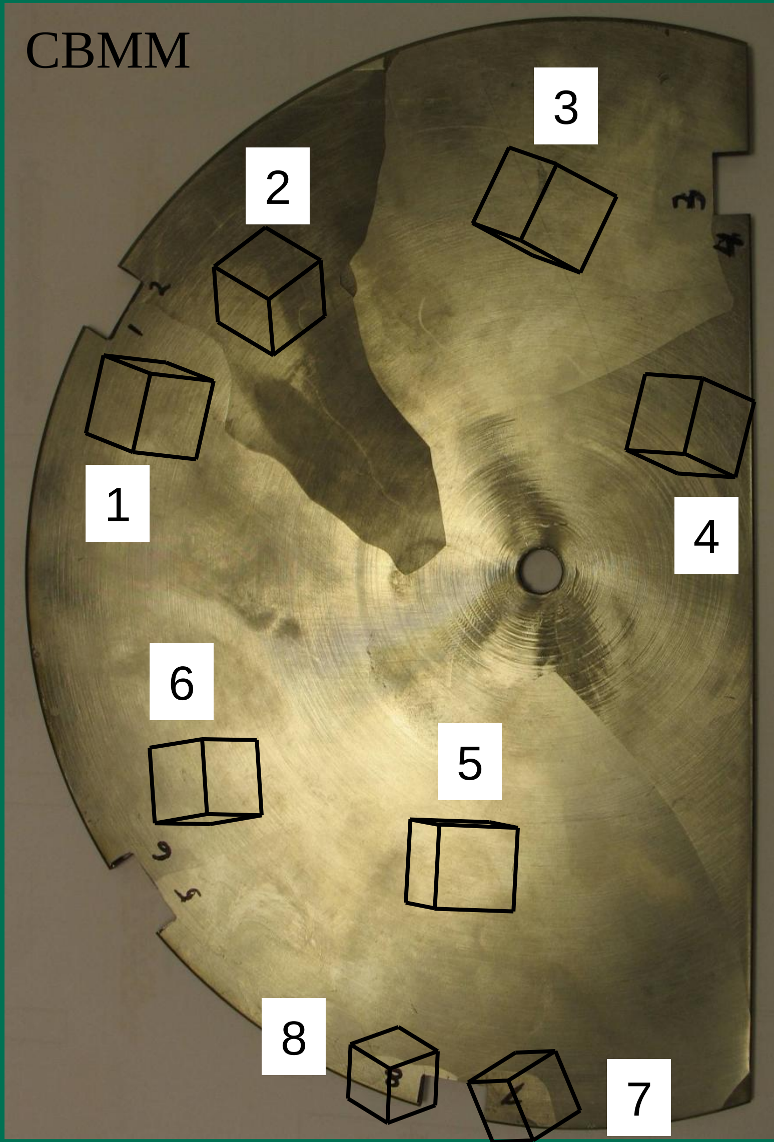


30 deg rotation about <111>

Comparing grain orientations of ingots

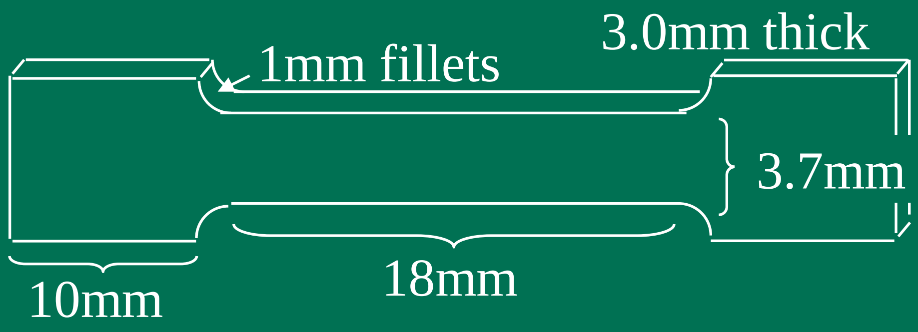
- More similar than different
  - two other ingots to be examined soon
- Both tend to have <110> parallel to ingot axis





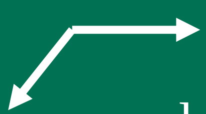
Orientations chosen to resolve shear stress τ onto specific slip systems

Alternating chemical etching and mechanical polishing, then electropolished for initial crystal orientation determination via OIM



then 40% strain in uniaxial tension

(similar to strain at iris during deep drawing)



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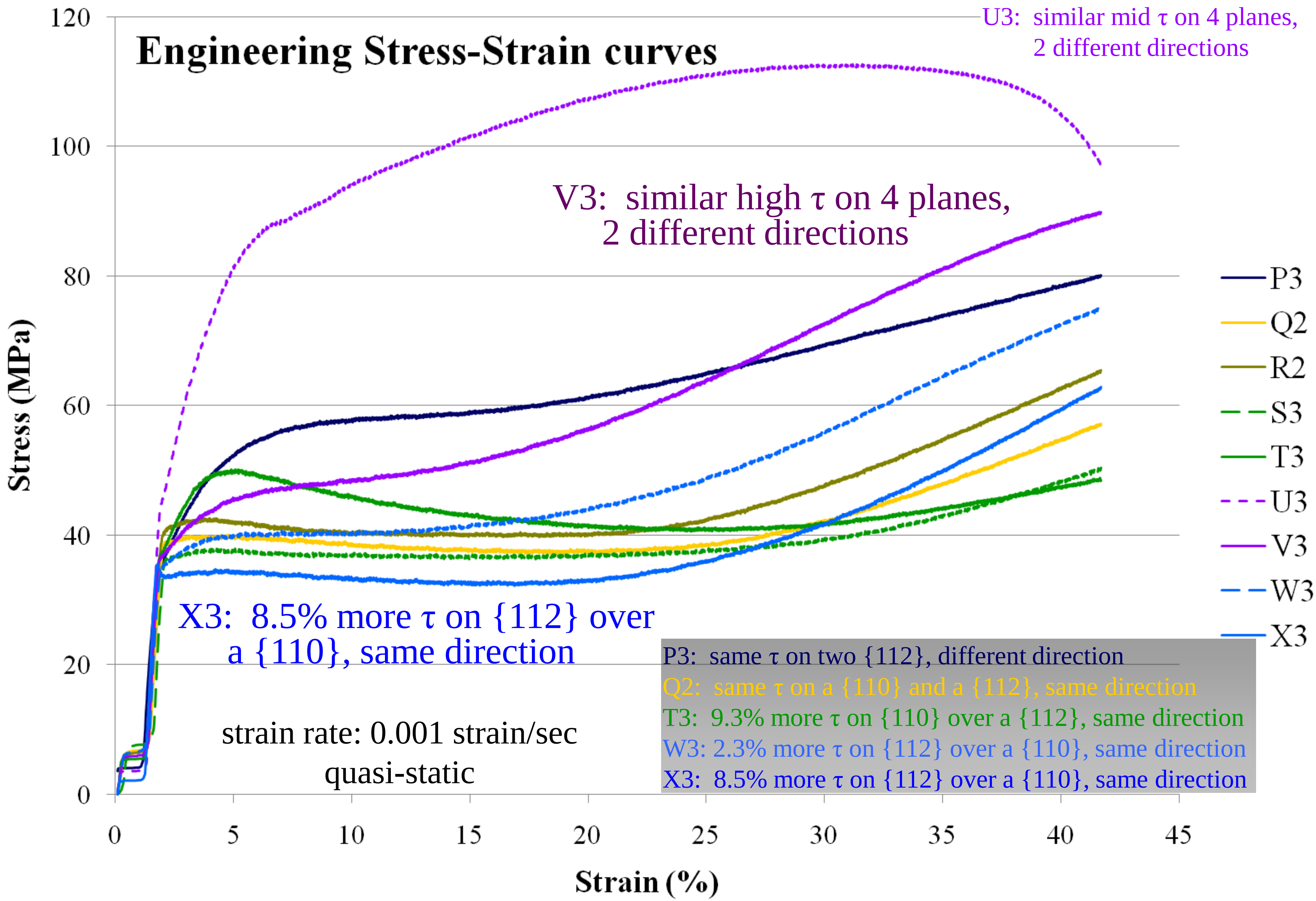
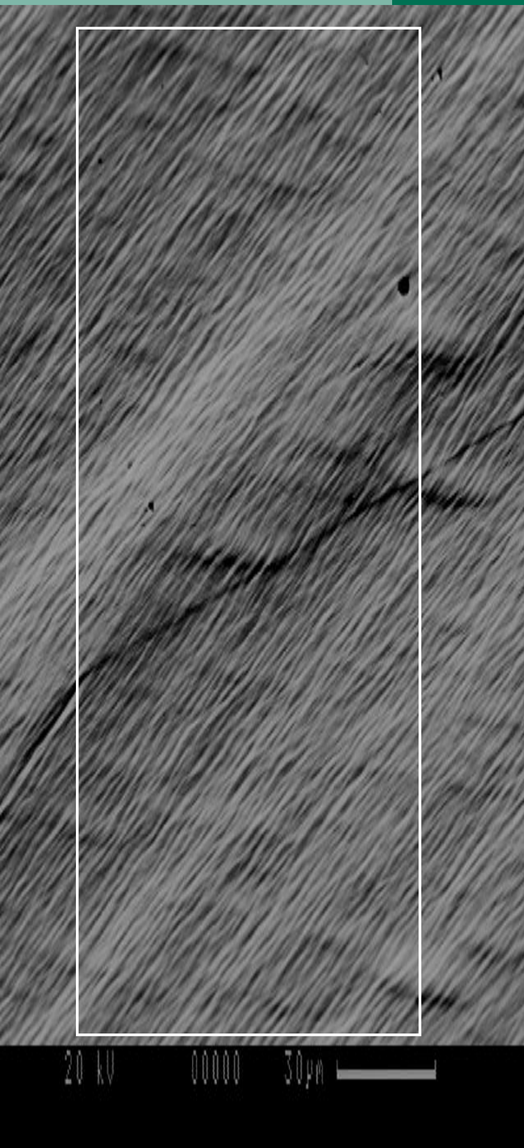
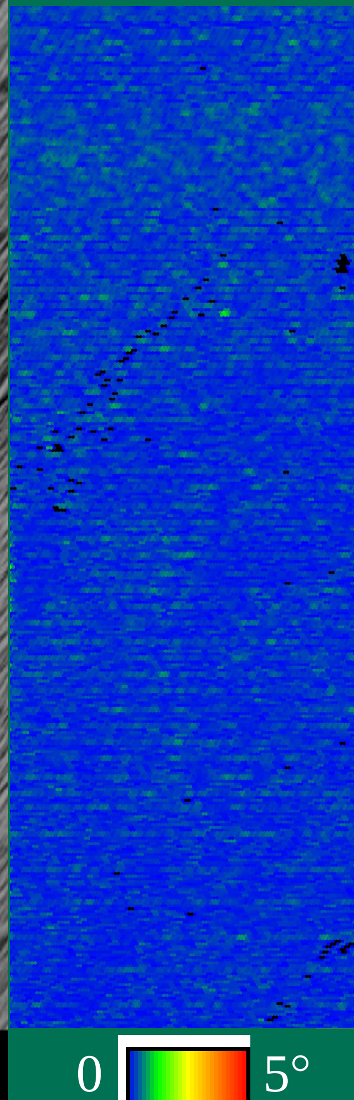
Tensile Test results



OIM data

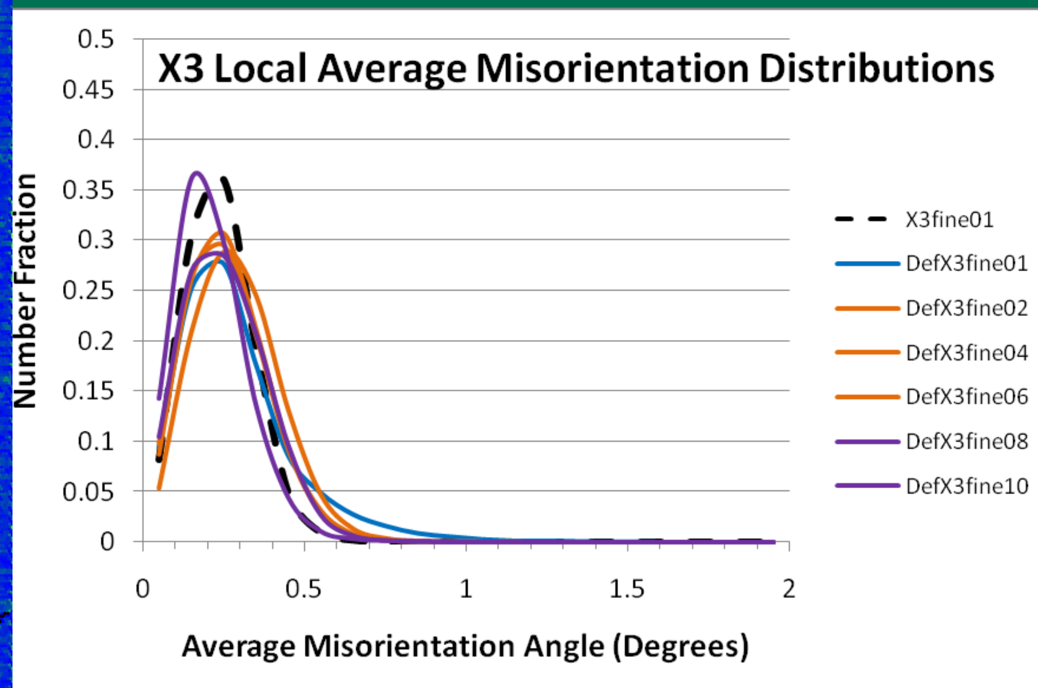
X3 Deformed





0 5°

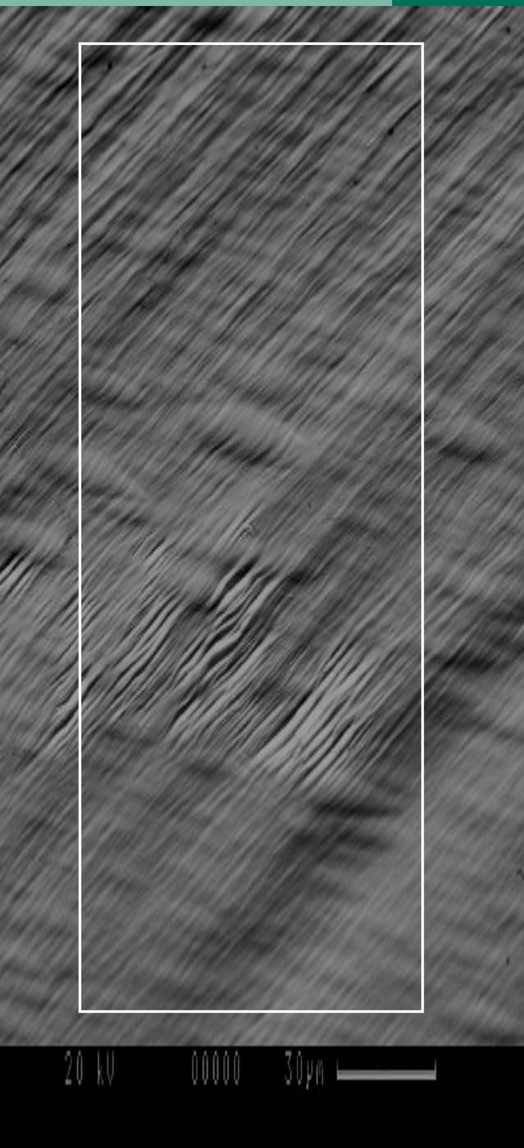
Less local lattice rotation (implies fewer GND) consistent with lower work hardening



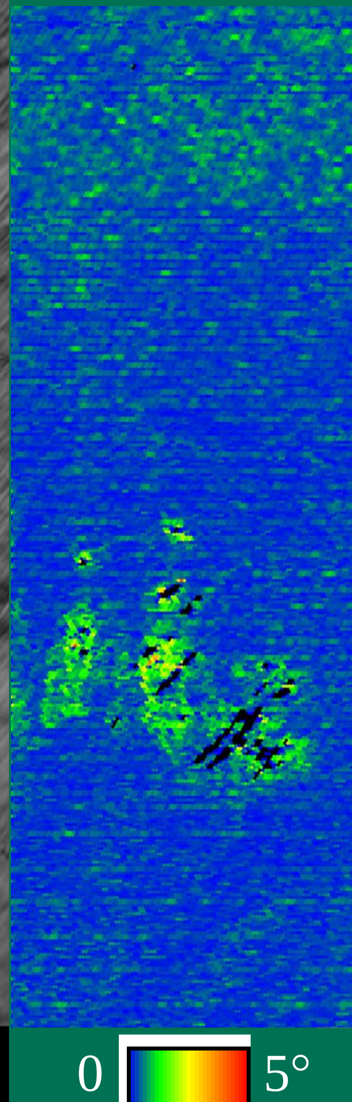
1 µm step size,
112x327 µm area

Local average misorientation
First neighbor (1 µm)

V3 Deformed

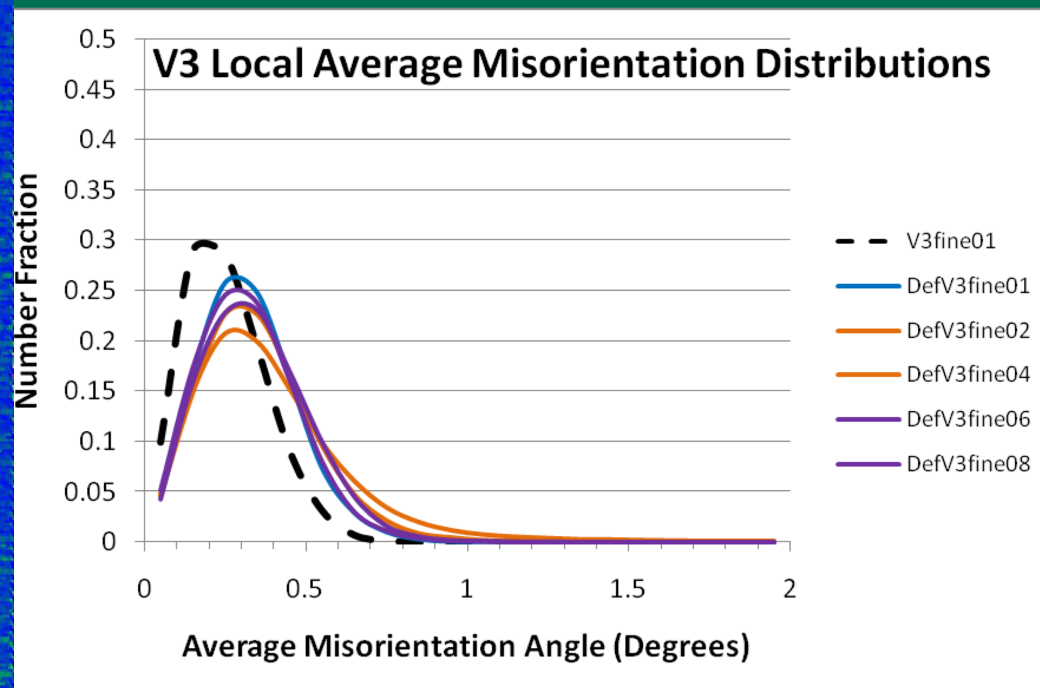


1 µm step size,
112x327 µm area



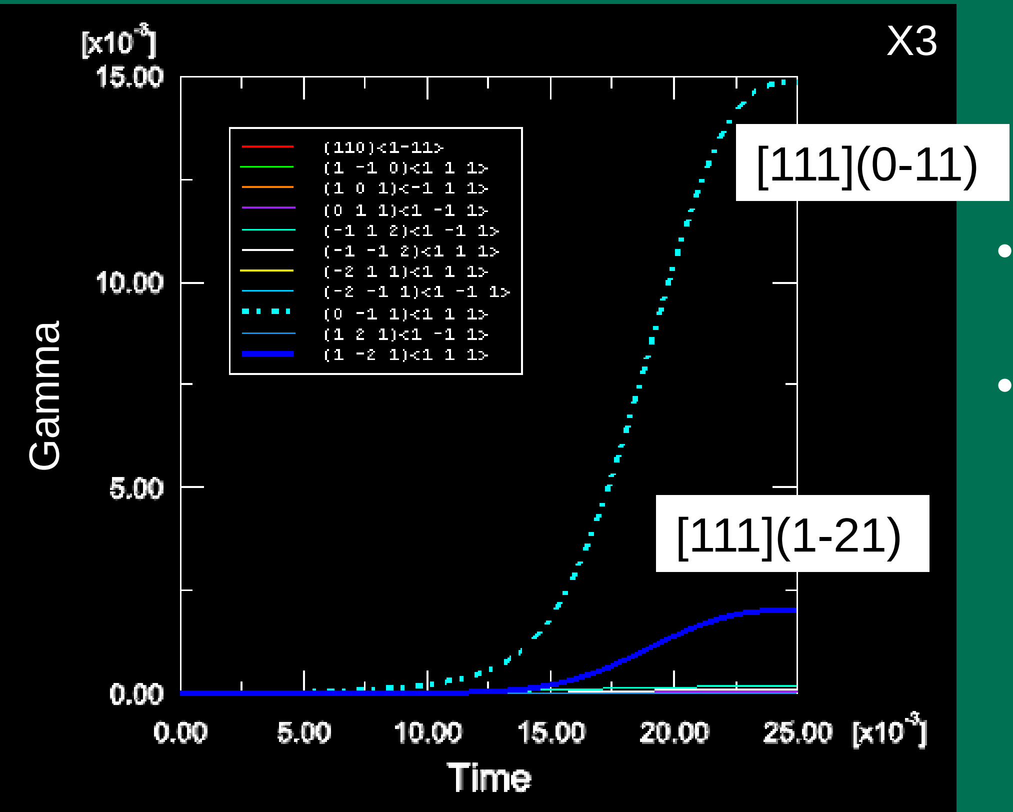
Local average misorientation
First neighbor (1 µm)

More local lattice rotation (implies more dislocations) consistent with higher work hardening



Predicted slip systems using crystal plasticity model

- Input initial crystal orientation, parameters from curve fitting real stress-strain data
- Imposes a fixed displacement for each time step, determines the stress-state
- Determines which slip system(s) require least potential energy to accomplish shear
- Updates work hardening of slip systems used, re-determine stress-state
- Iterate to optimize for deformation of each element in finite element mesh
- In this case, set {112}'s yield stress 20% higher than {110}; merely a starting point



- Model agrees with initially biased slip systems
- Careful adjustments to material parameters must be made based on physically observed systems

(P. Darbandi 2009)

Summary

- Chose niobium single crystal orientations biased toward activating certain slip systems
  - Simulate deformation using optimized finite element crystal plasticity model, to predict active slip systems
  - Directly determine active slip systems using slip traces (some orientations not so cooperative)
  - Directly determine active slip systems by microdiffraction of depth-resolved synchrotron x-rays, compare to simulations
- Determine if rotated regions persist to become recrystallized grains during welding/heat treating
- Relate active slip systems to recrystallized orientations
- Careful microstructure study from 'simple' real test → Getting reasonable material parameters for CPFEM → simulations of more complicated forming (hydroforming etc.)