Engineering Diffraction 102 : Understanding Polycrystalline Deformation

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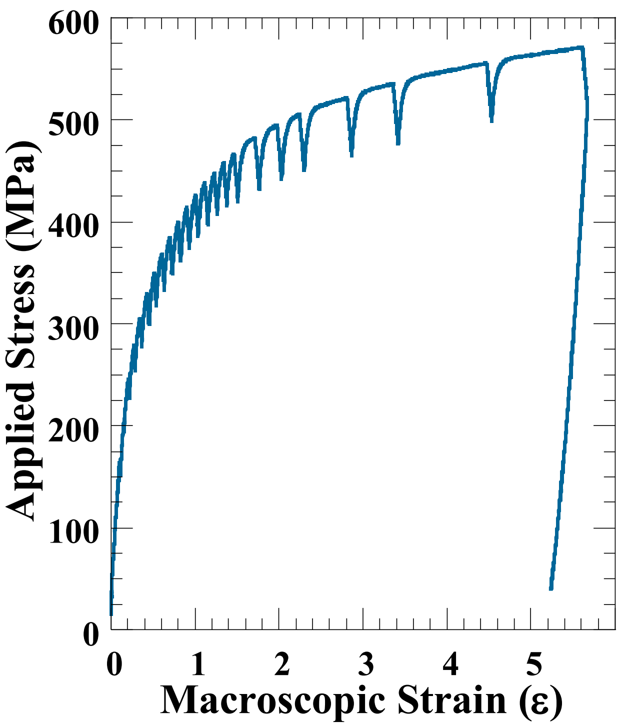
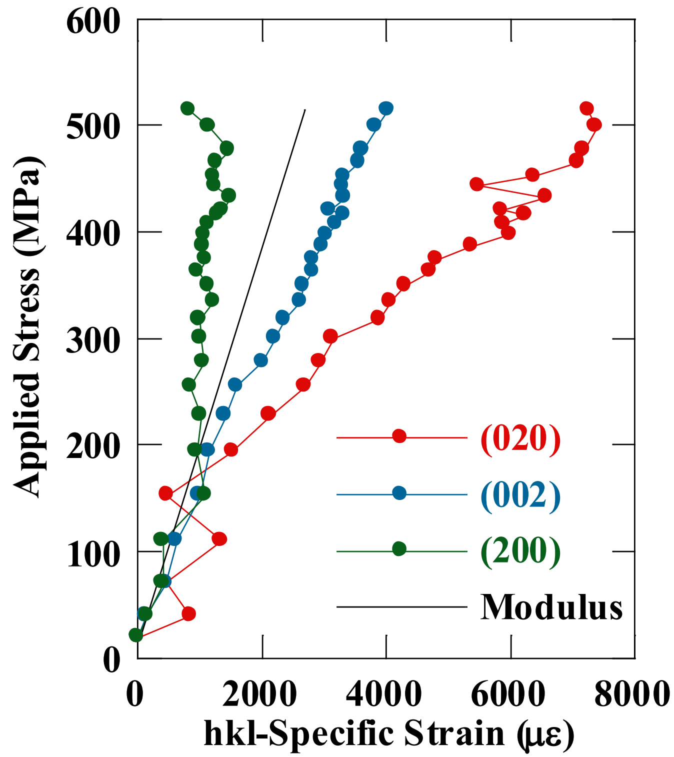
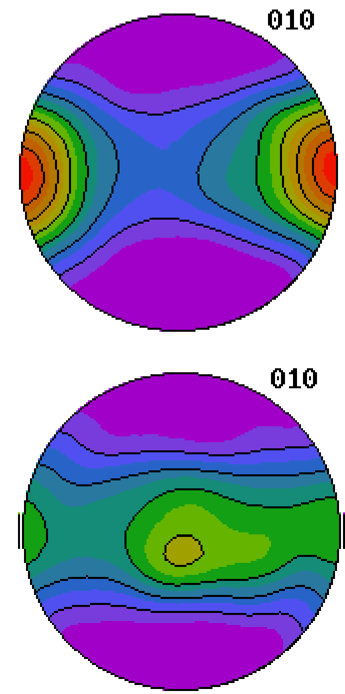
Why Perform Measurements of Internal Stress and Texture During Deformation







- Macroscopic flowcurve tells us about mechanical properties.
  - Yield strength, hardening…
  - Nothing about what is happening microscopically.
- The evolution of internal stresses and texture are signatures of the micro-mechanical deformation modes.

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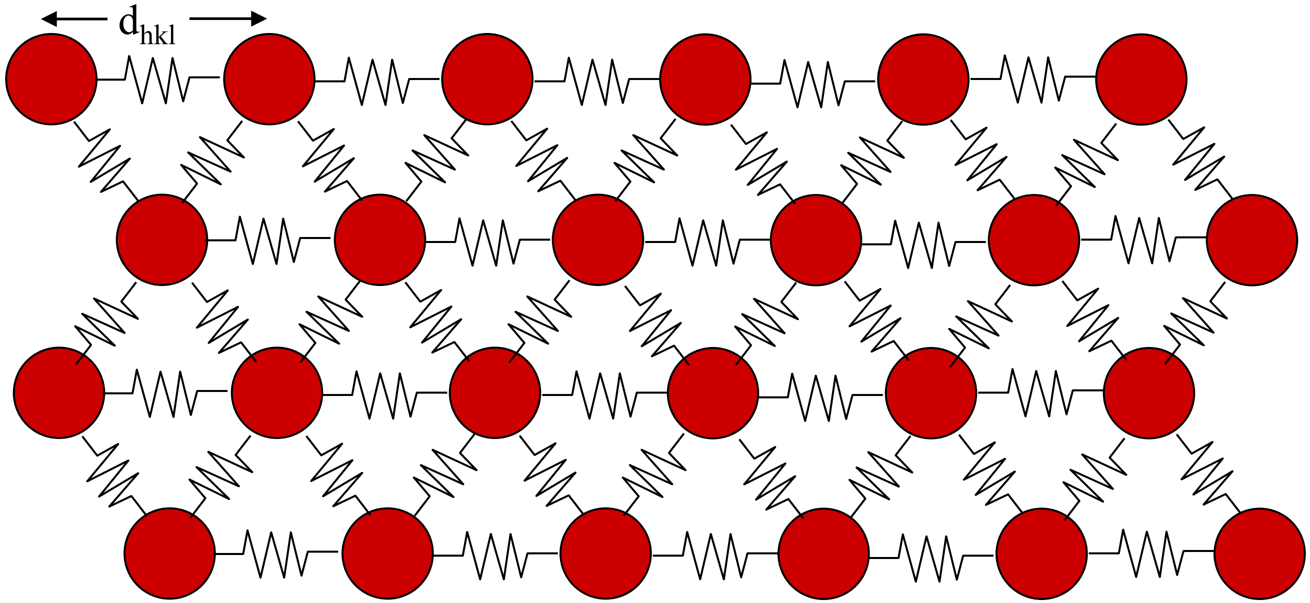
SMARTS is a 5 Million Dollar Bathroom Scale



- We measure the spacing between atoms very precisely: ~1 part in 10^5.
- Calculate lattice strains (elastic) from change in atomic spacing.

$$\varepsilon = \frac{d_{hkl} - d_0}{d_0}$$

- This is the elastic strain only !!! We cannot directly measure plastic strain.
- It is important to note that the lattice strain is necessarily proportional to the stress on the grain, not the macroscopic stress.
- Two types of measurement:
  - You know the stiffness tensor and want to determine unknown stress
  - You control the stresses and you want to learn about the springs (bonds)

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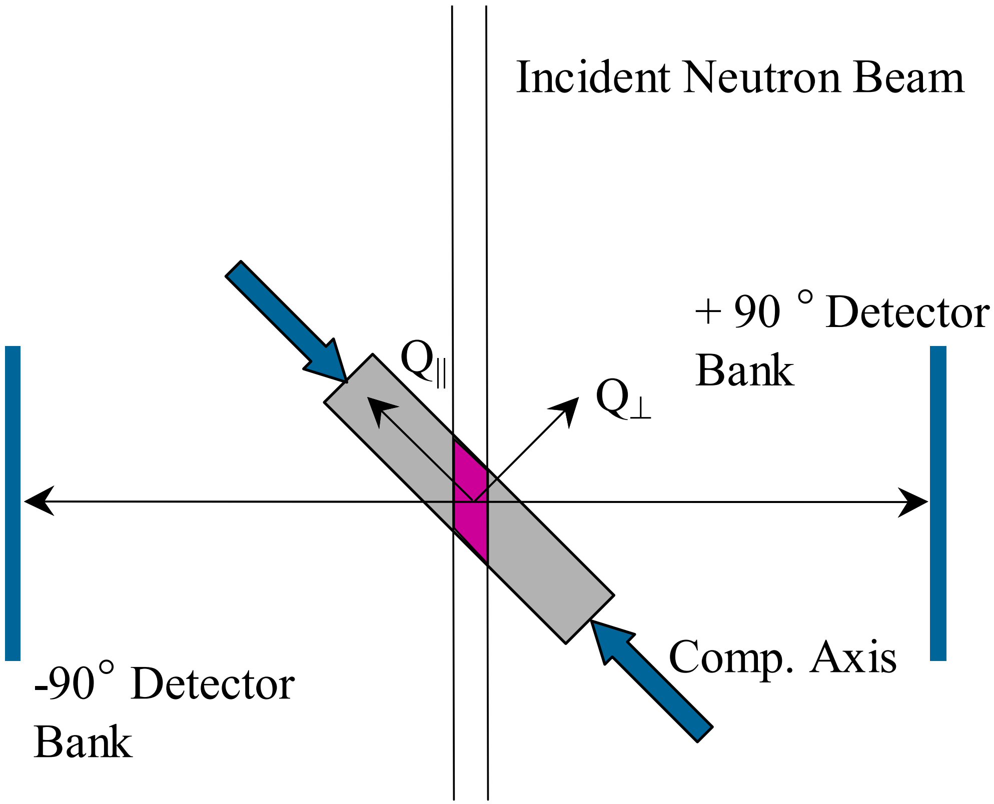
Reminder of the SMARTS Geometry



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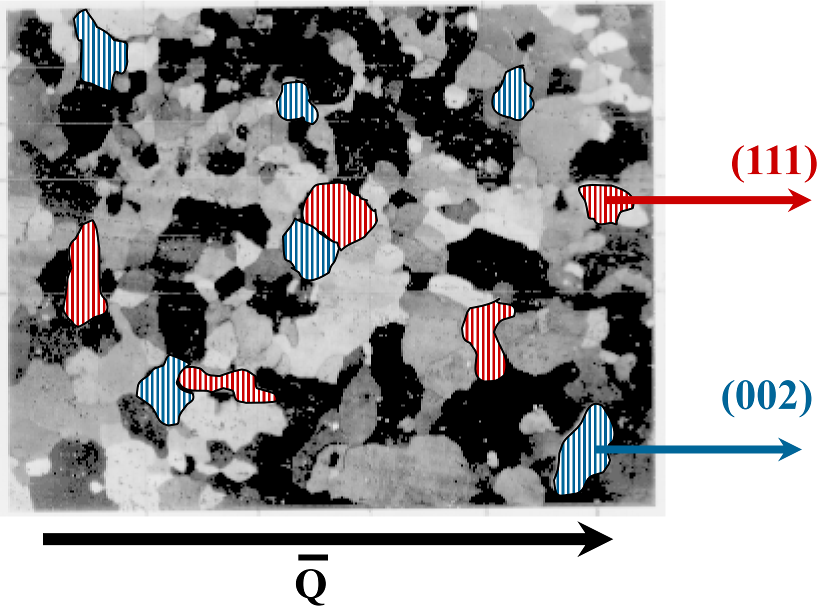
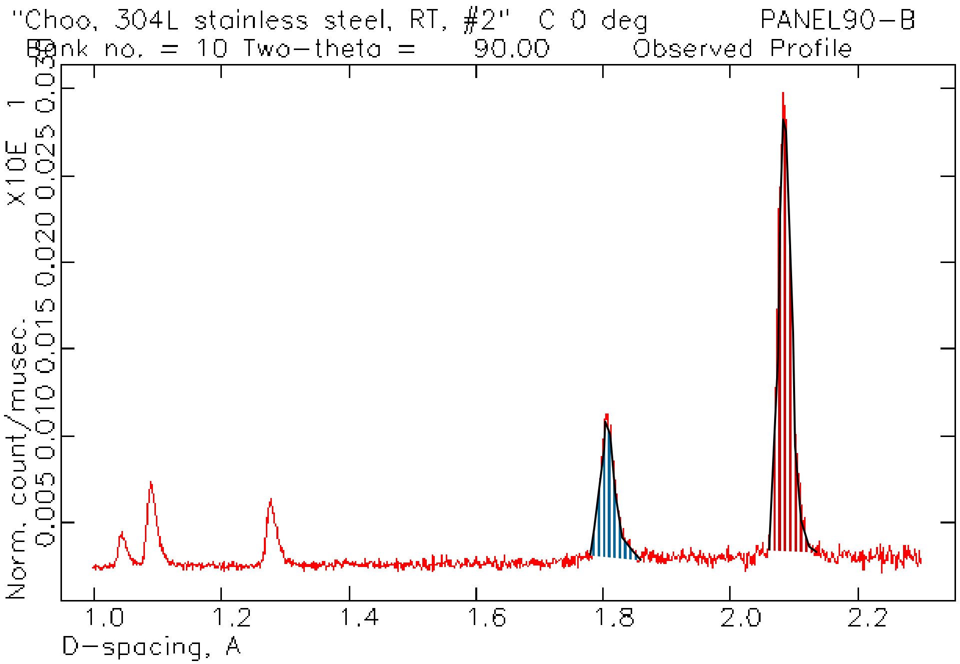
Diffraction Separates Response of Grain Orientations

Polycrystalline Aggregate



Stainless Steel



- **Grains with plane normals parallel to the diffraction vector defined by the instrument geometry diffract into a detector.**
- **Each grain orientation (hkl), or phase, contributes to a distinct peak, given by the interplanar spacing.**
- **We explicitly make the assumption that a family of grains can be used to represent the macroscopic stress field.**

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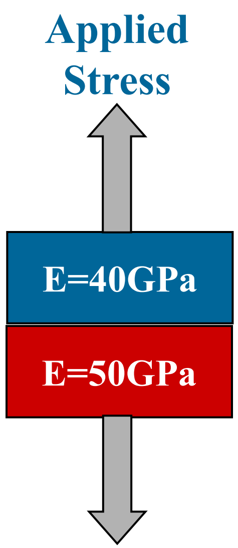
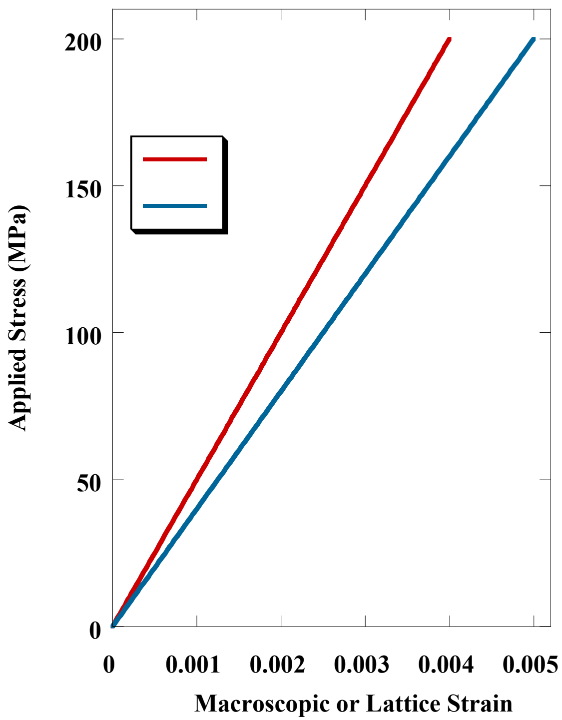
Consider the case of a bi-metallic sample : Elastic loading in series

*(rubber band demo)





Lattice strain $=(d-d_0)/d_0$

Macroscopic strain $= (l-l_0)/l_0$

- Stress on each is the same, but strain varies.
- Lattice and macroscopic strain are equivalent.

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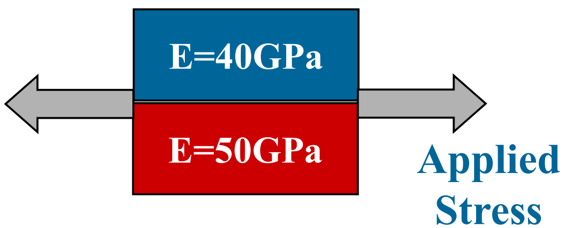
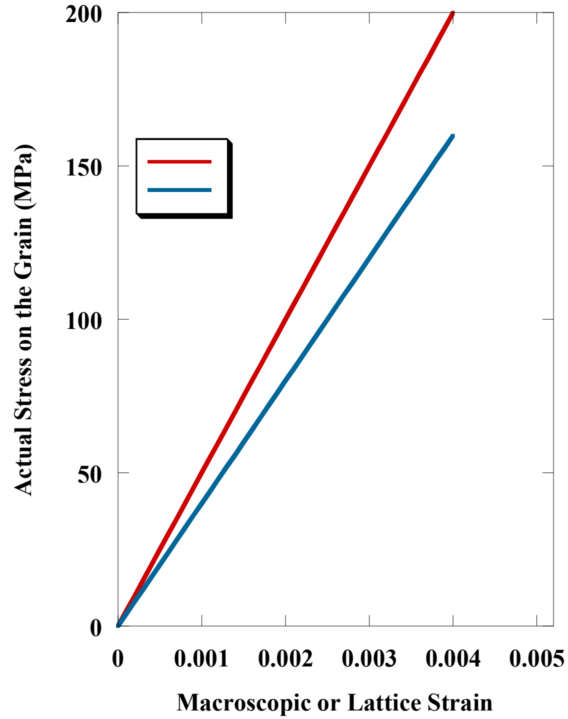
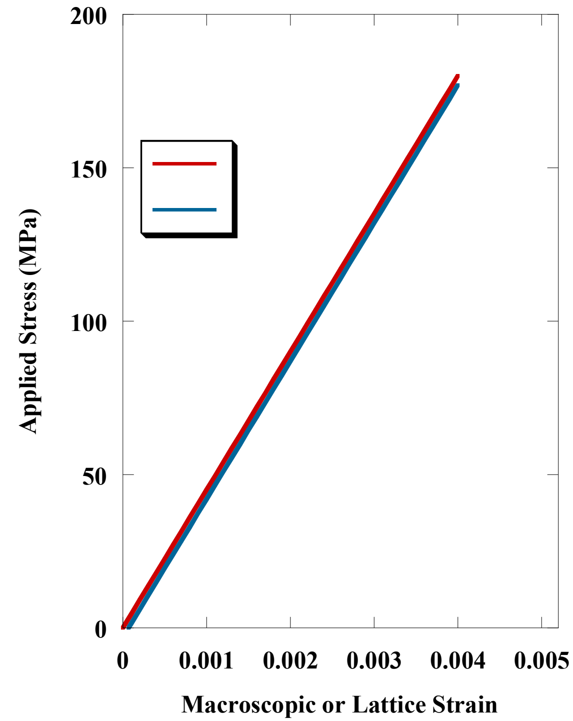
Consider the case of a bi-metallic sample : Elastic loading in parallel







- Stain on each is constant.
- But Stress=Modulus*Strain
- ∴ Actual Stress on each "grain" varies.
- We can only measure Applied Stress macroscopically.

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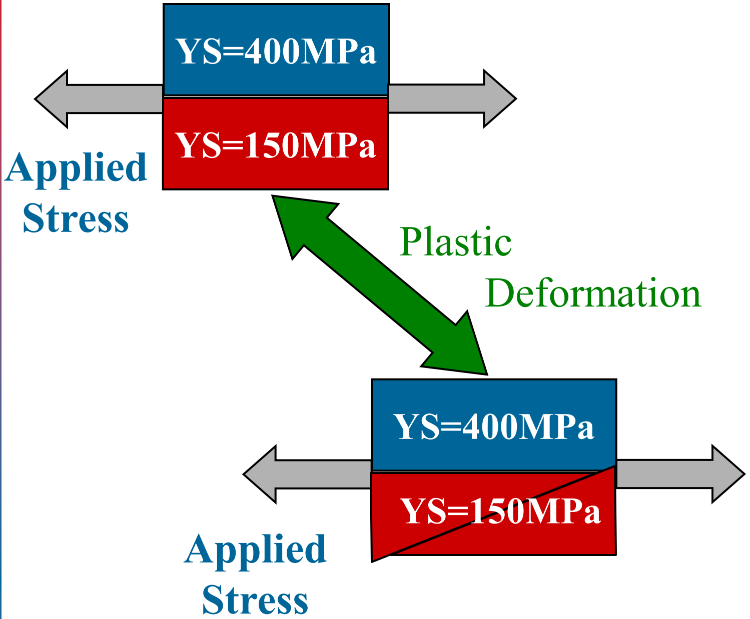
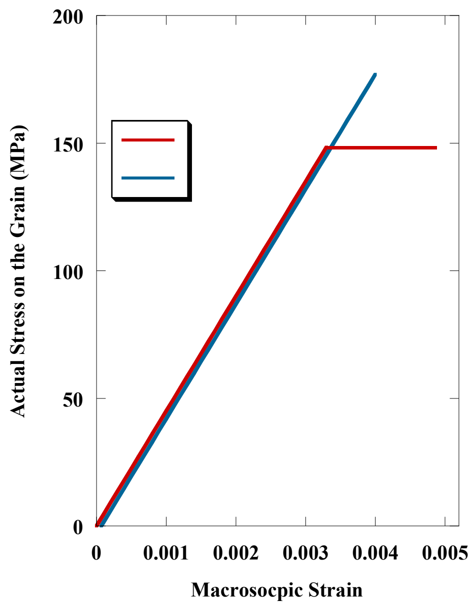
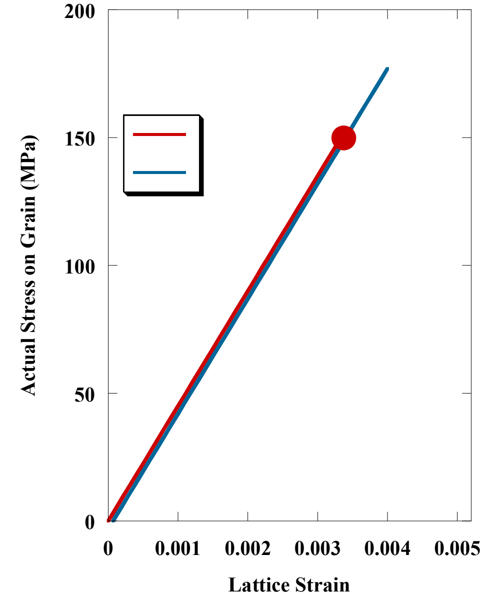
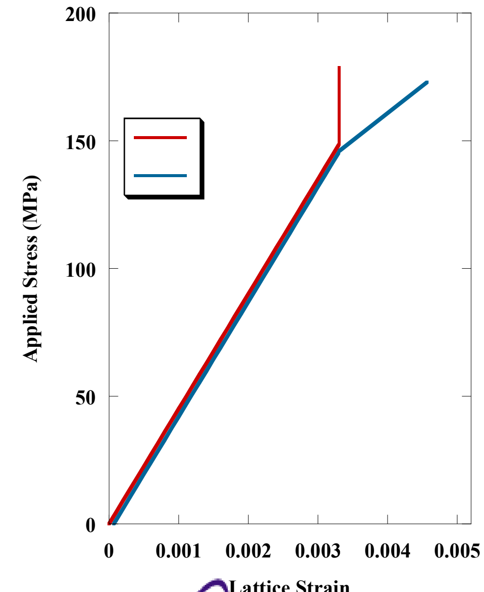
Consider the case of a bi-metallic sample : plastic loading in parallel.









- Plasticity : Macrocopic strain and lattice strain are no longer equivalent.
- Once one component yields, the stress on that component is fixed while macroscopic strain increases (perfect plasticity).
- Similarly, lattice strain is also fixed.
- Again, we can only measure Applied Stress vs Lattice Strain.
  - Characteristic Y shape associated with plastic deformation.
  - Deviatiation of lattice strain from linear is the “Intergranular Strain”.

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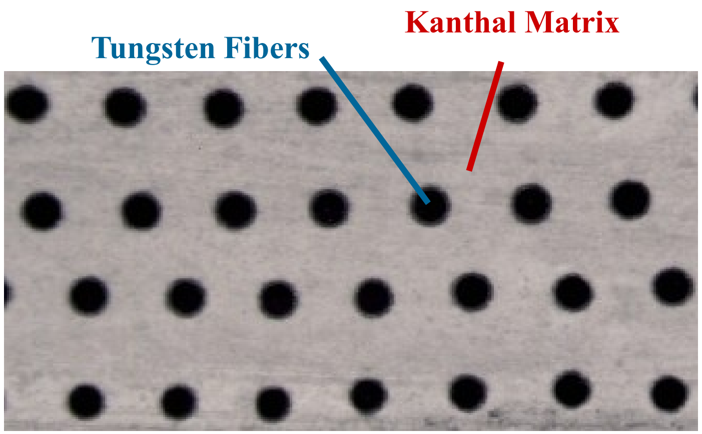
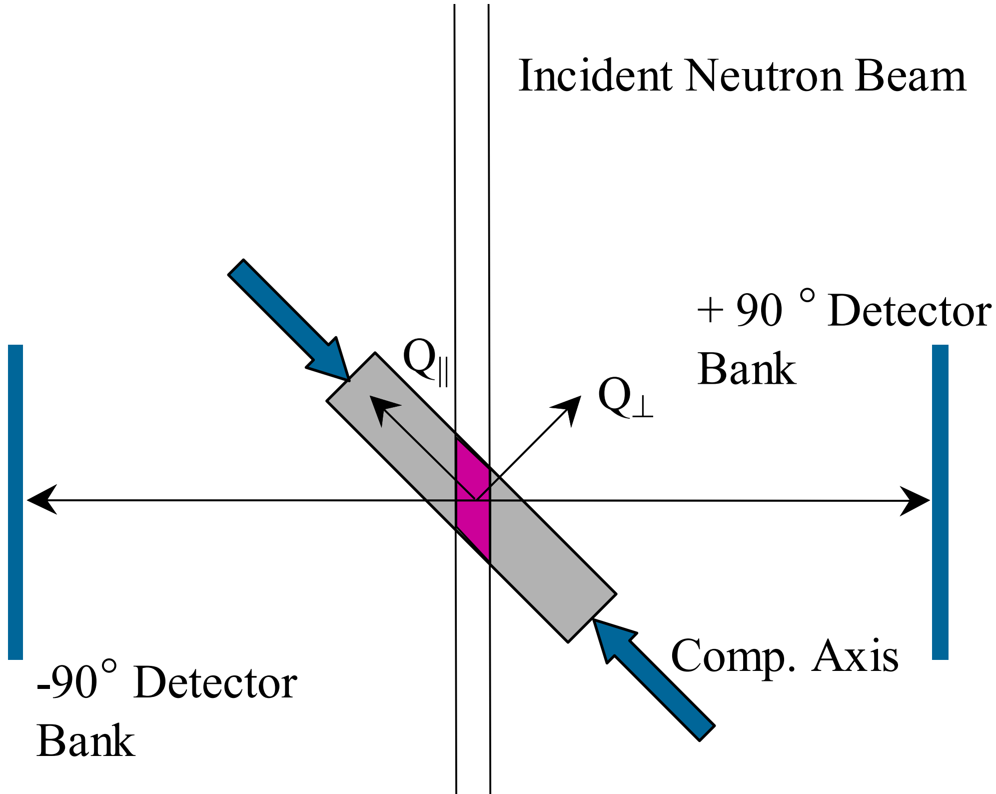
Lets Consider How a Composite Responds to Deformation



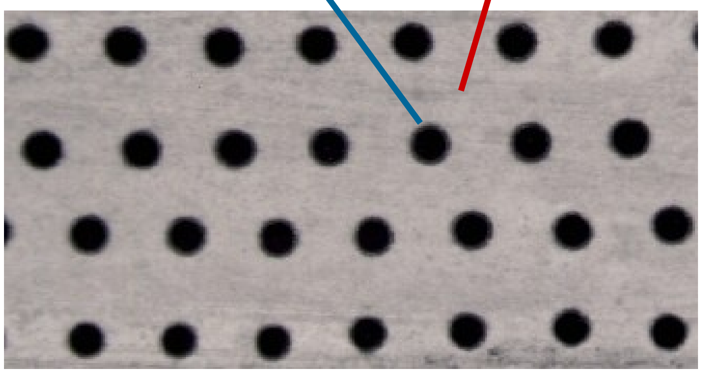


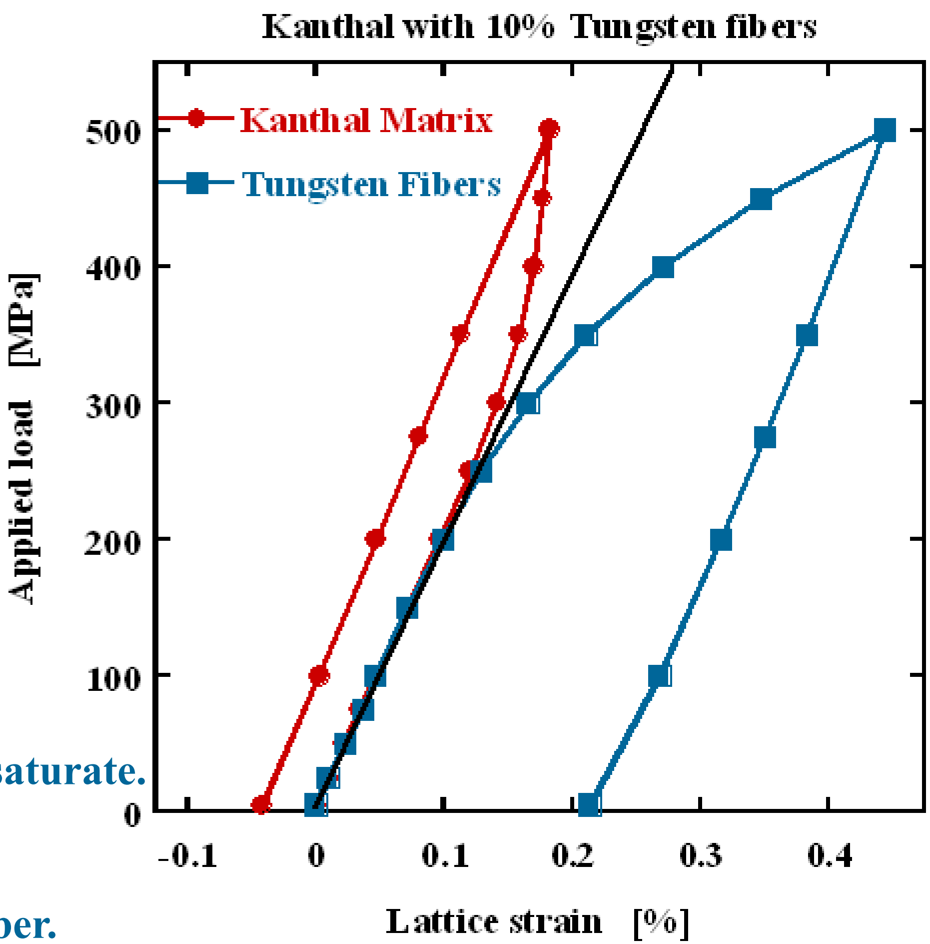
- Microstructure represents loading 2 constituents in parallel, total strains must be equal.

$$\varepsilon_T = \varepsilon_e + \varepsilon_p$$

- In elastic regime, lattice strains are equivalent.
- Once plasticity begins in one phase, the elastic lattice strains are no longer constrained to each other.

Understand Anisotropy in Terms of a Composite





- In elastic region, the strains are the same.
- Above yield point, elastic strains in Kanthal saturate.
    - It is yielding.
    - Load is redistributed to the Tungsten fiber.
- This is how a composite is designed to work.
- With release of the macroscopic stress, there is a residual stress in each constituent.
    - The phases stresses are not representative of the macroscopic stress state.
    - However, a weighted average would be representative.

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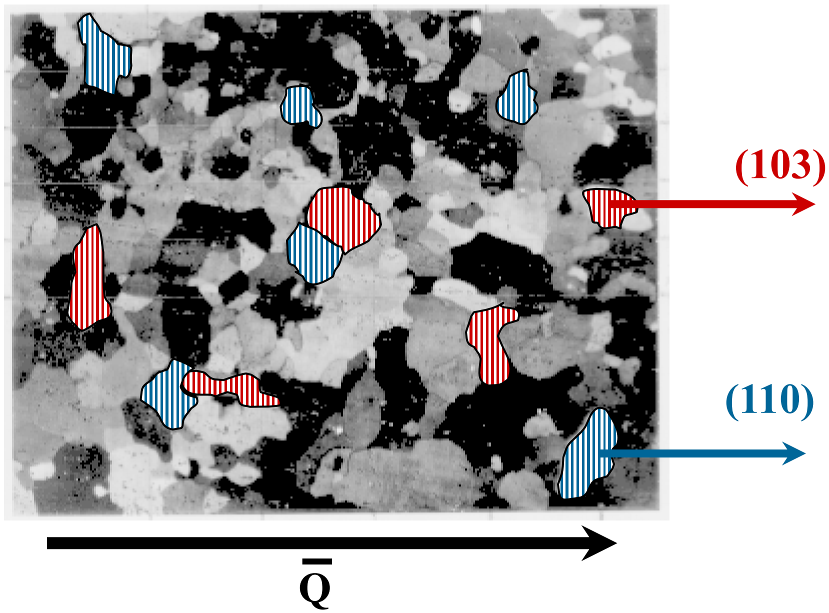
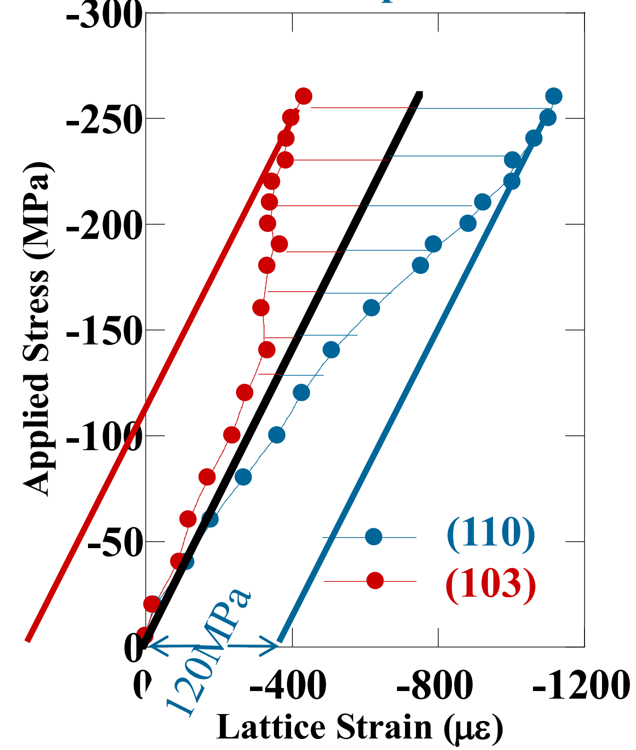
Polycrystalline Samples : “The Mother of All Composites”.

Polycrystalline Aggregate



Be in Compression



- Above yield point, elastic strains in (103) orientation saturate.
  - Grains with (103) parallel to the load direction are yielding.
  - Load is redistributed to the (110) orientations.
- With release of the macroscopic stress, there is a residual intergranular stress in each grain set.
  - The (hkl) stresses are not representative of the macroscopic stress state.
  - Moreover, the size of the intergranular stress changes with plastic strain

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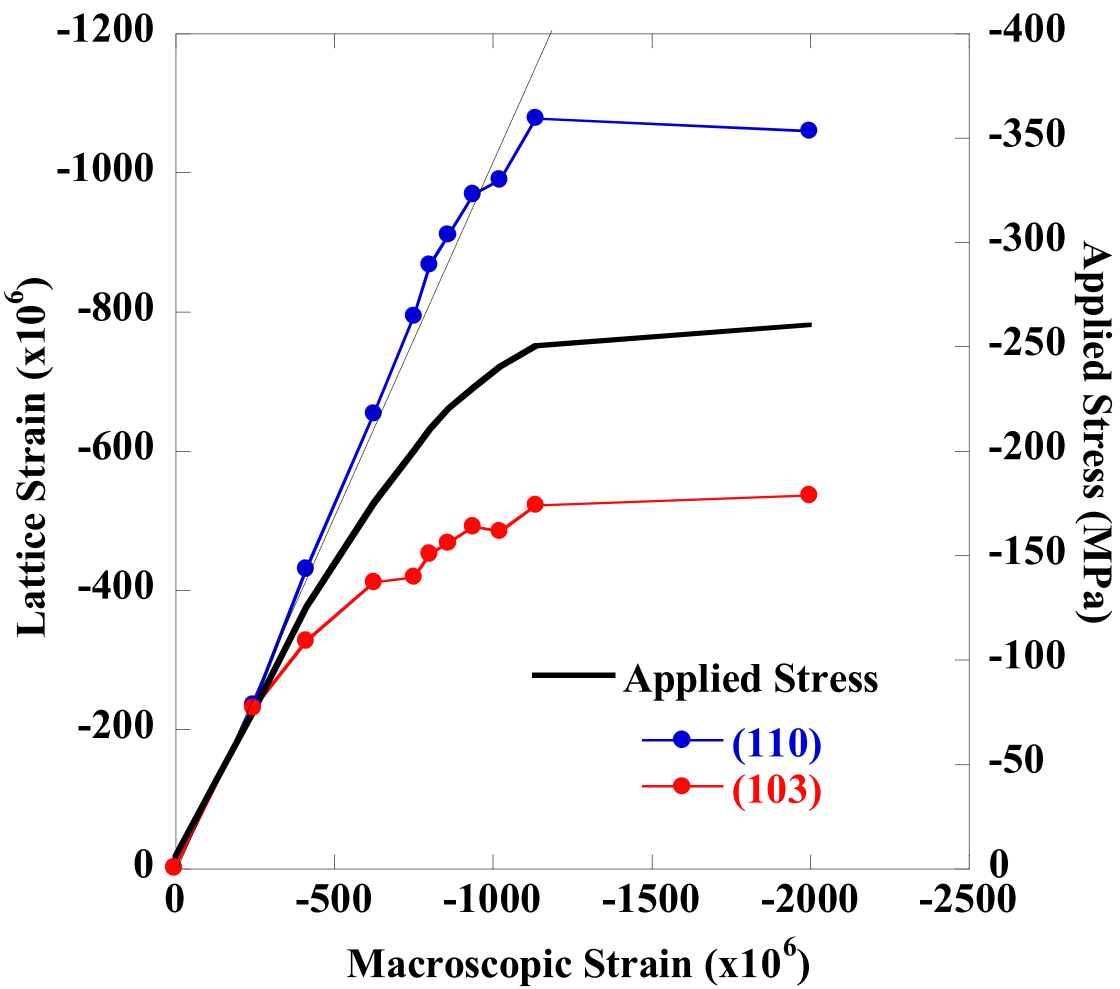
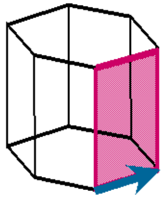
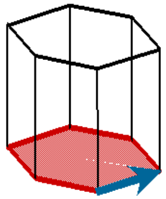
Lets Look at the Data in a Different Way.







- There is an heterogeneous distribution of stresses.
- Grains with (110) parallel to applied stress support more of the load once plasticity begins.

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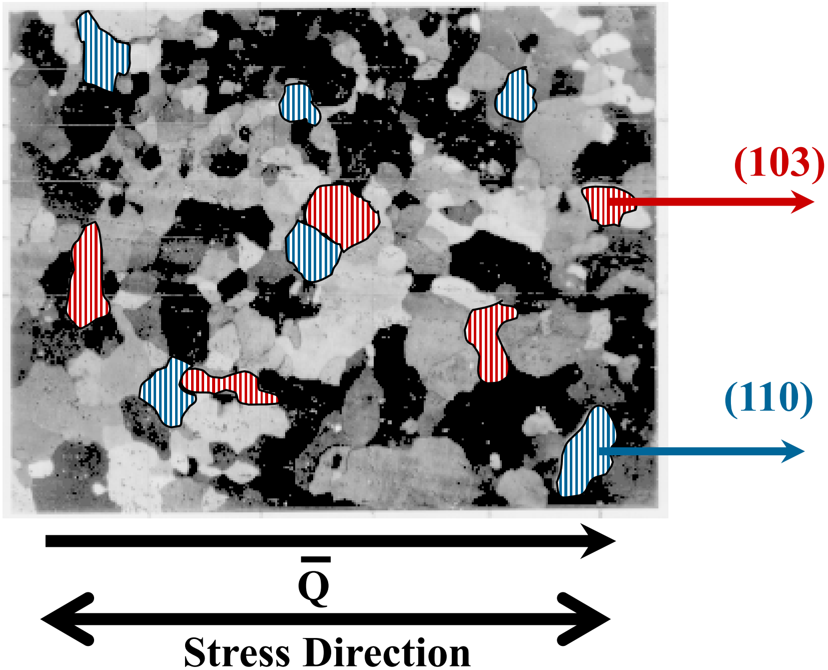
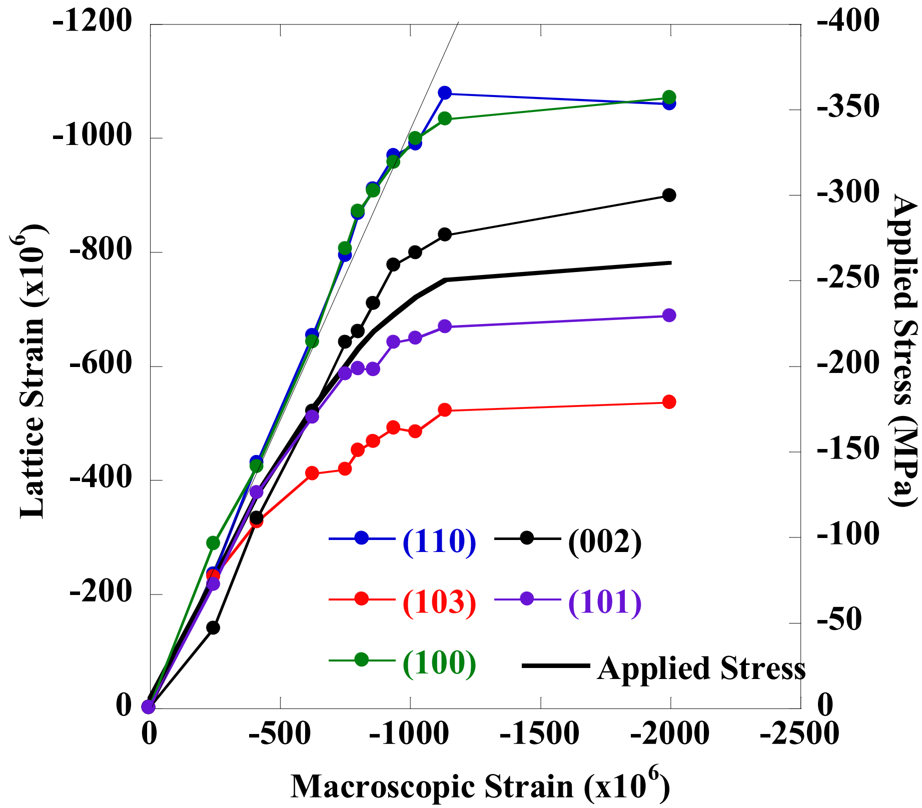
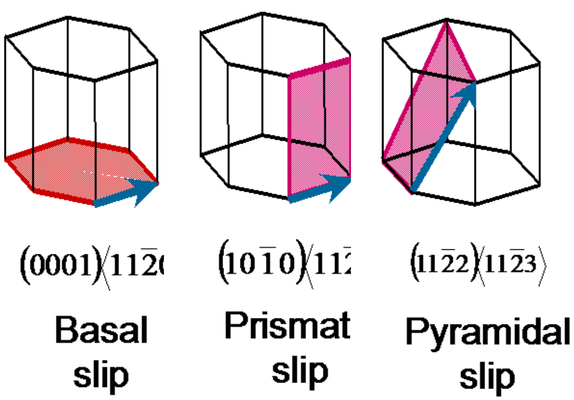
What Can We Infer About Deformation Modes ?

Polycrystalline Aggregate



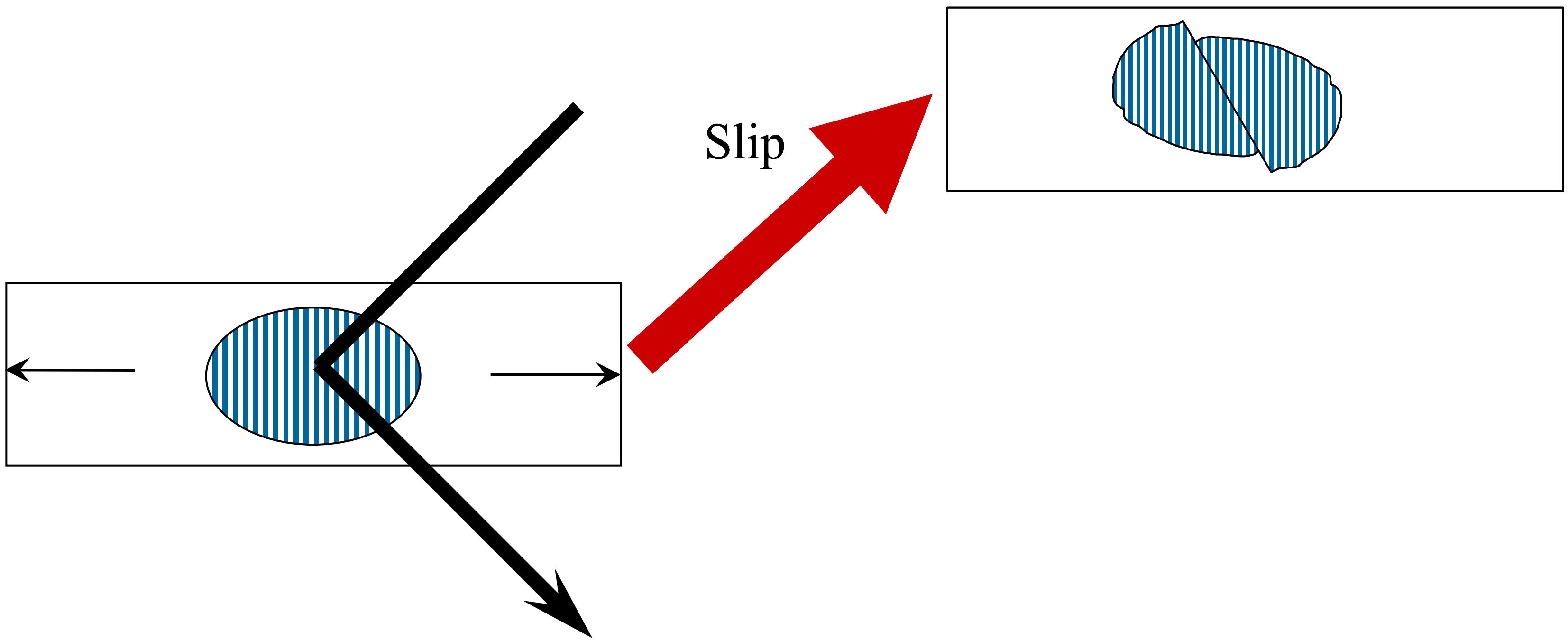




Basal slip is activated first.

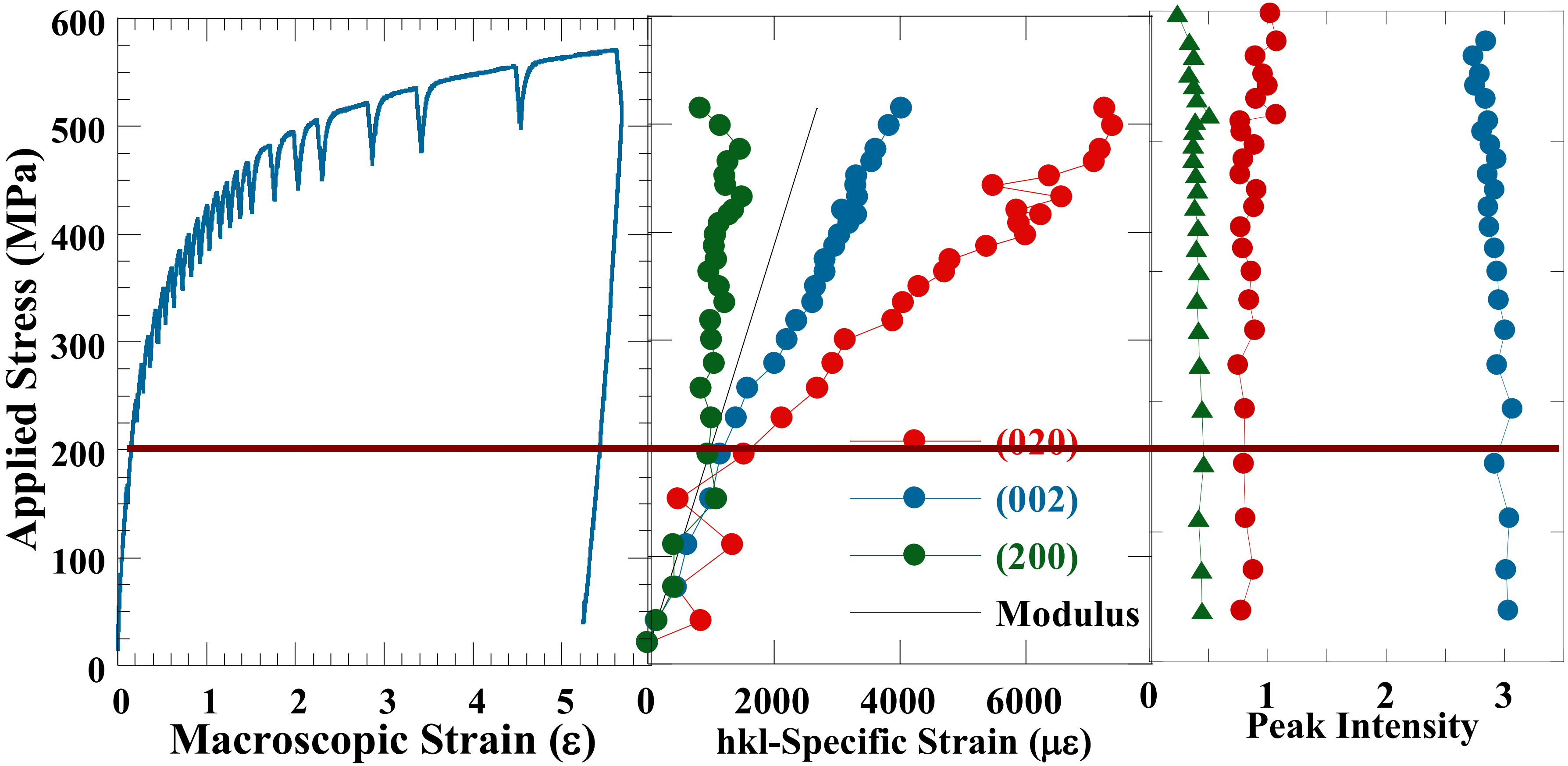
Pyramidal slip (or some mode that relaxes the c-axis) ıust activate.

Plastic Deformation Mechanisms Have Distinct Diffraction Signatures



• Slip : little if any change in peak intensity, broadening proportional to dislocation density.

Plastic Deformation of Uranium



- Deviation of lattice strains from linearity indicates that plastic deformation has initiated.
- Lack of change of peak intensity suggests it is slip dominated.

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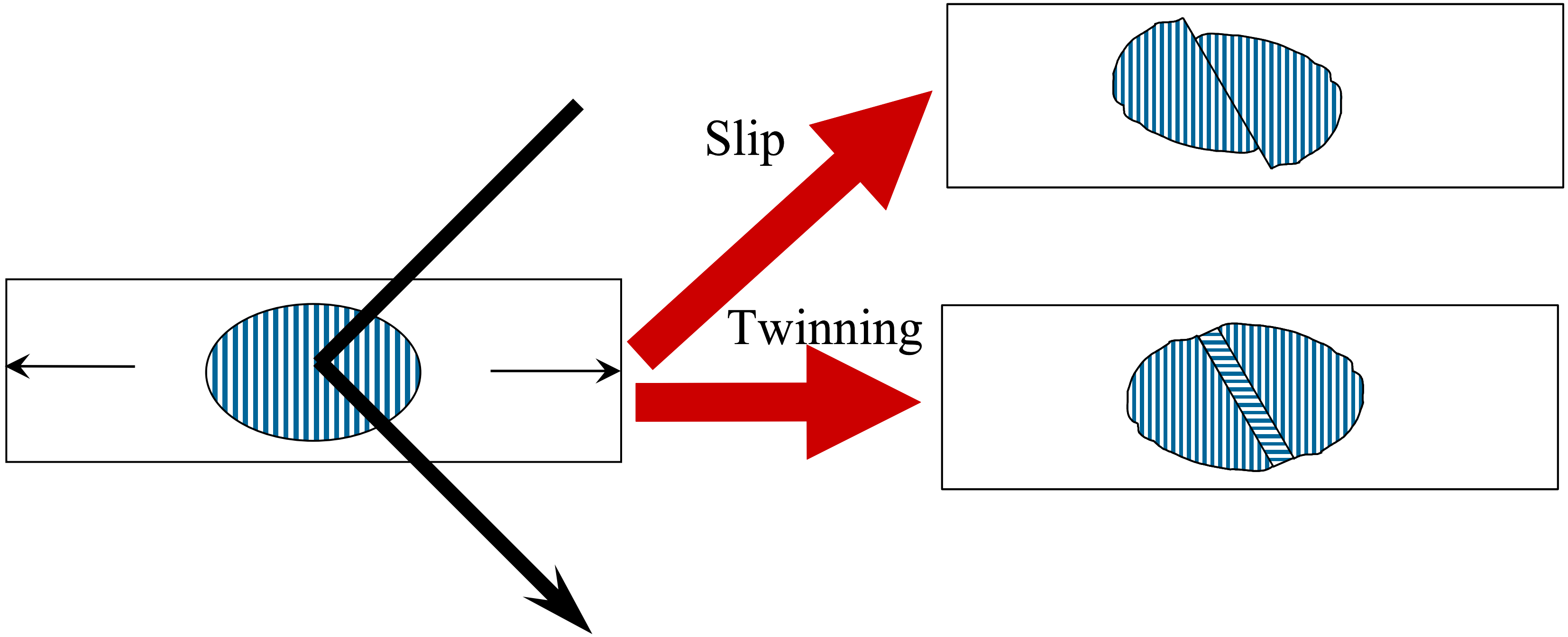
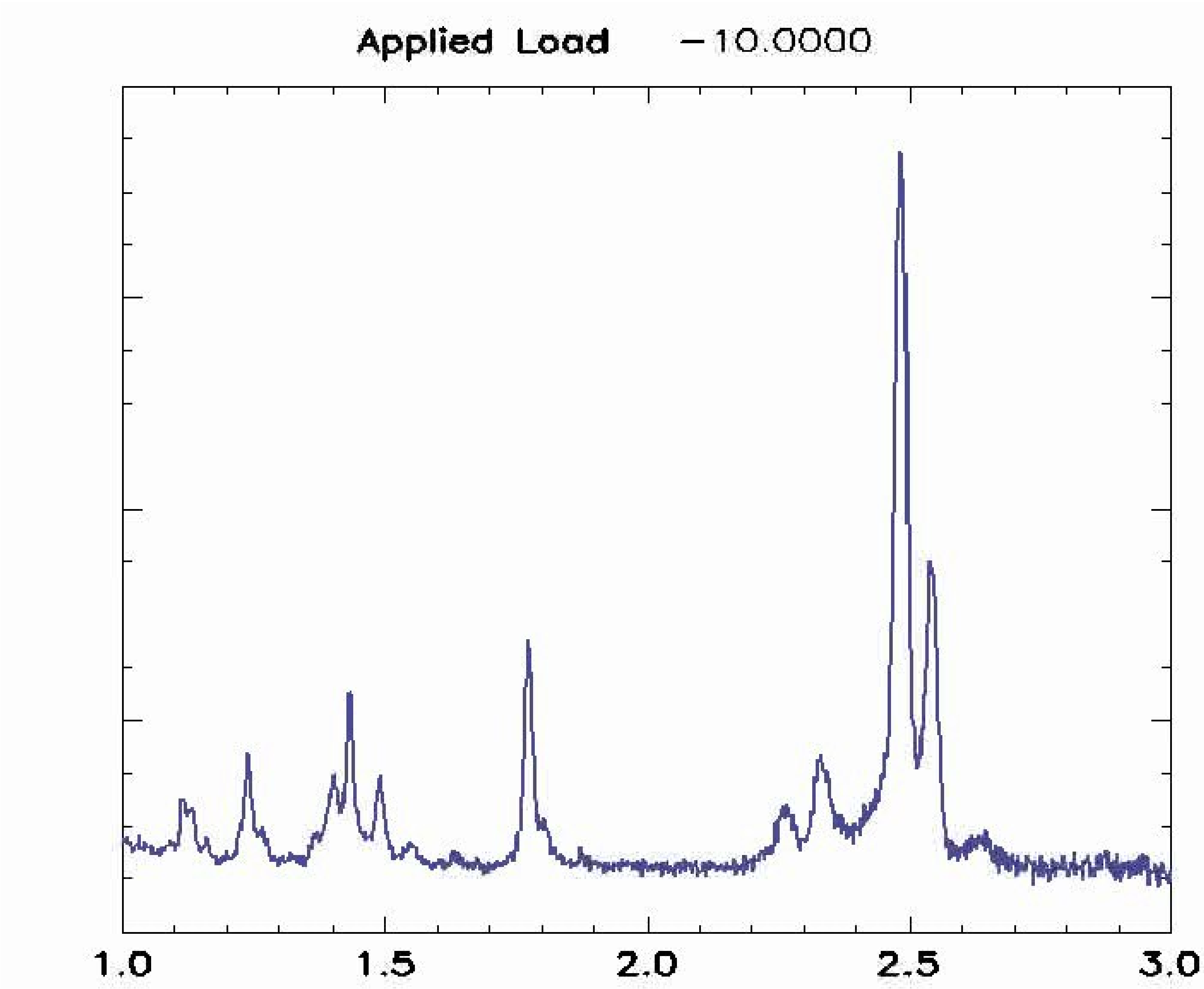
Plastic Deformation Mechanisms Have Distinct Diffraction Signatures

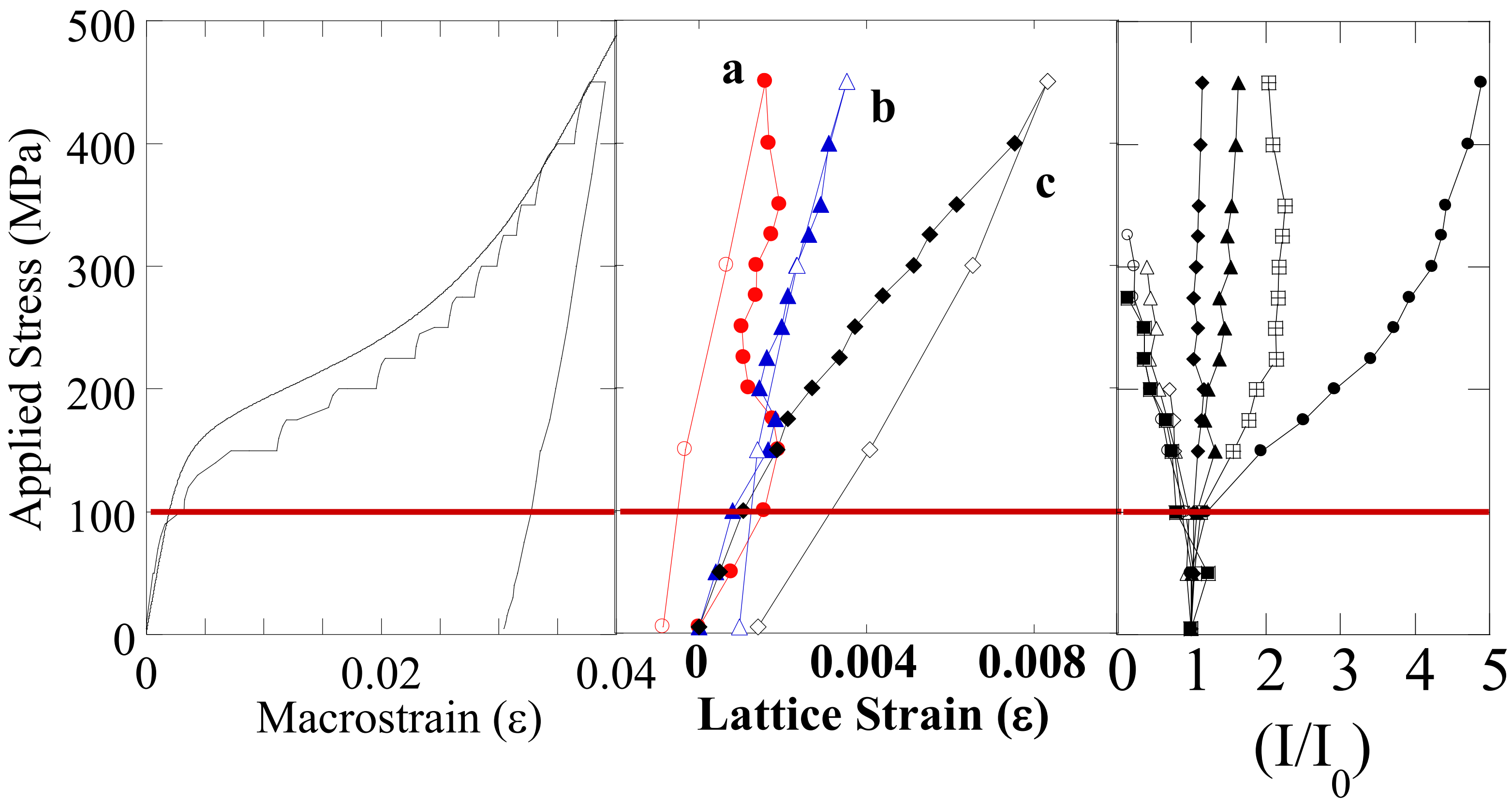


- Slip : little if any change in peak intensity, broadening proportional to dislocation density.
- Deformation twinning : large changes in single peak diffraction intensity.

Neutron Diffraction Indicates Twinning Reorientation During Deformation of U6Nb



Deformation Twinning is a Relaxation Mechanism for Twinning Grains

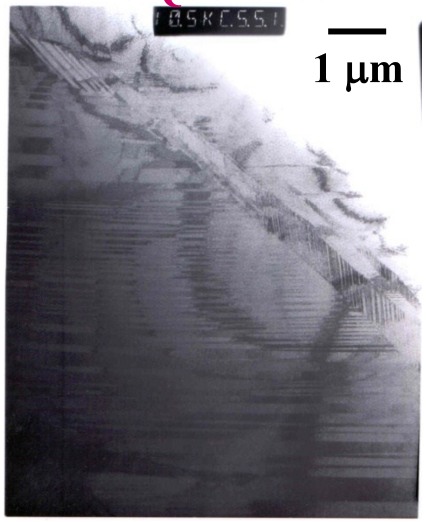


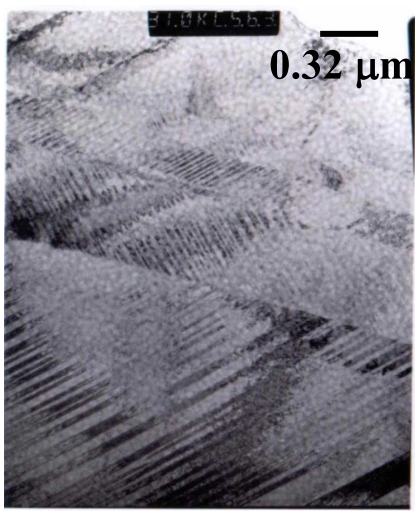
•Deviation of lattice strains from linearity indicates that plastic deformation has initiated.

• Significant change of peak intensity suggests it is twinning dominated.

TEM Provides Details of Deformation of U6Nb.

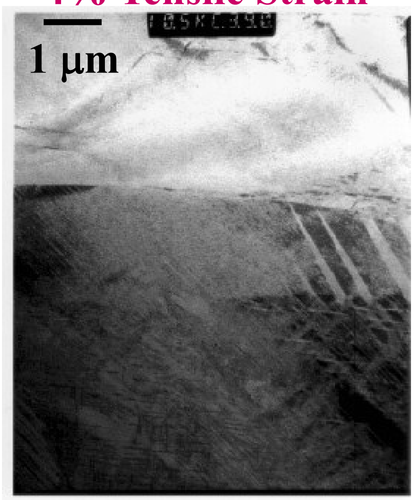
As-Quenched

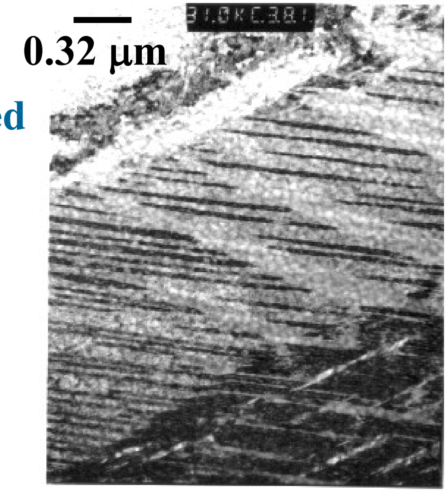




- As-Quenched
  - U6Nb Heavily Twinned.
  - (-130) Twin Boundaries
  - (021) Lath Boundaries.
- Post 4% Tensile Strain.
  - Large Single Orientation Areas.
  - (-172) Fat Lenticular Twins.
  - (-130) Fine Lamellar Twins.
- Growth and Assimilation of Preferred Variant.
- Nucleation of Deformation Twins.

4% Tensile Strain





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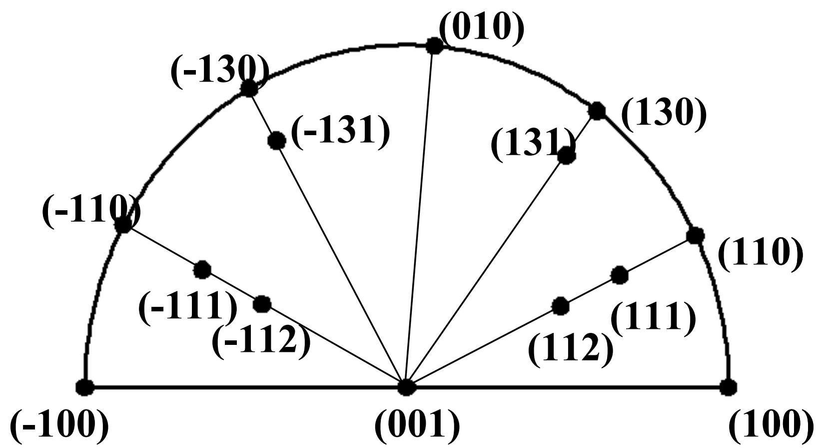
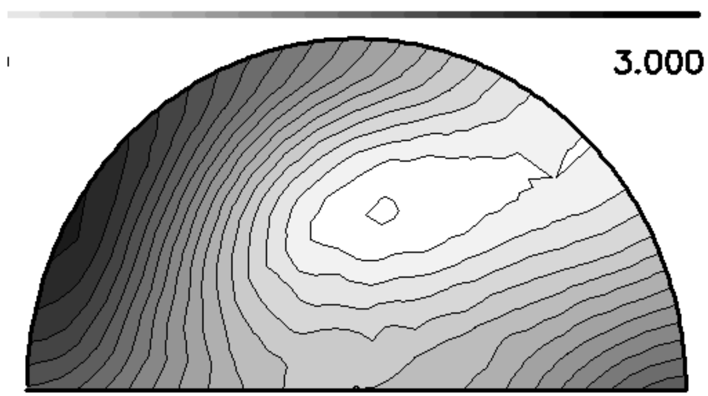
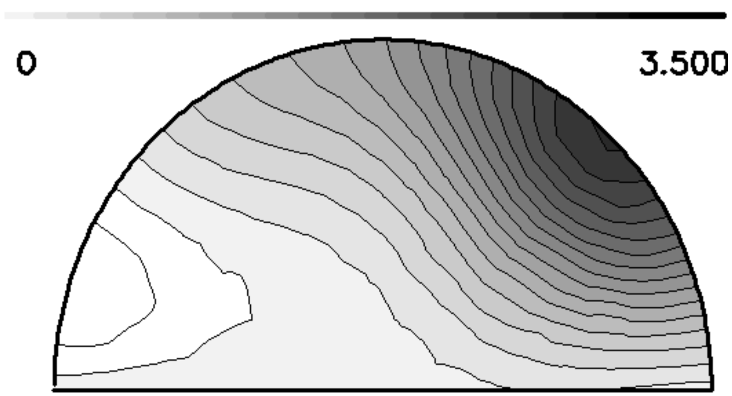
Texture Development During Deformation of U6Nb Indicates Deformation Twinning



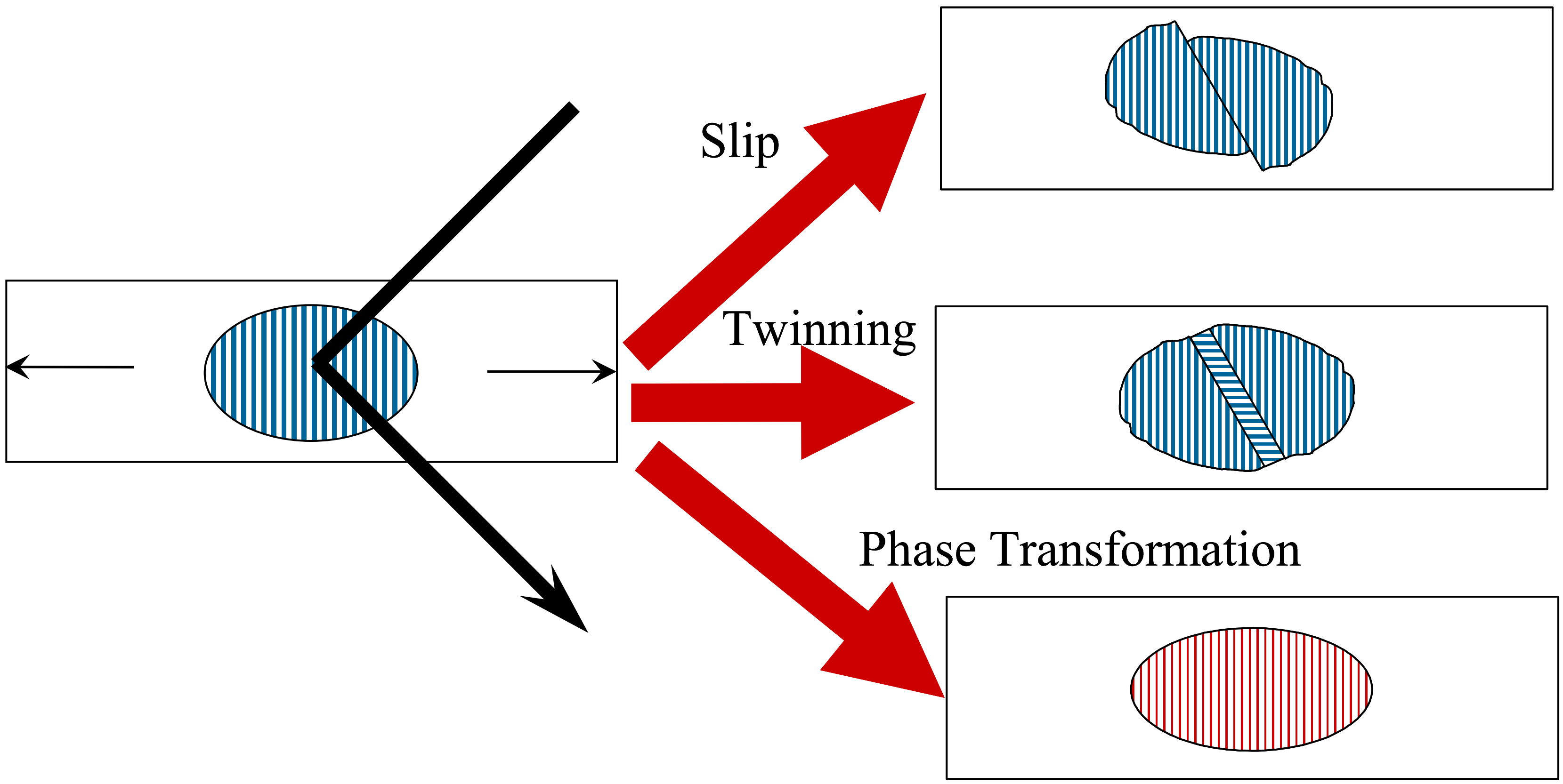


Compression



Tension

Plastic Deformation Mechanisms Have Distinct Diffraction Signatures



- Slip : little if any change in peak intensity, broadening proportional to dislocation density.
- Deformation twinning : large changes in single peak diffraction intensity.
- Phase transformation : appearance of new crystal symmetry.

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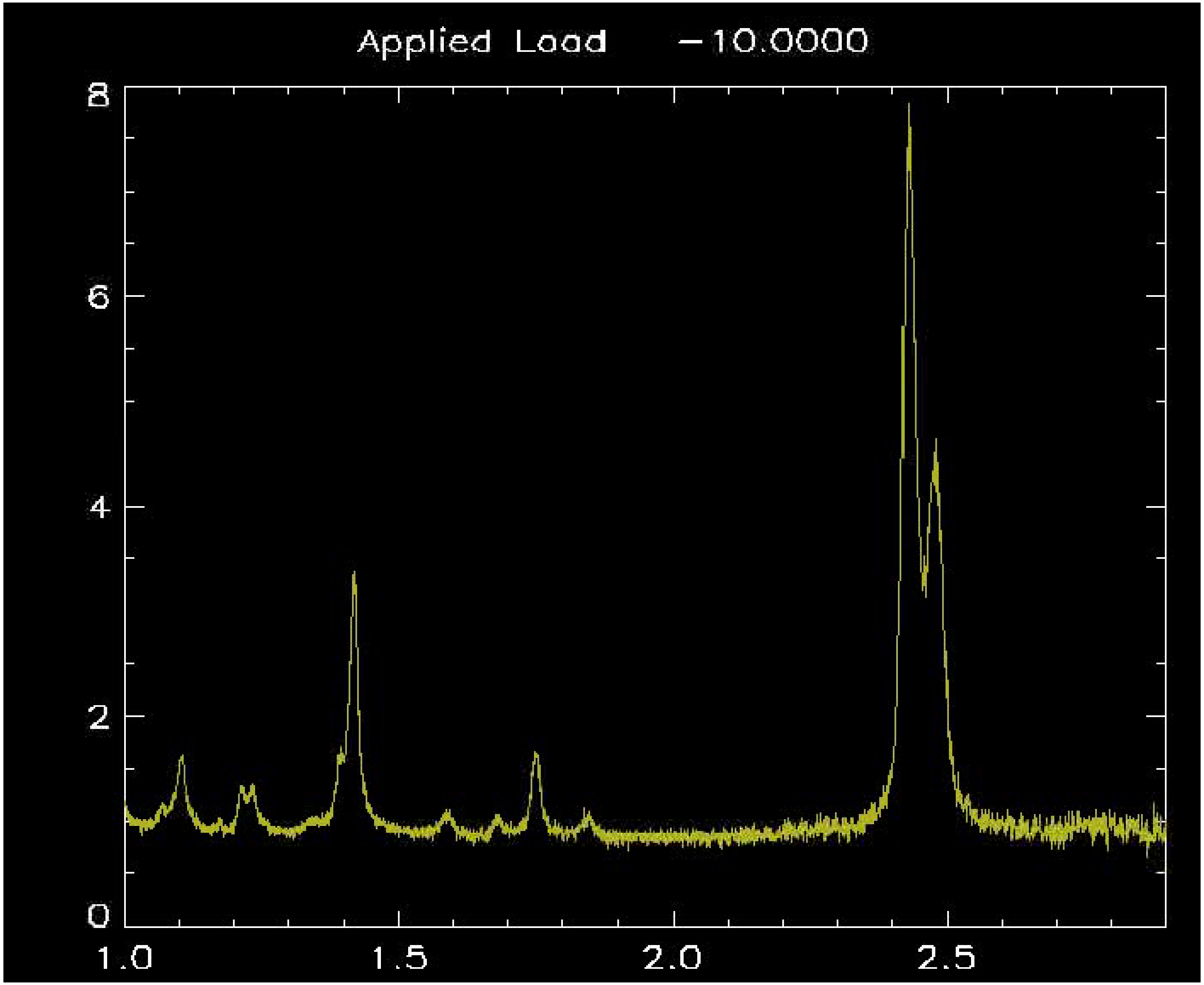
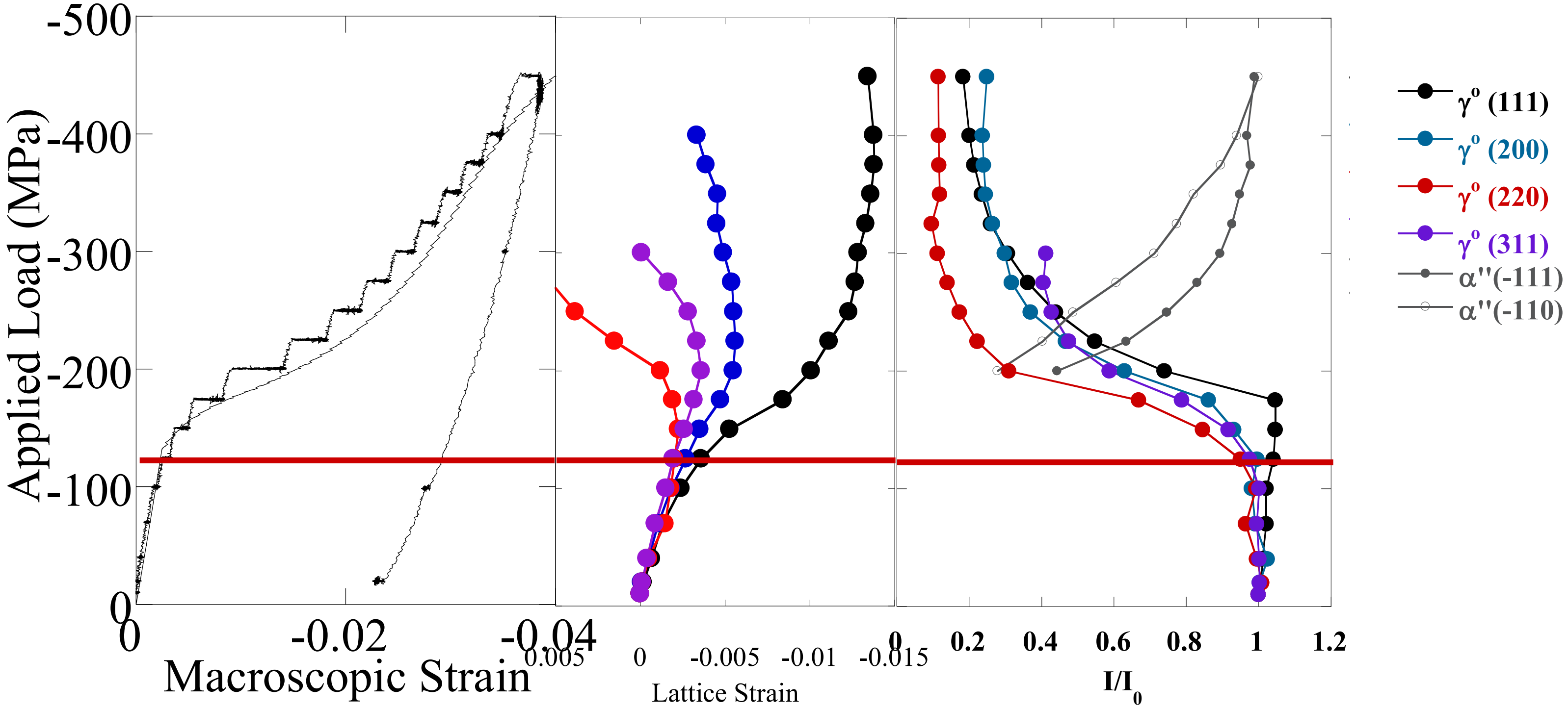
New Peaks In U7Nb Indicate Stress Induced Phase Transformation.



Deformation Twinning is a Relaxation Mechanism for Parent Grains



- Deviation of lattice strains from linearity indicates that plastic deformation has initiated.
- Addition of new peaks suggests stress induced phase transformation.

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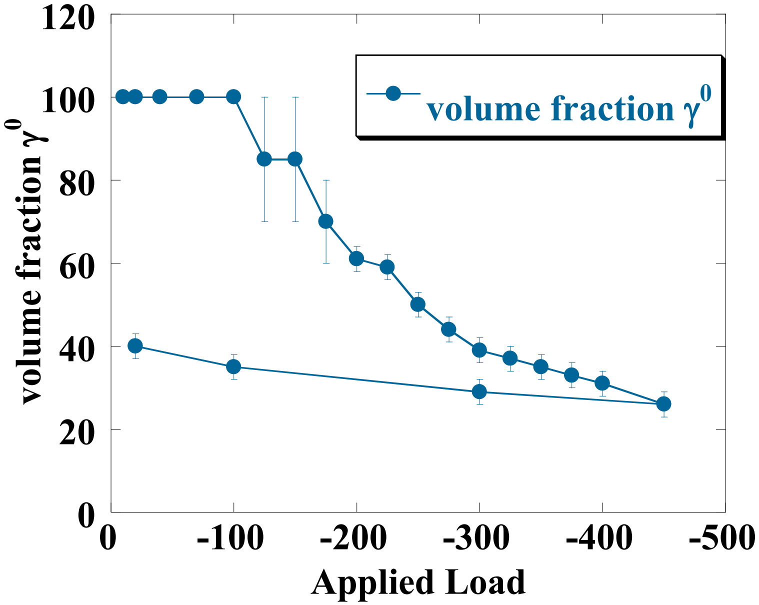
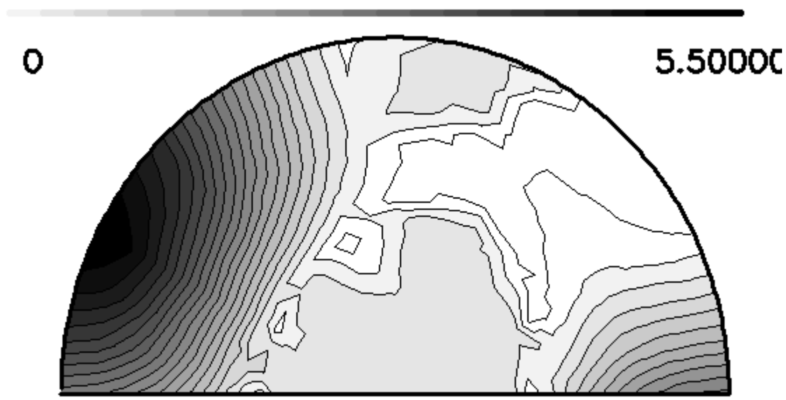
In-Situ Neutron Diffraction Monitors Volume Fraction and Texture During Deformation





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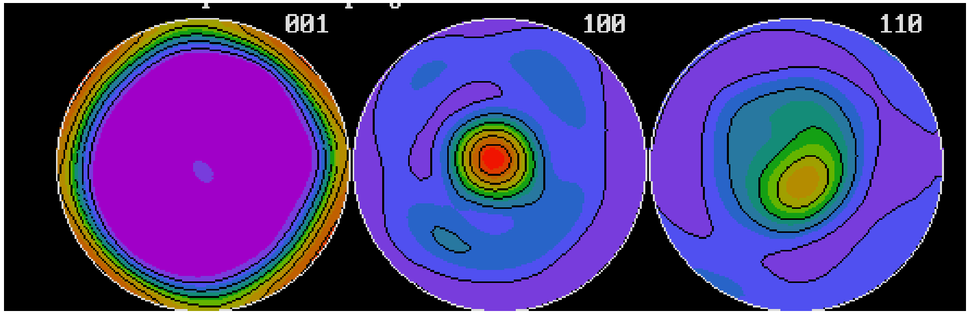
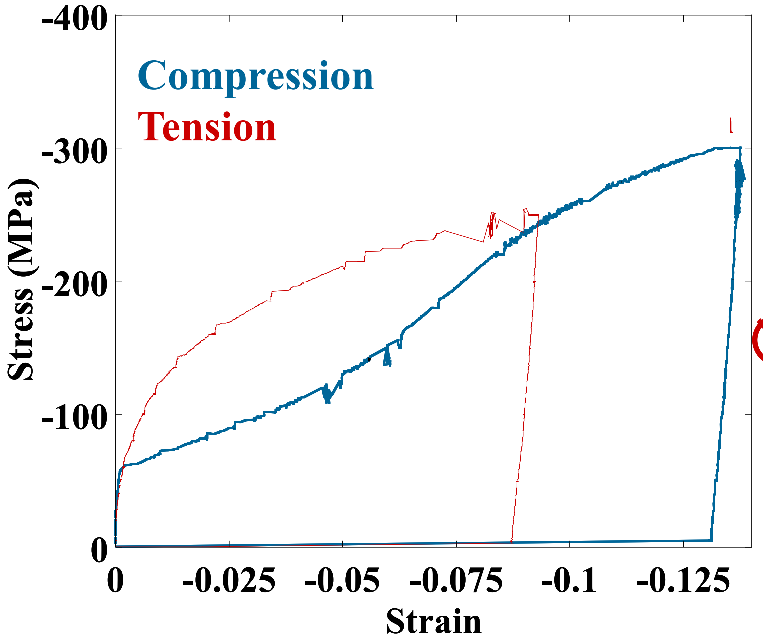
Example 2 : Deformation of Hexagonal Metals

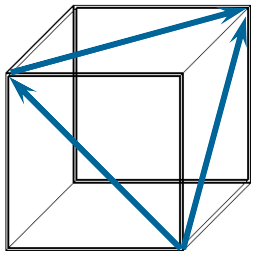


Load Axis Into Page

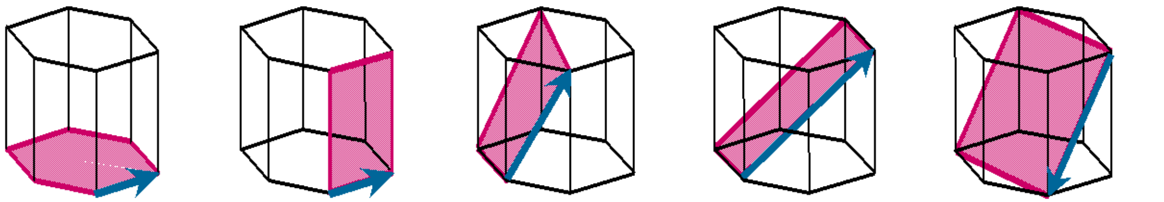


- **Atypical deformation in hexagonal metals drives our interest**
  - **Example : Tension / compression asymmetry in magnesium**
  - **Qualitatively different mechanical response**

Deformation of Low Symmetry Materials



- Face Center Cubic materials : Deform on {111}<110> slip system
  - 12 equivalent modes.
  - Can manipulate mechanical properties with texture.
    - e.g. strength, ductility, hardening...
- Hexagonal and lower symmetry materials often lack the necessary slip systems for arbitrary deformation by slip.
  - Can manipulate deformation mechanisms by choice of crystallographic texture.
    - e.g. slip, twinning, fracture…



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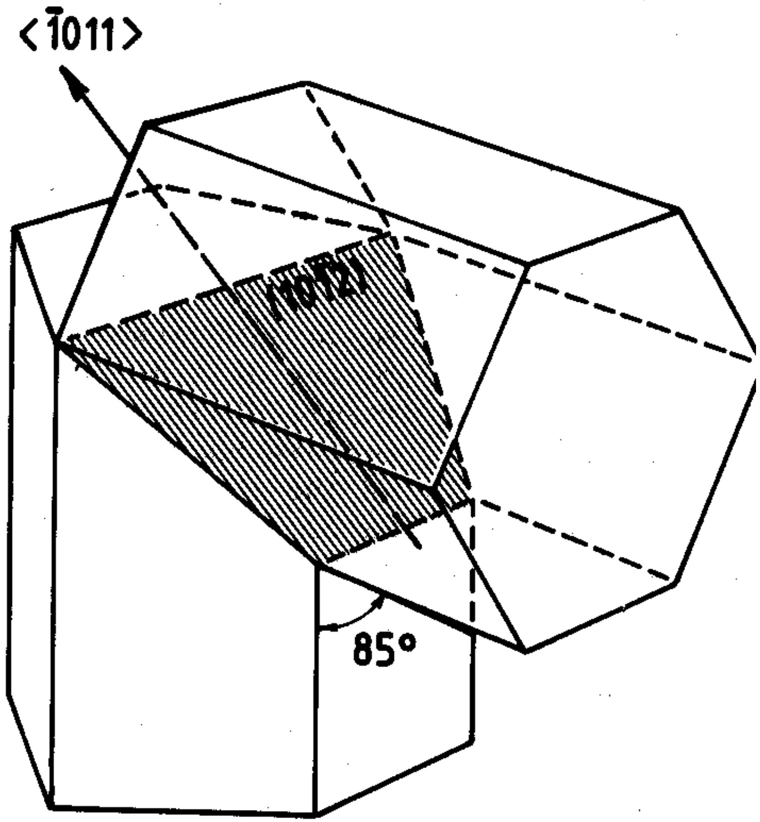
Deformation Twinning in Magnesium



- Twinning on (10.2) plane
  - results in a : c switching
- $c/a < \sqrt{3}$
- Twin extends grain along the original (parent) c-axis.
  - Twinning is polar!!!
  - Called extension or tension twin.
- Active when crystal pull along c-axis, or compresses transverse to c-axis.
- Source of the strength difference in tension vs. compression.

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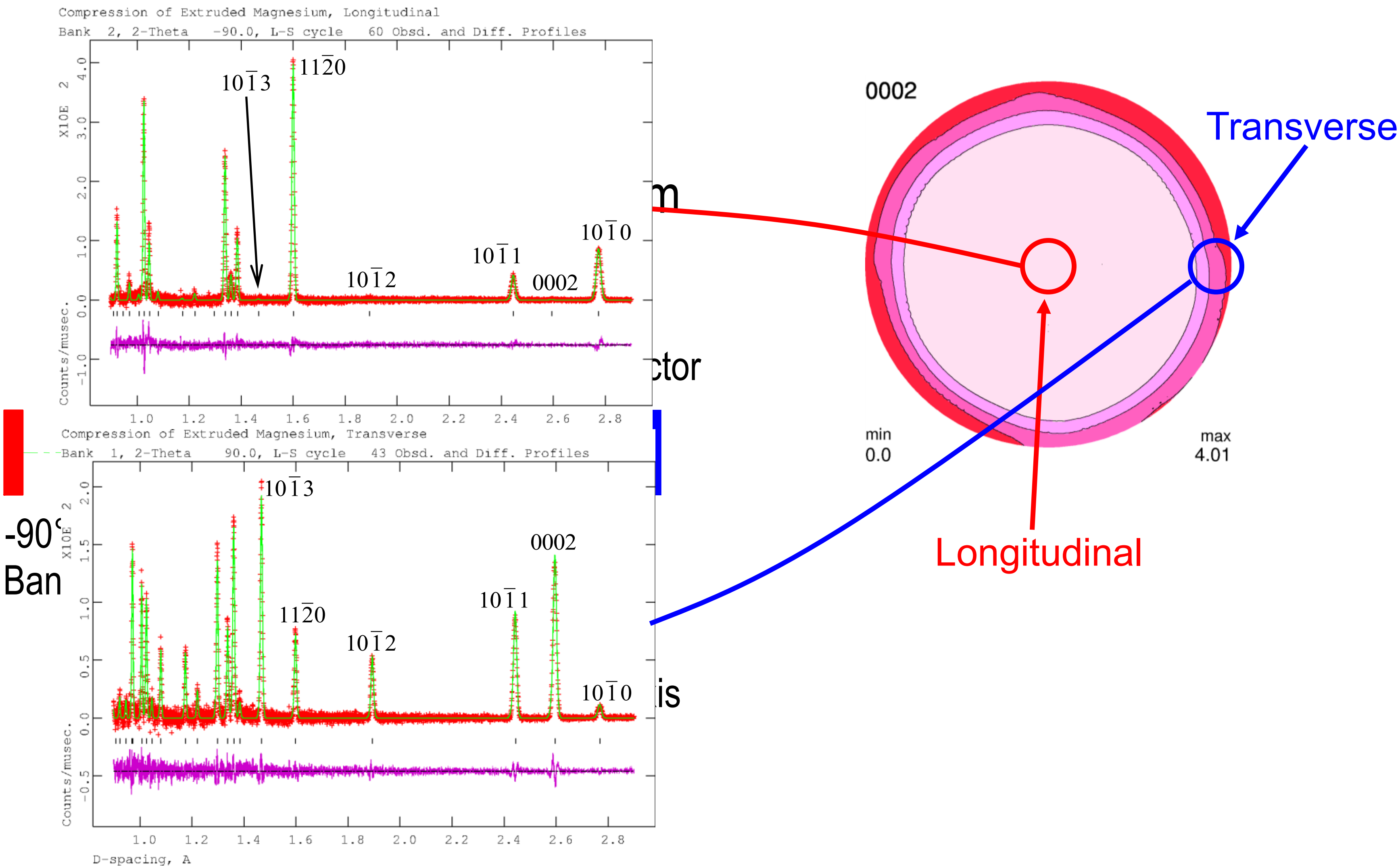
SMARTS Geometry Ideal For Study of Twinning in Mg



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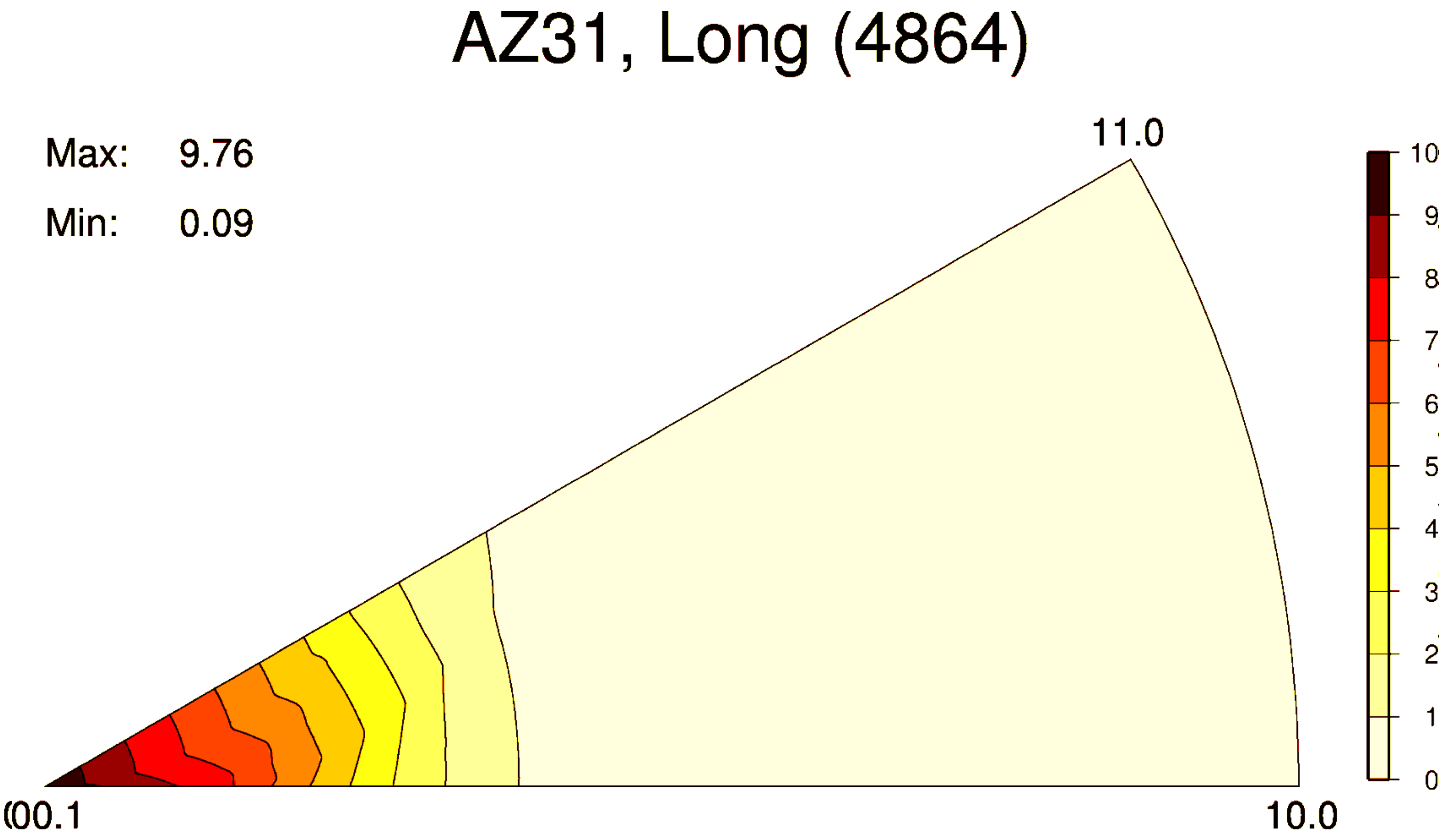
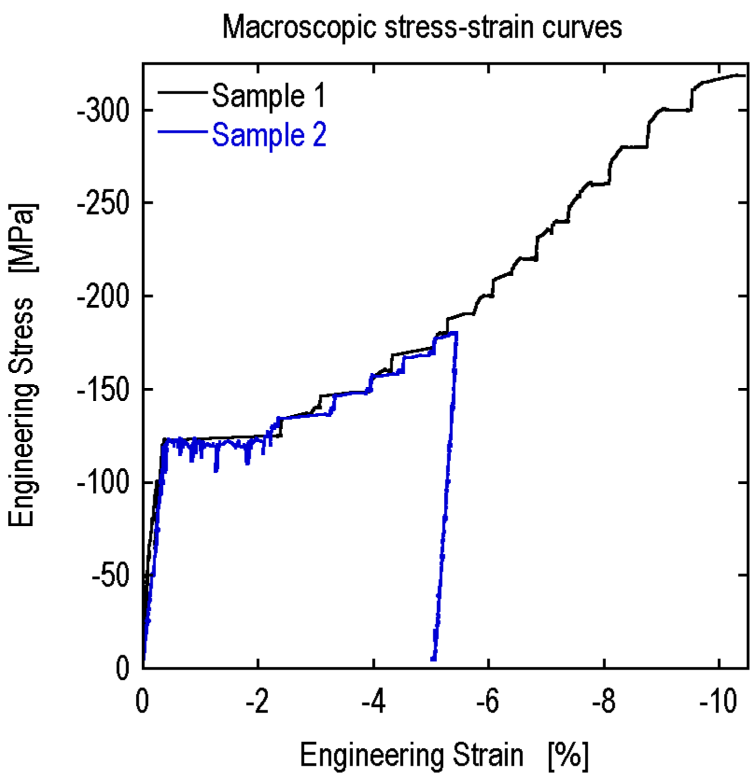
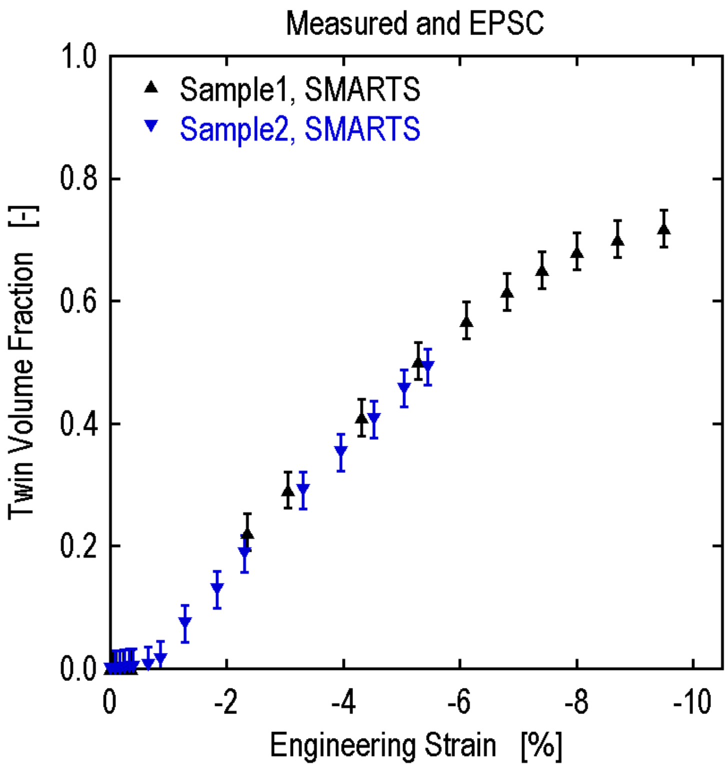
Evolution of Texture With Deformation



Extrusion Direction

Twin Volume Fraction Determined By Selectively Integrating the ODF





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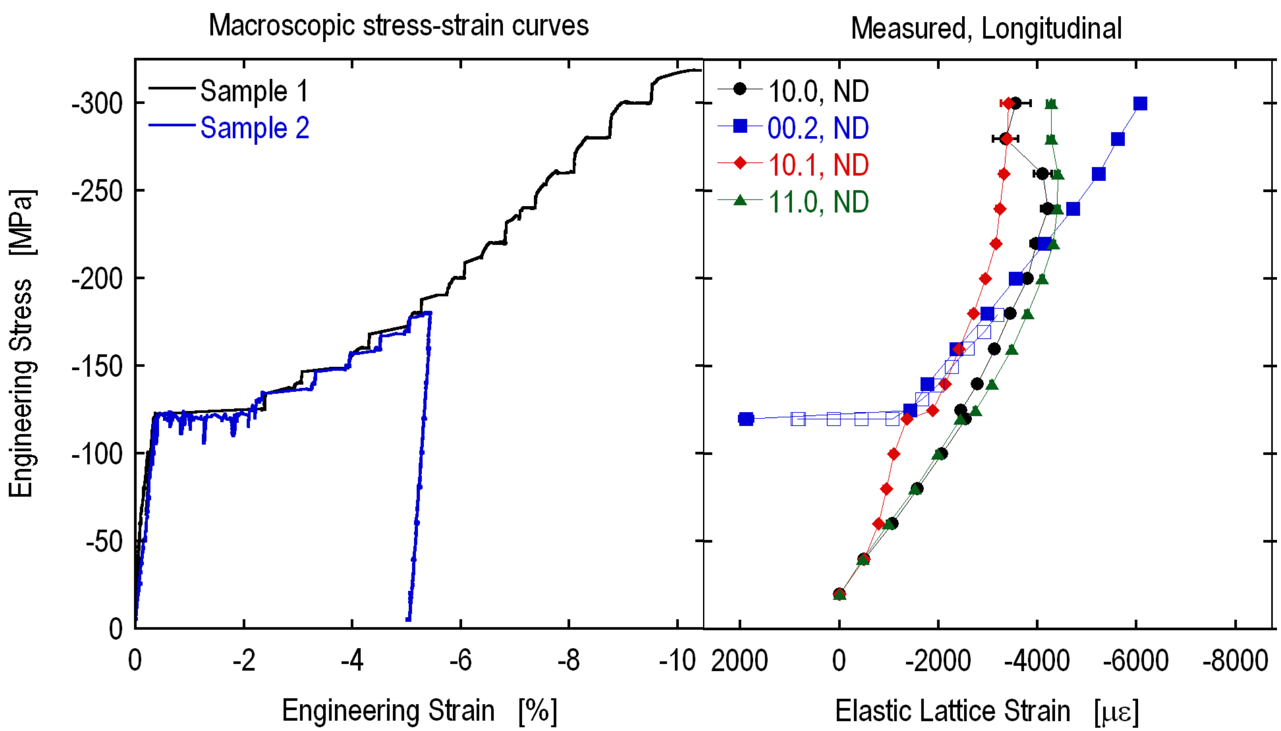
Neutrons Measure Internal Stress Development in Twins



- **Early non-linearity of the 10.1 reflection**
  - **Reflects basal slip in grains with (10.1) poles parallel to straining direction.**
- **Twins appear under tensile intergranular stress relative to aggregate.**
- **After arrival they rapidly accumulate strain.**
  - **Hard orientation.**
- **Parent grains relax when twinning.**

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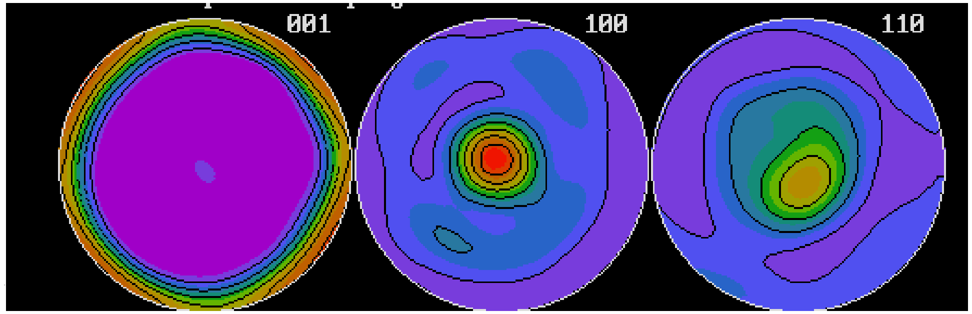
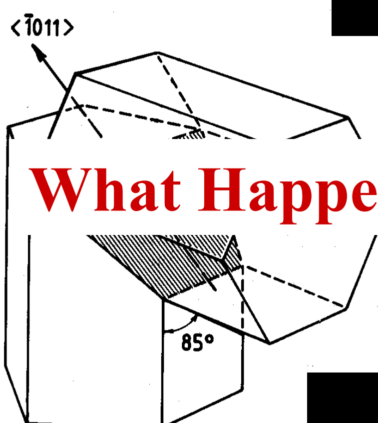
Compression Completely Reorients Microstructure

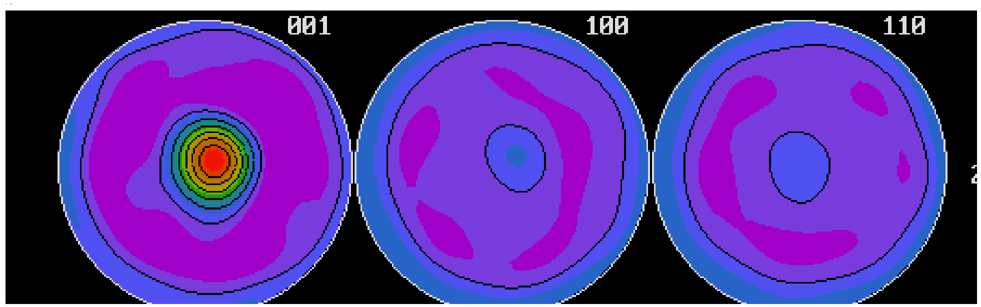
Starting texture : optimized for twinning in compression, will not twin in tension





What Happens If We Reverse Deformation Direction ???

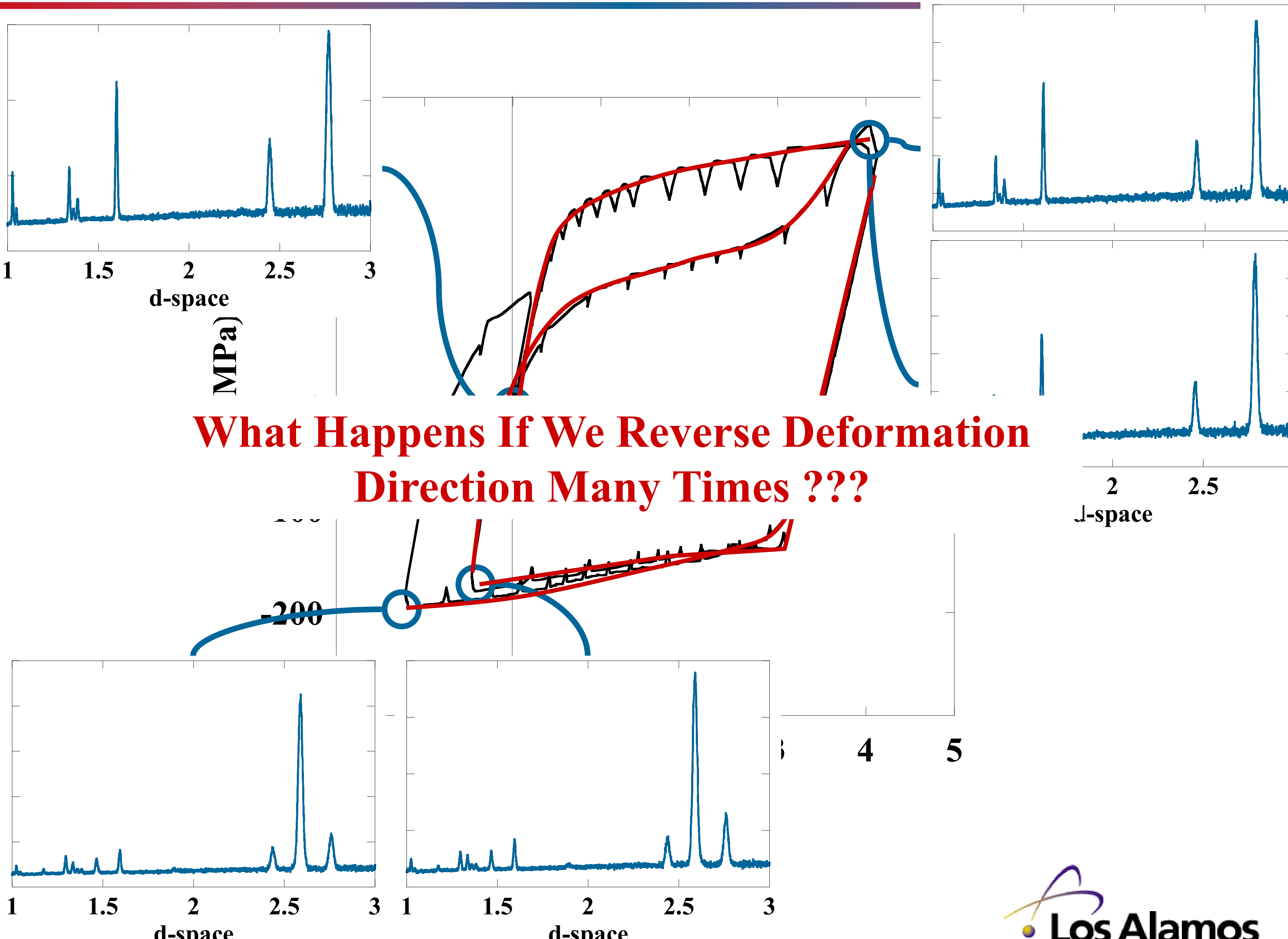
5%



Texture after 5% deformation, almost exhausted ability to twin in compression, but now aligned optimally for twinning in tension.

Los Alamos

Development of Diffraction Pattern With Reverse Loading : Tension First



What Happens If We Reverse Deformation Direction Many Times ???

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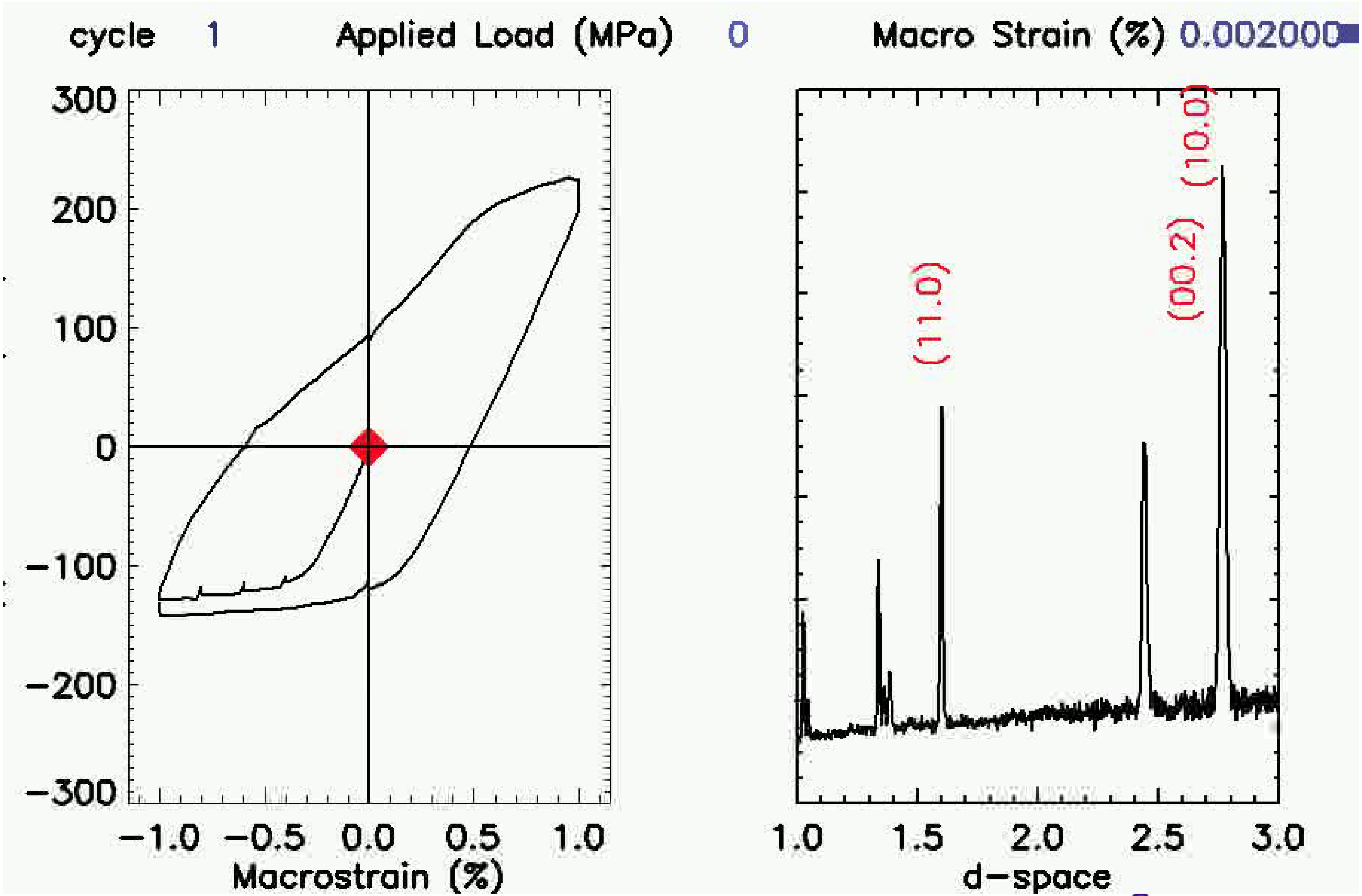
Development of Diffraction Pattern With Reverse Loading : Comp First



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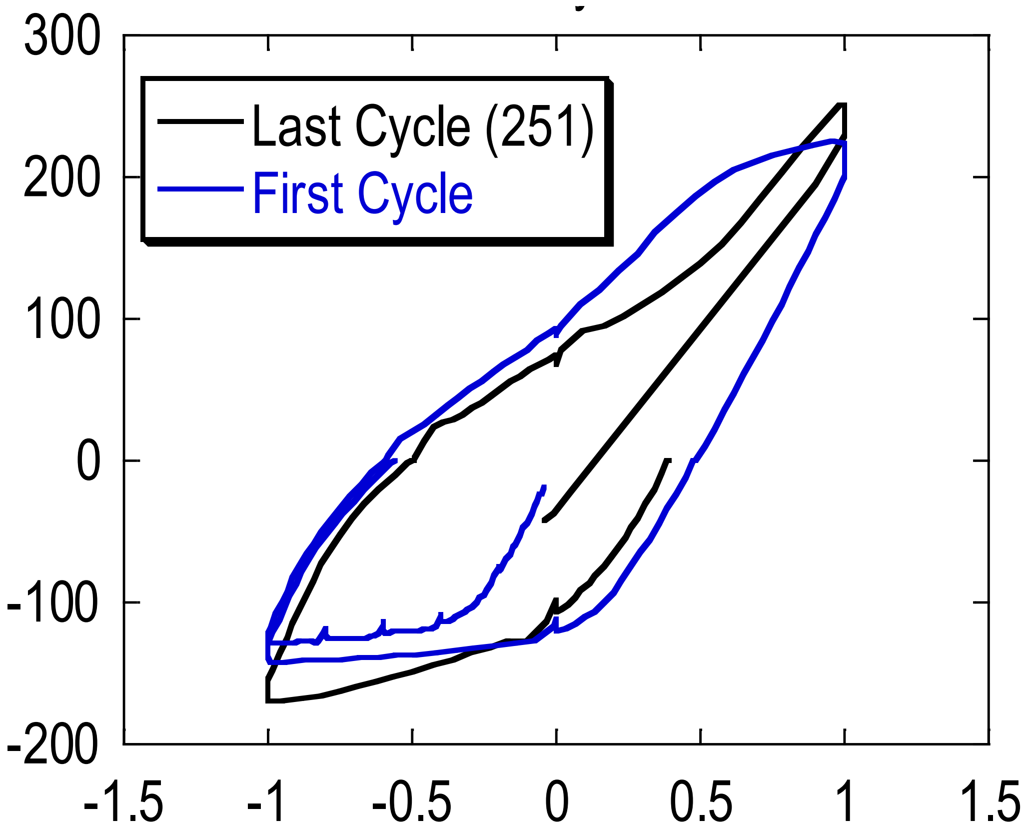
Development of Flow Curve With Cycling



- Broke at ~470 cycles.
- Last recorded cycle has significantly more hardening.
- Hysteresis loop has closed some.

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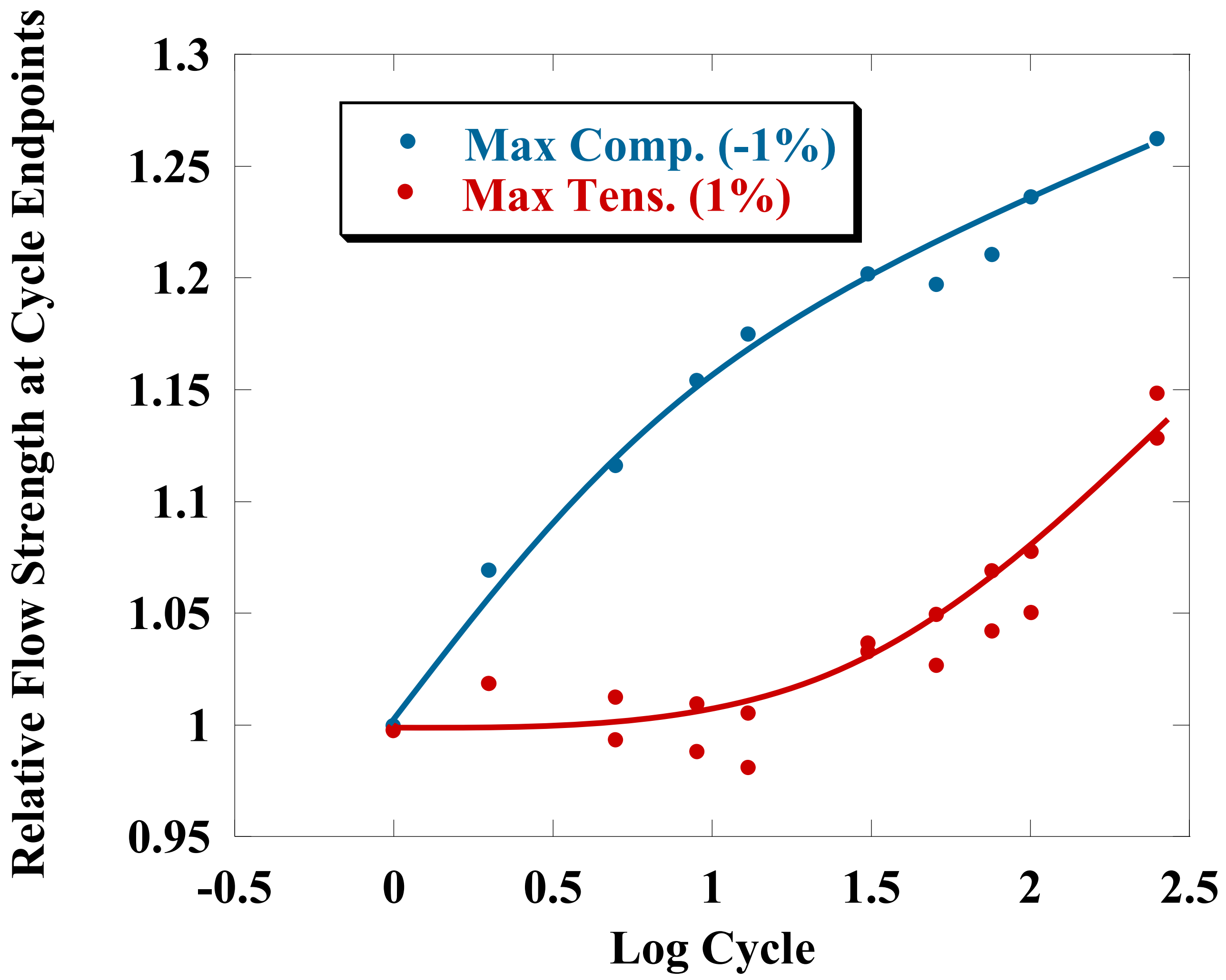
Flow Stress Increases With Cycling



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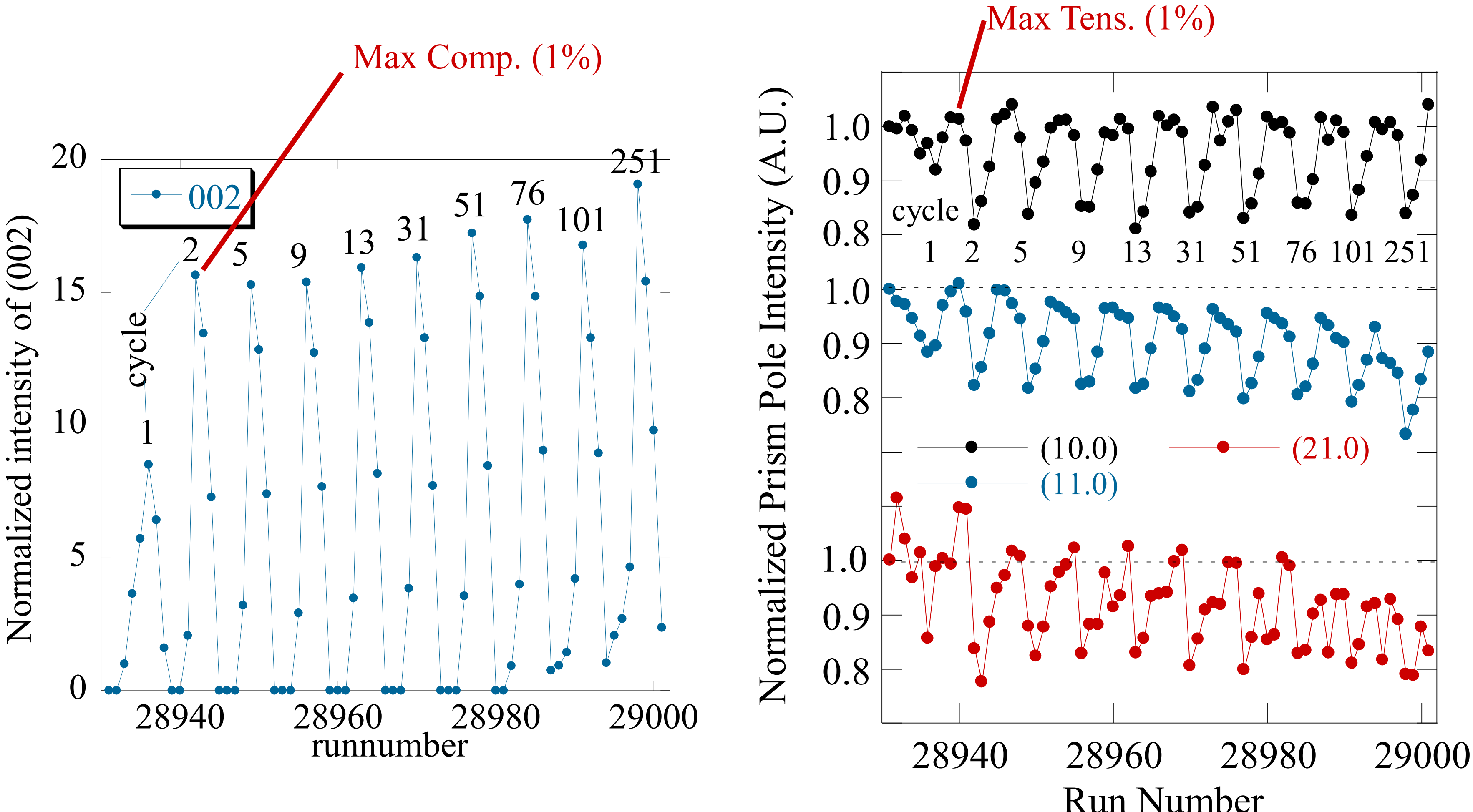
Twinning is Reversible During Cyclic Deformation of Extruded Mg



- (100) grains fully recover throughout measurement.
- (110) and (210) grains do not recover fully on cycling.
- Max resolved shear stress on the (100) grains.

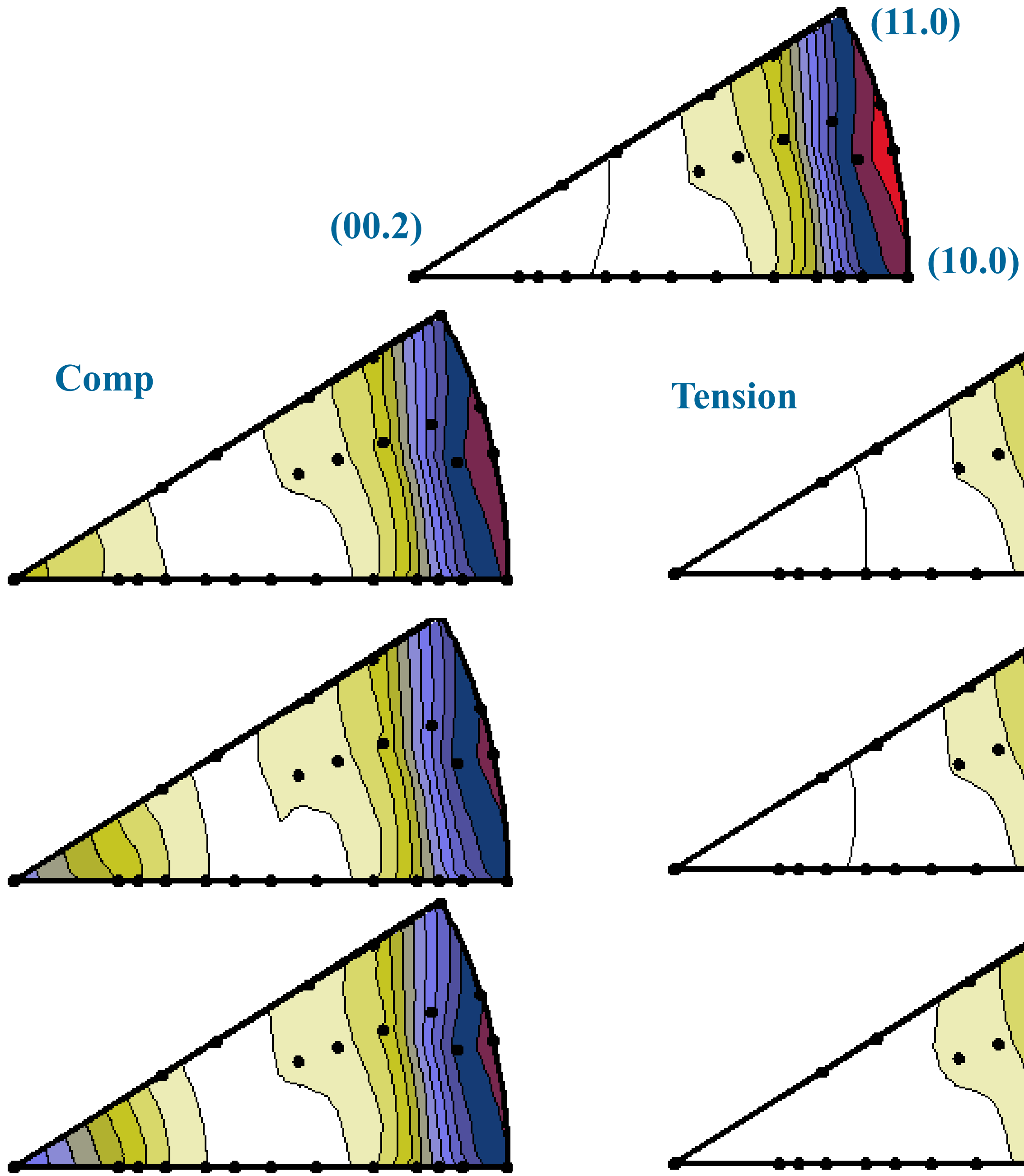
Development of Texture With Cycling

As-Extruded

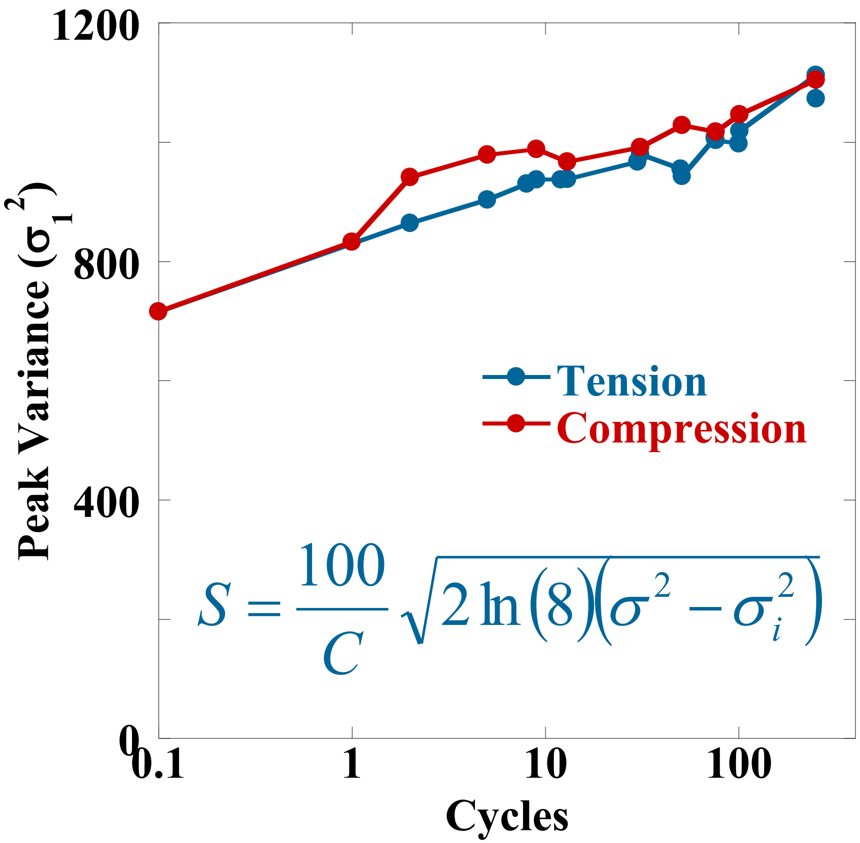
1st Cycle

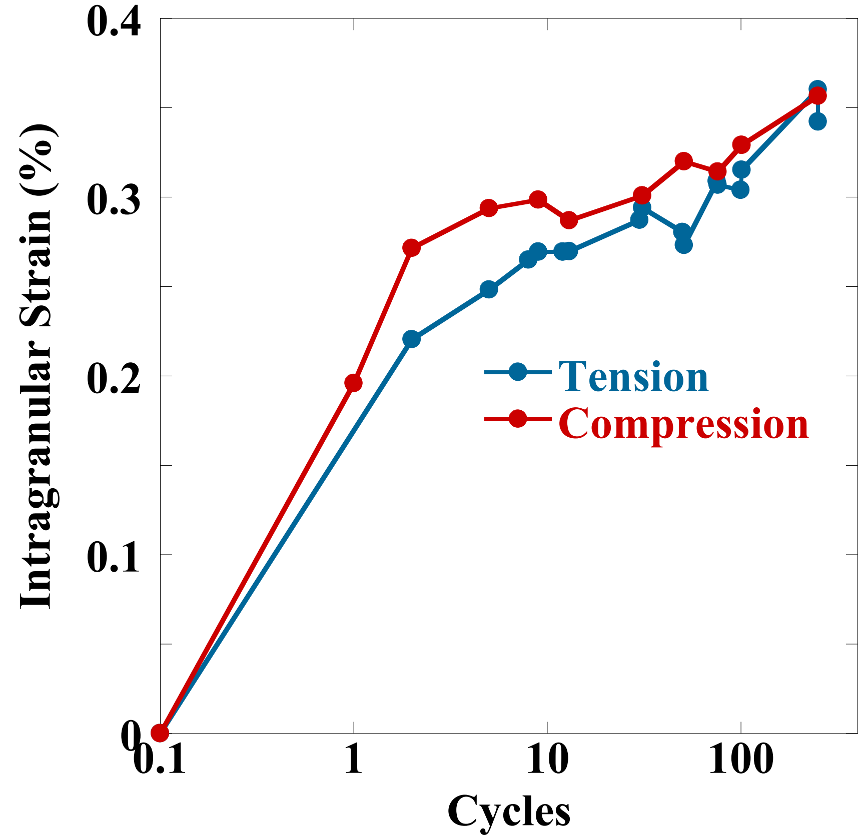
2nd Cycle

Final Cycle



Strain Broadening Increases With Cycling





- Peak broadening may be due to defects or dislocations which hinder motion of twin boundaries at higher cycles.

- Peak broadening is not unique to any one grain orientation.

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Summary

- Diffraction is an effective technique to monitor texture and internal stresses in structural material
- White beam neutron diffraction may be used to monitor evolution of microstructure *in-situ* during deformation or processing.
- Especially sensitive to twinning (or detwinning) and phase transformation.
- Monitor internal stresses in multiple grain orientations
  - Determine residual stress in anisotropic metals
- Monitor texture in-situ during deformation.
- Watch deformation twinning in magnesium
  - May be reversed by subsequent tension : detwinning.
  - May be cycled several hundred times.

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