# Engineering Neutron Diffraction

Don Brown

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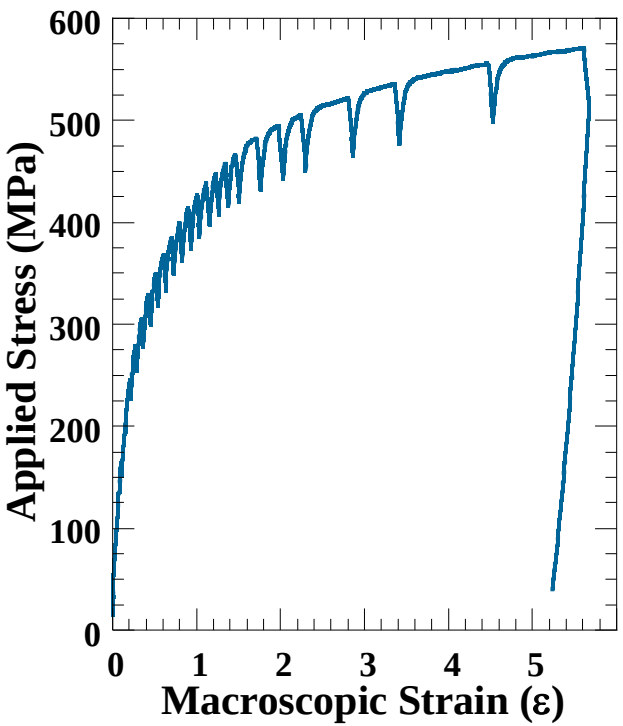
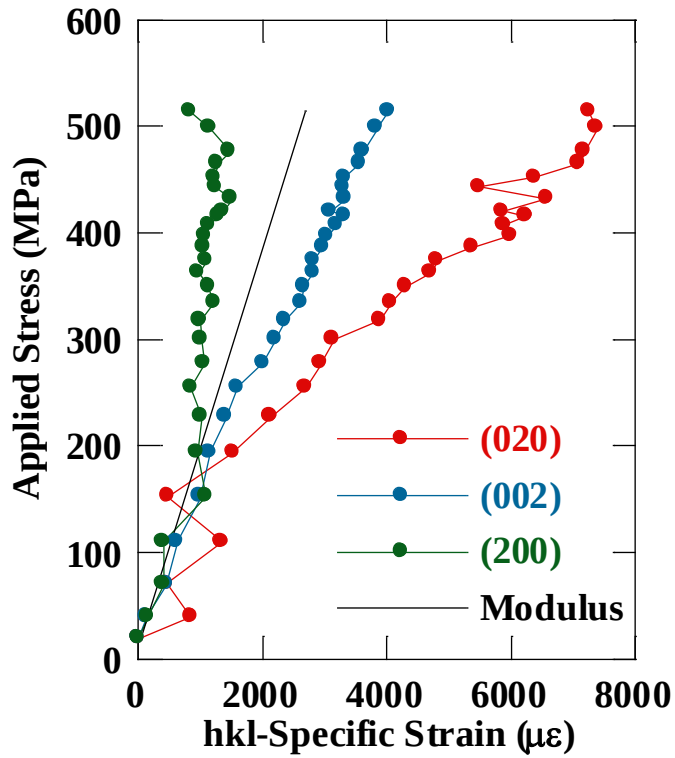
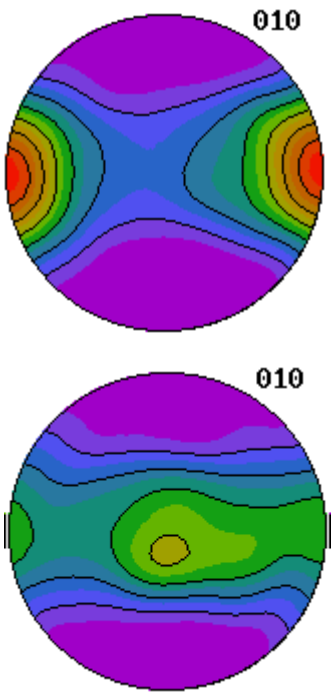
# Why Perform Measurements of Internal Stress and Texture During Deformation




- 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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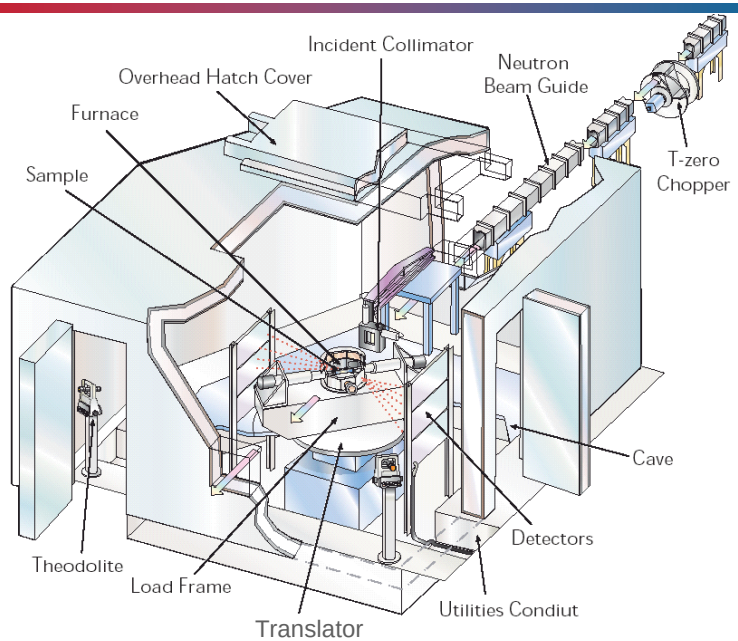
# SMARTS Designed to Study Engineering Materials Under Operating Conditions



- **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 sample capability: If you can ship it, we'll measure it !**
- **Next proposal call due 5/14: dbrown@lanl.gov.**

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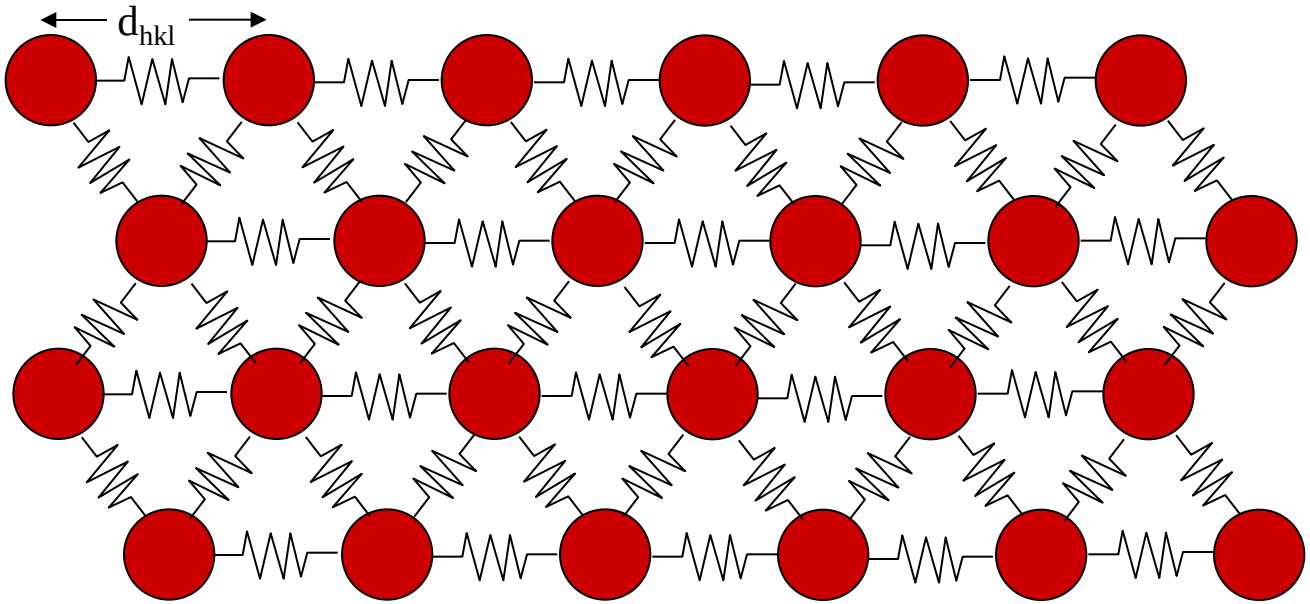
# SMARTS is a 5 Million Dollar Bathroom Scale




- 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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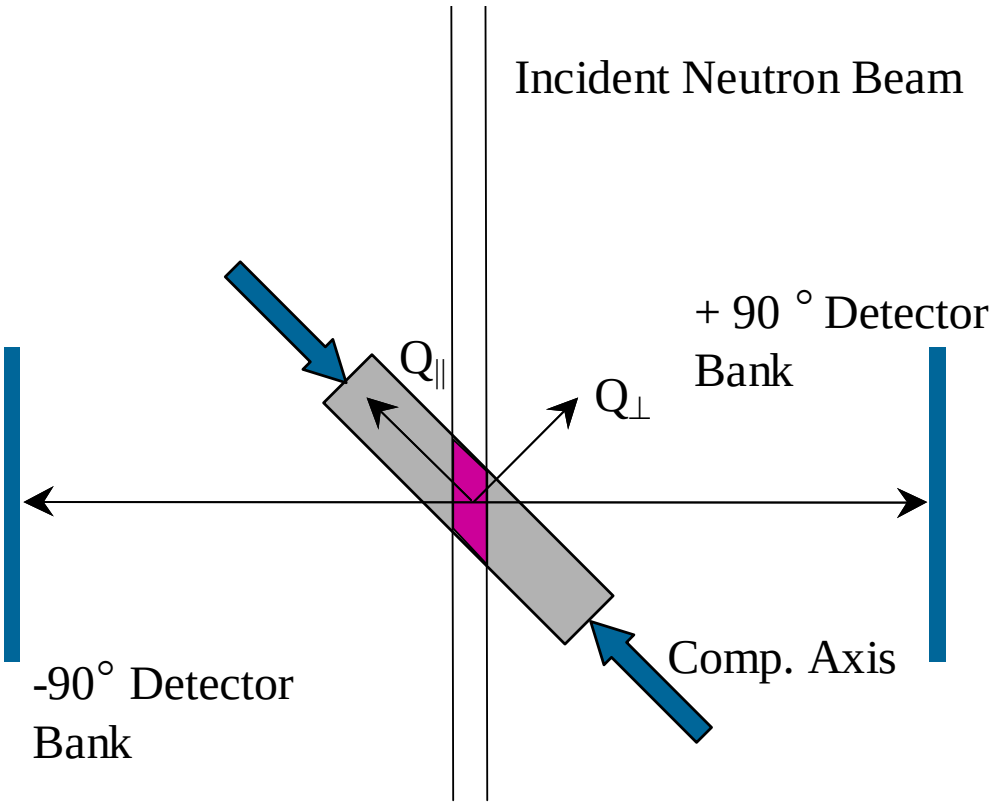
# Reminder of the SMARTS Geometry

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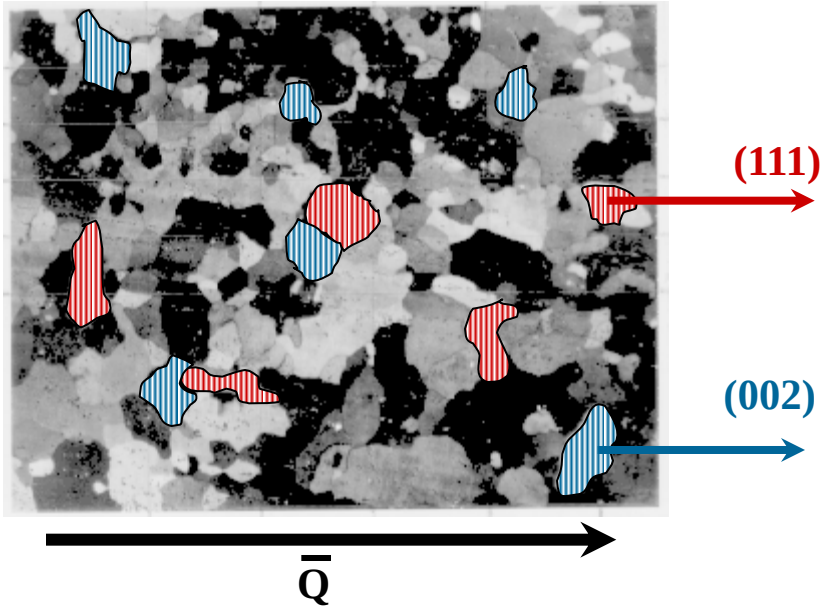
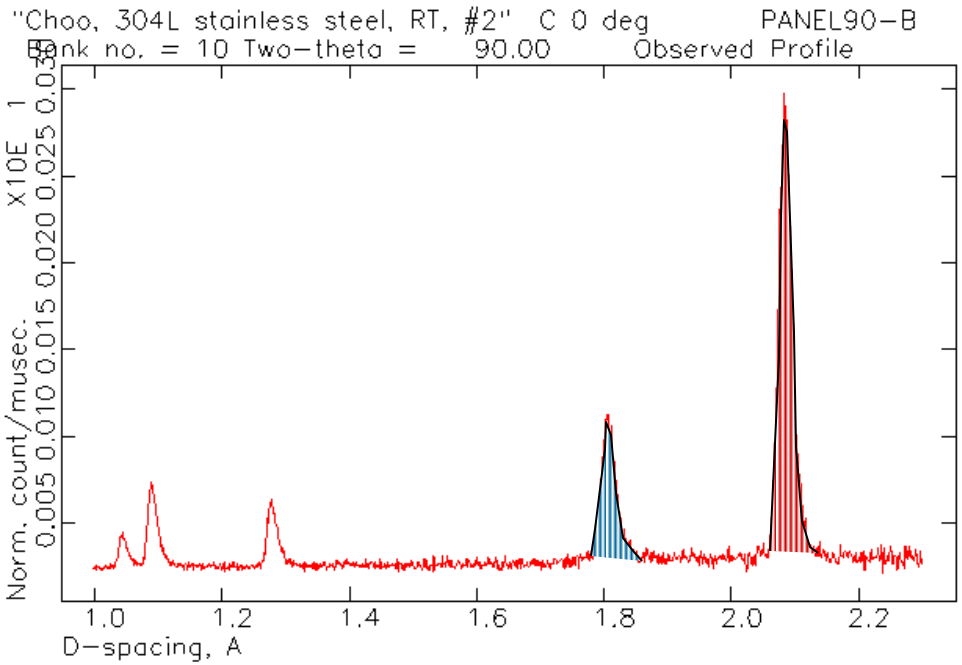
# Diffraction Separates Response of Grain Orientations

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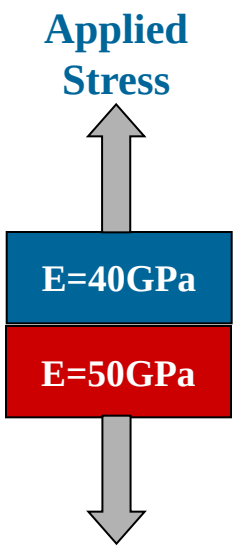
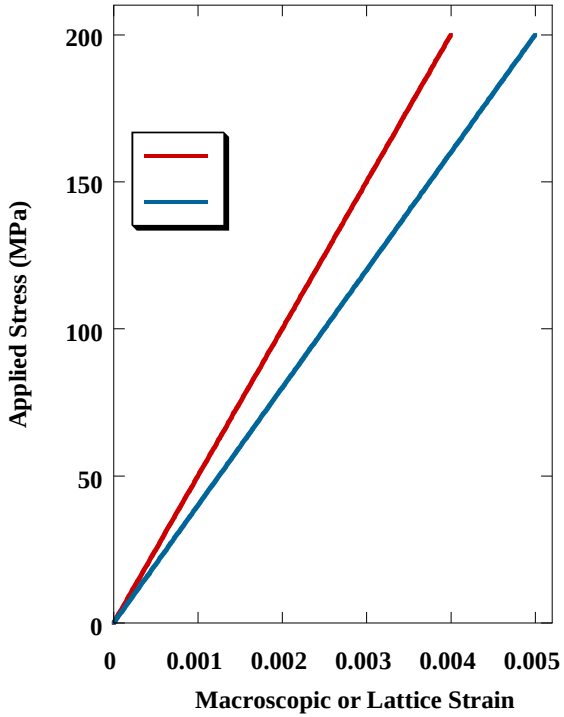
# Consider the case of a bi-metallic sample : Elastic loading in series

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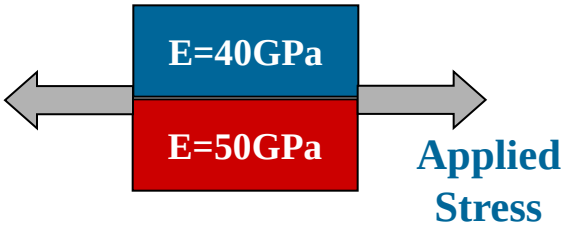
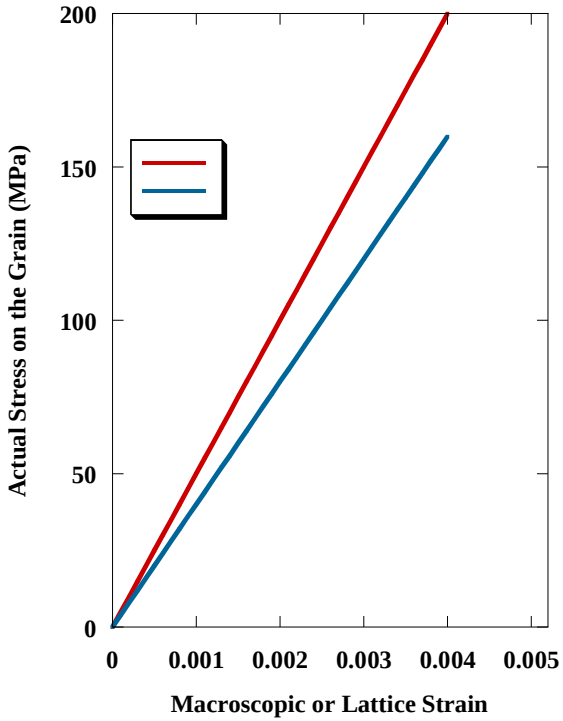
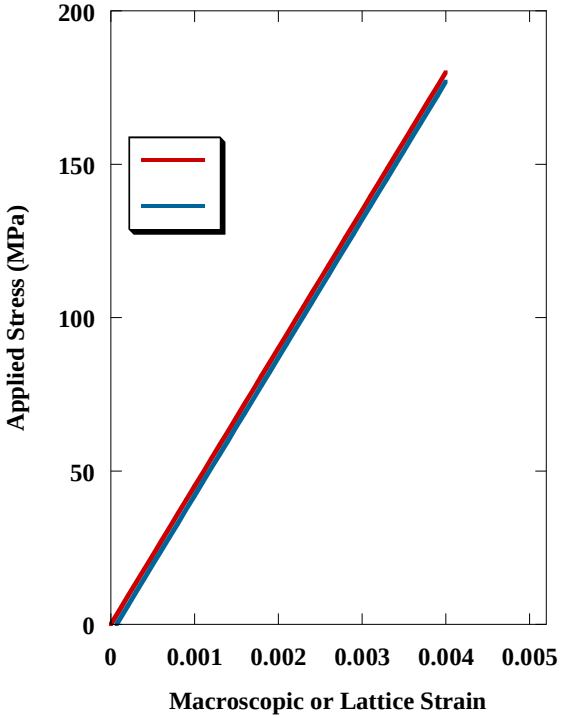
# Consider the case of a bi-metallic sample : Elastic loading in parallel




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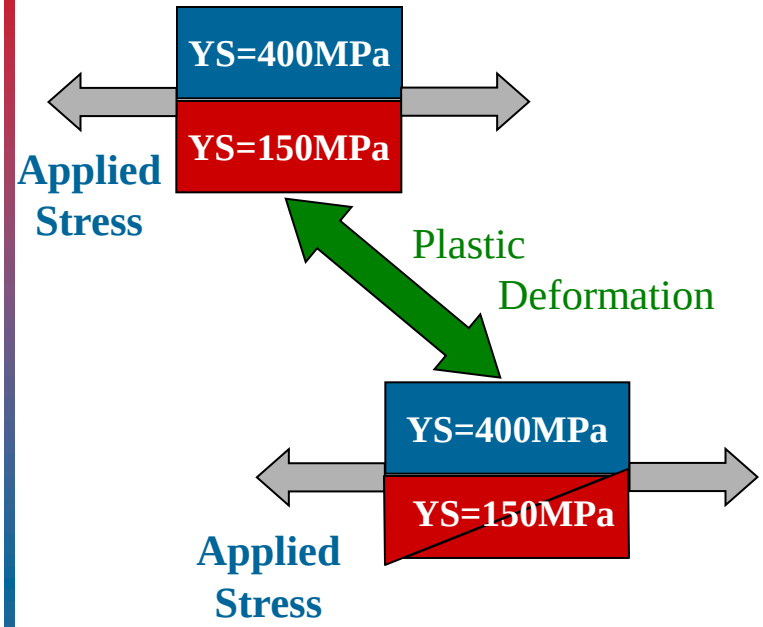
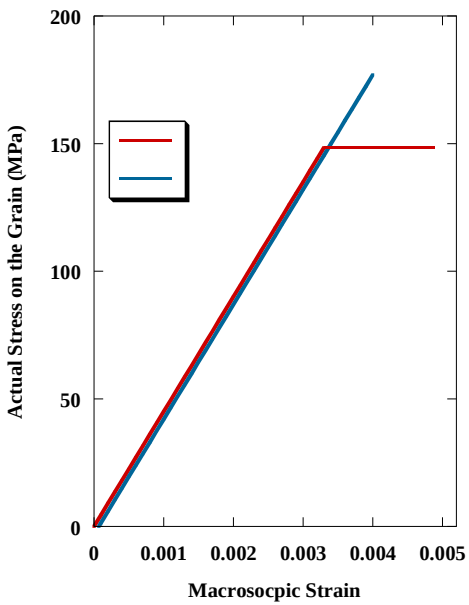
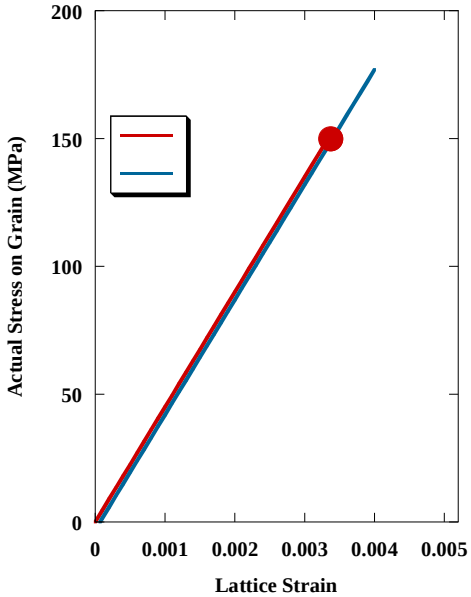
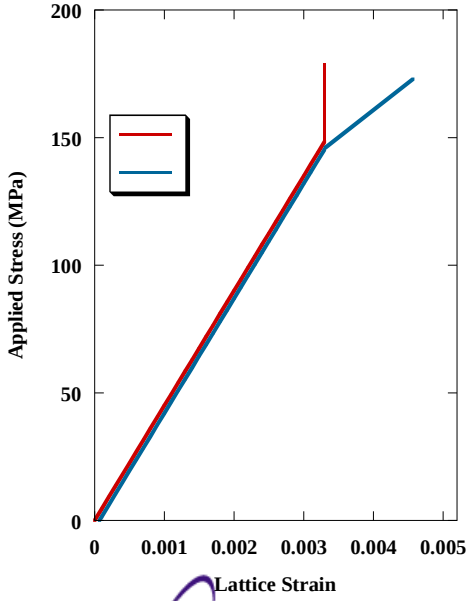
# Consider the case of a bi-metallic sample : plastic loading in parallel.




- 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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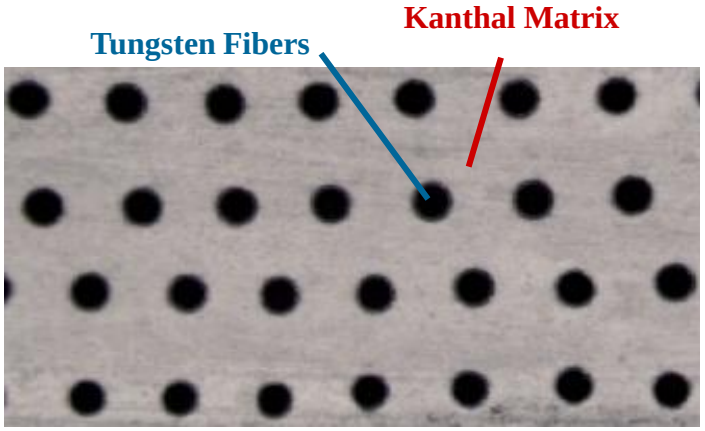
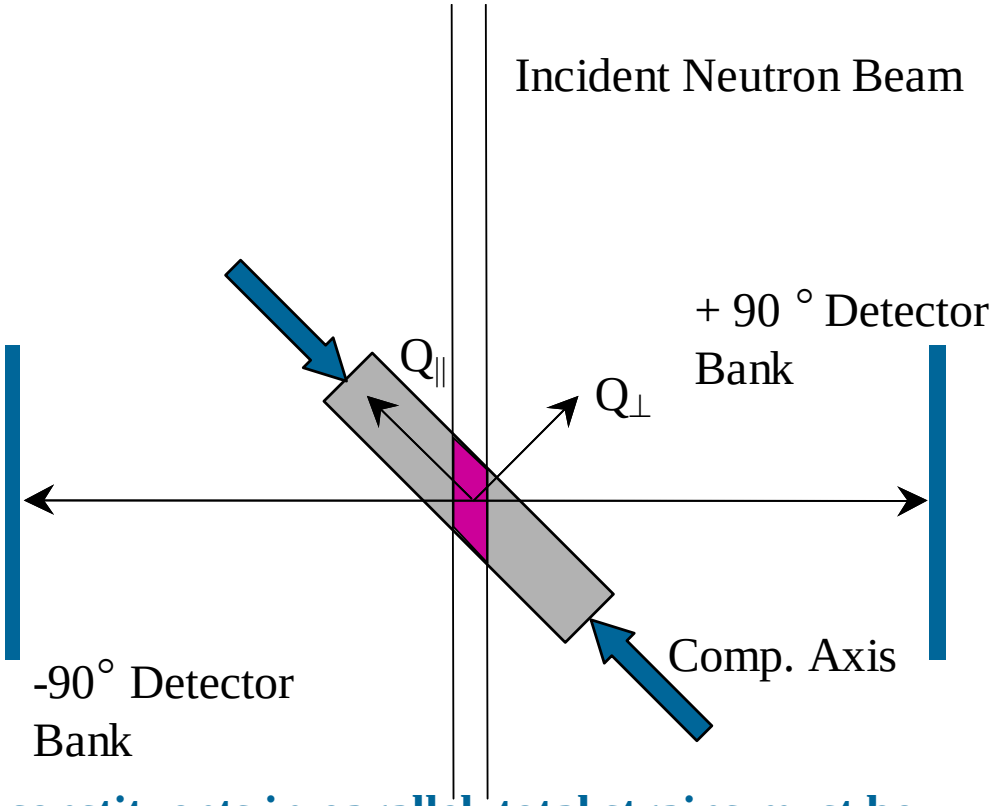
# Lets Consider How a Composite Responds to Deformation




- **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.**

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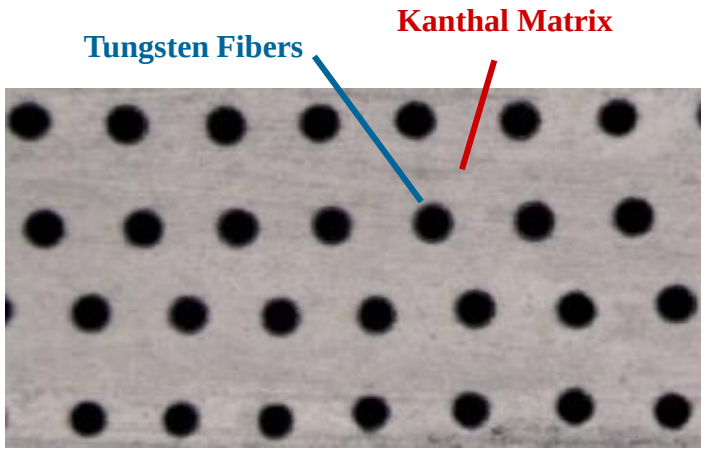
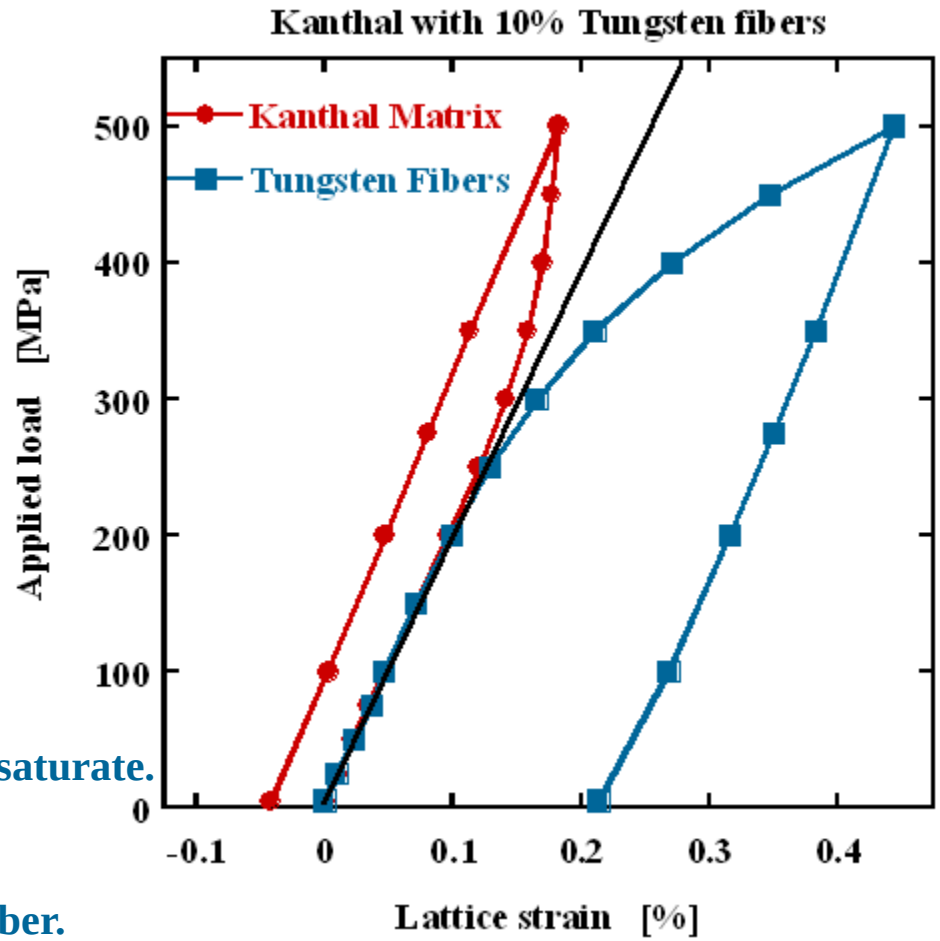
# Understand Anisotropy in Terms of a Composite




- 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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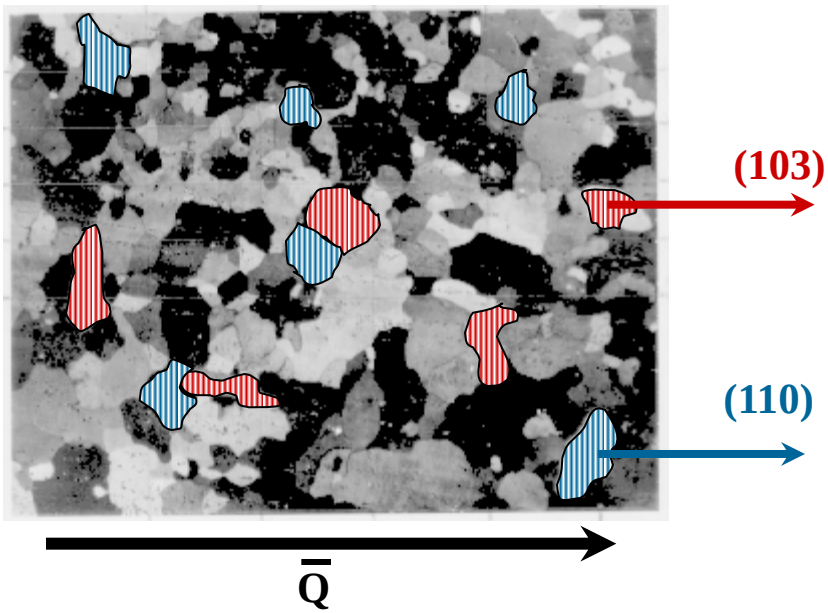
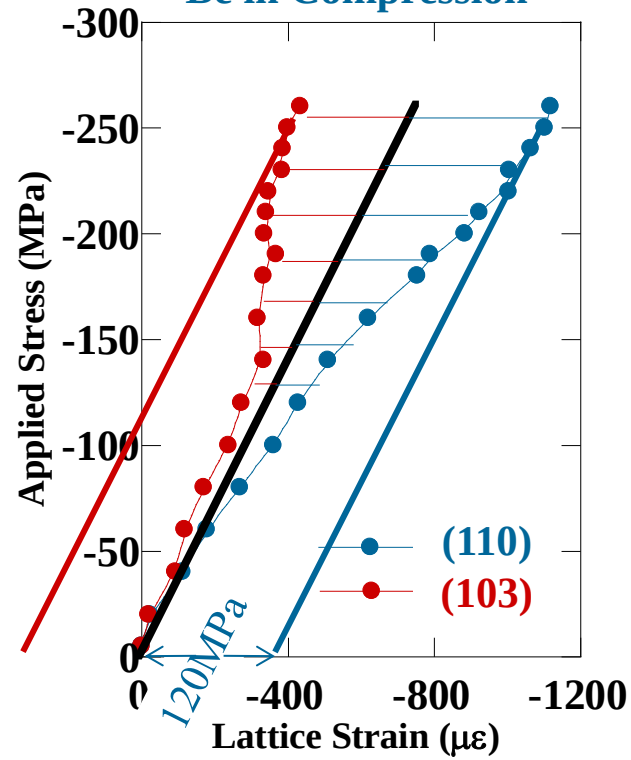
# Polycrystalline Samples : "The Mother of All Composites".

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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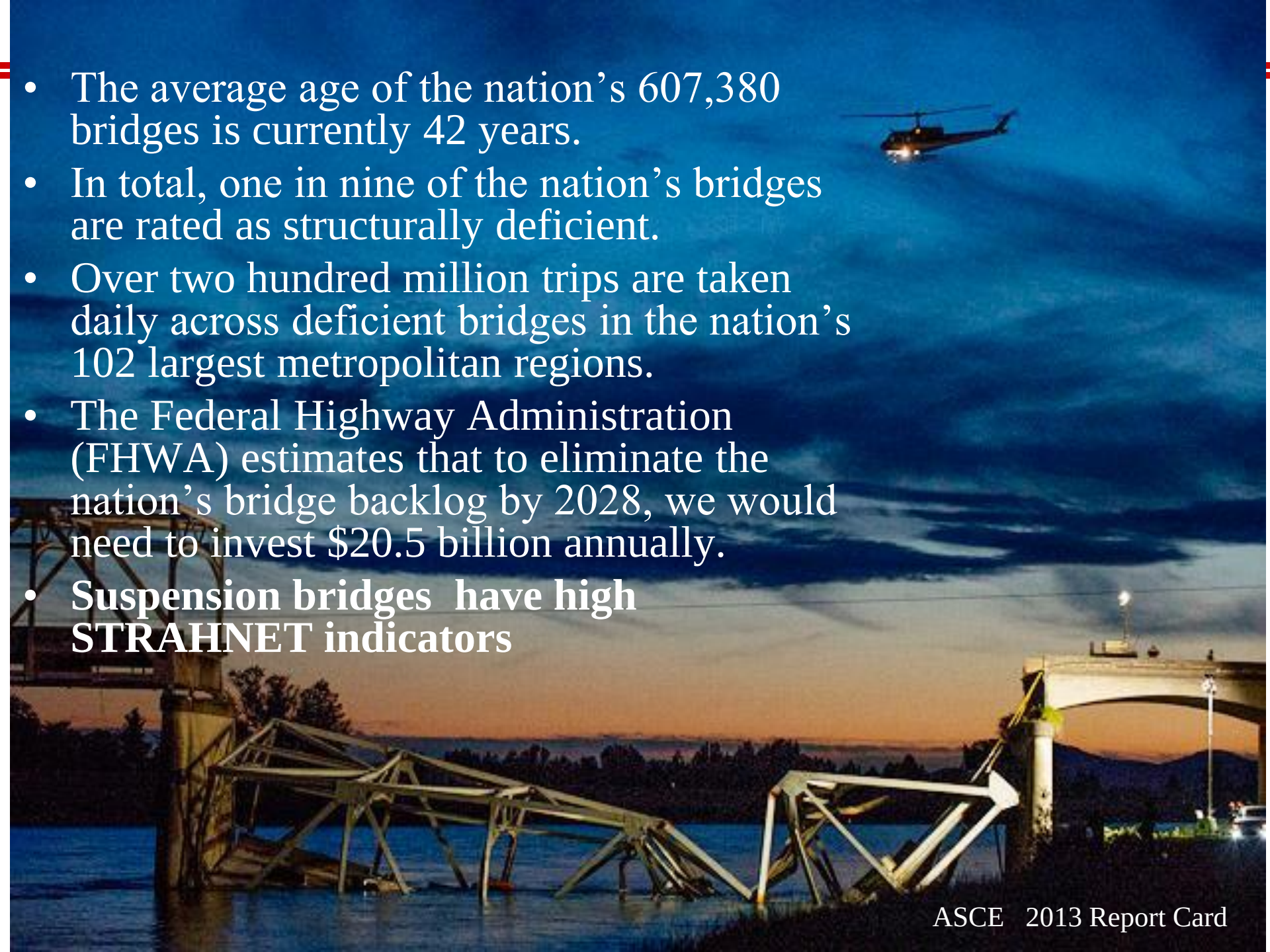
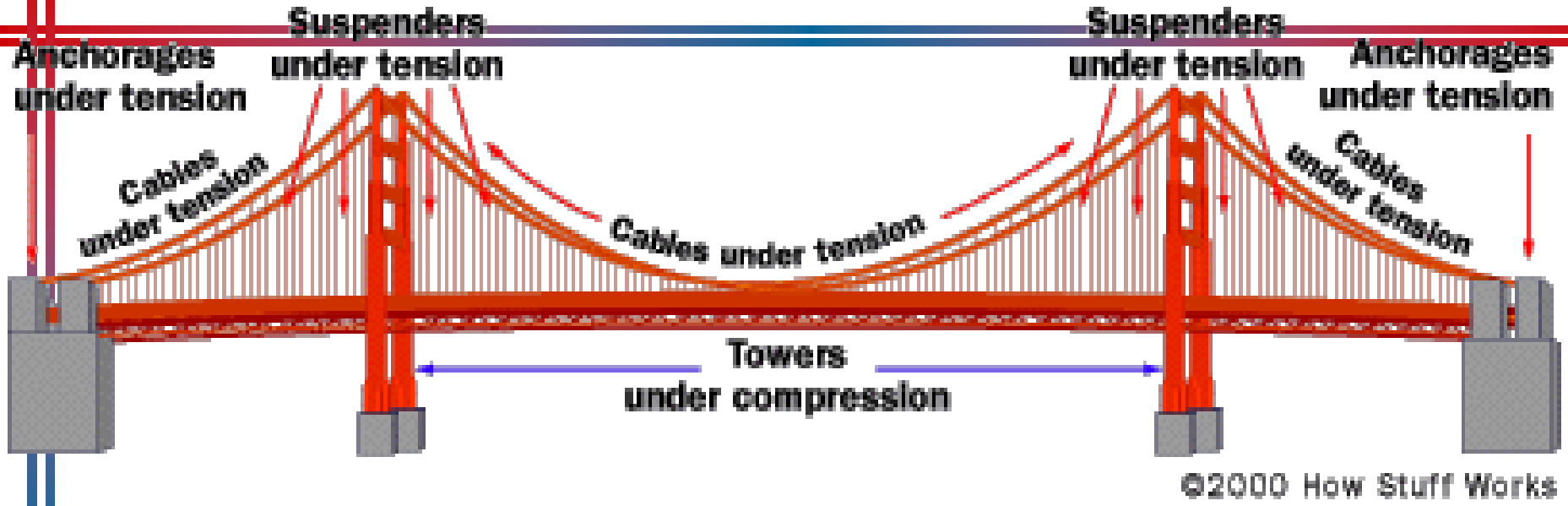
# I Have Intentionally Chosen Examples That Are a Bit…Atypical

1. Bridge Cables
2. Nuclear Fuel
3. Nurse Tanks

- The average age of the nation's 607,380 bridges is currently 42 years.
- In total, one in nine of the nation's bridges are rated as structurally deficient.
- Over two hundred million trips are taken daily across deficient bridges in the nation's 102 largest metropolitan regions.
- The Federal Highway Administration (FHWA) estimates that to eliminate the nation's bridge backlog by 2028, we would need to invest $20.5 billion annually.
- **Suspension bridges have high STRAHNET indicators**



ASCE 2013 Report Card




Length of main span: ~1km
Width: 36 m
Deck weight: 154 kton

Cable weight: 28.5 kton
Cable diameter:0.92 m
# of wires:26,472

The tensile stress in the cables can be quite high!

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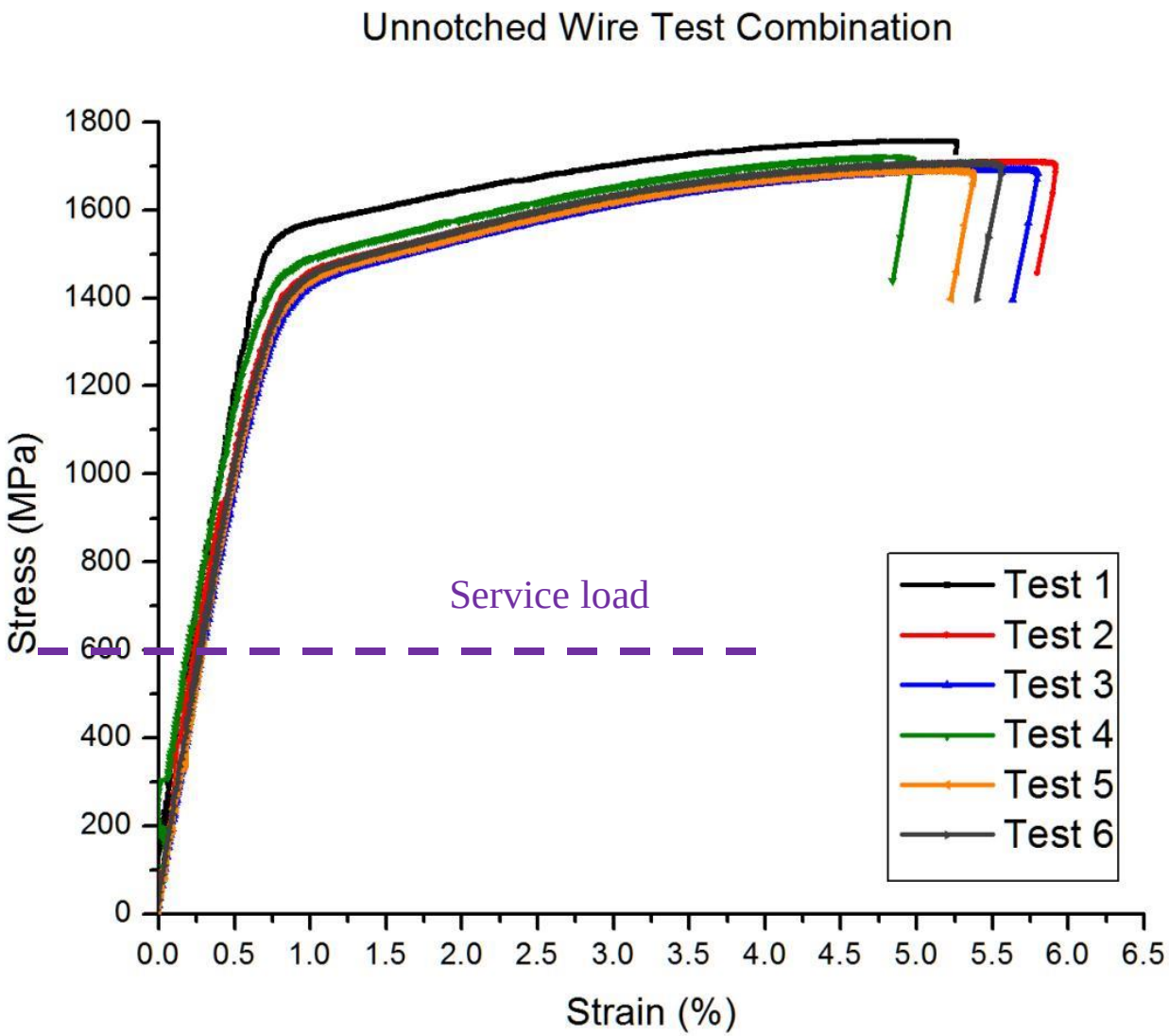
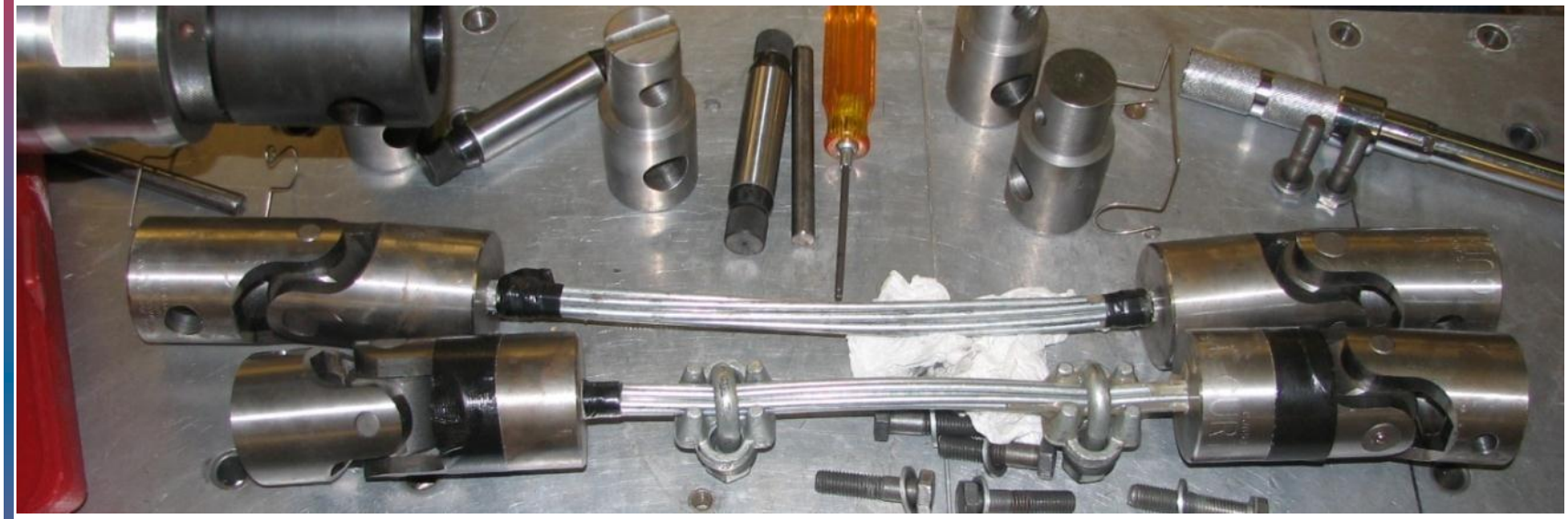
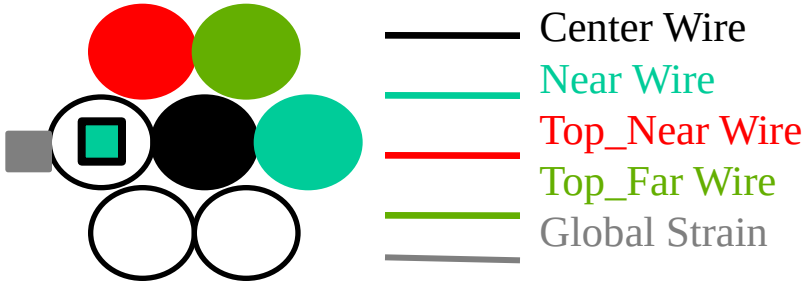
# Bridge Cables Are Strong…Not Tough



# Samples

Standard seven-wire parallel strands






Sample cross-section. The shaded rectangle is the neutron illuminated region.

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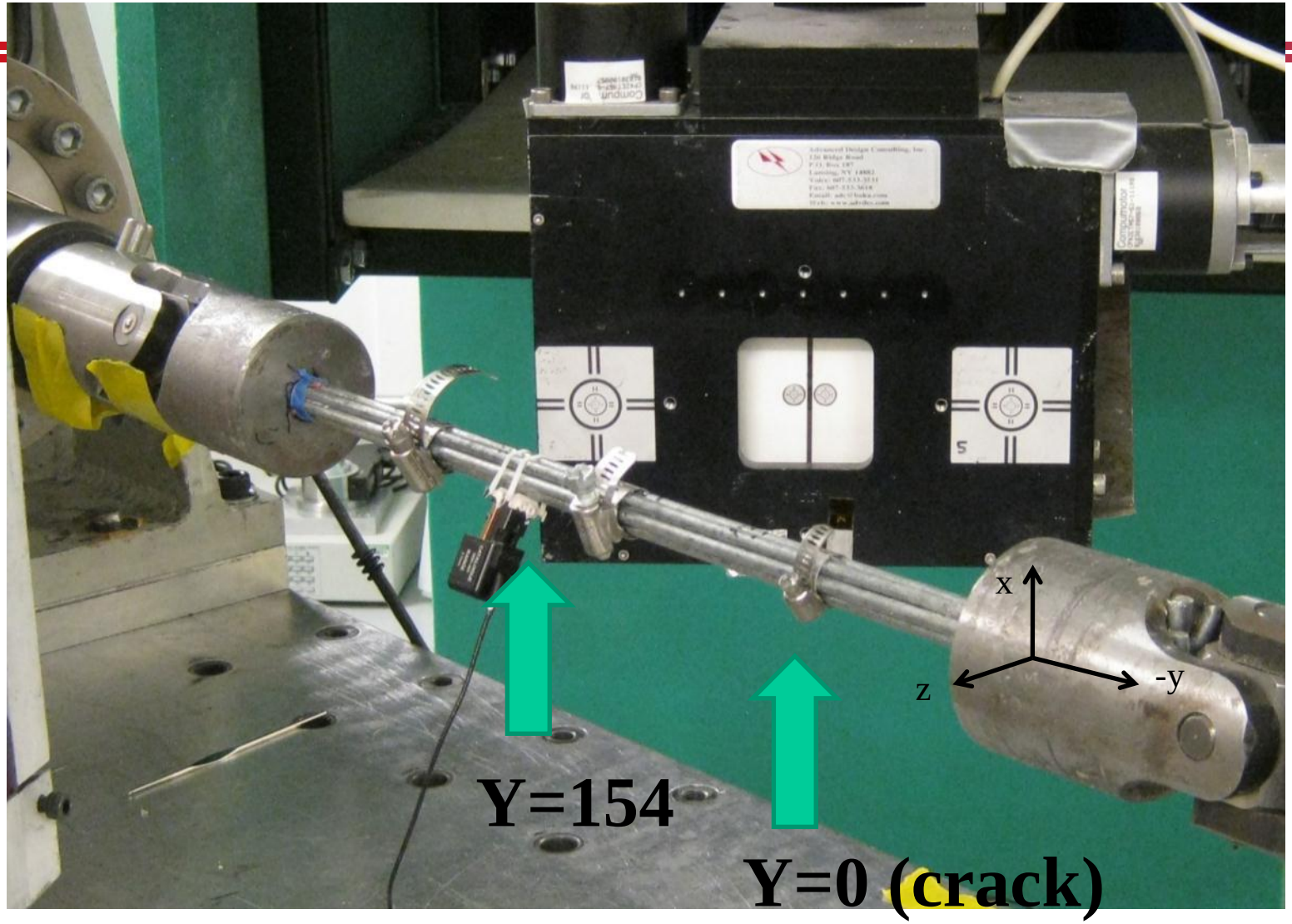


**Configuration: 7 parallel wires**
**Center wire cracked at y=0**

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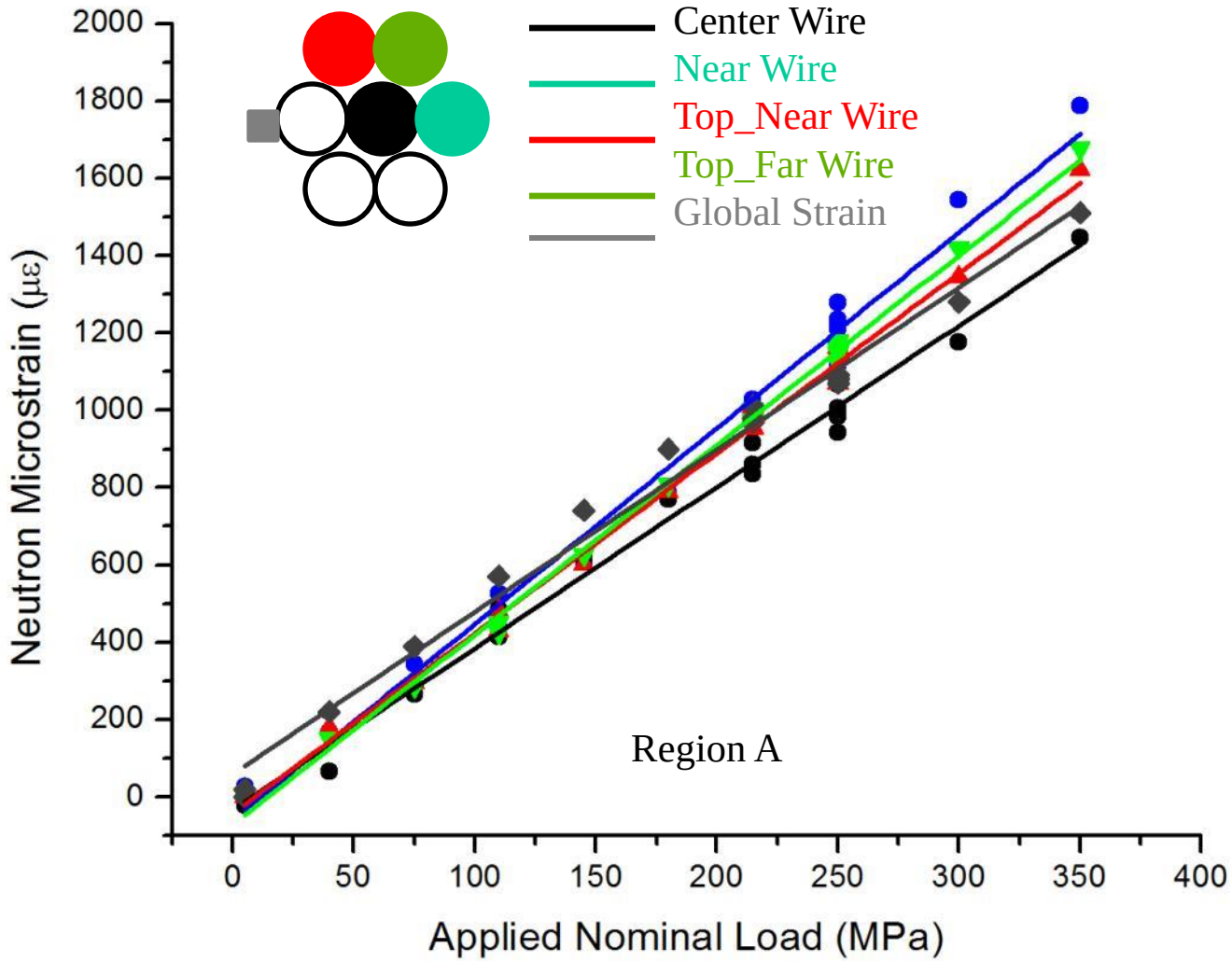
# Longitudinal Stress vs. Strain Plot of Sample III-I in Region A

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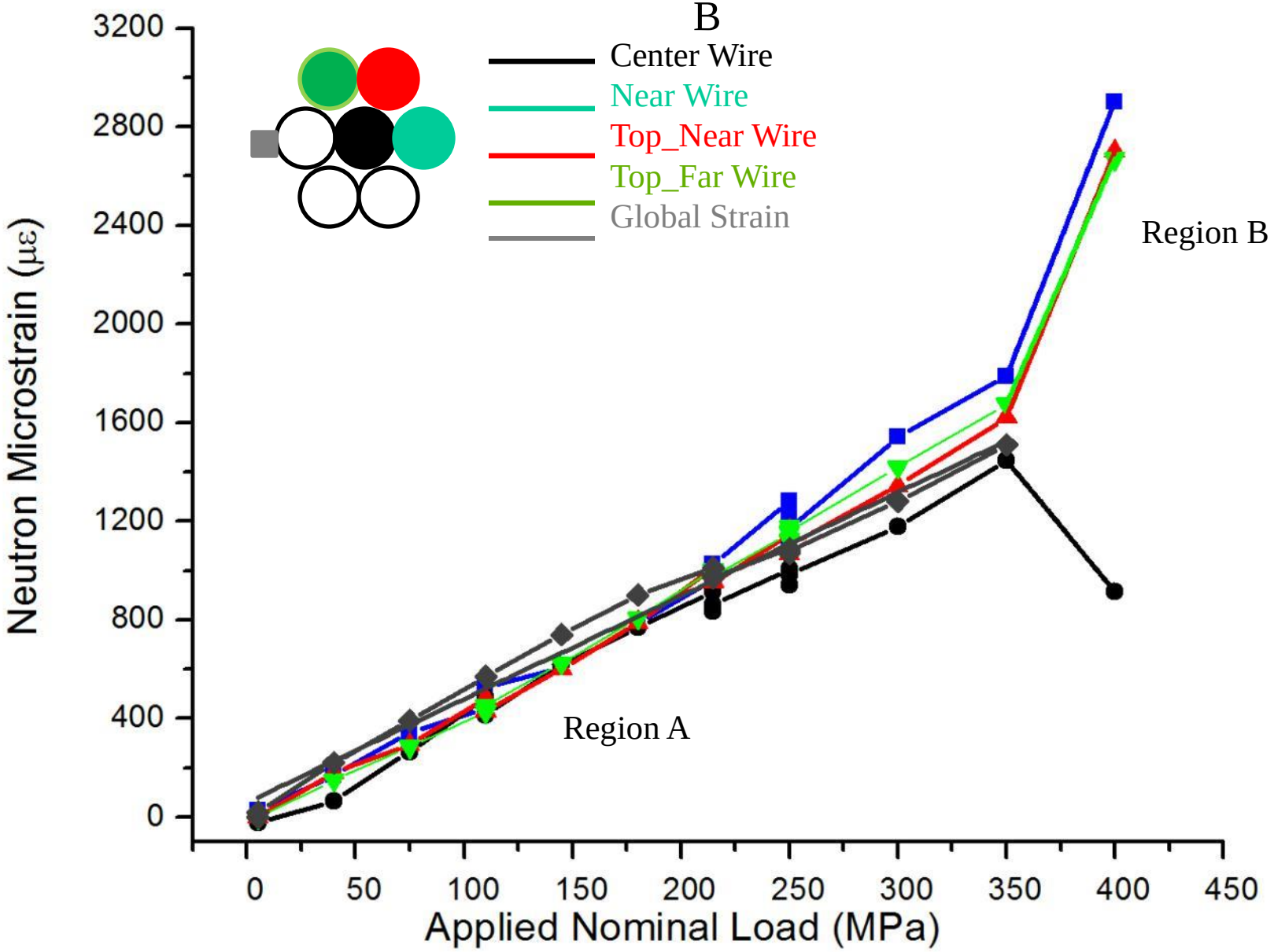
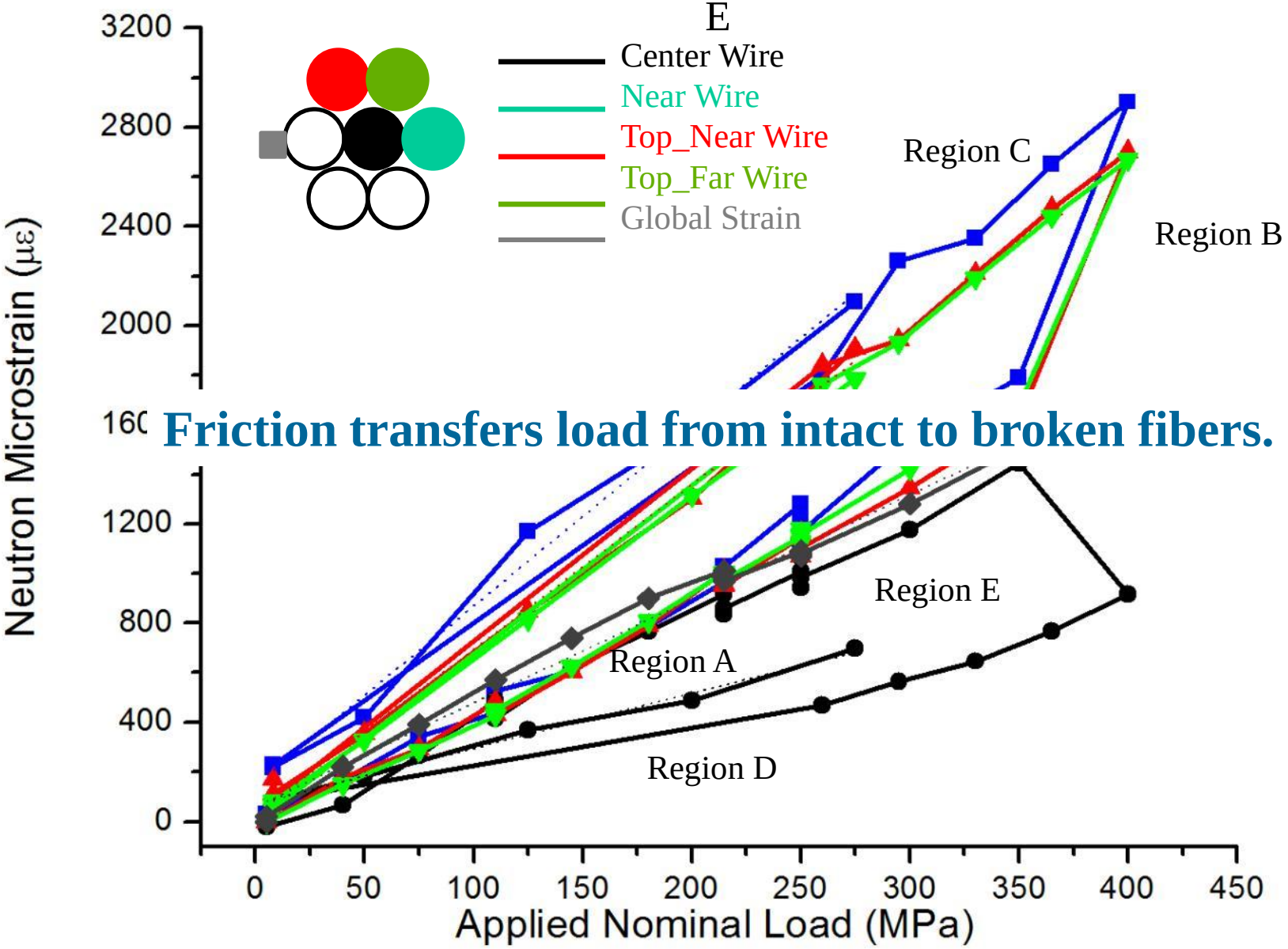
# Longitudinal Stress vs. Strain Plot of Sample III-I in Region B



Longitudinal Stress vs. Strain Plot of Sample III-I in Region E




**Friction transfers load from intact to broken fibers.**

# Nurse Tank Failure Resulted In Death Of Worker

- built in 1976 to the ASME *Boiler and Pressure Vessel Code*
- 53.5 inch-long fracture along longitudinal weld



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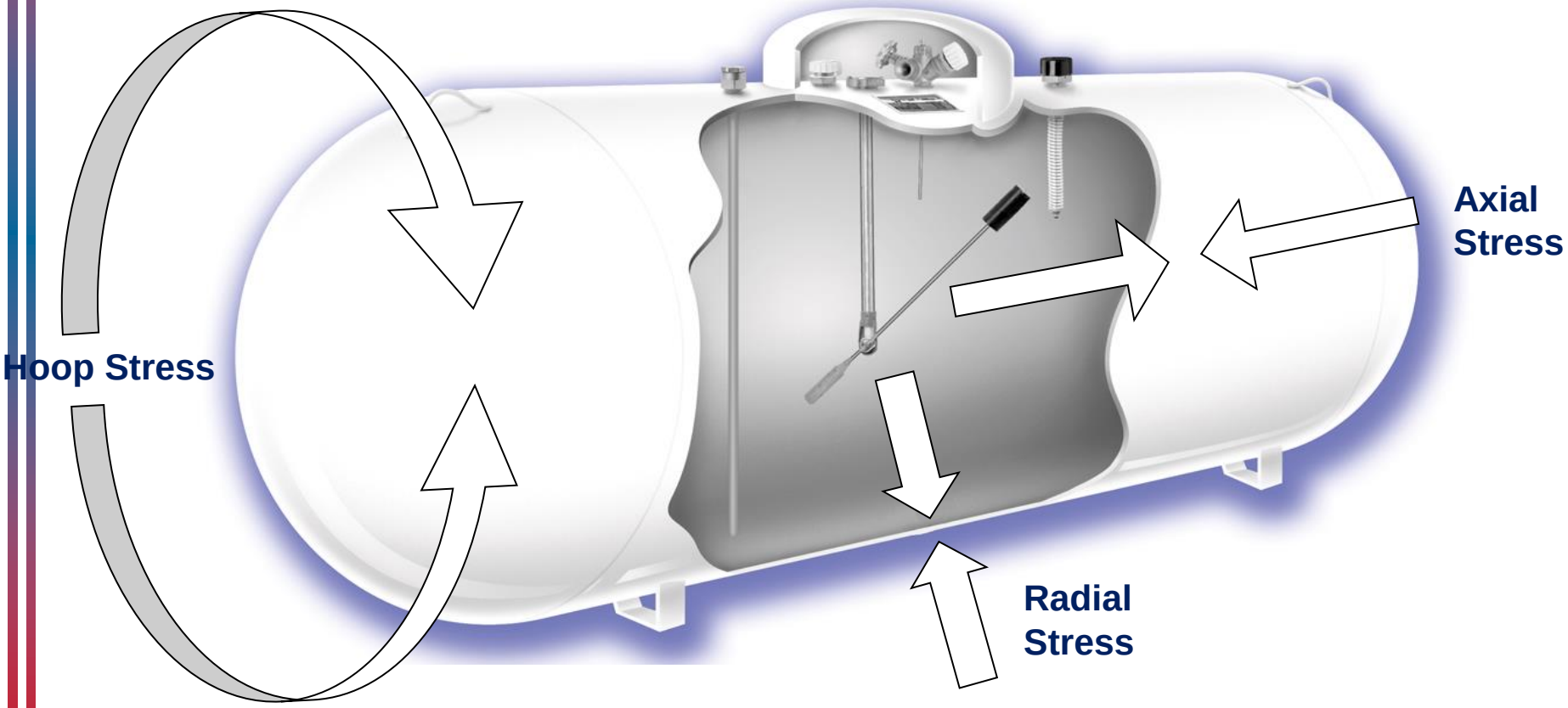
# Primary Stresses

- Affected by  Internal Pressure and Residual Stress from Weld
  - 1.74MPa (250psi) minimum working pressure

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# Circumferential weld taken near end of tank

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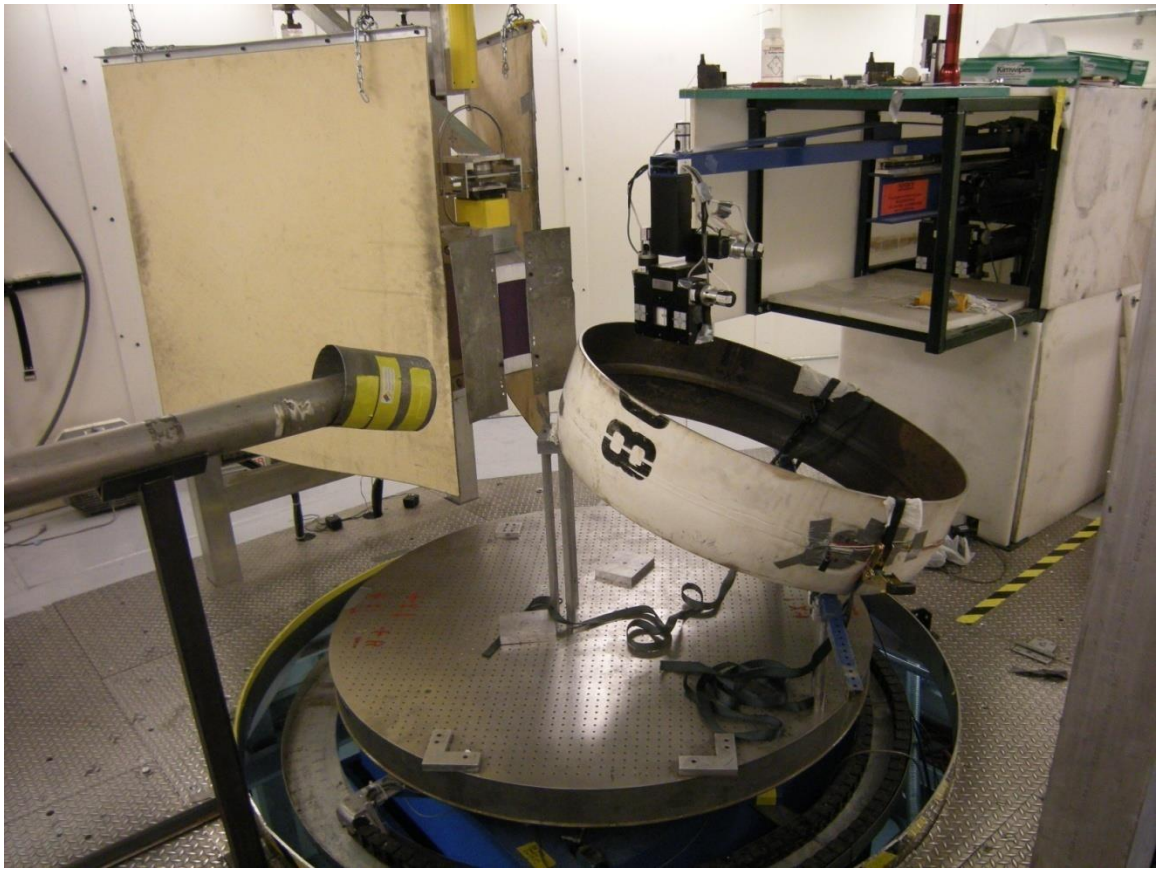
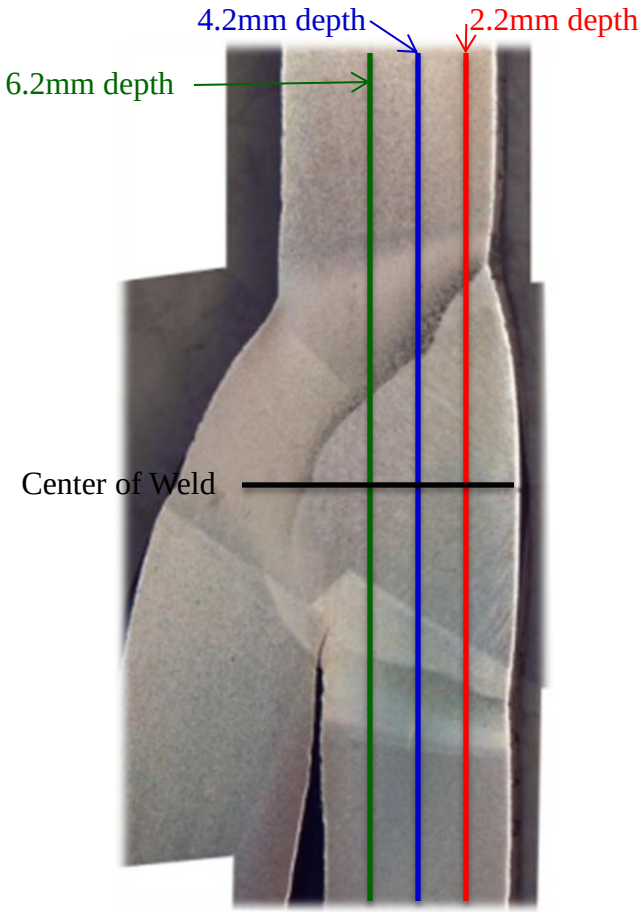
# Spectrometer for Materials Research at Temperature and Stress





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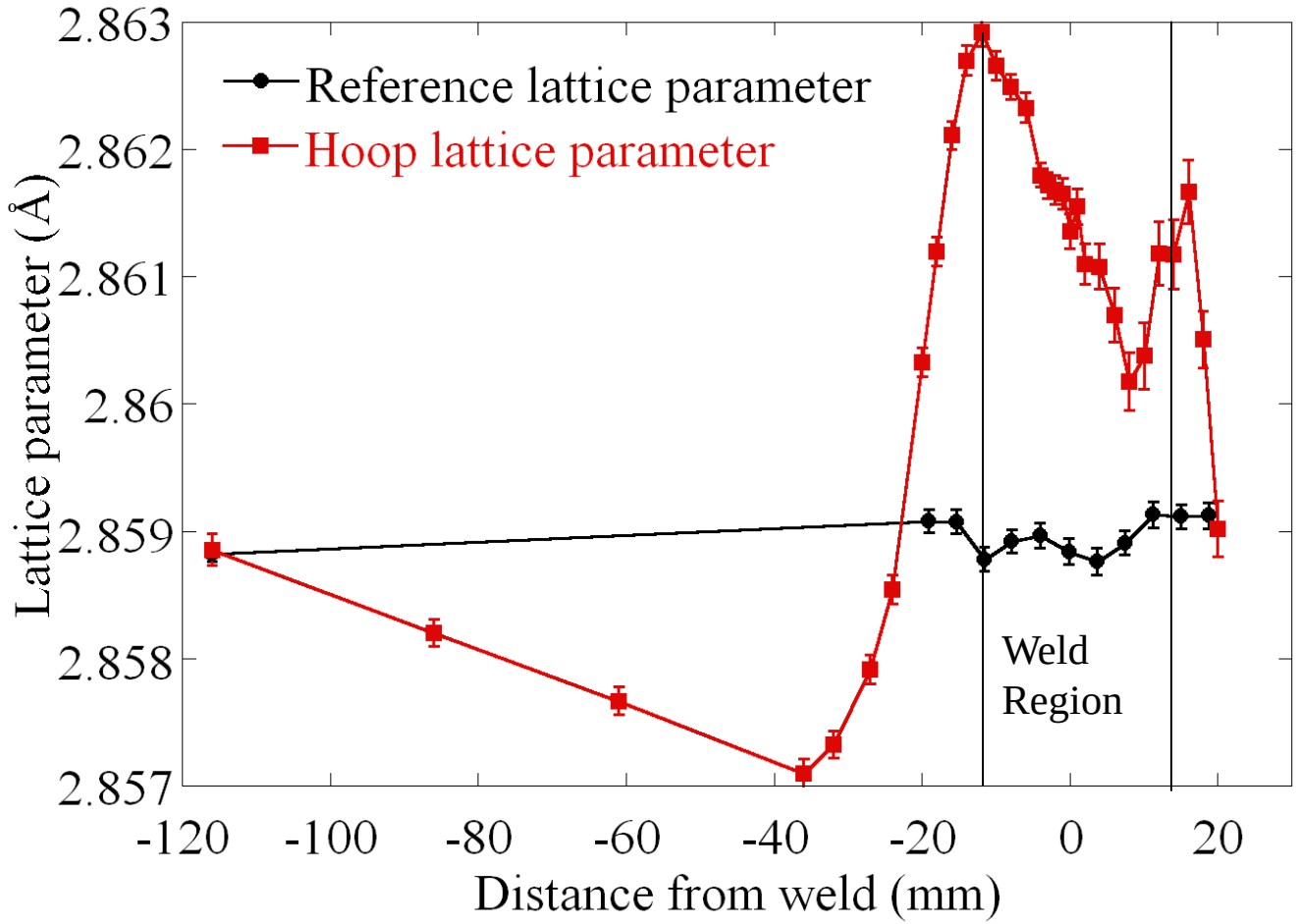
# d0 variation is insignificant

$$\varepsilon = \frac{(d - d_0)}{d_0}$$

$$\sigma = \varepsilon E$$



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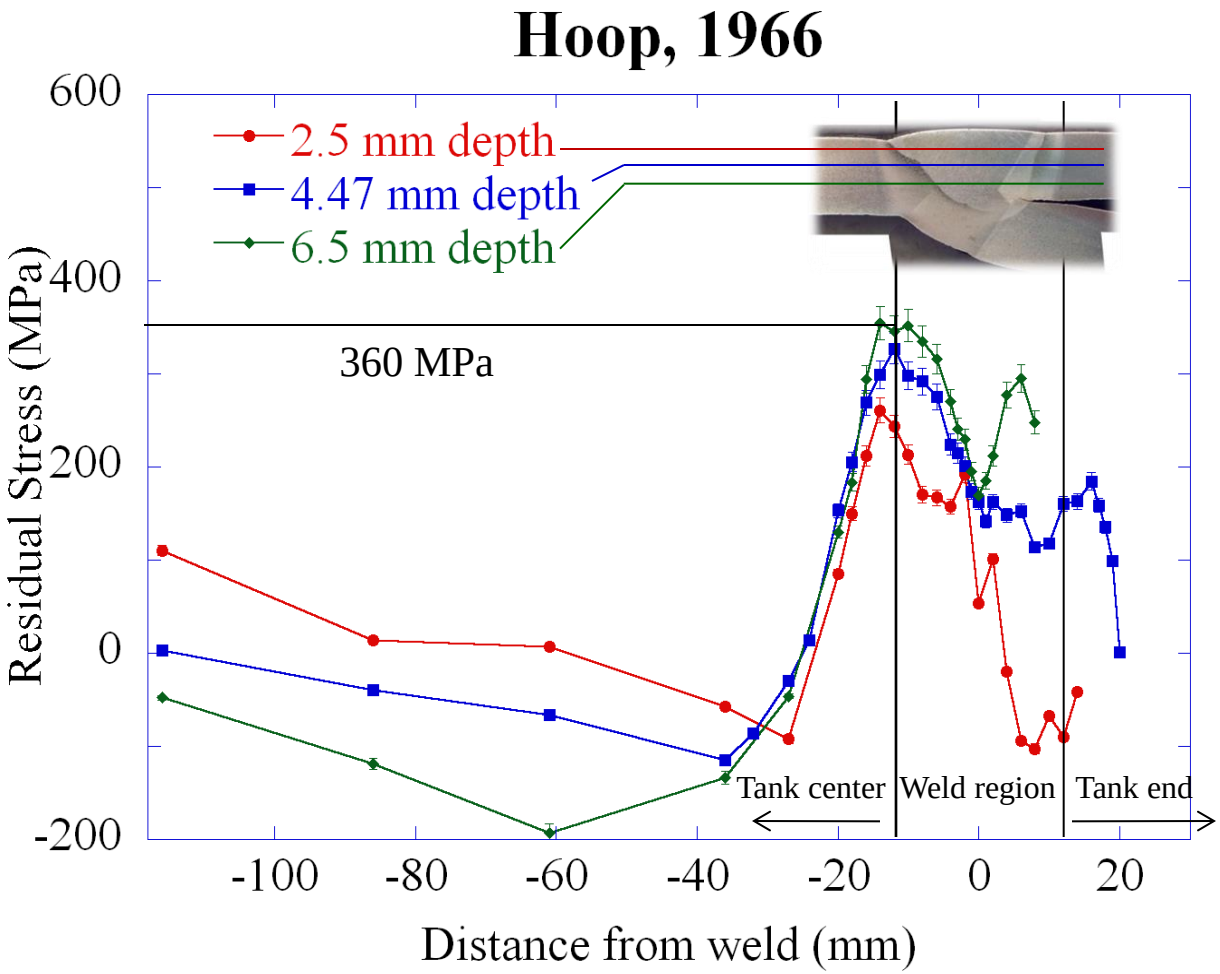
# Hoop Stress 2/3 of UTS



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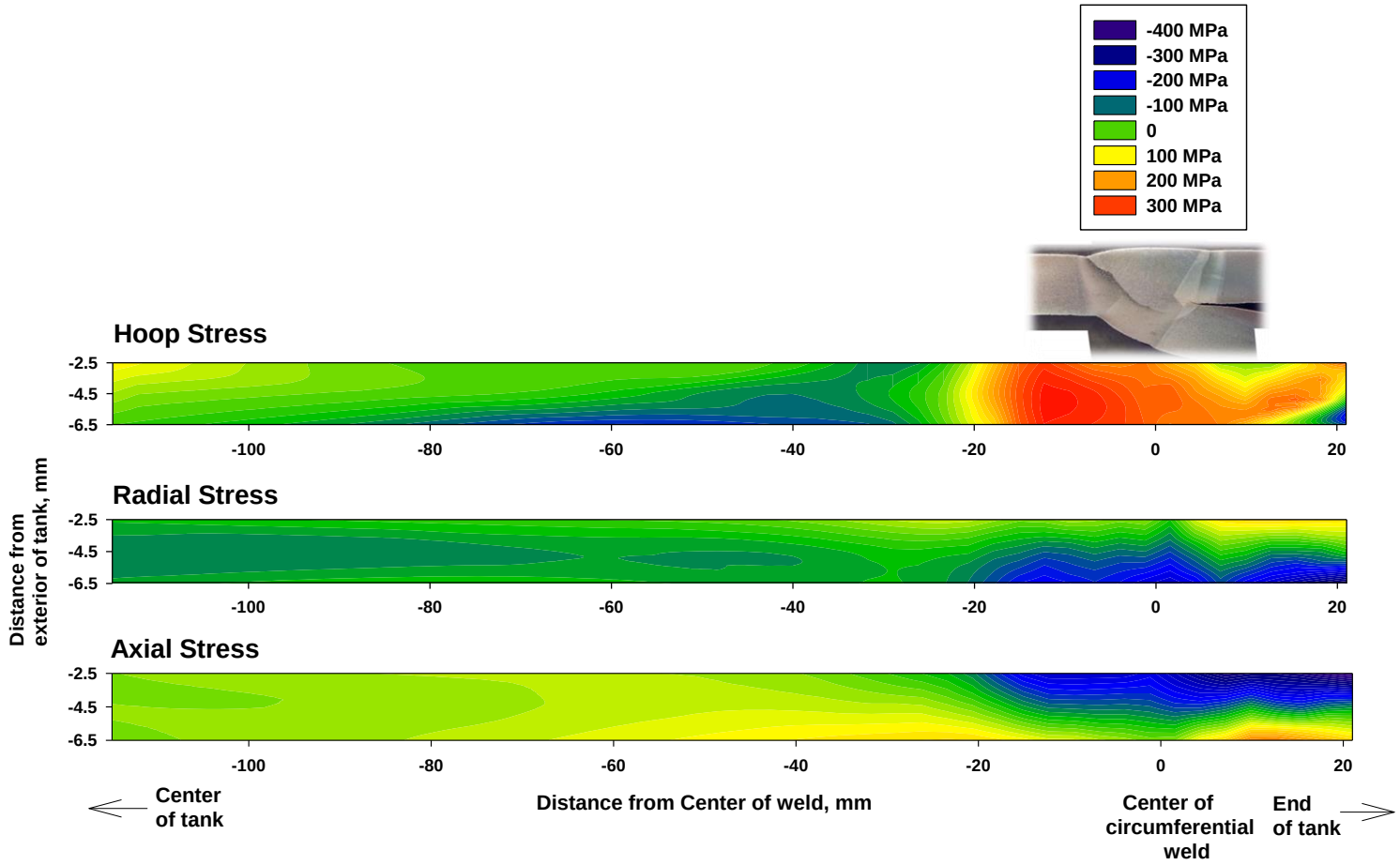
# Residual Stresses In Circumferential Weld Of 1966 Tank



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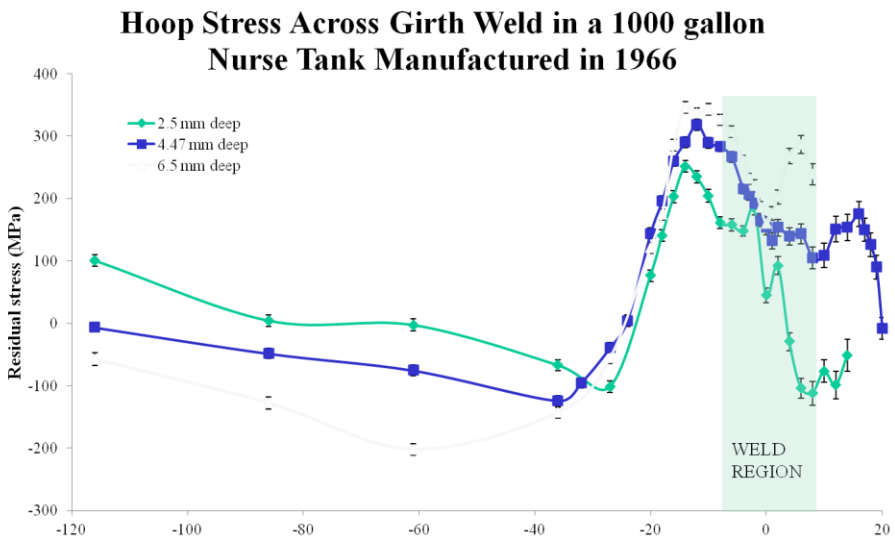
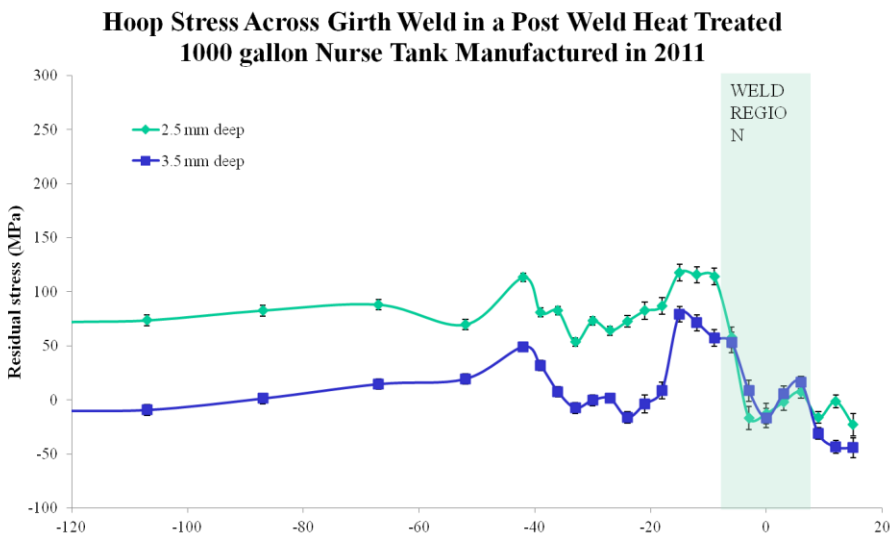
# Heat Treating Reduces Hoop Residual Stress by ~3x

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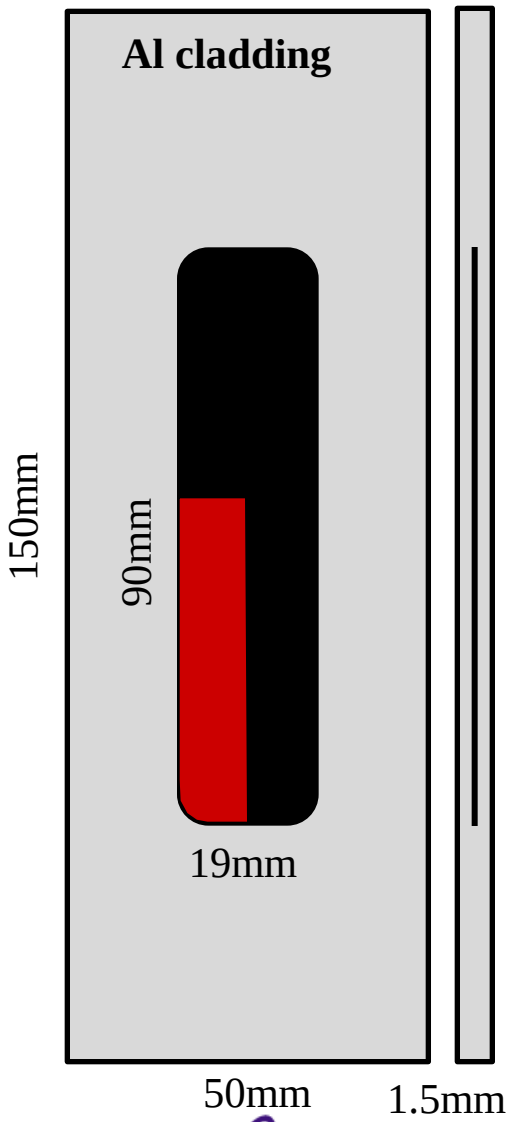
# Residual Stress in Al-Clad U10 WT% Mo Fuel Plates

**Don Brown, Maria Okuniewski, Bjorn Clausen, Jon Almer, John Okasinski**

- Uranium 10wt% molybdenum foil clad with aluminum, Zr diffusion barrier.
- CTE mismatch results in residual stresses which can cause distortions during processing and use.
  - CTE U10Mo ~$10x10^{-6}$/C
  - CTE Al ~$22x10^{-6}$/C
  - CTE Zr ~$5x10^{-6}$/C
- 1.5 mm total thicknes
- 0.25mm thick U10Mo foil
- 0.015mm Zr diffusion barrier.
- Foil was cold rolled, then HIP bonded.
- Diffraction residual stress measurements at the Advanced Photon Source.

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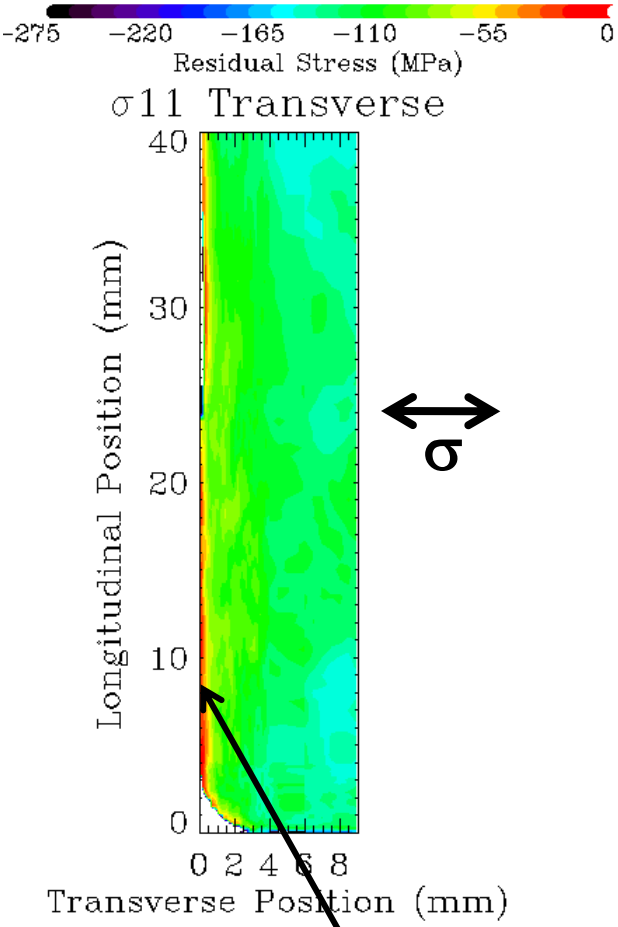
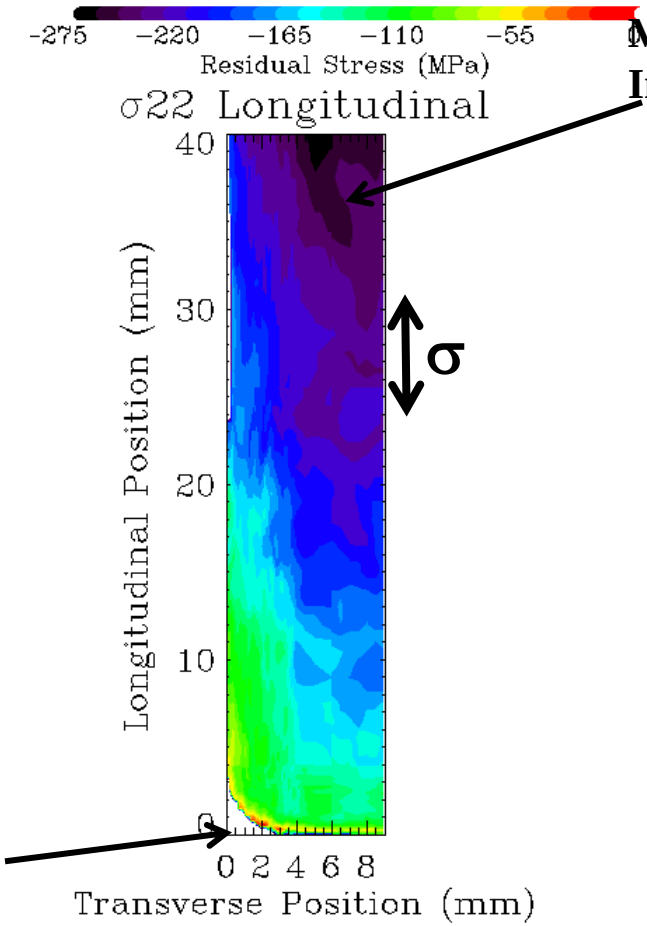
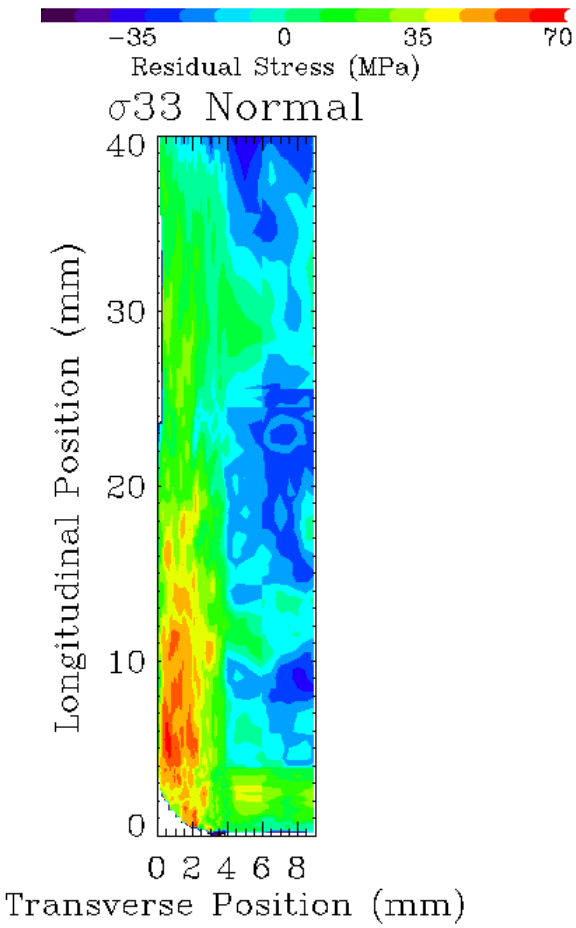
# Residual Stress Tensor Mapped Over ¼ of Foil Using APS



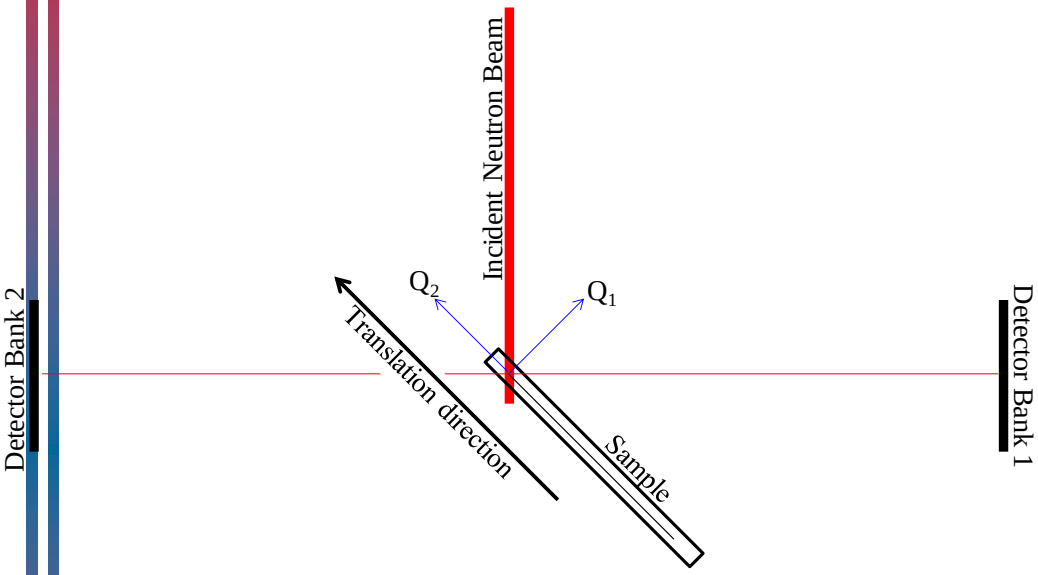

**Max comp > 250MPa**
**Implies large tension in Al cladding**




**Rapid variation near boundary**

**Poor grain statistics in both Al and Zr**

# Residual Stress Measurements in Al-Clad Monolithic Fuel Plates



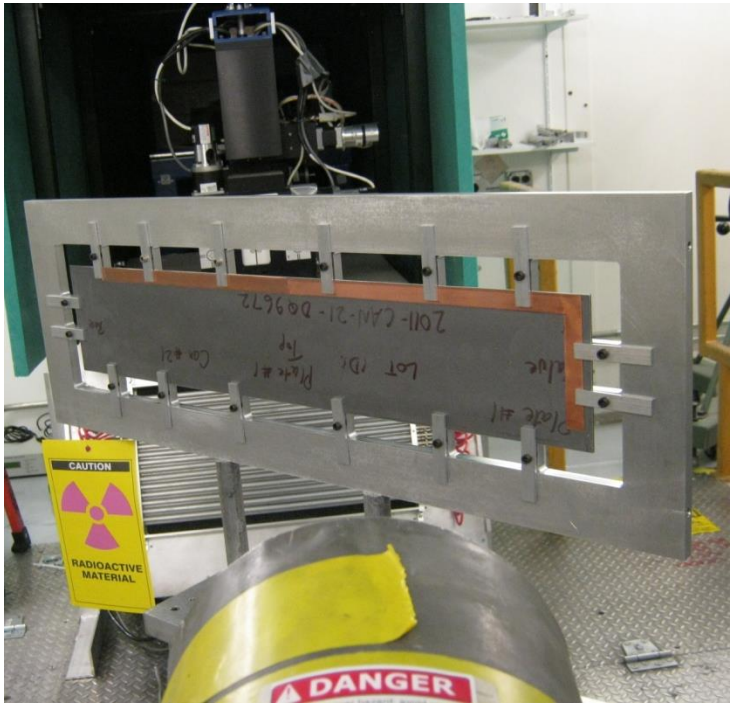




Figure 1. Schematic of diffraction instrument and picture of same.

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# On-Board Calibrant Allows For Mis-Alignment Correction

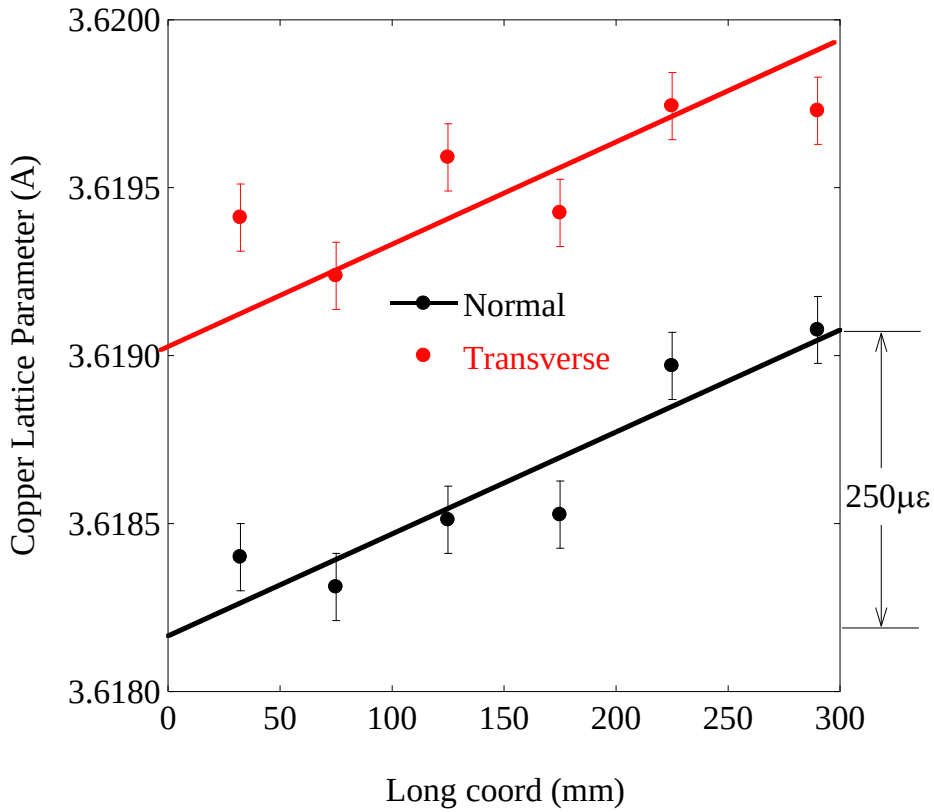
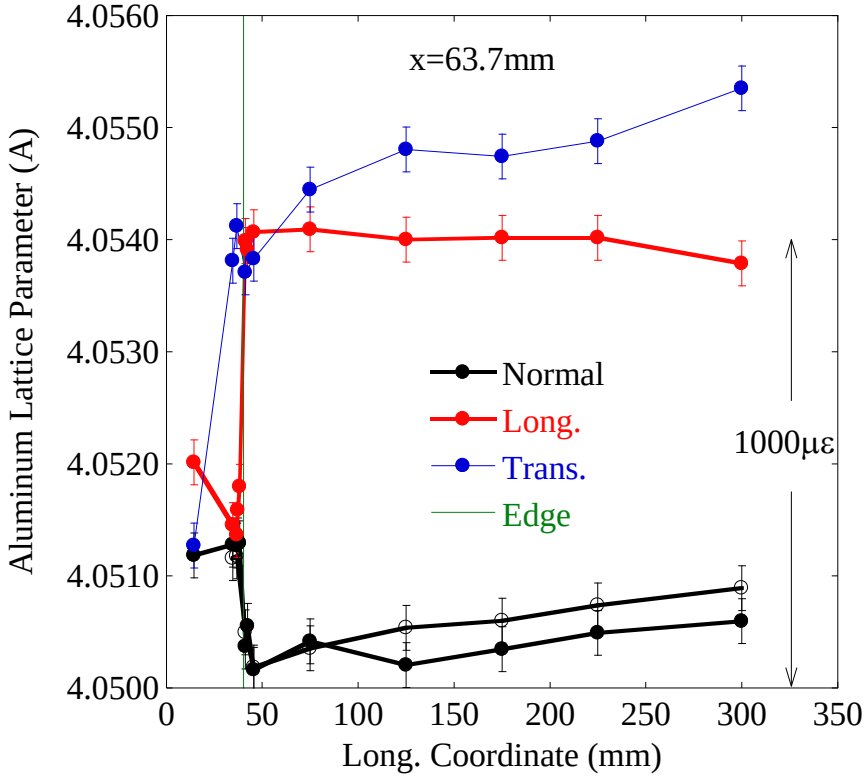
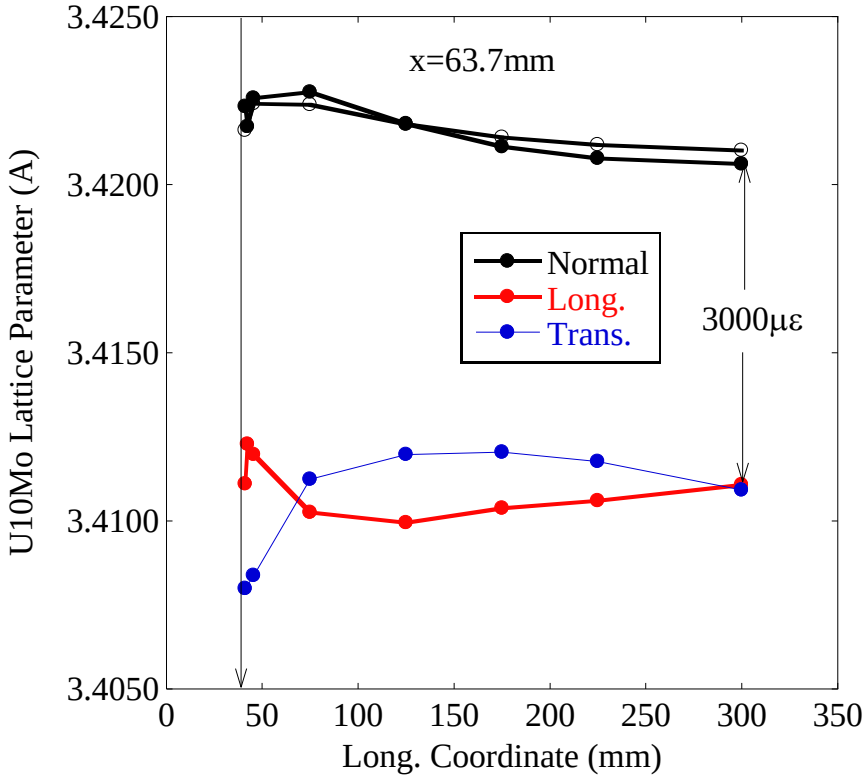


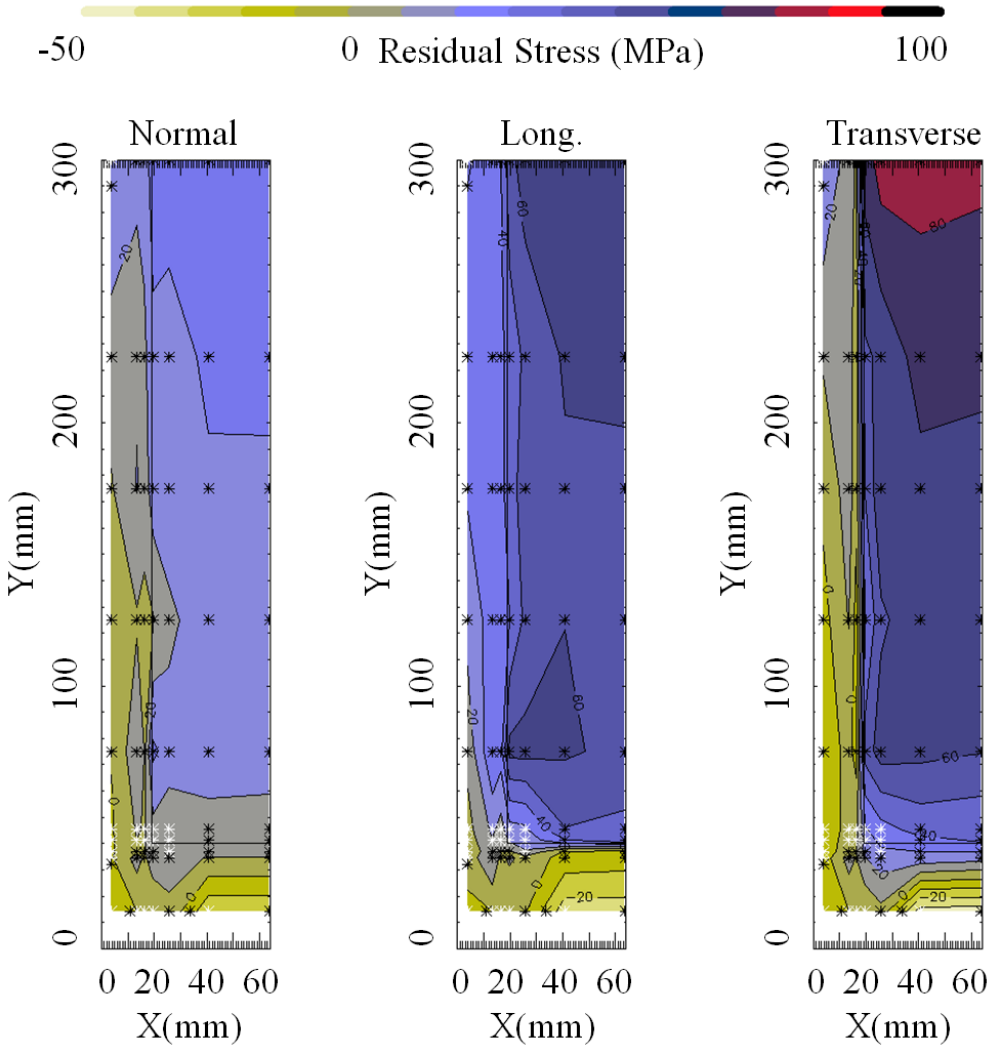

Figure 3. Lattice parameters determined from annealed copper foil used to calibrate the two detector banks and correct for mis-alignment. .

# Raw Lattice Parameters in Aluminum and U10Mo



# Predominantly Tensile Residual Stress in Aluminum Cladding…

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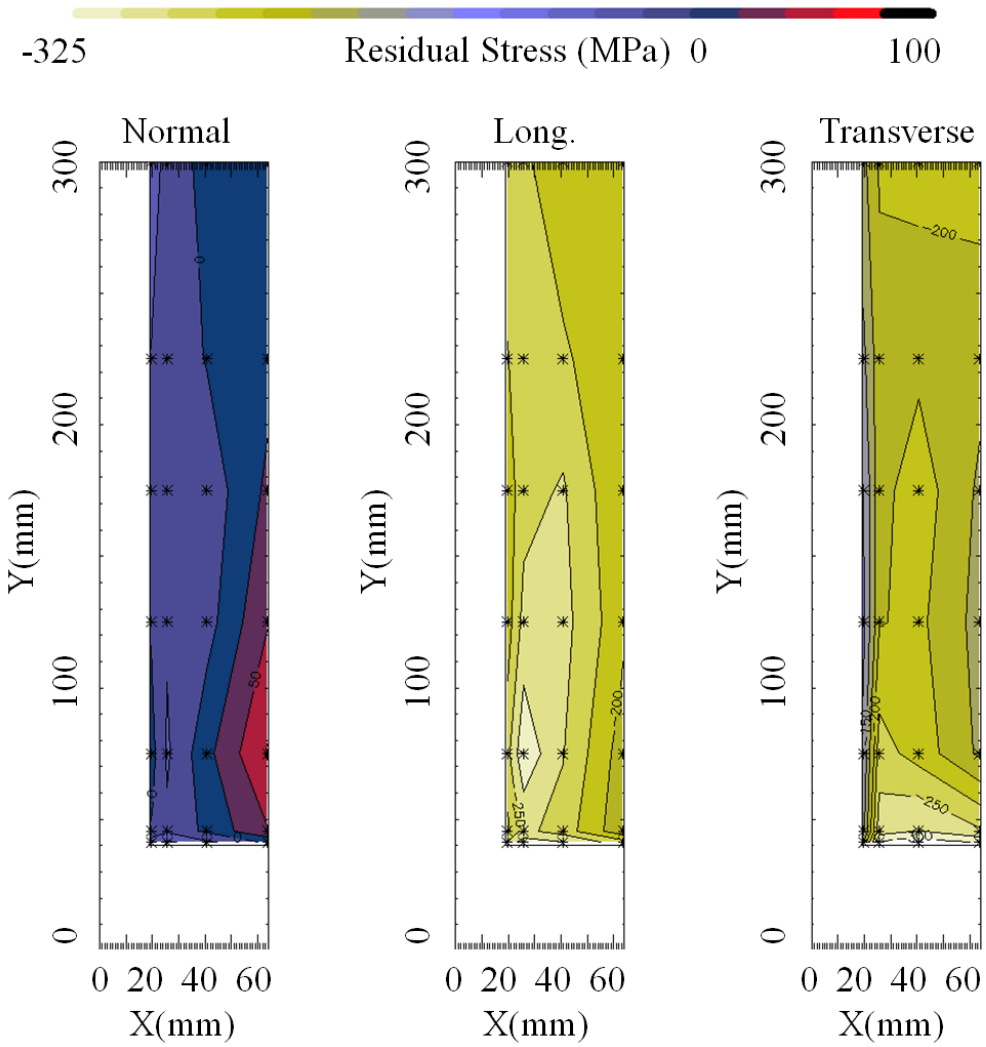
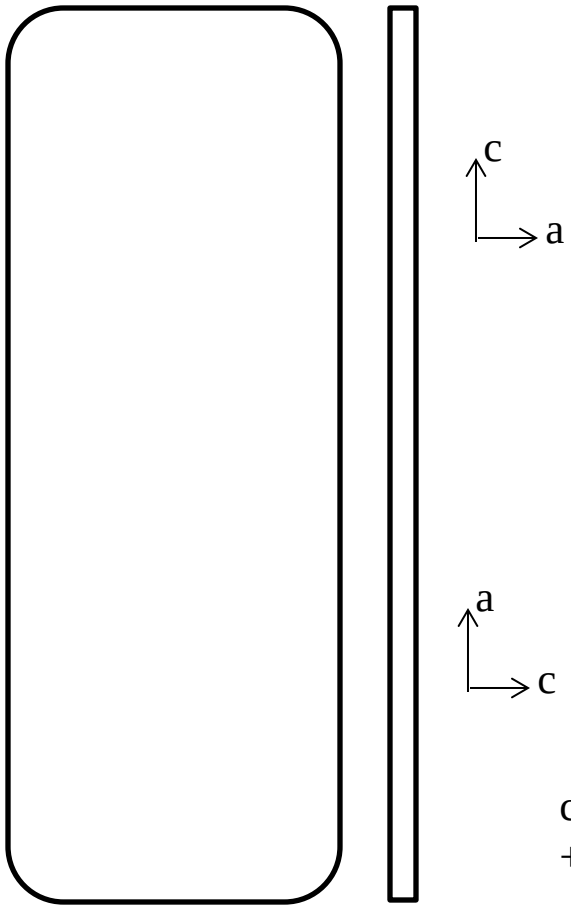
# …Compressive in U10Mo



# We Can Determine The C/A Ratio Of The Zr Layer.

Literature
c/a=1.59270




1.
c/a=1.589±0.0011A
-0.2%

2.
c/a=1.5972±0.0002A
+0.3%

1. Either c is too small or a is too large.
2. Either c is too large or a is too small.

Consistent with either in-plane compression or out-of-plane tension.

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# Conclusions

- Neutron diffraction can provide valuable information to help solve engineering problems.
- Ability to do measurements under extreme environments enables science under processing and/or operating conditions.
- Even under non-ideal conditions, neutrons can still give indications, even if they cannot give complete answers.

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