Neutrons for Study of Engineering Materials

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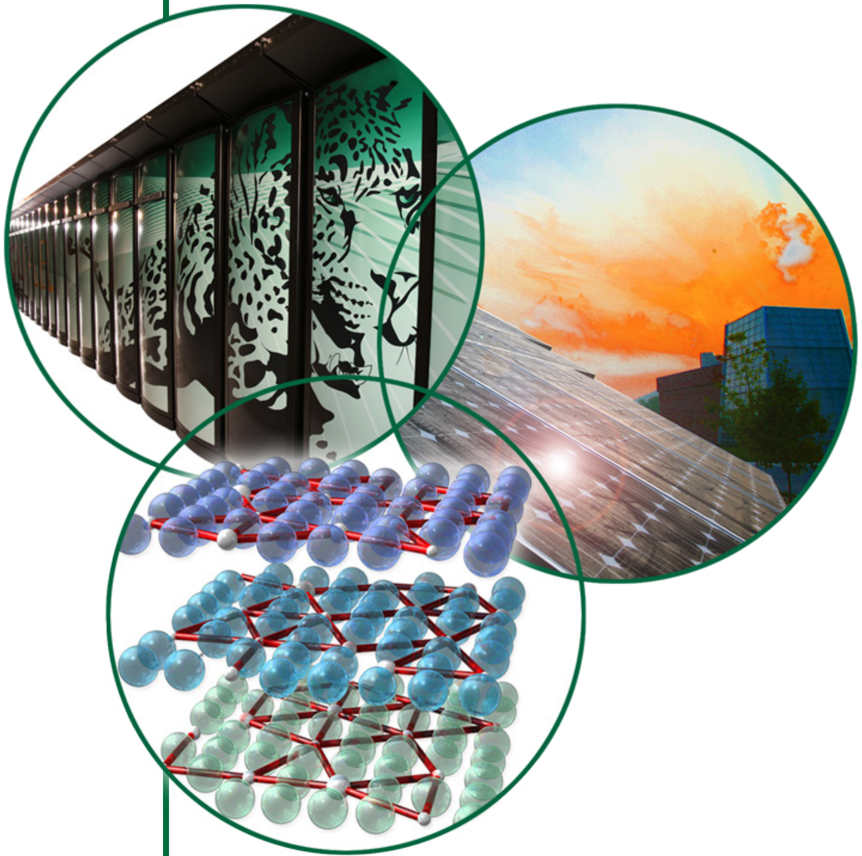
Chemical and Engineering Materials Division

"Neutron Lifecycle" seminar series

7 July 2016



U.S. DEPARTMENT OF ENERGY

OAK RIDGE NATIONAL LABORATORY

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Outline of Presentation

What are the typical measurements done by the scientists in CEMD at the SNS and HFIR?:

I am going to mainly focus on the sort of problems addressed by the Engineering Materials Group of CEMD

What do these measurements tell us?

In this presentation I am going to mainly focus on more ”applied science” problems and what we can learn

What are some highlights from the research program from CEMD?

Again I am going to present highlights from the Engineering Materials Group of CEMD

Neutrons have properties that make them useful for materials science

Only short-range nuclear interactions: so **very penetrating (in most materials)** and they do not heat up your sample

Wavelengths are comparable to inter-atomic spacings, so **good probe of crystal structure**

Energies are comparable to normal modes (phonons, diffusive modes, molecular vibrations), so **good probe of dynamics**

Interactions are strongly dependent on isotope, so labeling is possible, contrast can be varied, etc., to **study specific constituents** in a phase

Magnetic moment so can **study magnetic structures**

Neutrons can also be reflected from surfaces at low glancing angles

What sort of things can you learn using neutrons for chemistry, materials science, and engineering?

Enhanced understanding of structure – property relationships

Impact of microstructure on properties

Better understand materials synthesis and processing

Study materials under simulated "real world" conditions

What sort of instruments do we find in CEMD?

Diffraction (POWGEN, NOMAD, SNAP, TOPAZ)

Spectroscopy (VISIONS, SEQUOIA, BASIS)

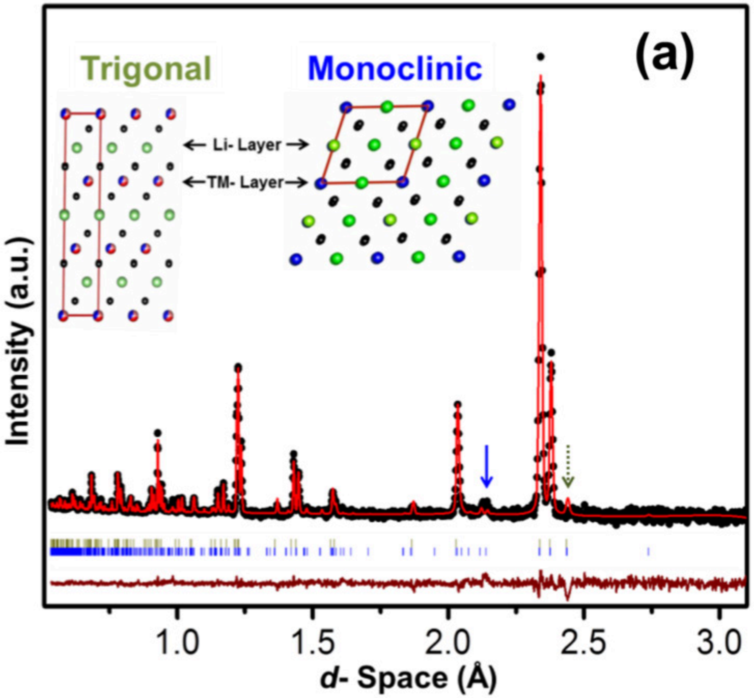
Small Angle Scattering (GPSANS, USANS, LiqRefl)

Imaging and Tomography (Imaging, VENUS*)

Engineering (NRSF2, VULCAN)

So what sort of materials science problems can I solve with neutrons?

1. Structure and Quantitative Phase Analysis



Quantitative phase analysis of neutron diffraction data from $Li_{1.2}Mn_{0.55}Ni_{0.15}Co_{0.10}O_2$ lithium-ion battery cathode conducted on POWGEN (SNS)

Mohanty et al., Chem Mater 25 4046 (2013)

Conventional Methods

XRD, TEM

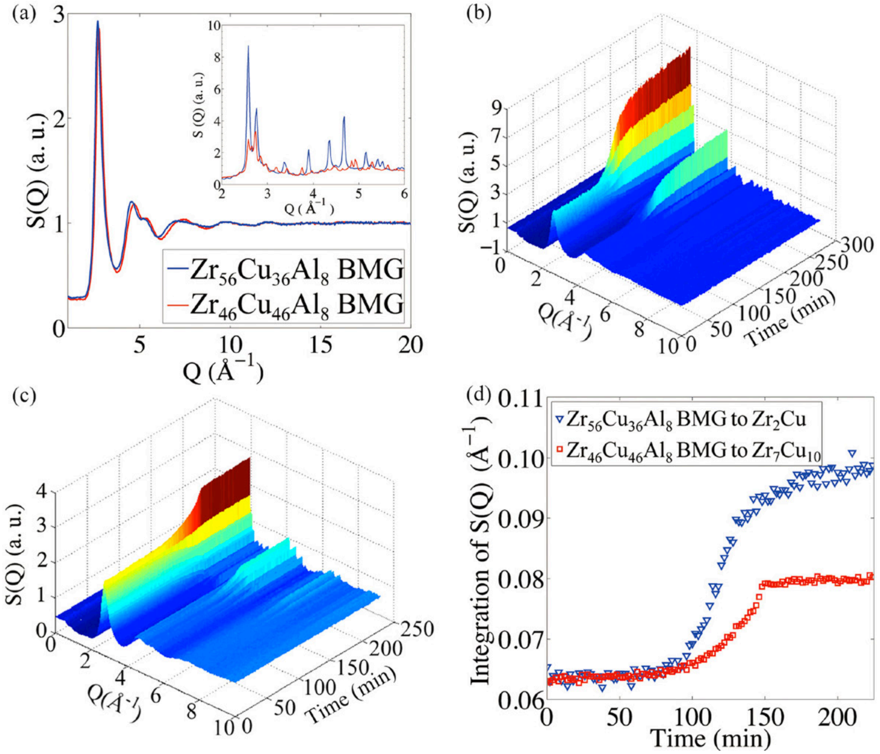
Why neutrons?

Nondestructive
Not limited to near-surface region
Spatial-resolved mapping (VULCAN)
Not dominated by heavy elements (great for H, Li, etc.)
Large sampling volume = better statistics

Potential instruments

POWGEN (SNS)
NOMAD (SNS)
NPD (HFIR)
WAND(HFIR)

2. Reaction Pathways and Kinetics



In-situ crystallization study of $Zr_{56}Cu_{36}Al_8$ and $Zr_{46}Cu_{46}Al_8$ conducted on NOMAD (SNS)

Lan et al., APL 105 201906 (2014)

Conventional Methods

XRD

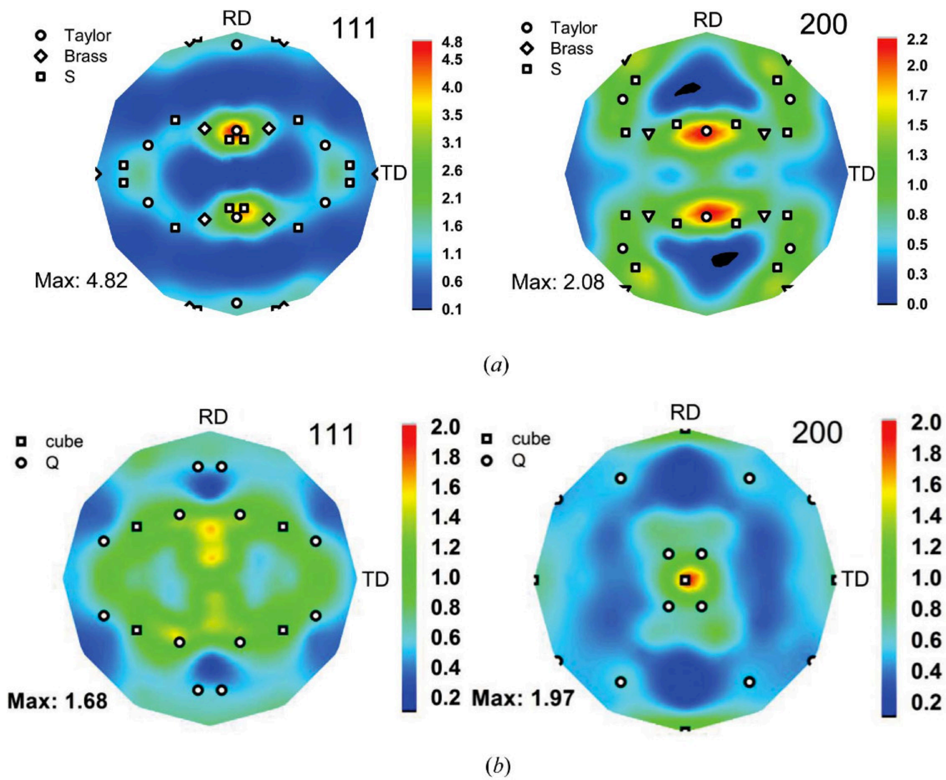
Why neutrons?

Sample environments (furnaces, cryostats, pressure cells, magnets, rheometers, etc.) can be large/complex and still not compromise the data

Potential instruments

POWGEN (SNS)
NOMAD (SNS)
VULCAN (SNS)
NPD (HFIR)
WAND(HFIR)
GP-SANS (HFIR)

3. Preferred Orientation (Texture)



Pole figures for Al-2%Mg: (a) cold rolled and (b) annealed showing the recrystallization texture collected on VULCAN (SNS)

Stoica et al., J Appl Cryst (2014) 47 2019

Conventional Methods

XRD (Schultz geometry), EBSD

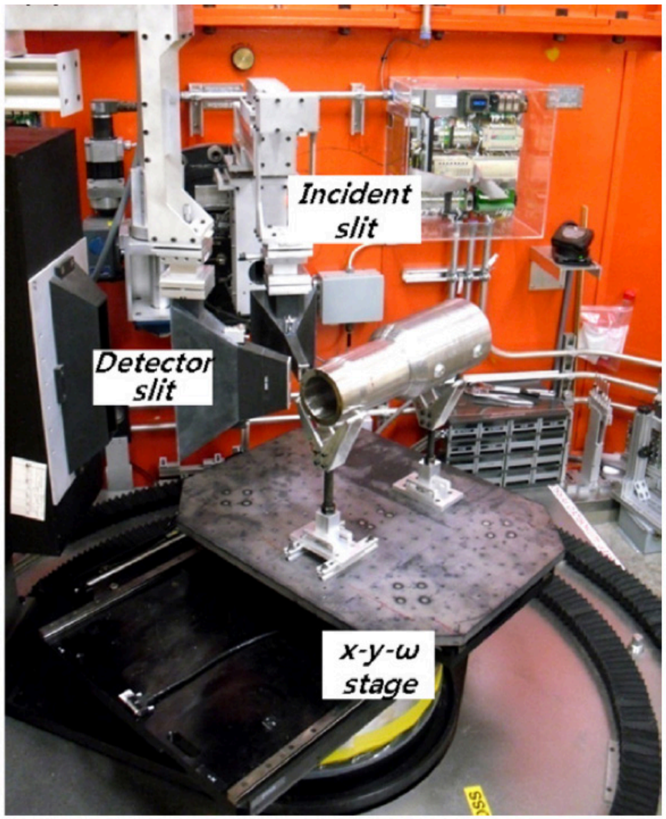
Why neutrons?

- Nondestructive
- Not limited to near-surface region
- Spatial-resolved mapping
- Multiple crystallographic directions in one measurement
- Large sampling volume = better statistics

Potential instruments

- POWGEN (SNS)
- NOMAD (SNS)
- VULCAN (SNS)
- VENUS (future SNS)

4. Residual Stresses



Residual stresses mapped in pipe with dissimilar metal weld and overlay (ferritic and austenitic) using 4mm cube gauge volume on NRSF2 (HFIR)

Woo et al. MSEA <u>528</u> 8021(2011)

Conventional Methods

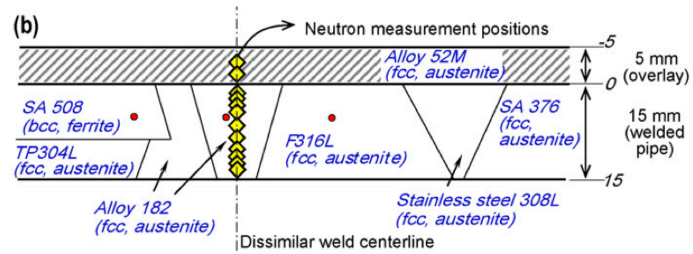
XRD ($\sin^2$ psi method), hole drilling, contour method

Why neutrons?

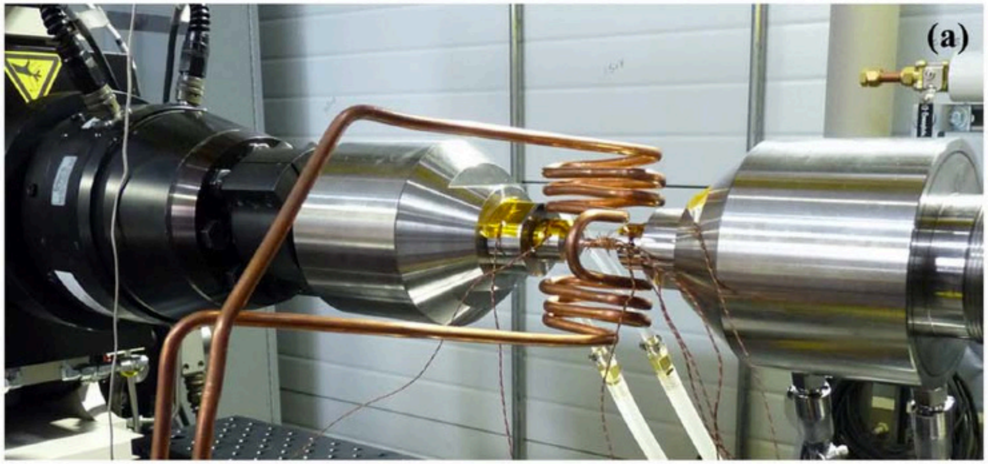
- Nondestructive
- Not limited to near-surface region
- Spatial-resolved mapping
- Multiple crystallographic directions in one measurement

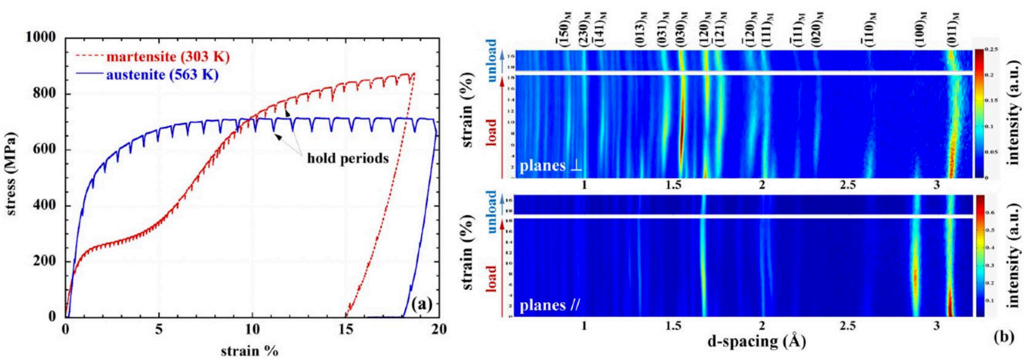
Potential instruments

- VULCAN (SNS)
- VENUS (future SNS)
- NRSF2 (HFIR)



5. Mechanical Response





Effect of temperature and multiaxial (torsion + tension) loading on NiTi shape memory alloy – data collected at VULCAN (SNS)

Benafan et al. Rev Sci Instrum 85 103901 (2014)

Conventional Methods

limited

Why neutrons?

Nondestructive

Not limited to near-surface region

Spatial-resolved mapping

Multiple crystallographic directions in one measurement (measure elastic response of all hkl for a given load vector)

Specialized sample environments (load frames, pressure cells)

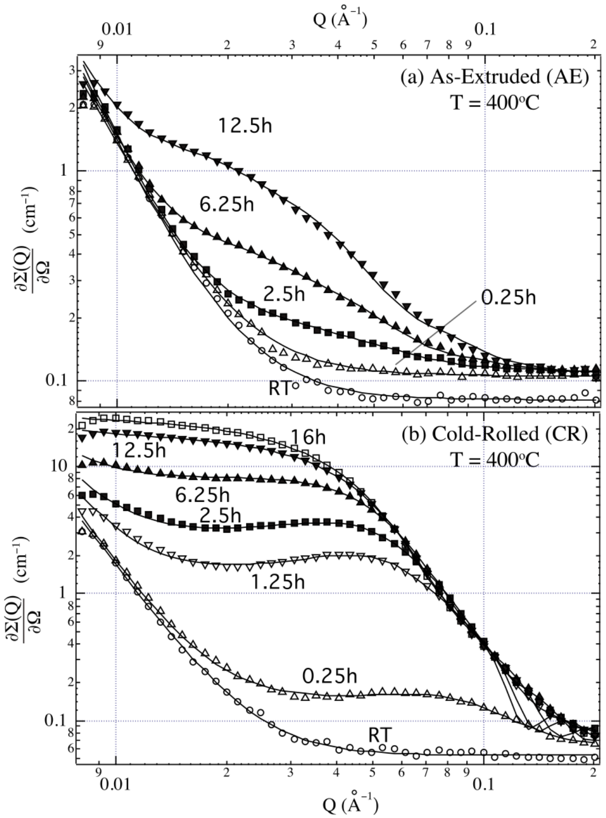
Potential instruments

VULCAN (SNS)

VENUS (future SNS)

NRSF2 (HFIR)

6. Porosity, Precipitates, etc



Evolution of nanoprecipitates in a beta-Ti alloy – data collected at GP-SANS (HFIR)

Coakley et al. J Alloys Compounds <u>623</u> 146 (2015)

Conventional Methods

microscopy

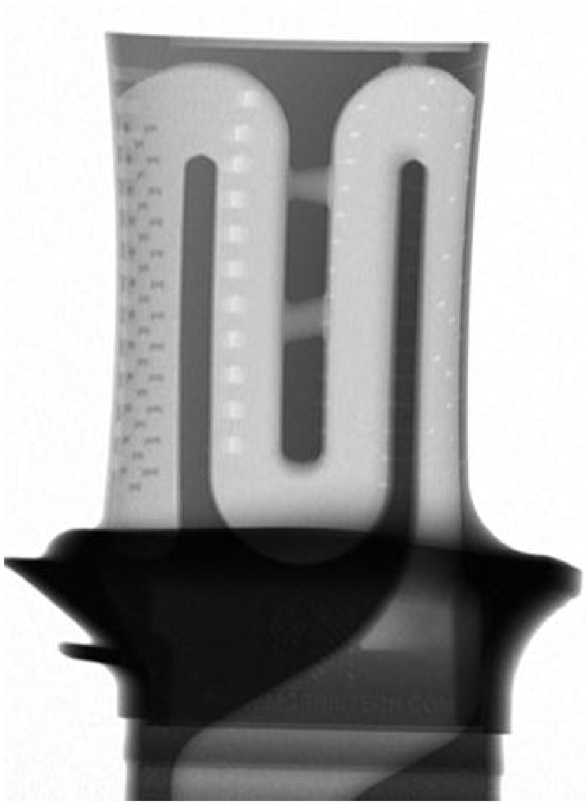
Why neutrons?

Nondestructive
Not limited to near-surface region
Samples do not need to be crystalline
Specialized sample environments (load frames, pressure cells)

Potential instruments

USANS (SNS)
VENUS (future SNS)
GP-SANS (HFIR)

7. Microstructure



Neutron radiograph of an Inconel turbine blade made by additive manufacturing – data collected at Imaging (HFIR)

Watkins et al. AM&P 171 23 (March 2013)

Conventional Methods

Microscopy, x-ray radiography/tomography

Why neutrons?

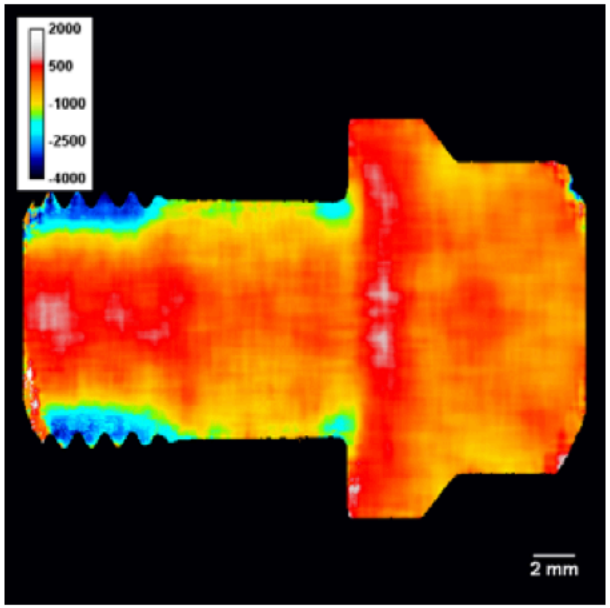
Nondestructive
Not limited to near-surface region
Samples do not need to be crystalline
Different contrast (compared with x-ray)
Specialized sample environments (load frames, pressure cells)

Potential instruments

VENUS (future SNS)
Imaging (HFIR)

8. Multiscale Characterization and Multimodality

Strain 4.16A edge



TOF image of bolt. Colors indicate position of Bragg edge, which is in this case an indicator of strain.

Anton Tremsin (UC Berkeley) unpublished data

Conventional Methods

???

Why neutrons?

Combine imaging/tomography with Bragg Edge and Resonance to get full 3D characterization of relatively large and complex samples with high (10 micron) spatial resolution

Potential instruments

VENUS (future SNS)

Strain mapping appears simple, but often is not

NRSF2 and VULCAN

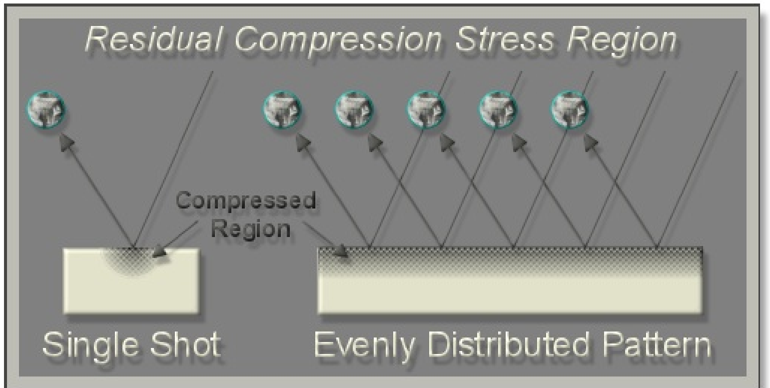
Method assumes a large number of randomly oriented and dispersed grains within the “gauge volume”

Potential for peak shift artifacts near sample edges (internal and external)

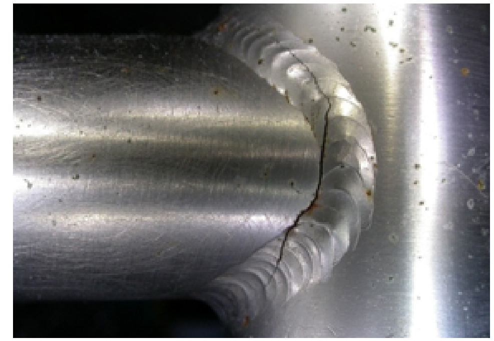
What is really measured is “d-spacing”. Elastic strain is determined by reference to a “d0” sample. Plastic strain is not (easily) measured.

Stress determination requires measuring the strain in multiple directions (at least the three principle axes, if these are known) and knowing the elastic constants and Poisson’s ratio. Therefor the sample must be reoriented on the goniometer at least once, unless special symmetry conditions can be assumed

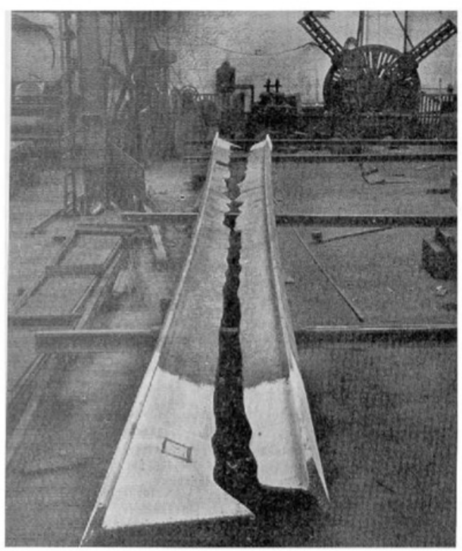
Residual Stress: What is it good for? ~~Absolutely nothing!~~



Good: Shot-peening is a method of introducing wanted compressive stress at the surface of materials.



Bad: Sometimes, processing materials (e.g. welding) introduces stresses that induce cracking and failure

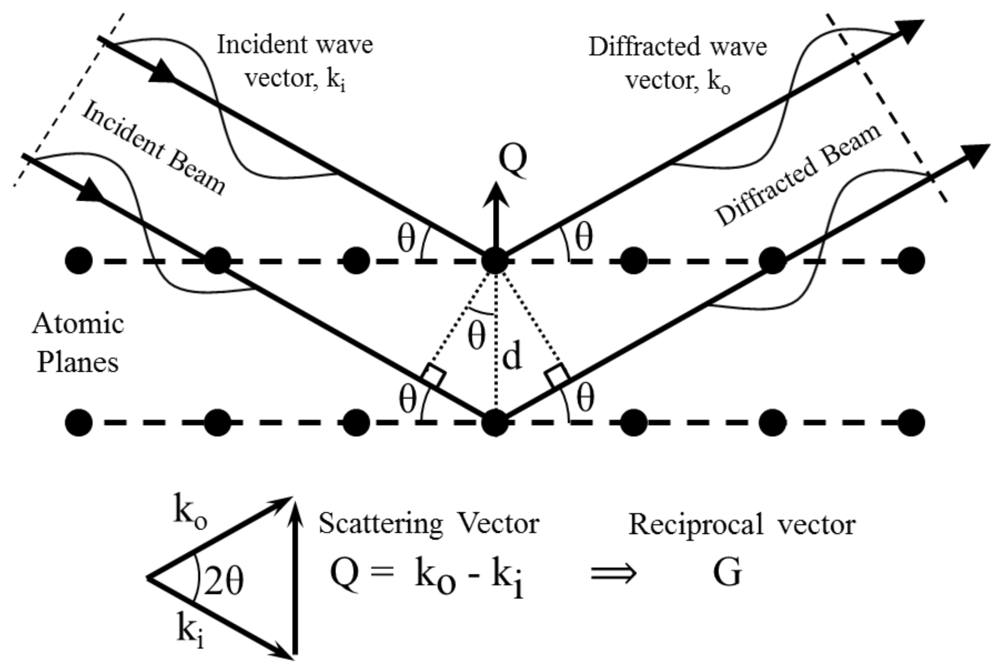


Ugly: relaxation of residual stress can also cause failure. This is a giant I-beam that split from this effect

We use Bragg's Law to determine the lattice spacing

- **Bragg's law relates the wavelength, crystallographic lattice spacing and the scattering angle.**

$$\lambda = 2d_{hkl}\, sin(\theta_{hkl})$$

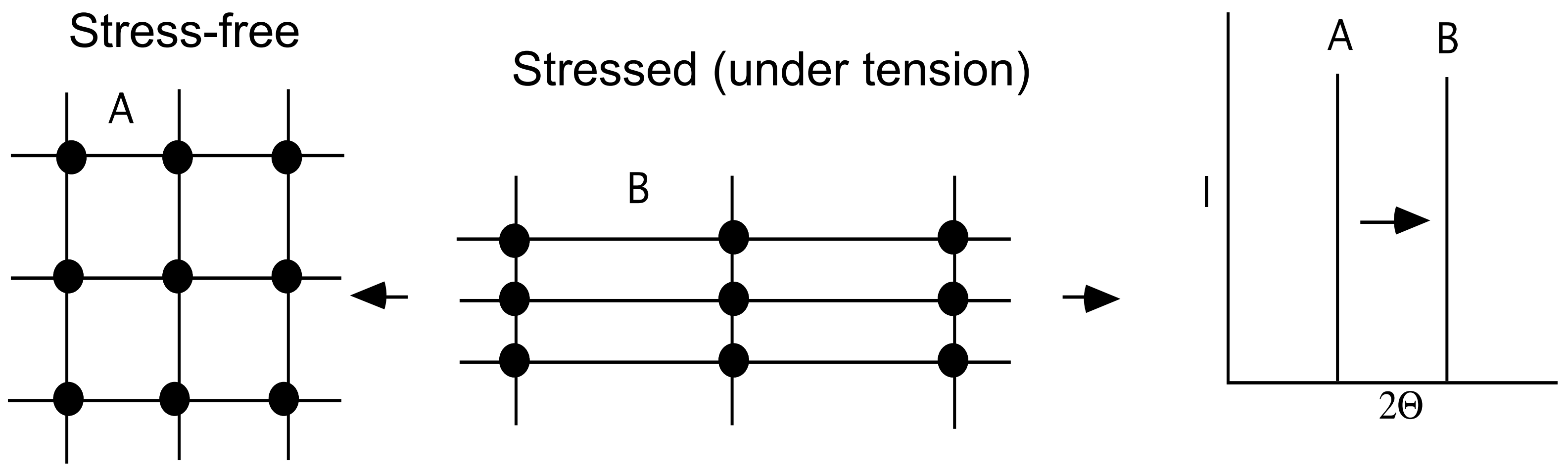


Residual Stress:
Where does it come from, how to measure it?

Many processes lead to residual stress:

- The inhomogeneous distribution of plastic deformation
- Differences of physical properties of phases/materials
- Mechanical properties

- **In diffraction methods, we can measure it by changes in atomic d-spacing.**



We use Hooke's Law to determine strain from lattice spacing

- Strains* determined from the ratio of the d-spacing (d_0) to the stress-free d-spacing:

$$\varepsilon_{ij} = \frac{d_{ij} - d_0}{d_0}$$

- Residual stresses are determined from the 3-D strain as follows:

$$\sigma_{ij} = \frac{E}{(1+\nu)}\left[\varepsilon_{ij} + \frac{\nu}{(1-2\nu)}(\varepsilon_{11} + \varepsilon_{22} + \varepsilon_{33})\right]$$

Where:

$$\nu = Poisson's\ Ratio \qquad E = Young's\ Modulus$$

$$\mathcal{E} = strain$$

* Diffraction methods only measure elastic strains

NOTE: in order to determine residual stresses for neutron diffraction, you need a MINIMUM of 3 directions of strain. Also, in the equation the strains ε_{11} , ε_{22} and ε_{33} are principal strains, but three orthogonal strains are not necessarily equivalent to principal strains.

From a diffraction perspective, what is the difference between reactor and spallation neutrons?

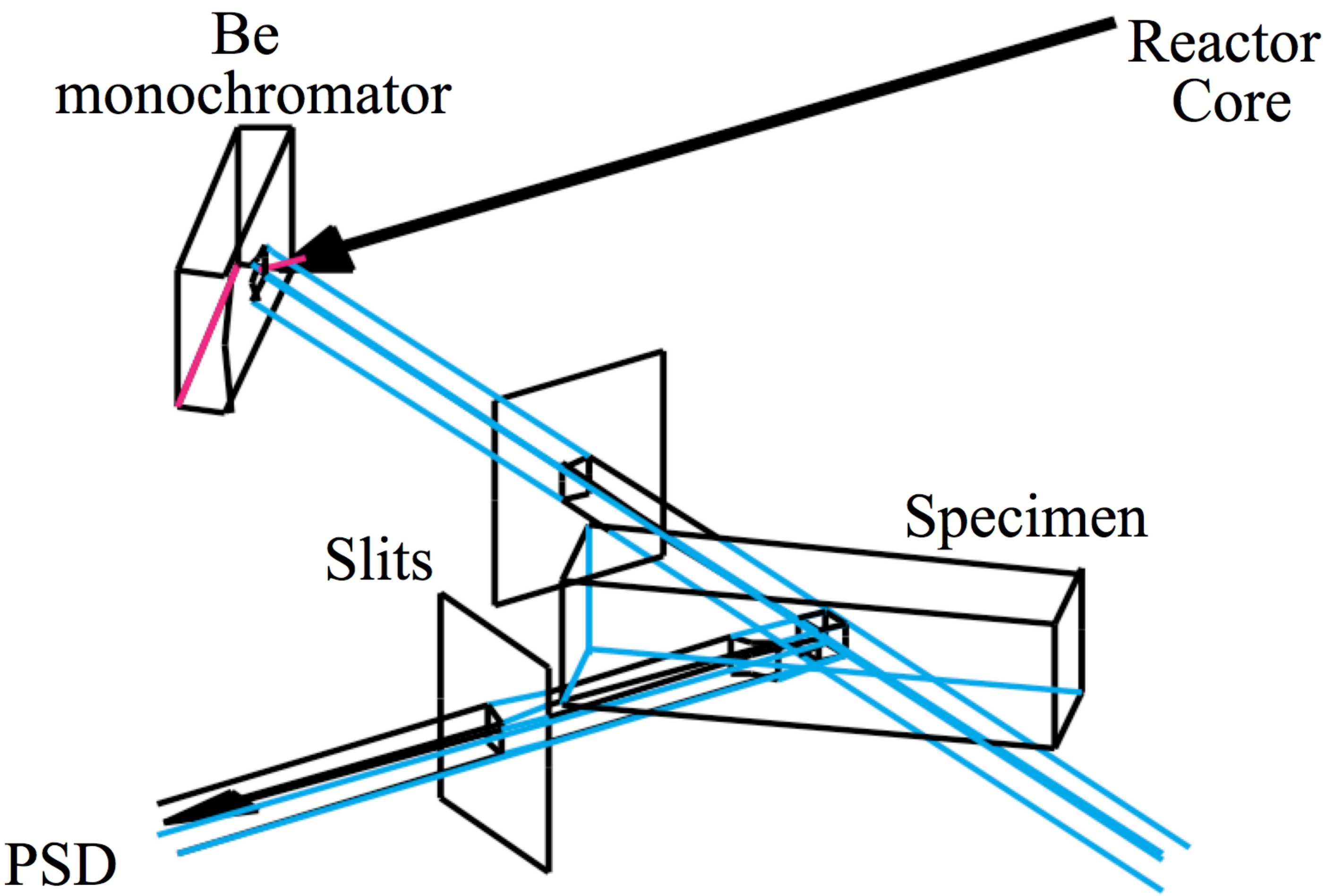
- Reactor sources generate a constant flux of moderated neutrons (usually thermal) that are subsequently made monochromatic (constant wavelength). Diffraction patterns resemble XRD (intensity versus 2-theta) spectra, since:

$n\lambda = 2d\sin\theta$ or $d = \lambda/2\sin\theta$ so for fixed λ, we measure $d = f(\theta)$

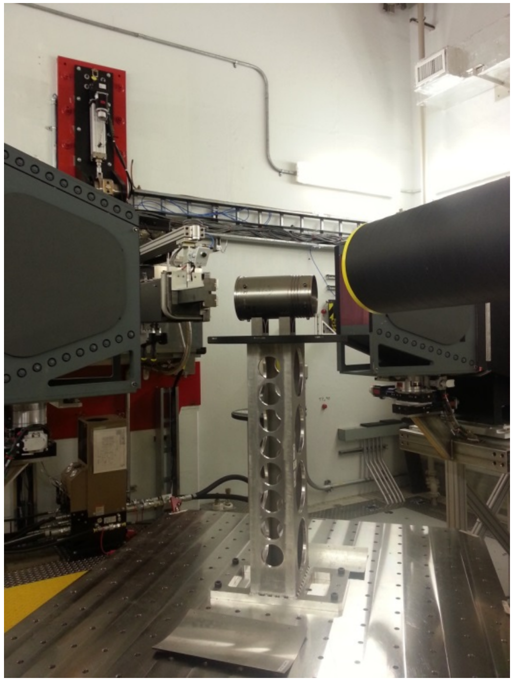
- Spallation sources generate neutrons from impacting a high-energy proton, electron, or positron beam (>500MeV) onto a heavy metal target (W, U, or Hg). The interaction produces many neutrons per event, and the accelerator is operated in a pulsed mode (10 - 120 Hz). The resulting pulses of neutrons enable time-of-flight (TOF) analysis on the neutron scattering experiments. The patterns look a bit different from XRD because it is the velocity of the neutrons that is important, and m,L and θ are constant:

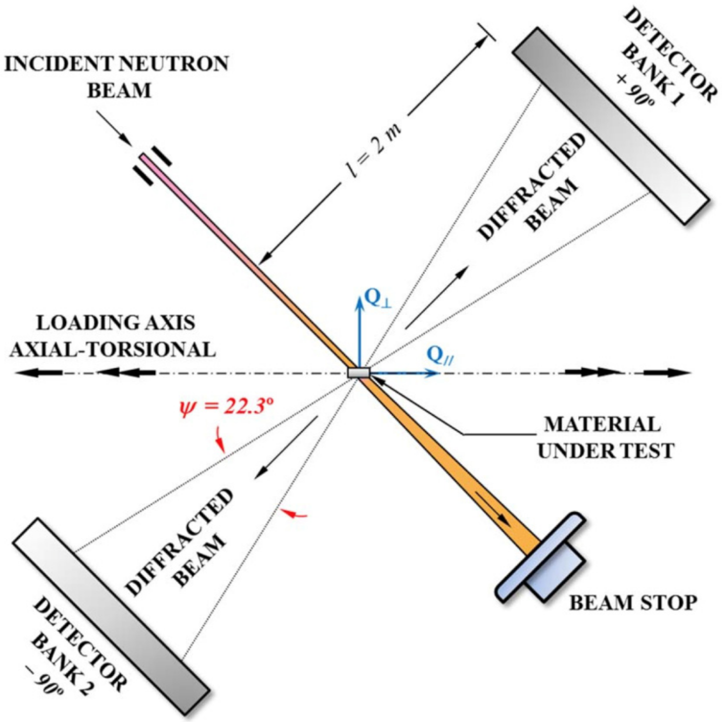
$\lambda = h/mv$ or $d = ht/2mL\sin\theta$ so for fixed θ, we measure $d = f(t)$

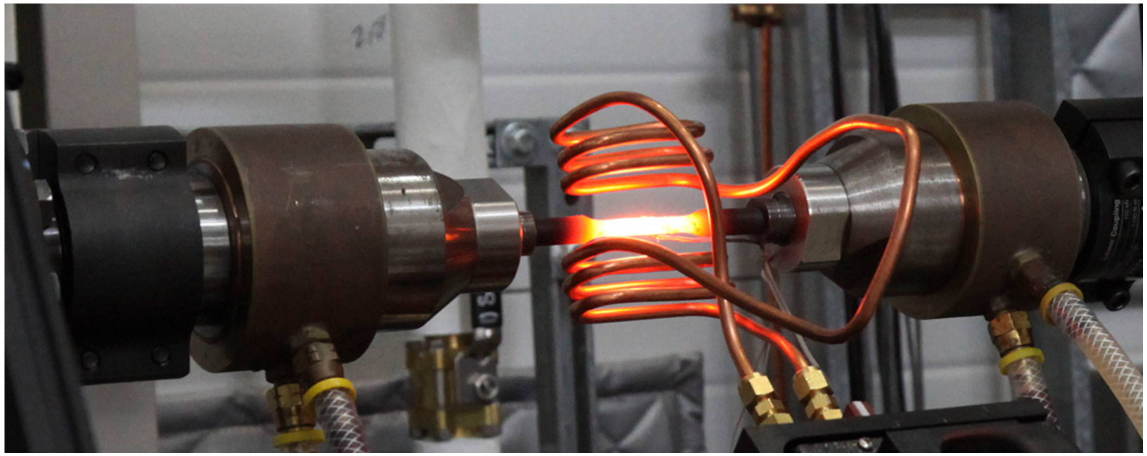
So NRSF2 uses slits to define a small sampling volume inside a solid object



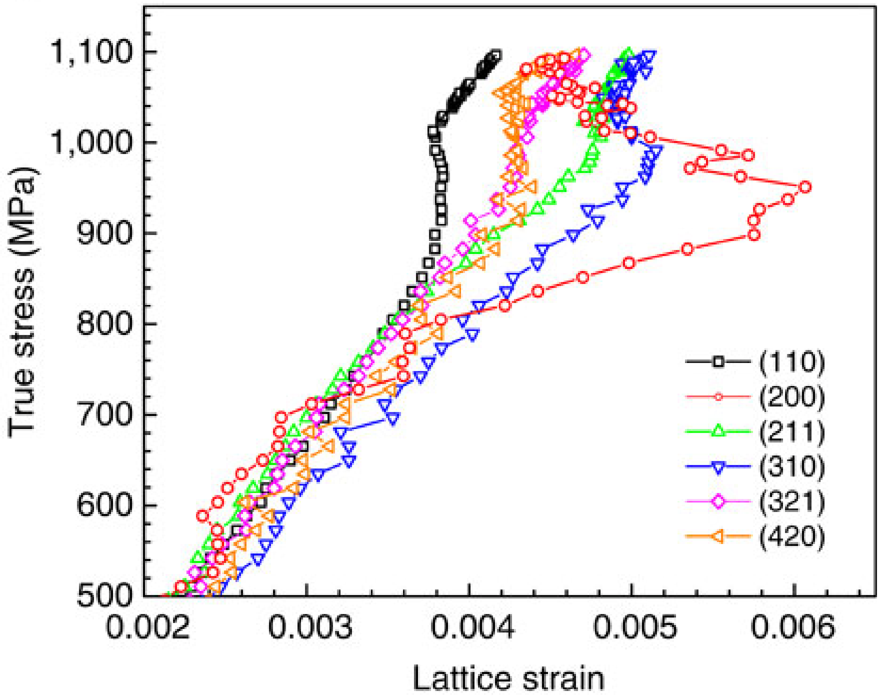
VULCAN uses radial collimators to define the sampling volume at a fixed angle

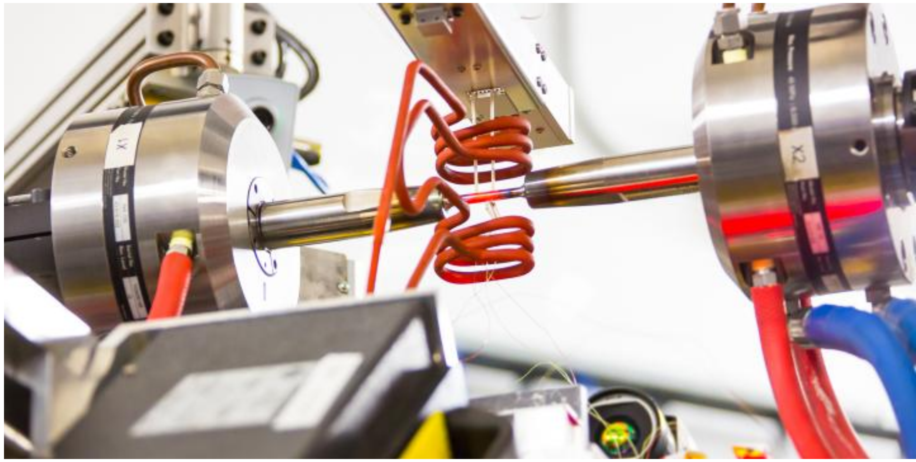






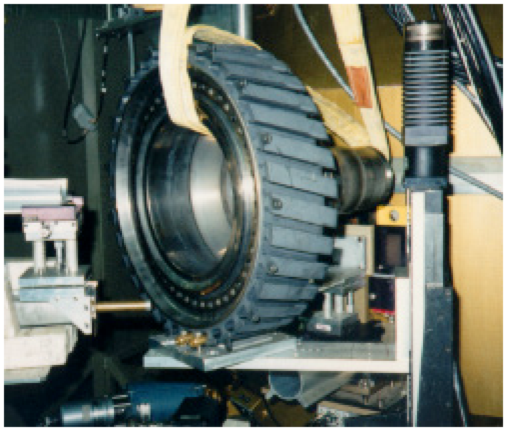
VULCAN is capable of collecting full diffraction patterns at two fixed angles



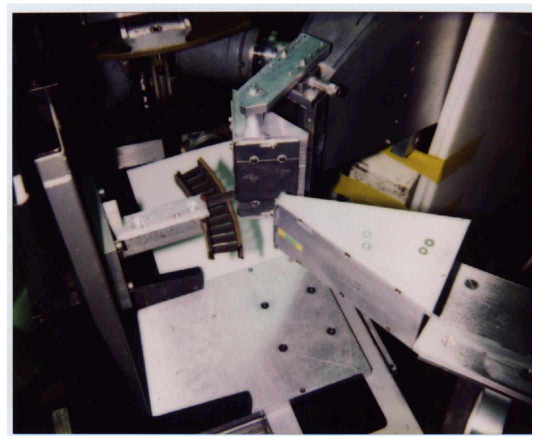


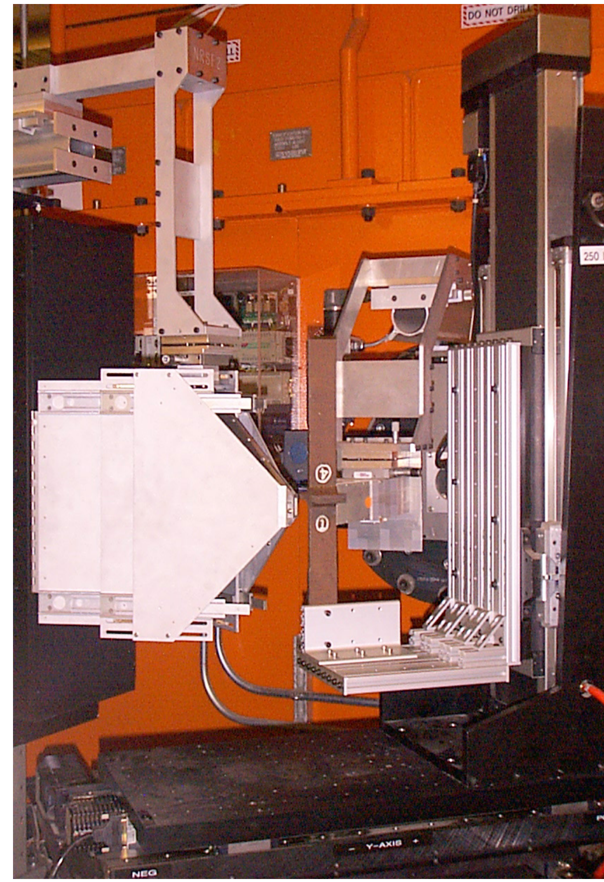
HFIR's residual stress beamline: two decades of engineering research

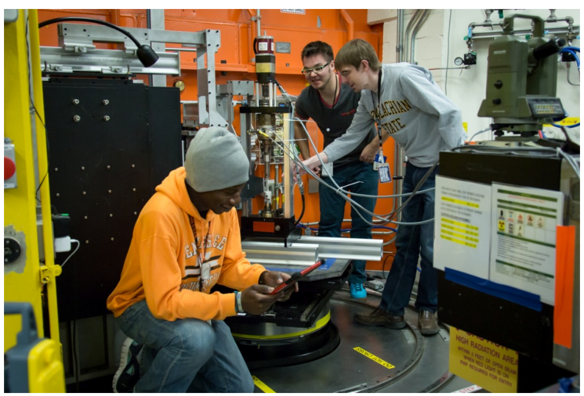
400 lb Ti-6Al-4V jet engine fan blade hub



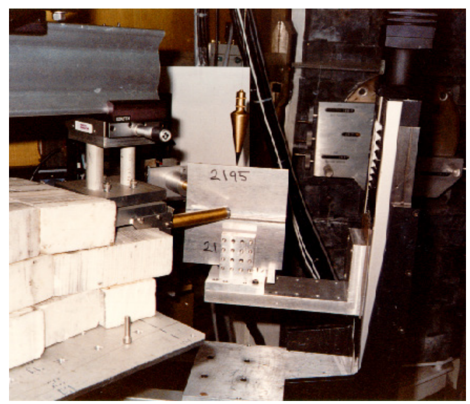
F414A jet engine nickel superalloy stator vane segment







Shape memory alloys under applied multiaxial loading

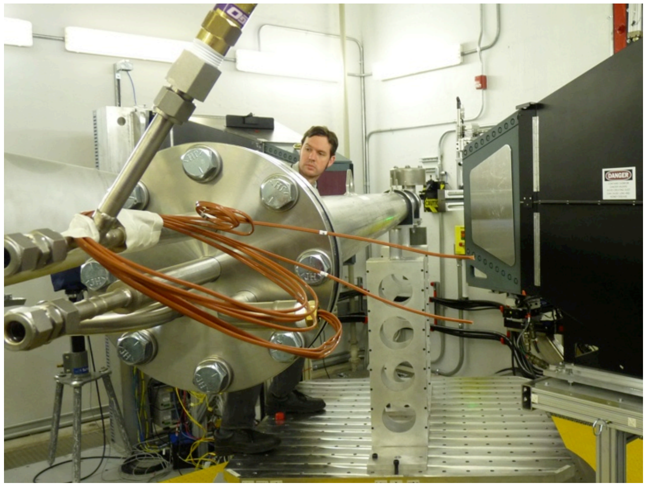


Welded 2195 aerospace aluminum-lithium alloy

Welded steel cruciform samples

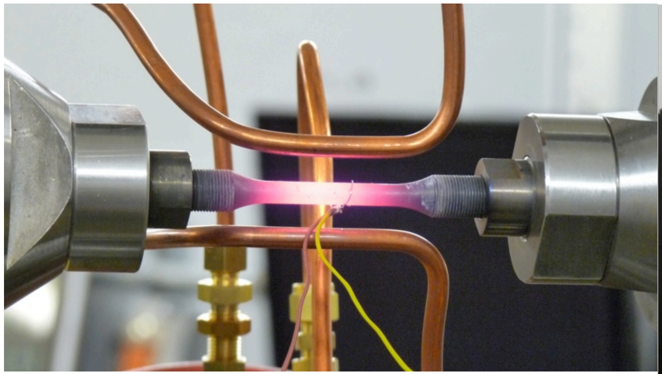
SNS's VULCAN beamline provides new experimental capabilities for MatSciEng

Residual stress / phase mapping



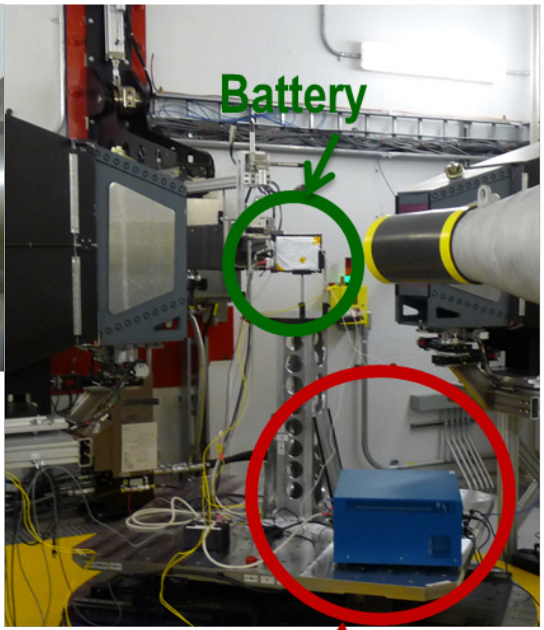
Residual stress determination of large engineering components. Picture shows mapping an ITER cable in a cryogenic chamber.

In-situ diffraction under thermomechanical loading.



The temporal evolution of lattice strain during in-situ thermomechanical testing provides insights into the micromechanism at the local levels.

In-operando measurement.

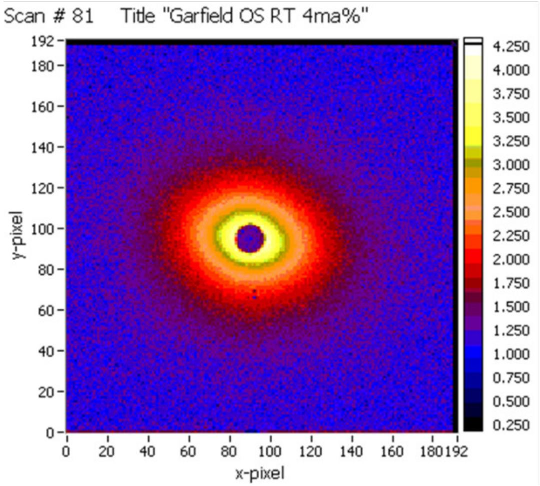


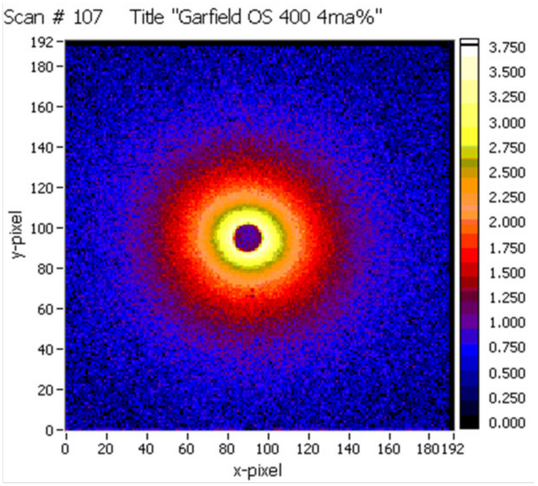
Electrochemical instruments

Neutron diffraction probes the degradation of large format batteries.

HFIR's GP-SANS – applications in materials engineering

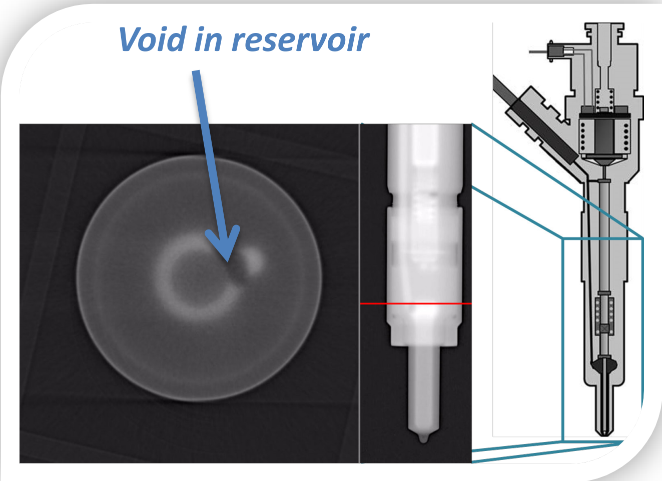
- **Beyond soft matter:**
- phase separation, grain growth, and orientation in metallurgical alloys, nanocomposites, advanced ceramics
- porous catalytic and adsorbent materials.
- Sample environments include magnets, load frames, furnaces





Oil Shale SANS showing effects of sedimentary layering (K. Littrell, CG-2)

HFIR's Neutron Imaging - direct imaging and tomography for materials engineering



(1) Neutron Computed Tomographic Slice, (2) Neutron Radiograph and (3) schematic of a diesel injector.

Scientific Achievement

New details of fluid density and dynamics in commercial fuel injectors are being revealed using neutron radiography.

Significance and Impact

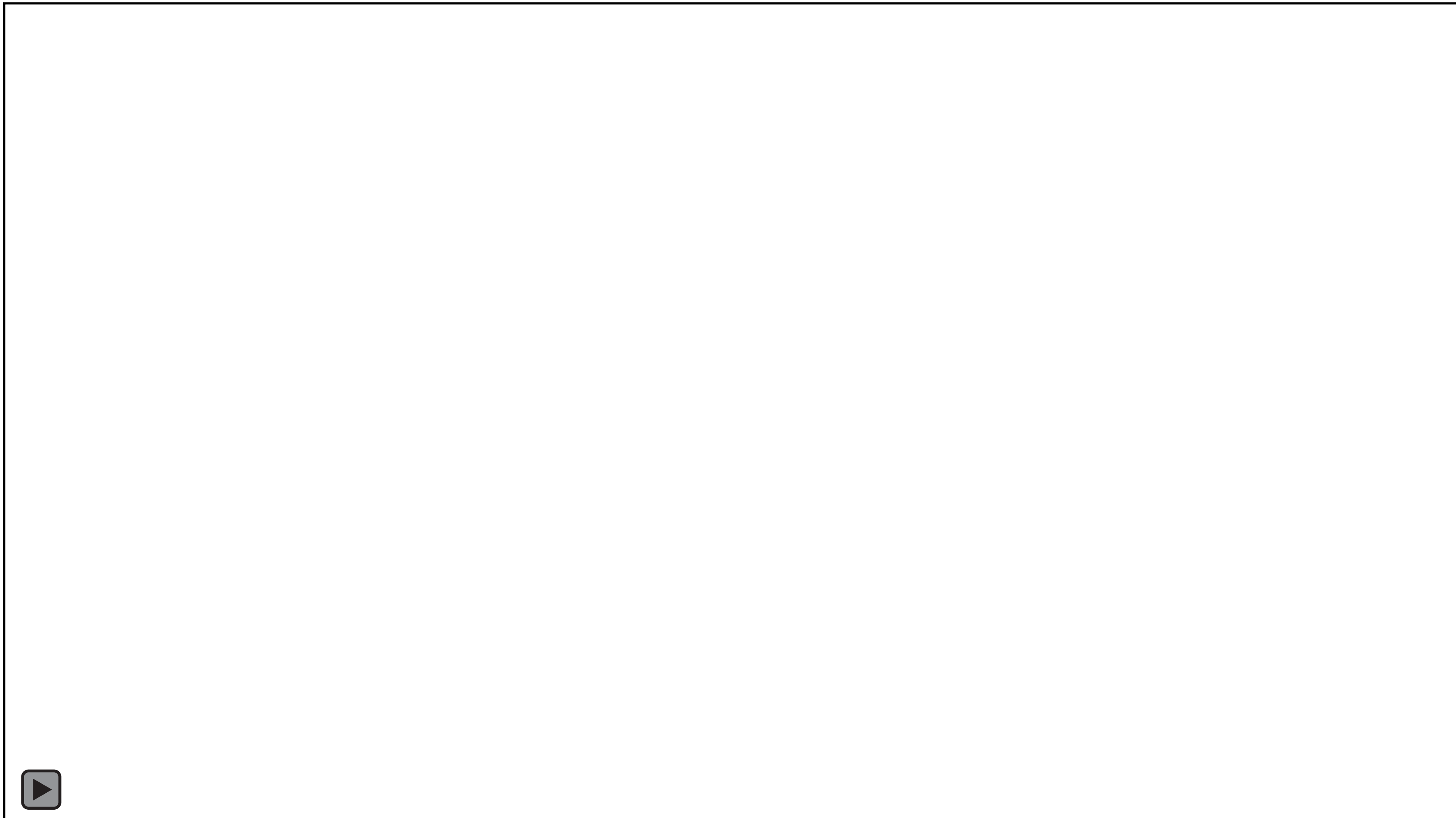
Development of high efficiency injectors is critical to improve combustion efficiency and reduce emissions

Research Details

- Efforts are focused on diesel and gasoline injectors
- Void areas in fuel reservoir are clearly visible
    - Fuel interacts with neutrons more than metal exterior
- Future efforts are focusing on internal fluid flow and understanding hydrodynamic cavitation behavior
- GM currently collaborating with ORNL on injector fluid dynamic modeling with HPC
    - Efforts aimed at employing this technique to guide and validate model
    - Bosch fuel injectors being employed in study

Work was performed at HFIR CG-1D Imaging Beamline

Finally: a short movie break!



Neutron research on engineering materials can be really ~~fun~~ interesting!

Not all samples are a few grams of powder!

Sometime full-scale problems are better studied at full scale than laboratory scale

Not all studies on "engineering diffractometers" are residual stress or in-situ loading studies – the photo shows a spatially-mapped quantitative phase analysis study on real components from a working hydrogen fuel cell design

VULCAN diffractometer studies on full-scale (3 kg) hydrogen storage media in steel tubes for General Motors