Neutron Scattering for Studies on Engineering Materials at NRSF2
-- Status and Opportunities --

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Residual Stress User Center, HTML

Materials Science and Technology Division

Oak Ridge National Laboratory

Neutrons for Stress, Texture and Phase Transformation for Industry

SNS, Oak Ridge, Tennessee

19 April 2007

Residual Stress User Center's Unique X-ray, Synchrotron & Neutron Facilities for Stress Mapping

RSUC is one of six user centers in the HTML User Program sponsored by DOE-EERE-OFCVT (Office of FreedomCAR and Vehicle Technologies)

- **Large or small specimens**
- **Flat or curved**
- **Thin films or bulk**
- **Ambient or elevated temperature or load**

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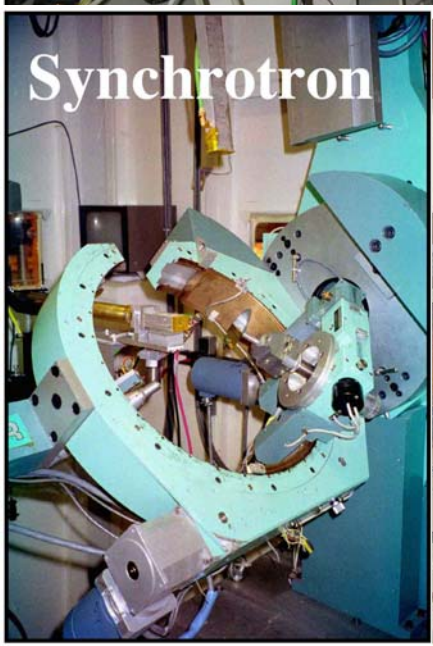
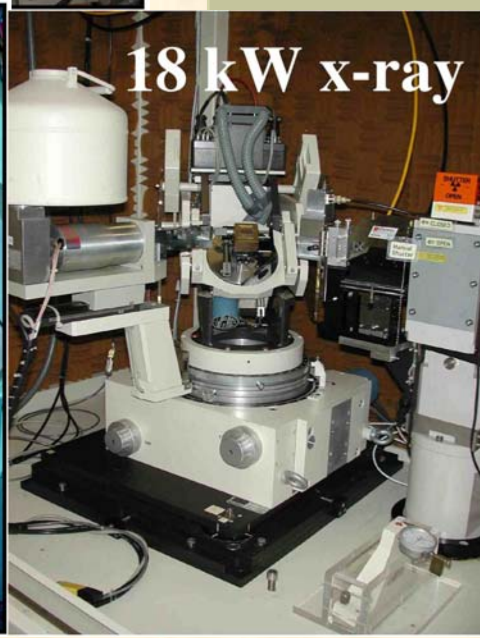
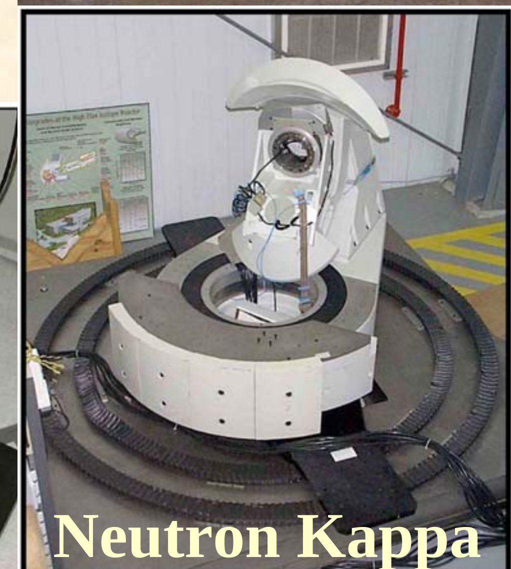


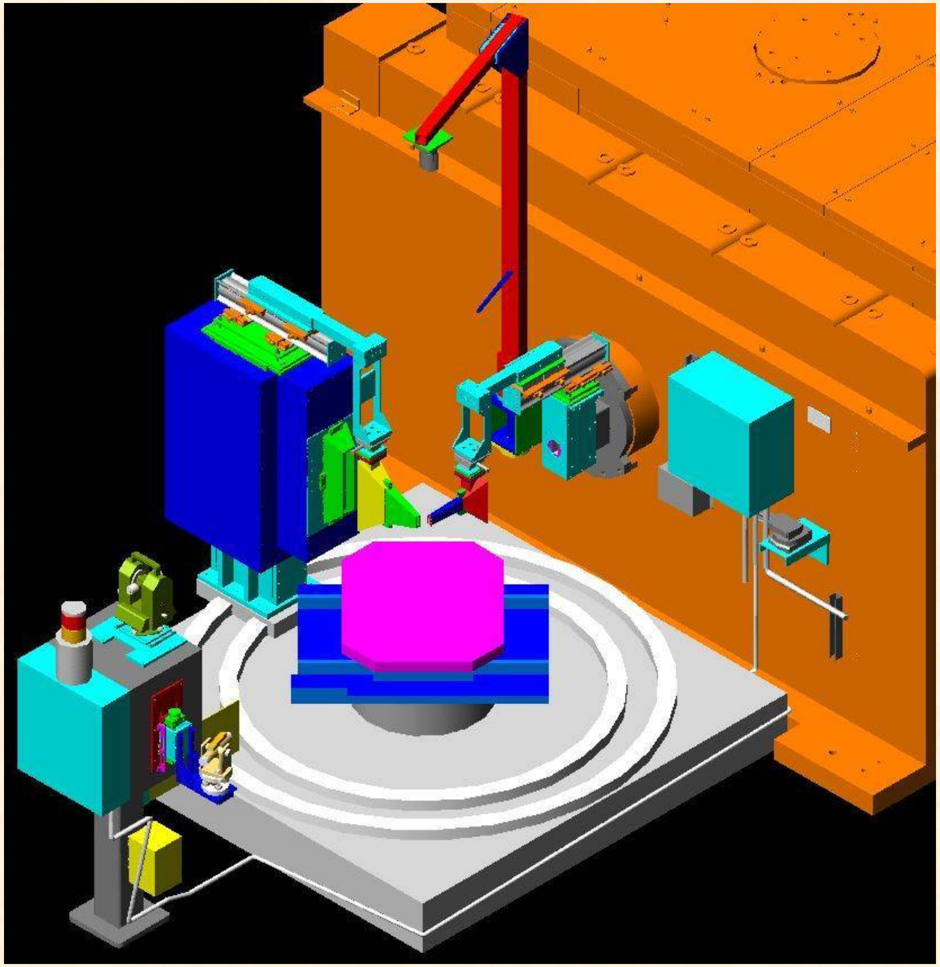




Outline

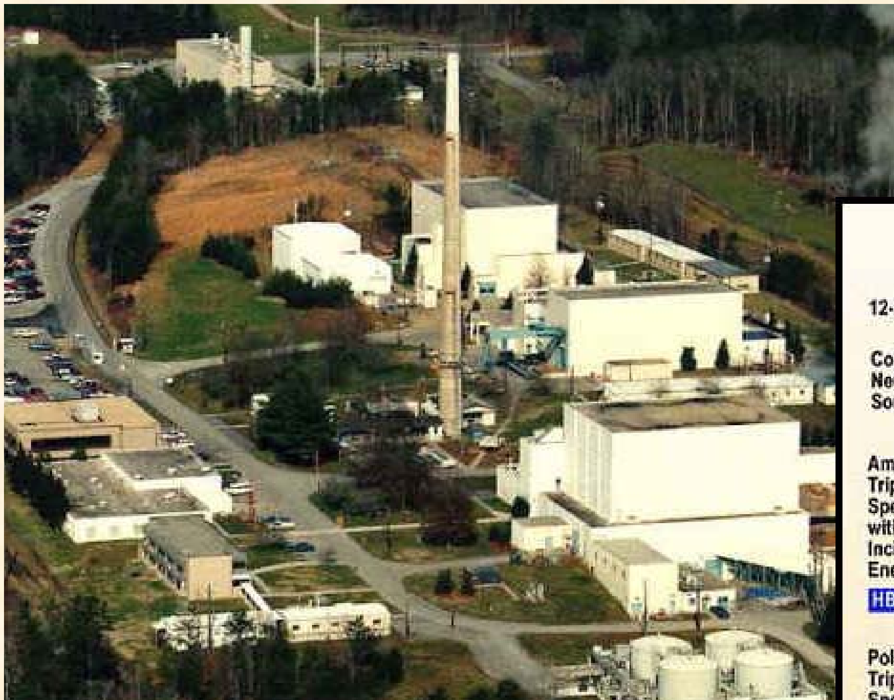
- **HFIR upgrades and instruments**
- **Design and performance of the engineering diffractometer NRSF2 at HFIR**
- **What's new at NRSF2#?**
- **Schedule and call for proposals**
- **Highlights of selected studies**

#Throughout the presentation the items in purple are new since January 2006

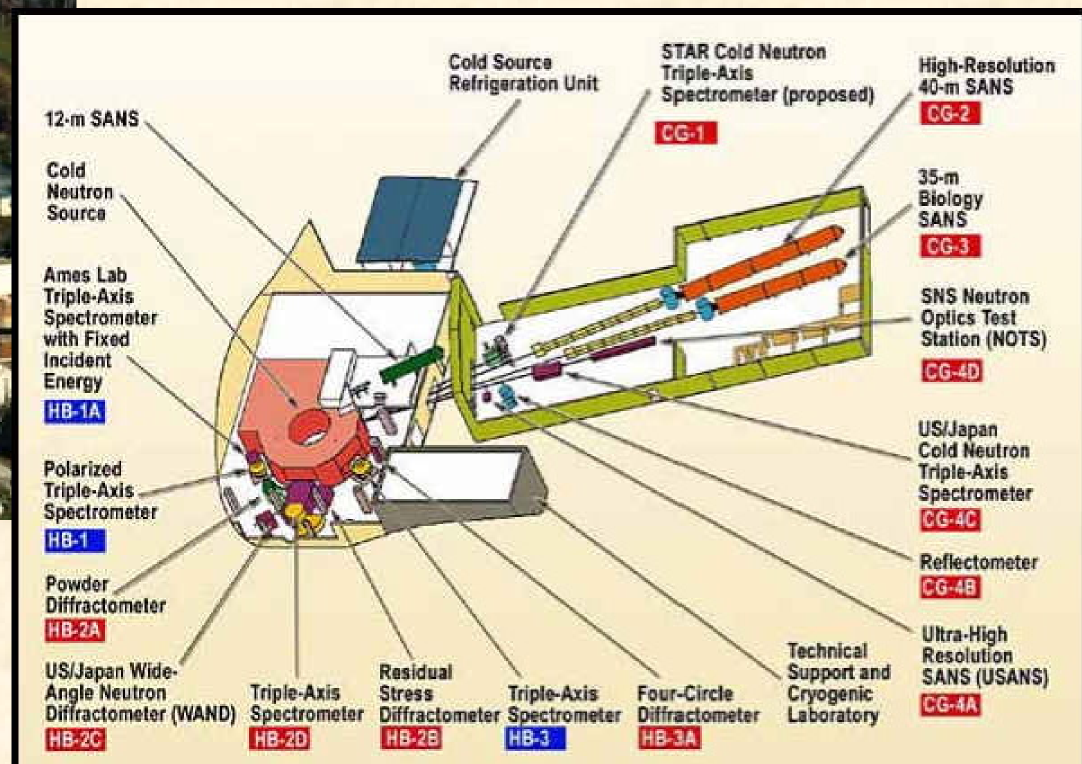


Neutron Residual Stress Mapping Facility at HFIR

The High Flux Isotope Reactor at ORNL is One of The Two Highest Flux Reactor Facilities Worldwide



Commissioning of **new scattering instruments** began in 2004. Goals is 15 new, world class instruments.



HFIR Upgrades:

- Be reflector replacement
- new beam tubes
- **addition of cold source**

Installed Instruments at HFIR as of May 2007

- **Thermal Neutrons**
  - **Triple axes instruments (HB1, HB1A, HB3)**
  - **Neutron Residual Stress Mapping Facility (NRSF2)**
  - **Wide Angle Neutron Diffractometer (WAND)**
  - **Four Circle (Single Crystal) Diffractometer**
- **Cold Neutrons**
  - **Small Angle Neutron Scattering (40 and 35-m SANS)**

Operating mode

Commissioning mode

Learn more at **http://neutrons.ornl.gov/**

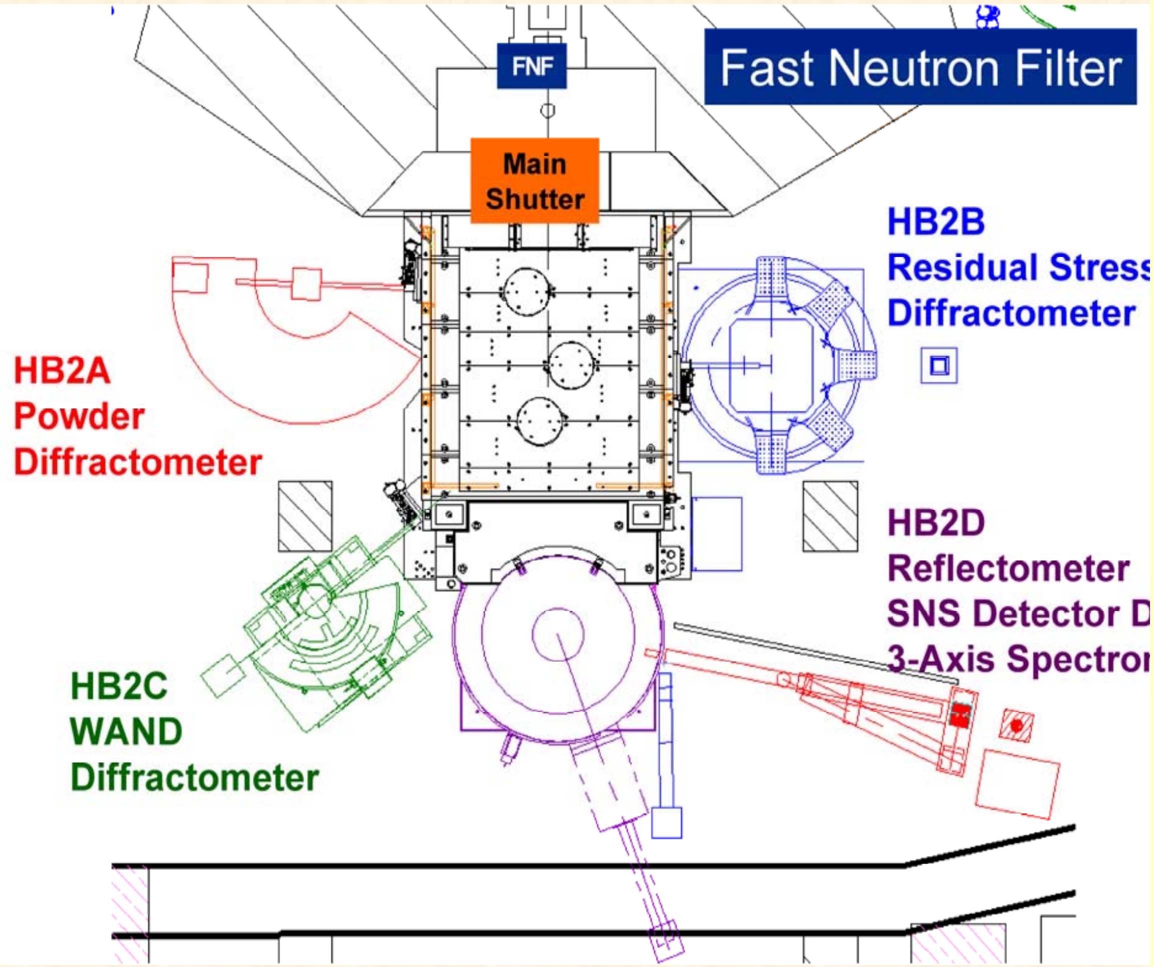
NRSF2 Design Goals Based on Feedback from Nine Years of RSUC User Interactions

- **Capacity to measure wider range of specimen**
  - Small research specimens (e.g. crack tips, rivets, interfaces)
  - Large engineering specimens (e.g. engines, castings, blades, tubes and panels)
  - Highly textured, those with “large” individual grains, single crystals

- **Capable to study many materials (Fe, Al, Zr, Be, ZrO_2, ...)**
  - Greater range of d-spacings and hkl
  - In-situ measurement of intergranular strains (type II)
  - Resolution (~0.3° 2θ FWHM)
  - Working 2θ range of 70° to 110°

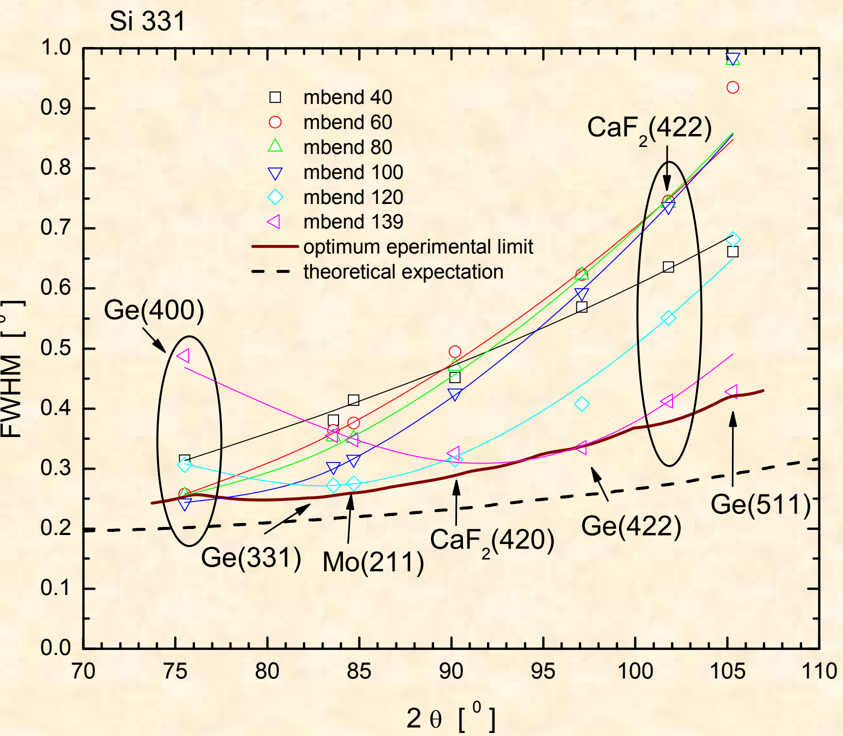
- **Faster measurement with higher accuracy**
  - Higher flux (> 10^7 n/cm^2/sec)
  - Cubic and match stick sampling volumes from ~0.3 to 100 mm^3
  - Precision of measurement to ~0.002° 2θ
  - Real-time measurements
  - In situ measurements - e.g. load, fatigue, temperature, fields
  - Determine strain tensor

NRSF2 Was Designed to Take Full Advantage of the large, radial beam at HB-2



- ✓**100% of time for materials science & engineering**
- ✓**Doubly focusing Si wafer dual monochromator**
  - **Higher flux**
  - **Five wavelengths**
- ✓**Interchangeable XYZ and KAPPA goniometers**
- ✓**Seven PSD detector array**
- ✓**Sample environments**
  - **Load frame**
  - **Furnaces**
  - **5T magnet**
- ✓**Advanced software**
  - **SScanSS**
  - **MAP, VIEW, CALIB**
  - **E-Notebooks**

The Popovici-Stoica Stacked Si Wafers, Two Monochromator Crystal Sets, Each Doubly Focusing, Provide Five Choices of λ from 1.45 to 2.67 Å with Major Flux and FWHM Enhancements



FWHM optimized by changing the bending radius

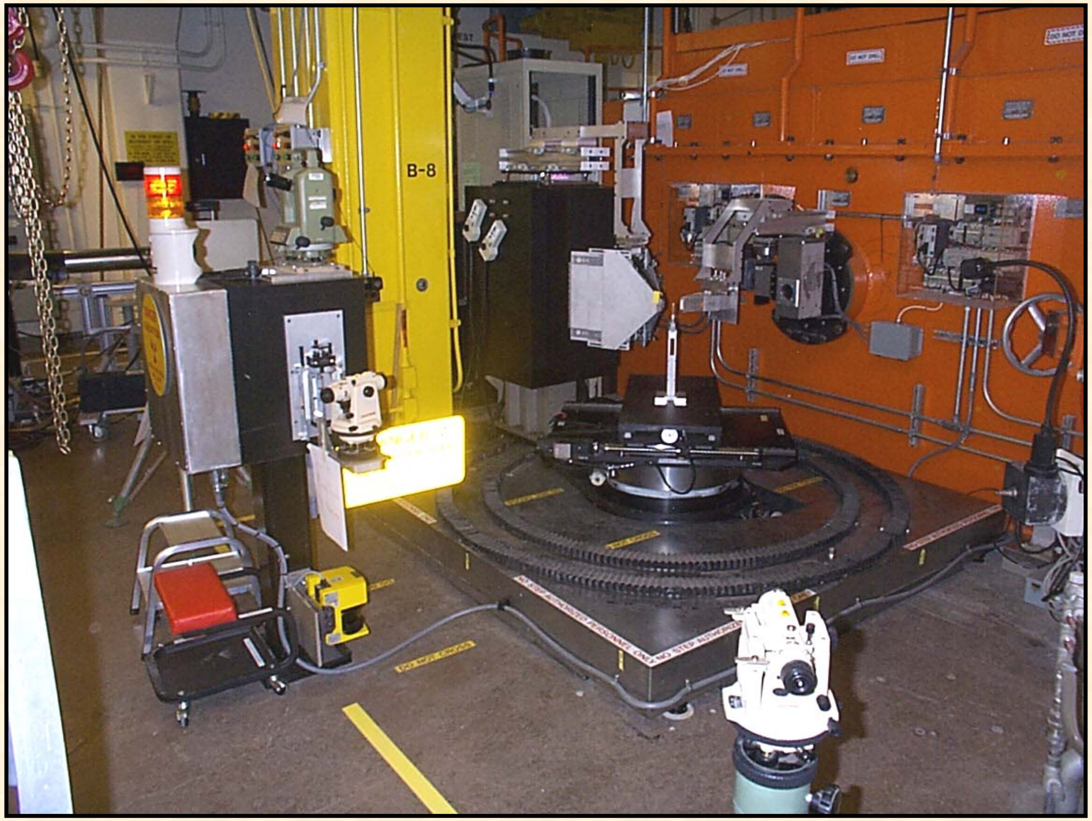
Profile Intensity and FWHM compared to first generation NRSF (year 2000)
(both used single PSD detector, same specimens)

Phase hkl	Peak Intensity Ratio	Integrated Intensity Ratio	FWHM Ratio
Fe 211	11	12	0.54
Ni 220	16	15	0.69
Al 311	6.3	6	0.63

With 7-detector array the gains will be 40-100x

X Y Z Ω & 2Θ Stages Provide High Accuracy with Flexibility for Samples and Accessories

- **Mechanically rigid, highly reproducible positioning**
  - **Rotation stages**
    - **2θ accuracy of 0.001°**
    - **Ω accuracy of 0.003°**
  - **Translation stages**
    - **X-stage - 400 mm**
    - **Y-stage - 200 mm**
    - **accuracy - <0.01 mm**
  - **500 kg load directly on XY**
  - **Larger Y table for more flexible mountings**
  - **Upgraded motor controller**

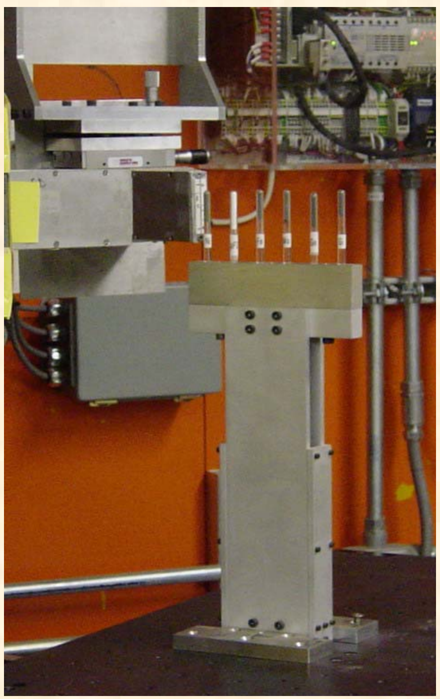


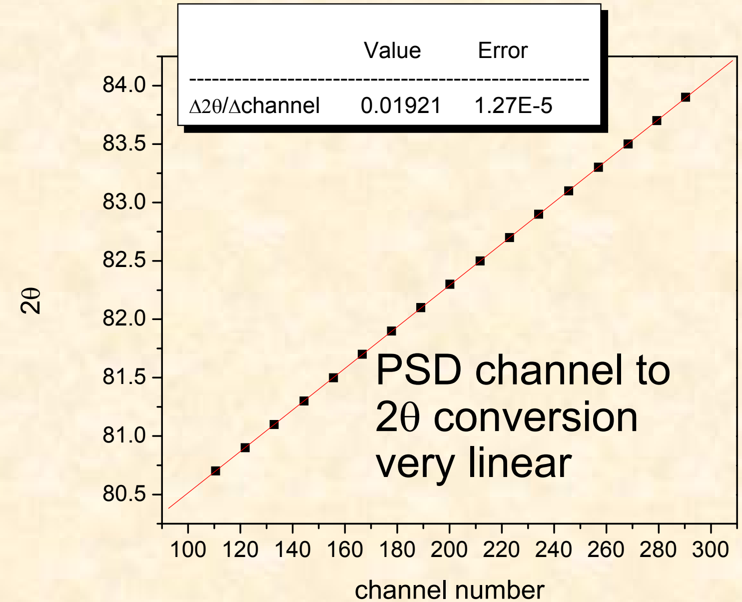
NRSF2's XYZ Goniometer at HB-2B

NRSF2 System Calibration is Precise and Accurate

Calibration powders:

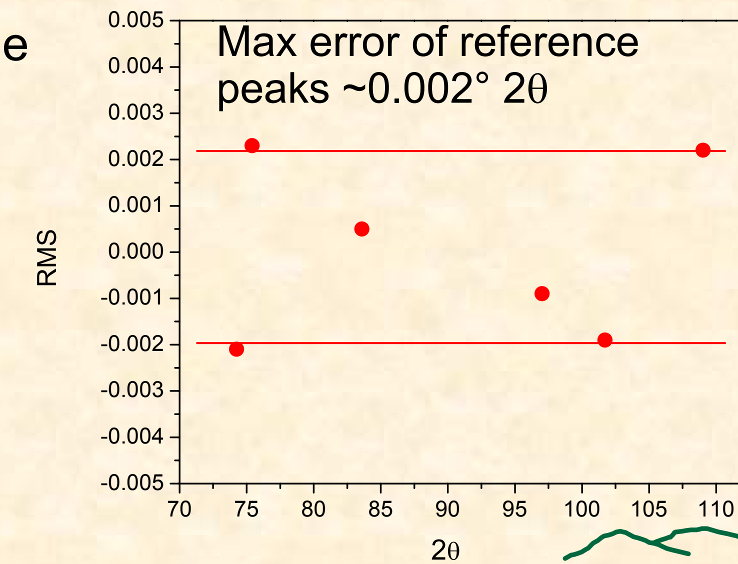
- Ni, Fe, Mo, Ge, CaF_2 reference powders
- Accurate lattice parameters for each from XRD with SRM Si standard
- Reproducible mount of calibration samples





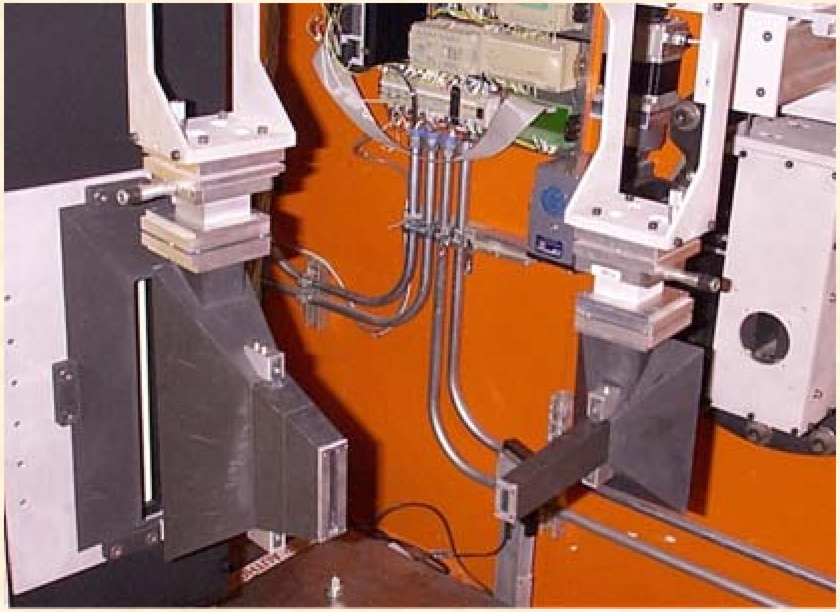
Calibration Steps

- PSD intensity response
- Conversion of MCA channel number to angle
- Out of plane detector angles
- **Finite detector height corrections**
- Fit $2\theta_0$ and λ
- ***Ready for 7 detectors***



New Incident and Diffracted Beam Snouts and Slit Systems Added

- **Doubly tapered B_4C/epoxy snouts fabricated**
  - **Reduces collision possibility**
  - **All 7 detectors will fully see gauge volume at any set up**
  - **Receiving side has 4 different snout lengths and slit sets**
  - **Incident side is continuously adjustable**
- **New slits and slit holders**
  - **Gd plates cut by EDM**
    - **High precision**
    - **Should enable changing without need to recalibrate**
  - **Widths: 0.3, 0.5, 0.7, 1, 2, 3, 4, 5 mm**
  - **Heights: 0.3, 0.7, 1, 3, 5, 10, 15, 20 mm**
  - **Offsets: 25, 50, 100, 150 mm**

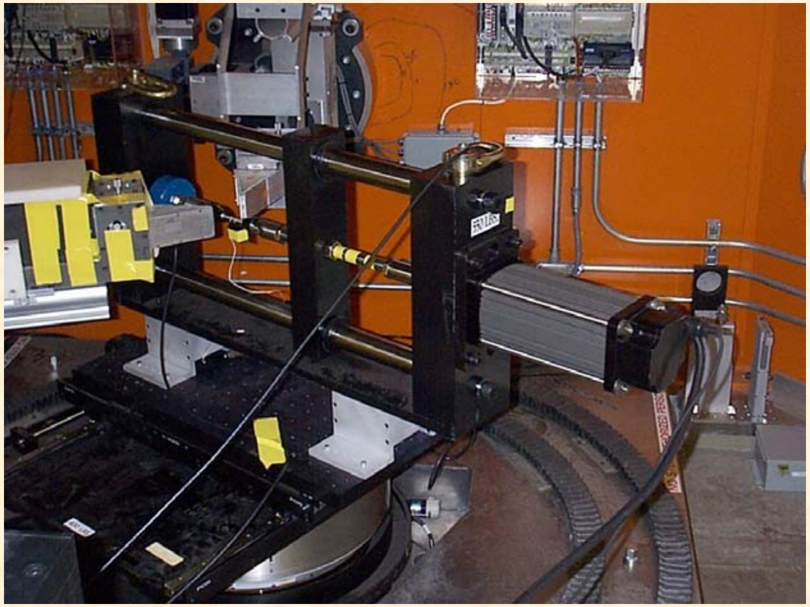


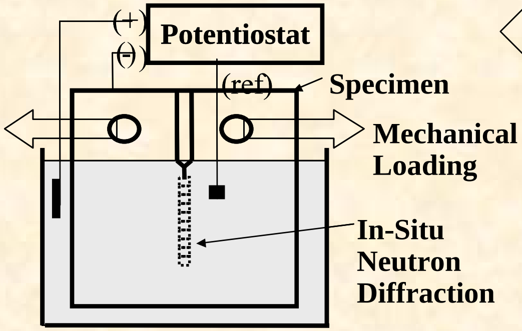
Load Frame Accessory for In-situ Deformation and Materials Behavior Studies

- **Automated load frame developed to study specimens under uniaxial loads for in-situ deformation studies**

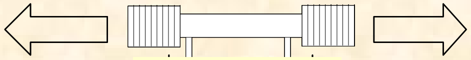
- **Key features include:**
  - **5000 lbf. loading capacity**
  - **Tension/compression (static)**
  - **Automated load & strain control and macro-strain recording**

- ***Mounts on new Z-elevator to take advantage of NRSF2's excellent mapping strengths***
  - ✓ ***e.g. map strain fields around crack tips in compact tension specimens under load***



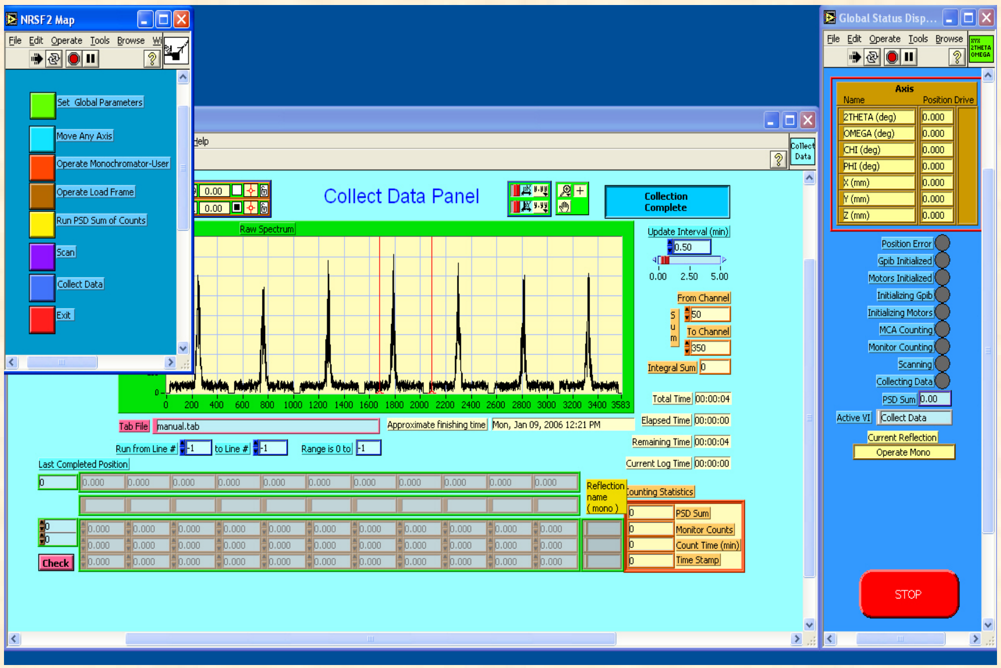


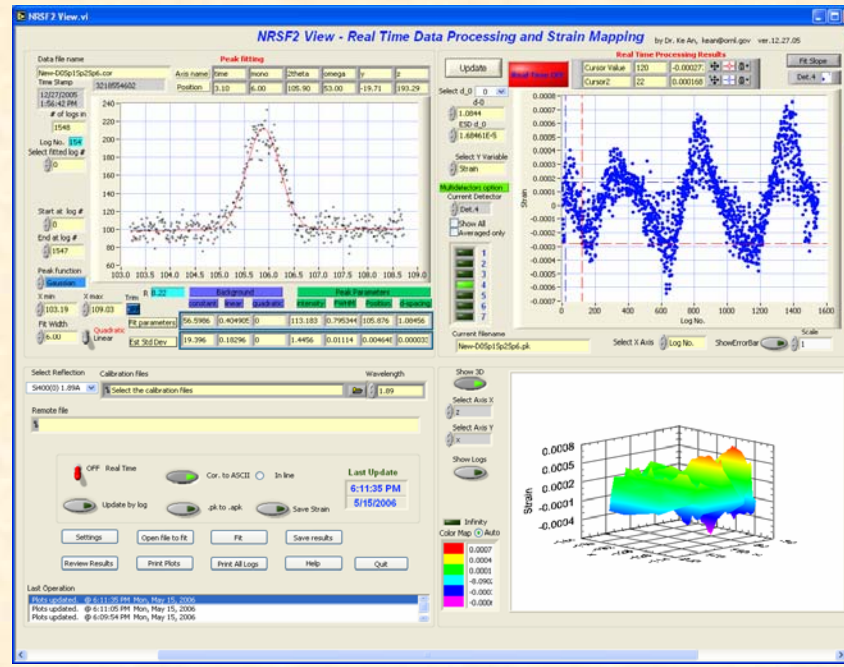
Environmental cell for electrochemical charging



Tensile testing sample geometry

LabView System Software was Designed for Flexibility, Reliability, and Real-Time Display





NRSF2-MAP controls the instrument and accessories such as load frame

NRSF2-VIEW provides real-time results and post processing

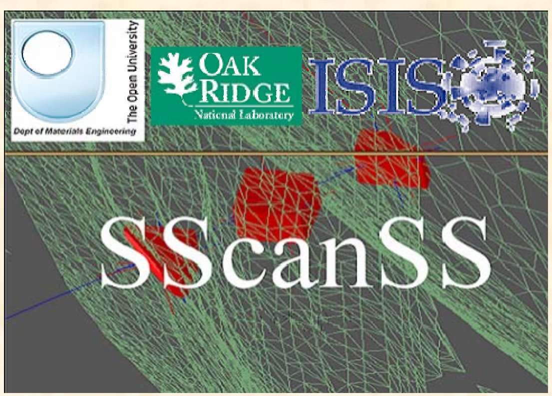
Several Sample Environments Are Available for Use at NRSF2

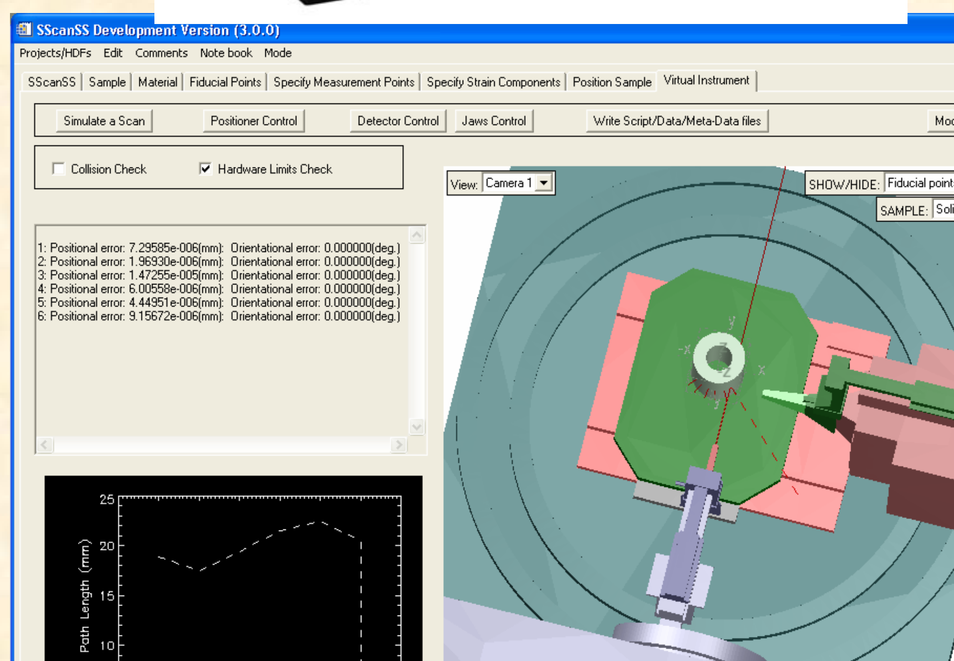
- **5000 lbs uniaxial load frame**
  - ✓ **grips for standard samples**
- **Z-elevator (500 kg, 100 mm travel)**
- **Z-stage (50 kg, 400 mm travel)**
- **Huber 2-circle orienter**
- **Sample rotation stage**
- **5T magnet with induction heater insert (1000°C)**
- **ILL vacuum furnace (1600°C)**
- **Controlled atmosphere furnace (1250°C, 1 atm)**

Tools for Experiment Simulation, Fast Sample Alignment, and Accurate Spatial Measurement

- **Laser tracker & laser scanner (proposed) for defining alignment and reverse engineering**

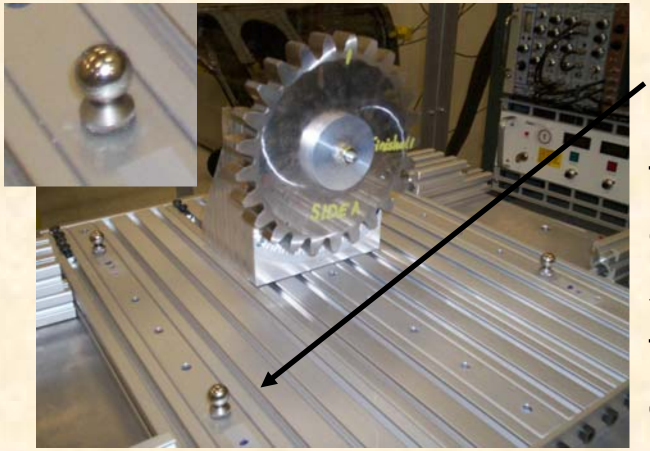
- **SScanSS (Open Univ.) to enable planning of mapping locations, path lengths and time. SScanSS also perform checks for interferences and collisions**





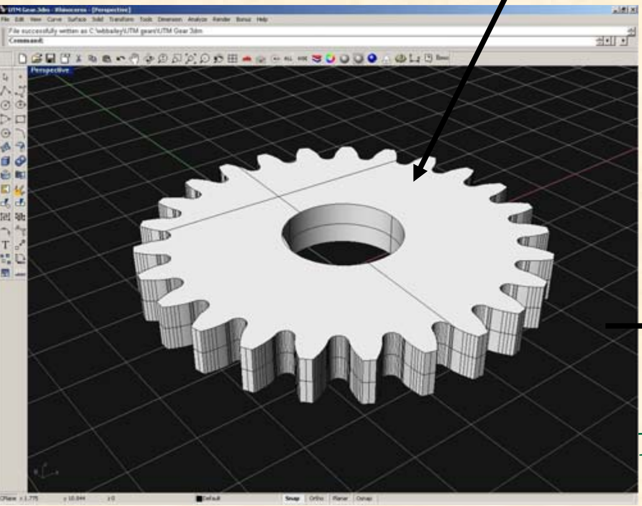
Laser Tracker and SScanSS Software Support Studies of Engineering Components

Sample and engineering drawings from user

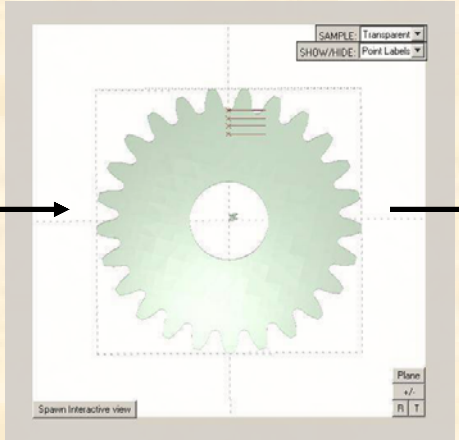


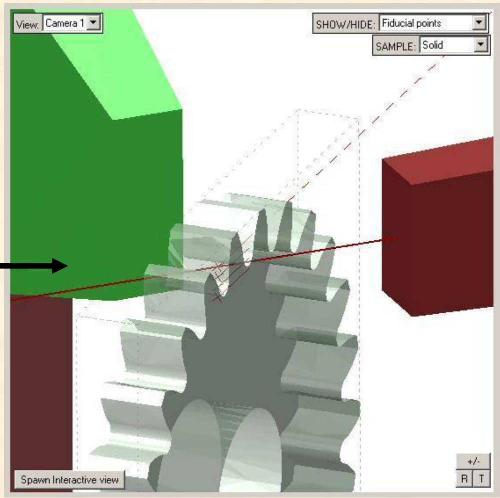
Fiduciary locations related to sample coordinate system via laser tracker enable quick set up on the instrument

SScanSS helps user determine mapping locations in sample coordinate system and orients it on the instrument



TORY





Comparison of NRSF2 with VULCAN

Instrument	Δd / d	Flux	Notes
	(%)	$n / cm^2 / s$	
NRSF2 **(λ=1.54A)**	0.24% measured	3x10**7 measured	fixed lambda,
		SNS at 1.3 MW	
VULCAN **(high resolution)**	0.20%	3x10**7 predicted	lambda band width
VULCAN **(high intensity)**	0.60%	1x10**8 predicted	lambda band width

NRSF2 ready for of a wide range of studies

- **Neutron strain mapping facility accepting user proposals for neutron time in summer 2007**
  - **Commissioning in HFIR cycles 408 & 409**
    - **Test and document**
    - **Demonstrate instrument capabilities**
      - **Local users, key OFCVT users, good science**
  - **Time in cycles 409, 410, 411, 412 --- Open for Proposals!**
- ***What challenging studies do you have?***
  - **High resolution mapping -- strains and phases**
  - **In situ experiments - load, temperature, magnetic field, …**
  - **Real-time and Quasi Steady State experiments**
  - **Studies of materials deformation behavior**
  - **Hydrogen content mapping**
  - ***Open to unusual requests from industry and academia***

User Access to HTML's NRSF2

As well as complimentary X-ray RS and other HTML user program characterization facilities

- Proposals reviewed monthly: http://html.ornl.gov
- Priority to projects on transportation materials
- Submit User Proposals now for June-September beam time

Contact: Camden Hubbard
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Questions ?

Early Projects during NRSF2's Installation and Commissioning

Four themes for studies during the fall 2005 and winter 2006 beam cycles 405, 406 and 407

- **Residual stress mapping**
- **Materials deformation behavior**
- **Real time and in situ experiments**
- **Phase and hydrogen content mapping**

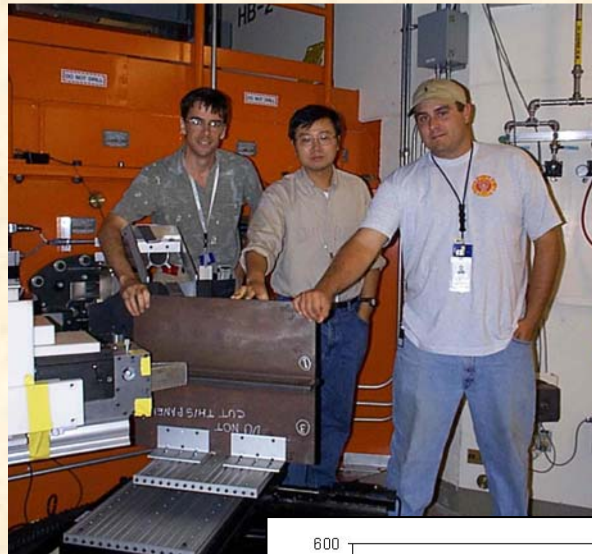
Note: During commissioning NRSF2 used just 1 to 3 detectors and had other limitations as to performance

Expect 2 to 5 fold count rate gains and many other important improvements starting HFIR Cycle 408

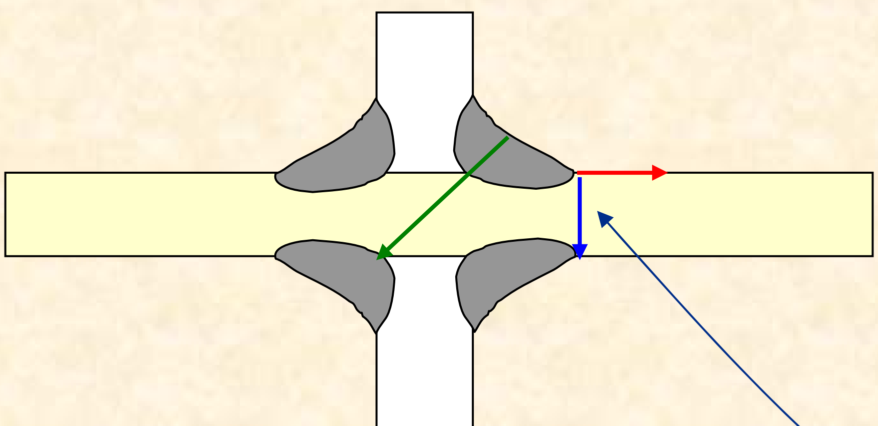
NRSF2's First Strain Mapping Project

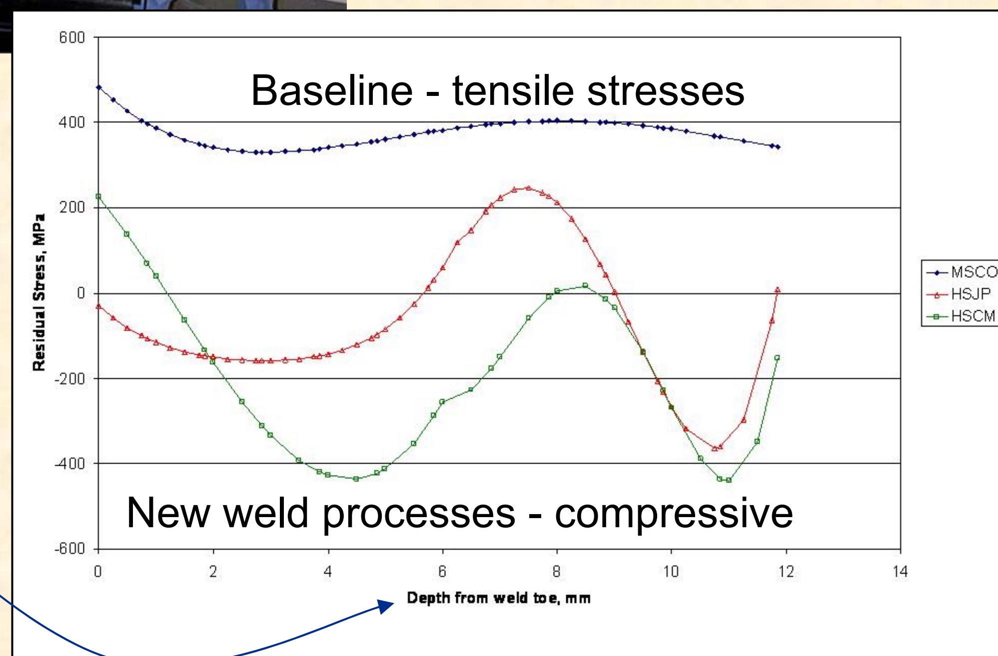
Characterize New Weld Processes and Filler Metals

- *Caterpillar developing new filler metals and weld processes*
- *Validation of finite element modeling was goal*
- *Cruciform steel plate samples*
- *Combined ND and XRD stress mapping*



- ➢ Results helped improve & validate stress models
- ➢ Impact is enhanced fatigue life!

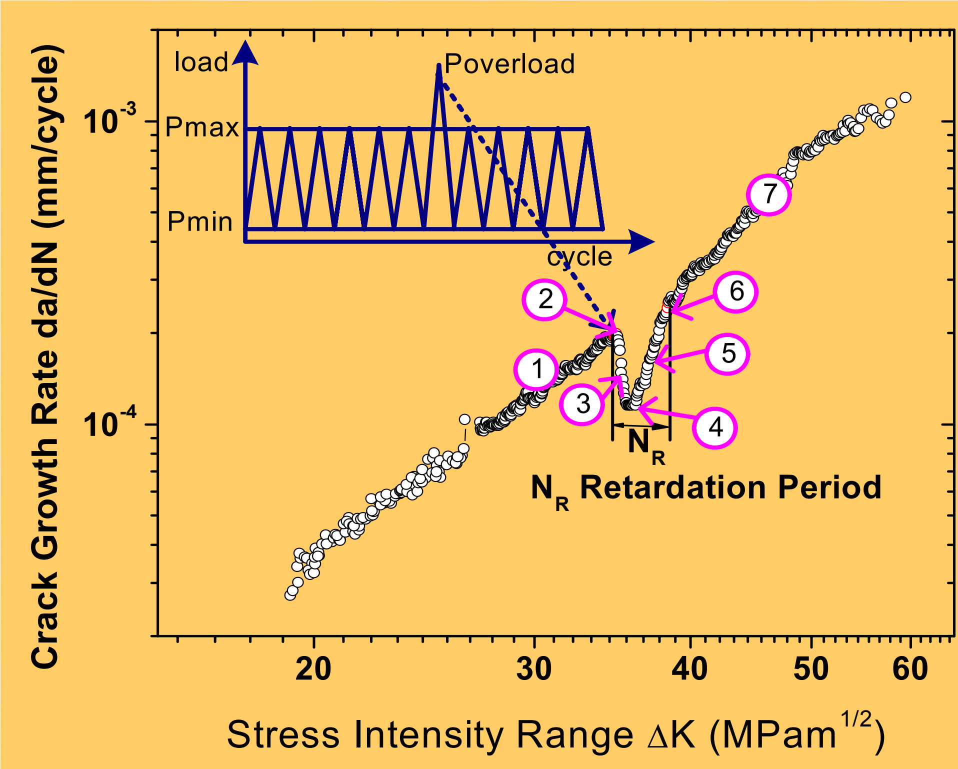




Changes in Strain Profiles around a Fatigue Crack Through the Retardation Period

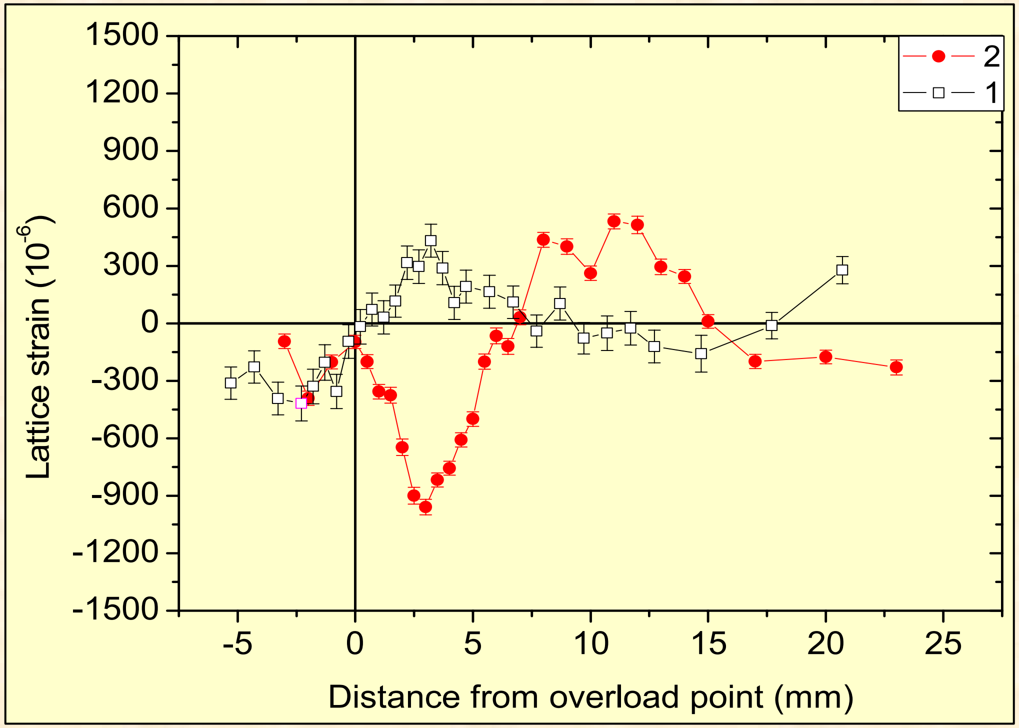
Y. Sun[a], C. R. Hubbard[b], K. An[b], F. Tang[b], Y. L. Lu[a], H. Choo[a,b], and P. K. Liaw[a]
[a]*Materials Science and Engineering Department, The University of Tennessee*
[b] *Metals and Ceramics Division, Oak Ridge National Laboratory*

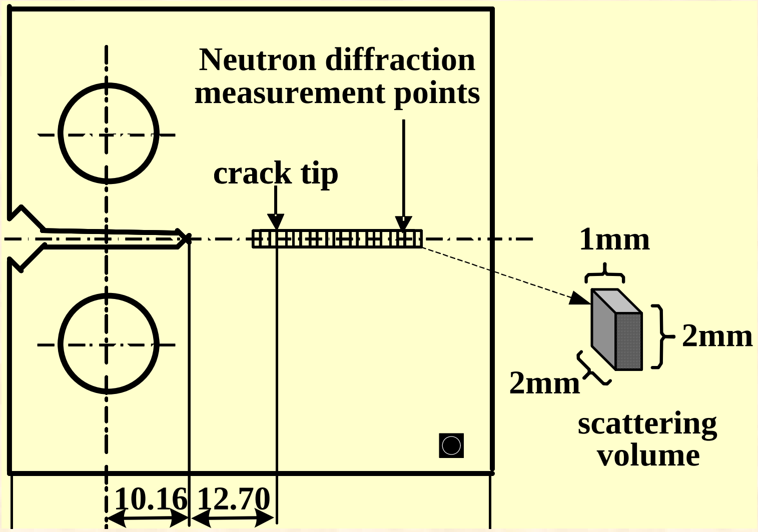
- After an overload during cyclic fatigue there is a period of crack-growth rate retardation that is related to the magnitude and number of overloads.
- What causes the crack-growth-rate to decrease and then increase?
- Approach
  - Study the changes in the residual strain/stress maps due to cyclic fatigue and overload around the crack
  - Type 316 stainless steel (SS), widely used in the nuclear industry
- Correlate strain maps with crack-tip plasticity transient effects, which affect fatigue-crack-growth rates & fatigue life.



Crack Opening (Transverse) Strains Mapped

1D mapping along line of crack

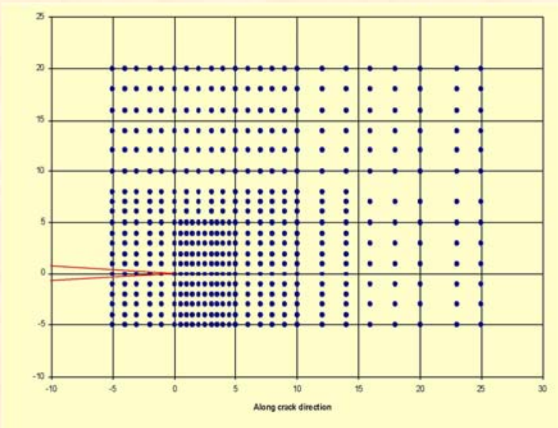


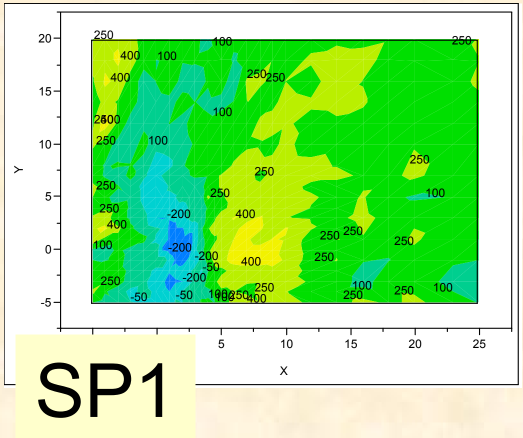


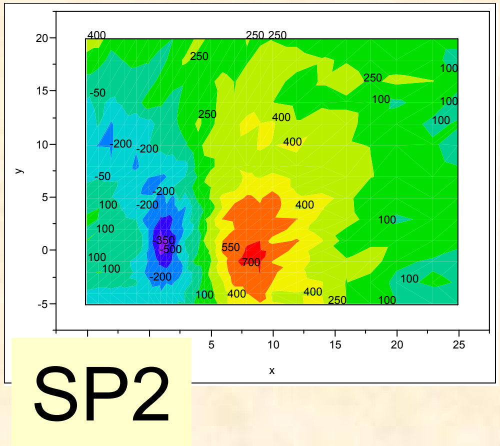
- ➢ Perturbed plastic zones associated with cyclic fatigue and the overload readily distinguished. Overload imposes compressive stress ahead of the crack tip.

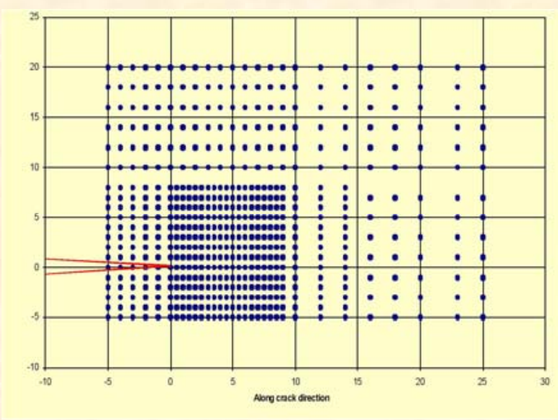
2-D Mappings of Transverse Strains in 304L Overload Specimens

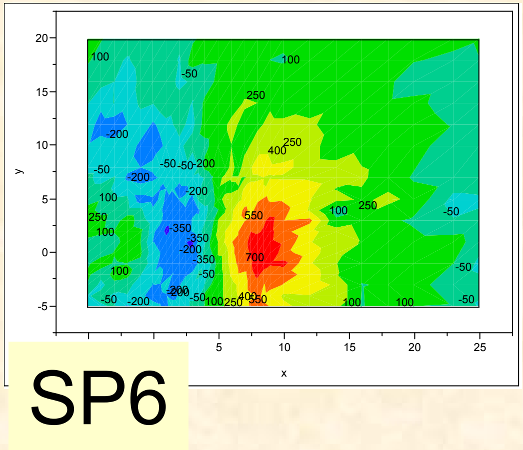
Gage volume 4 mm^3, 1700 data points in four 2-D maps collected in 4 days

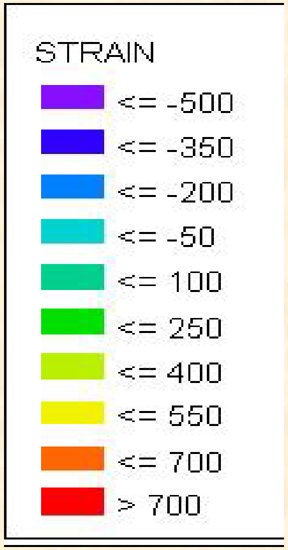








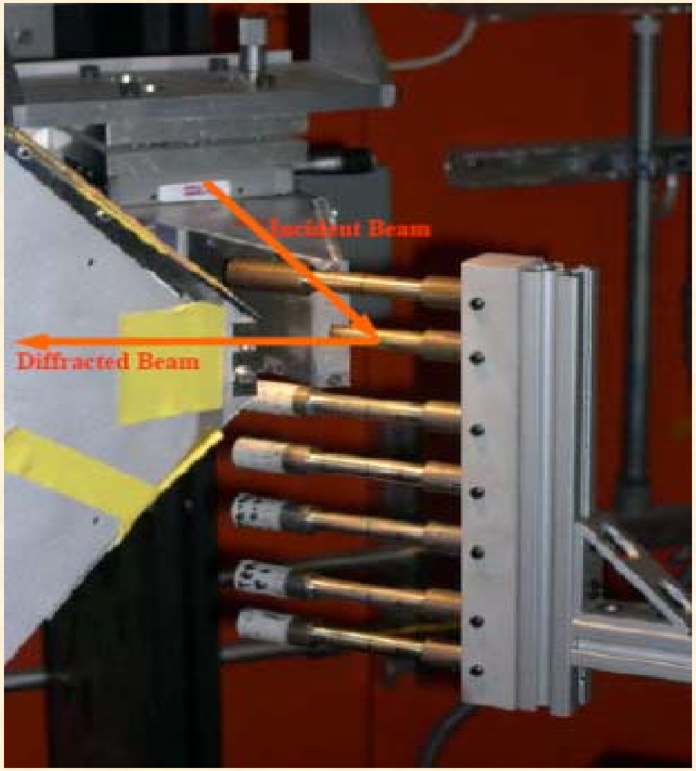




Residual Strains Due to Torsion Versus Tension Deformation using Hollow Cylinder Steel Specimens and Neutron Strain Mapping

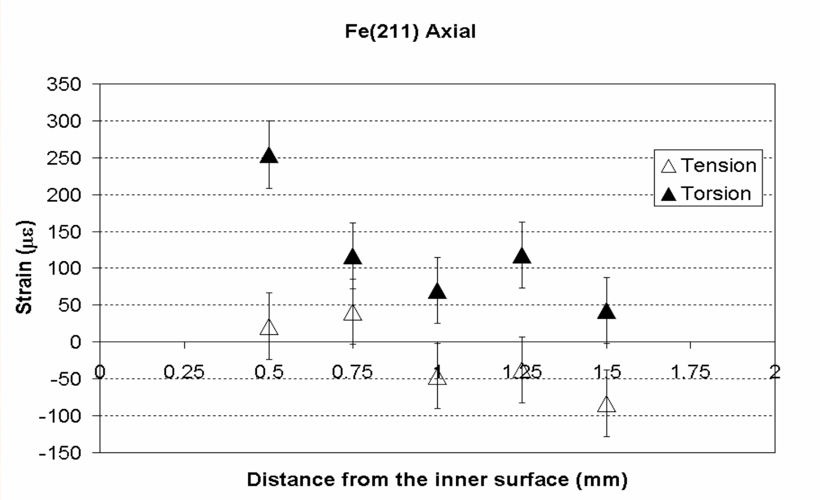
D. Penumadu[1], X. Luo[1], A. Dutta[1], C.R. Hubbard[2], K. An[2], F. Tang[2], H. Choo[1], P. K. Liaw[1]
[1]The University of Tennessee, [2]Oak Ridge National Laboratory

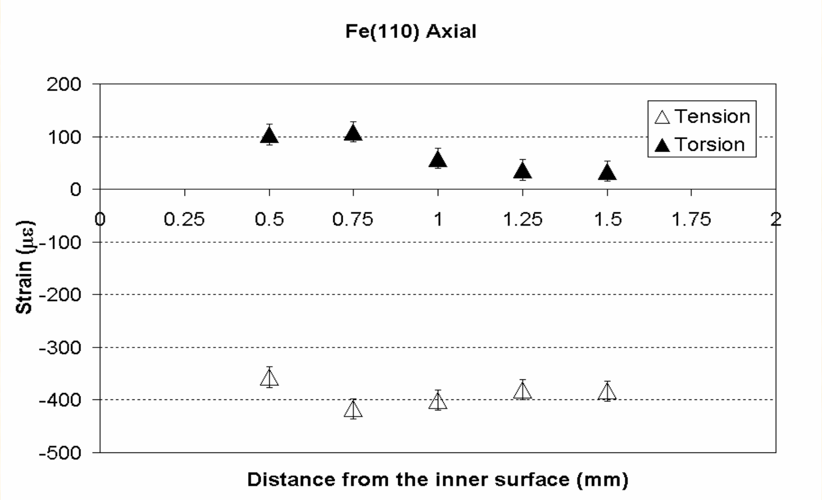
- Torsion provides unique opportunity to probe mechanical behavior of materials under pure shear stress
- In combination with axial load, torsion provides a mechanism to rotate principal stress axes in a controlled fashion
- Steel hollow cylindrical specimens with outer diameter 10.8 mm, inner diameter 6.8 mm, wall thickness 2.0 mm
- Subjected to either pure torsion or pure tension that corresponds to a target magnitude of equivalent octahedral shear strain



The hollow cylinders as mounted on NRSF2 for axial strain measurement

Residual Strains Due to Torsion Versus Tension Deformation Appear Different



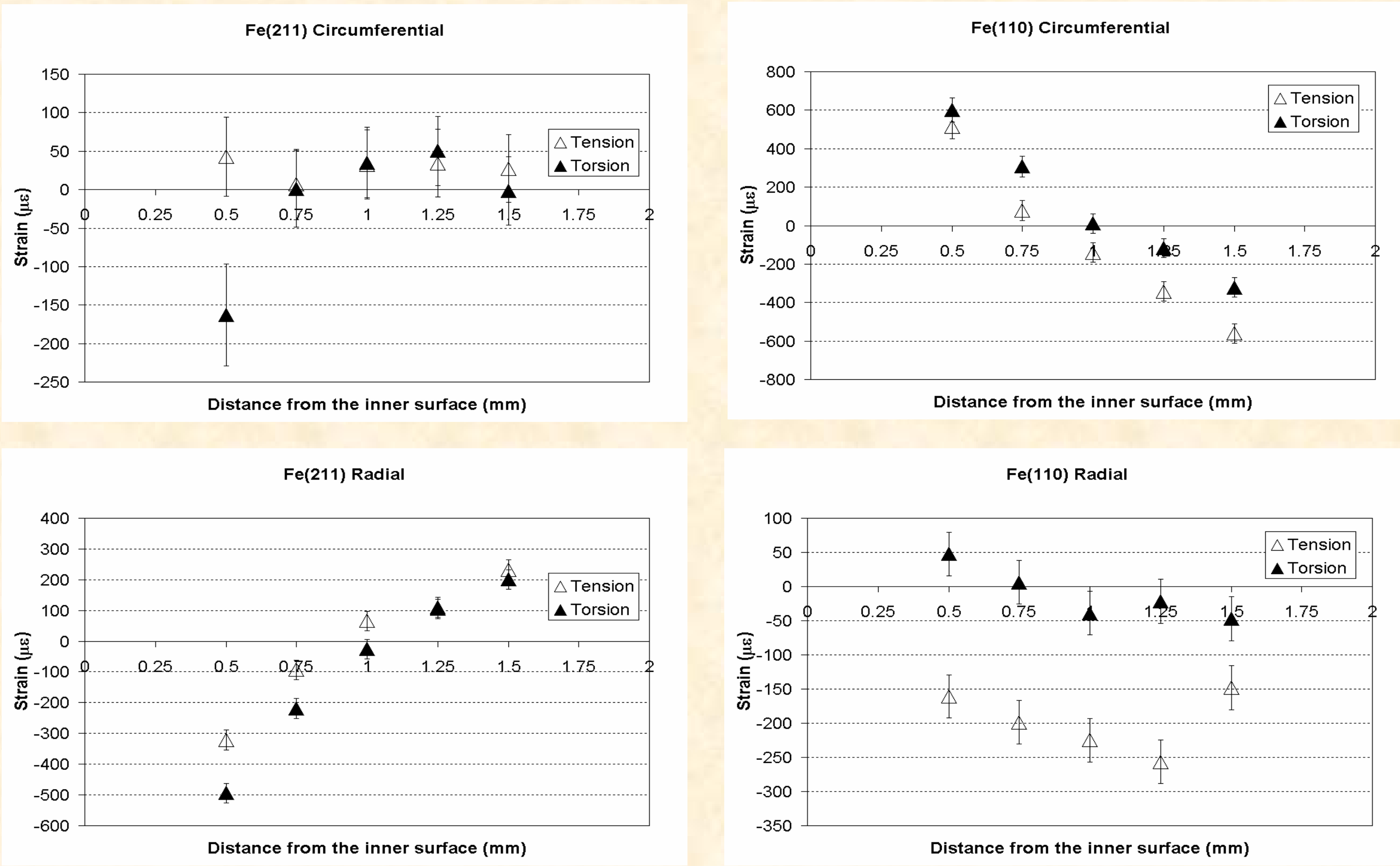


gage volume of 2×1×2 mm^3

- The d-spacings measured for both the Fe(110) and Fe(211)
  - Both reported to be weakly affected by intergranular strain for tension stress path
- Considerable differences exist in the measured residual strains

➢ Understanding of shear deformation on intergranular strains needed

Residual Strains Due to Torsion Versus Tension Deformation in Circumferential and Radial Directions



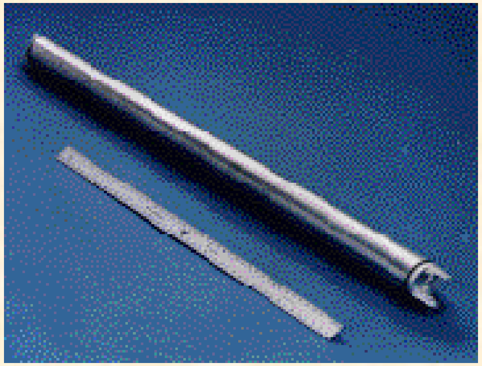
radial and circumferential direction gage volume of 1×2×1 mm^3

UT-BATTELLE

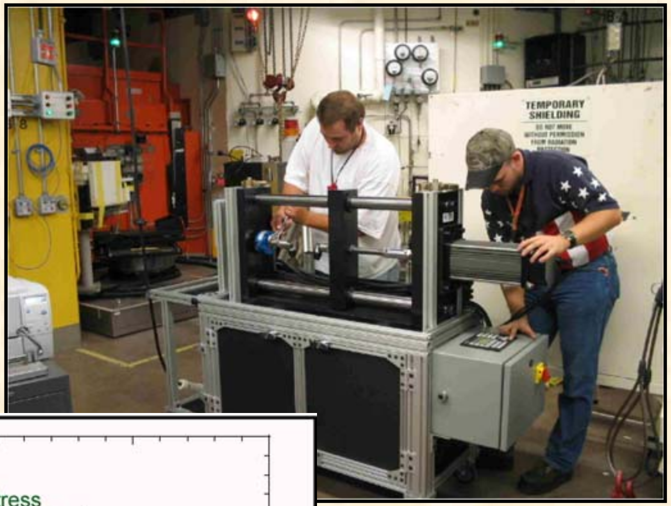
Arizona State Used In-situ Loading on NRSF2

ASU Goal is to Develop Models for Lightweight MMCs

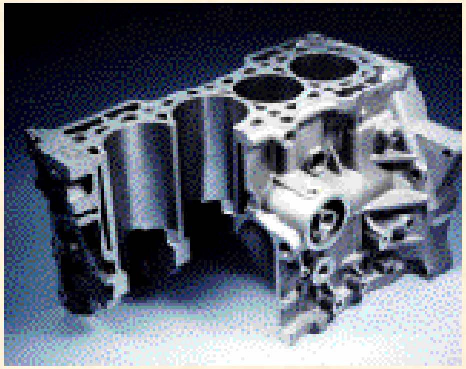
Driveshafts

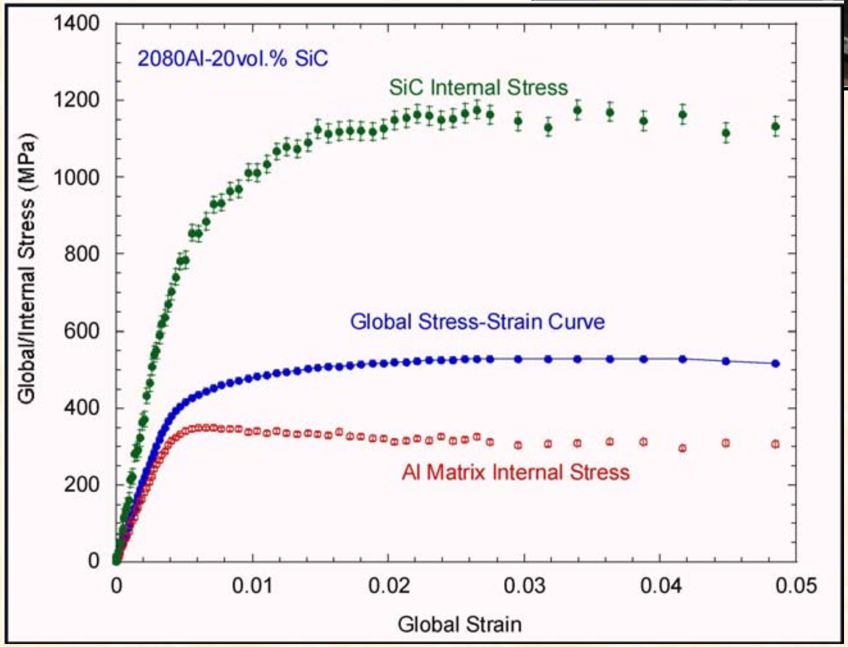


Al-SiC Composites



Cylinder liners



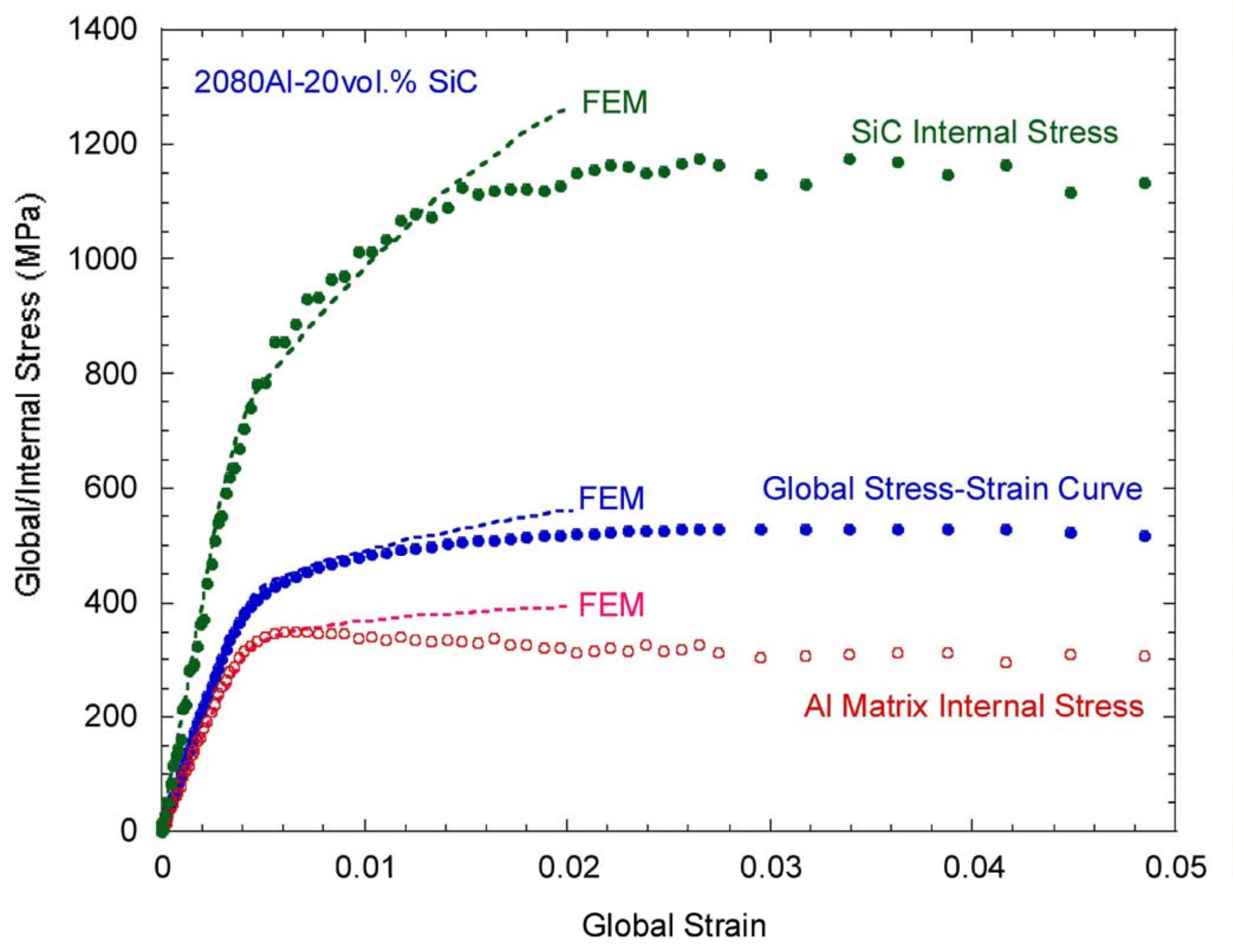


$5x5x5\ mm^3$

Diffraction is phase sensitive!

Load ⟶

Comparison of in-situ Loading Stresses from ND With *FEM* Model Shows Need to Improve Models



Neutron Scattering Measurements on Hydrogen-Charged Zircaloy-4 Using HB1A and NRSF2

E. Garlea[1,2], V. O. Garlea[2,3], H. Choo[1,2], C. R. Hubbard[2], and P. K. Liaw[1]
[1]University of Tennessee, [2]Oak Ridge National Laboratory, [3]Ames Laboratory and Iowa State University

- Zirconium - used in nuclear power application
  - high transparency to thermal neutrons
  - excellent corrosion resistance
  - good mechanical properties
- Mechanical behavior degrades during operation due to a combination of (Fig 1):
  - oxidation
  - hydriding
  - radiation damage
- Experiments were conducted on:
  - 10 mm diameter,
  - 50 mm long Zr-4 rods
  - Charged in H_2 in tube furnace
    - 430°C and 13.8 kPa
    - 30, 60 and 90 min.

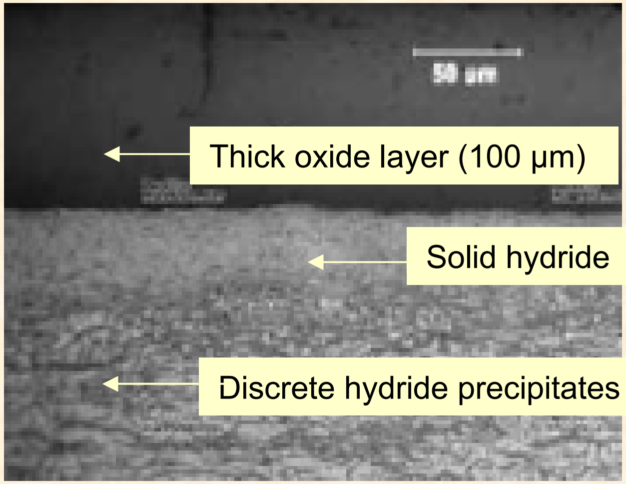


Fig. 1. A hydride layer and associated hydrides in the substrate beneath the layer in **irradiated Zr-4** cladding tube. O.N. Pierron, D.A. Koss, A.T. Motta, K.S. Chan, J. Nuc. Mat. 322 (2003) 21–35

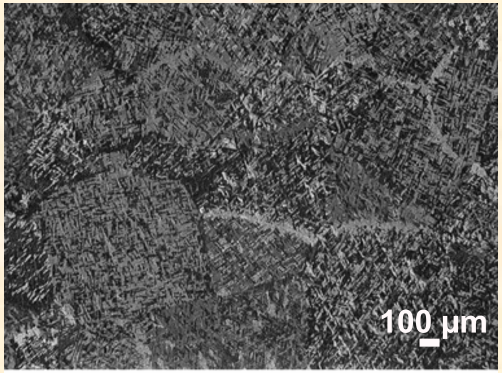


Fig. 2. Basket weave Widmanstatten hcp α-Zr microstructure, showing α-Zr grains with a mean diameter of approximately 700 µm and second phase precipitates.

δ –ZrH_2 Identified in the Bulk

- Zr-4 rods charged with H gas in tube furnace:
  - at 430 ˚C and 13.8 kPa
  - for 30, 60, and 90 min.
- A hydride layer at the surface of the specimen was observed for the 60 min. rod (Fig. 3)
- Neutron diffraction was used for identification of δ-ZrH_2 (Fig. 4):
  - increase in δ-ZrH_2 peak intensities with charging
  - decrease in the Zr peak intensities
  - systematic increase in the overall background
- Neutron incoherent scattering was used for measurement of total H content by quantifying the change of the background signal:
  - large incoherent scattering from H
  - low from Zr-4

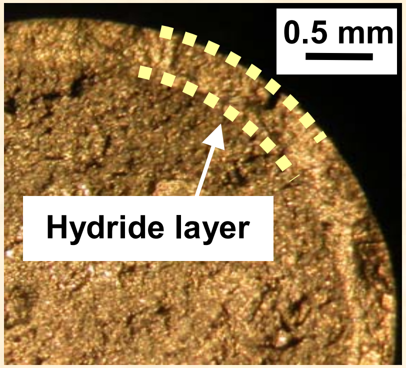


Fig 3. A cross-sectional fracture surface (quadrant) showing a hydride layer with a thickness of about 400 μm at the surface of the specimen.

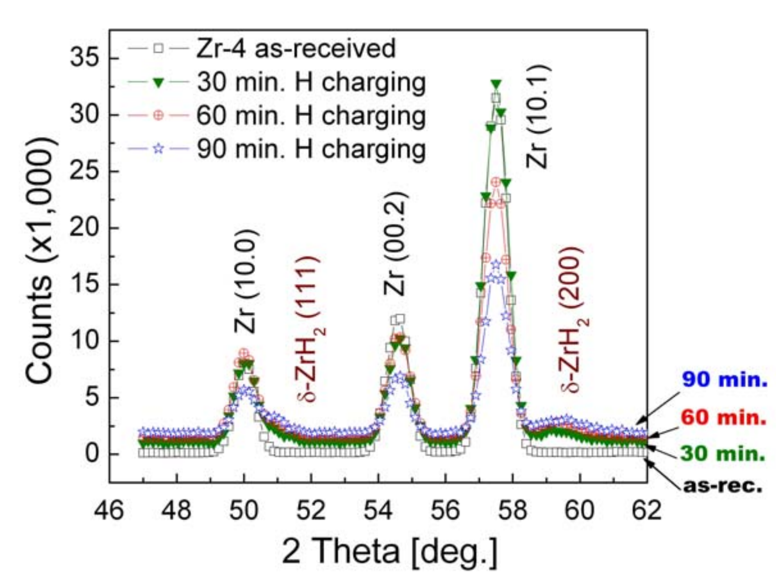


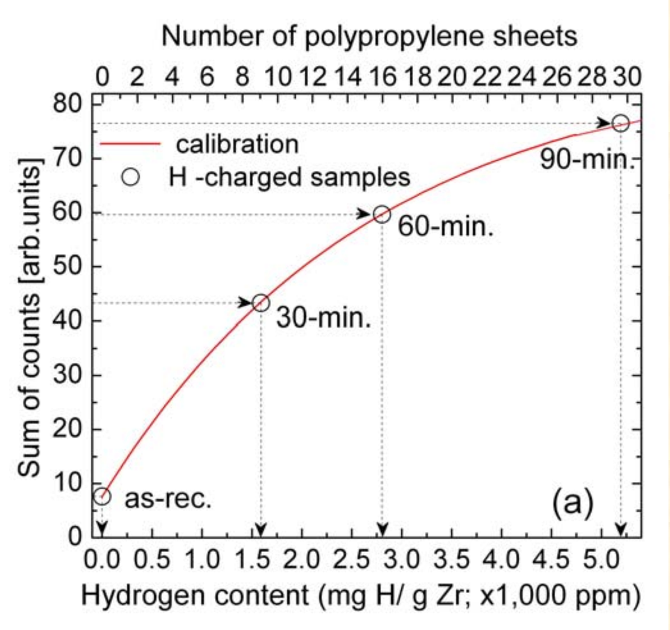
Fig 4. Neutron diffraction profiles of the as-received Zr-4 specimen and specimens charged with H for 30, 60, and 90 min.

HB1A: Calibration and Bulk Hydrogen Content

- Incoherent scattering of bulk and calibration samples
  - ➢ 2θ = 62 deg, λ = 2.367 Å
  - ➢ Flat background- no coherent scattering present
  - ➢ Sum of counts over energy scan is related to the total hydrogen content
- Calibration
  - ➢ Zr-4 rod wrapped with polypropylene sheets
  - ➢ Sets of 3 sheets for each measurement
  - ➢ Calibration curve (red line Fig. 5a and equation)
  - Not considered: attenuation and multiple scattering
- Hydrogen content in Zr-4 rods (Fig. 5b)

$$\boxed{I = I_0 + A[1 - \exp(-c / c_0)]}$$

V. V. Kvardakov, H. H. Chen-Mayer, and D. F. R. Mildner, *J. Appl. Phys.*, 83, 7 (1998): p. 3876-3879.



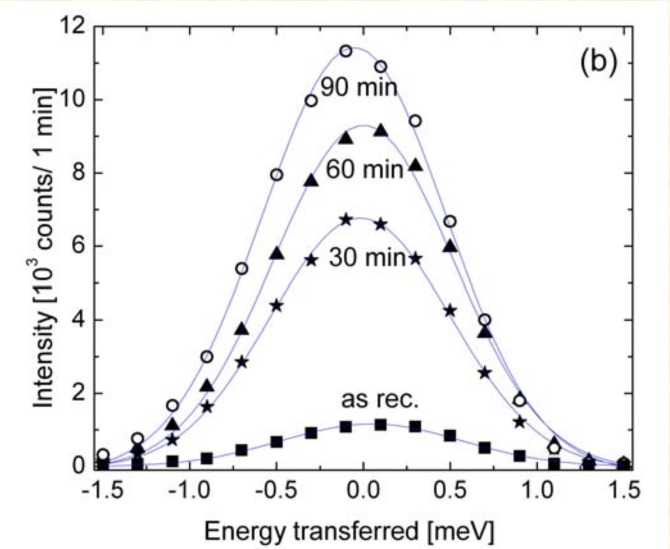


Fig. 5 a. Neutron incoherent scattering of the calibration specimens
Fig. 5 b. Specimens charged with H gas.

Distribution of Hydrogen within the Bulk Mapped

- NRSF2 neutron scattering
  - Gage volume: 1 x 20 x 1 mm^3
  - λ = 1.887 Å
  - 2θ = 74-78 deg., flat background region
- The mapping (Fig 6.) shows a H concentration maximum near the surface
- Future work
  - Quantitative evaluation of the data taking into account H attenuation, sample geometry, and the gage volume.
  - Mapping ZrH_2 phase fraction

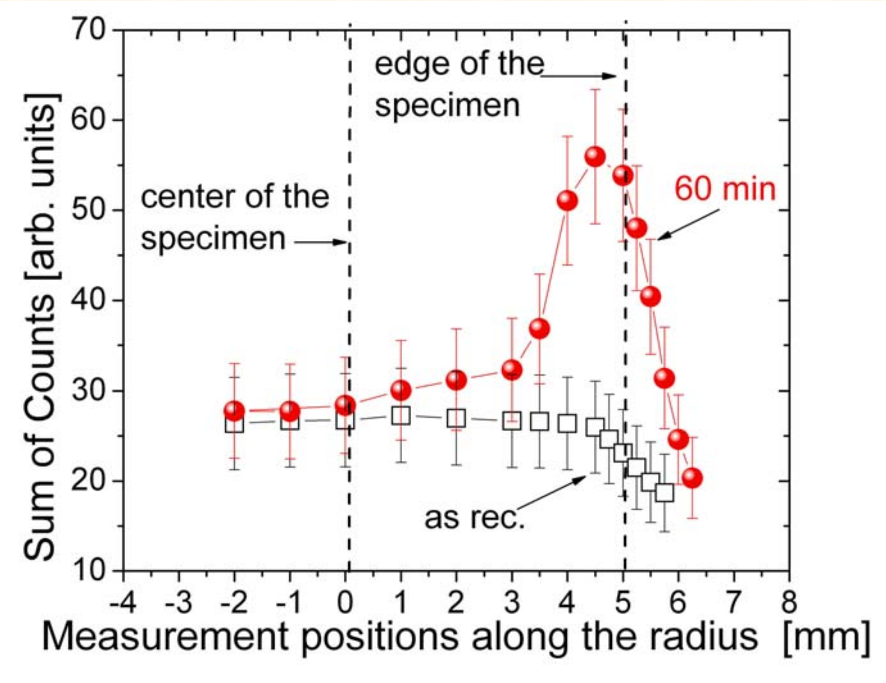


Fig. 6. Variation of the background intensity along the radius of as-received and 60-minute charged round-bar specimens.

E. Garlea, V. O. Garlea, H. Choo, C. R. Hubbard and P. K. Liaw, "Neutron Incoherent Scattering Measurements on Hydrogen-Charged Zircaloy-4", *Materials Science Forum* (2007), 1443-1448.

Next - study impact of hydrogen charging on stresses around cracks and relate to changes in fatigue life

Real Time and Quasi Steady State (QSS) Measurements -
Exploring Neutron Diffraction Mapping for Fast, Real-time Processes

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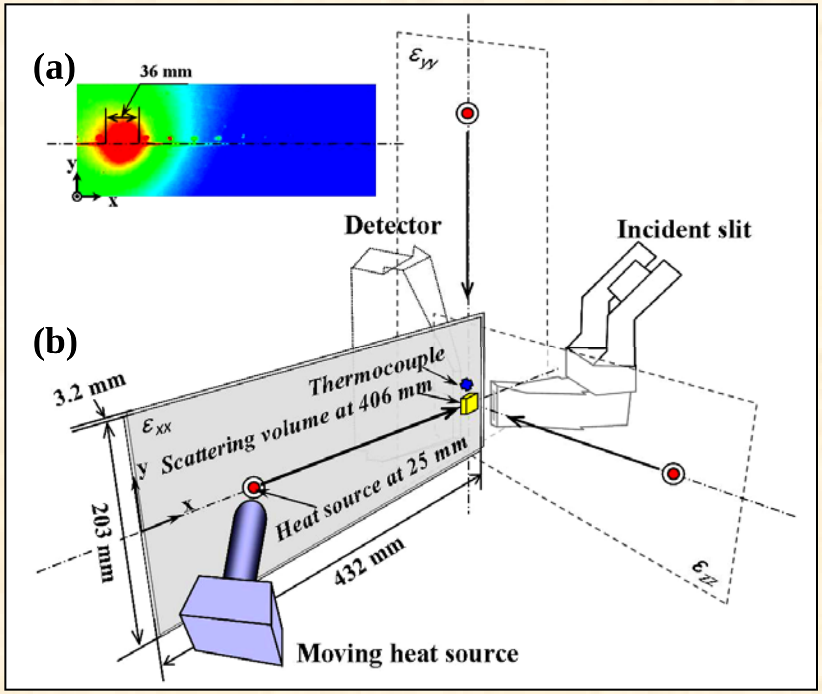
[2]Materials Science and Technology, Oak Ridge National Laboratory, Oak Ridge, TN 37831
[3]Spallation Neutron Source, Oak Ridge National Laboratory, Oak Ridge, TN 37830

- **Objective**: to study transient material behavior

- **Experimental**
  - Thermal stresses were imposed in a local area of a 6061-T6 Al alloy plate using an electric heating gun
  - A controlled heating source yields a uniform thermal distribution with the neutron scattering gage volume (5x5x2 mm).
  - Two different ND methods used:

  Direct real-time measurements: Moving heat source approaches the scattering volume

  A series of QSS measurements: Al plate moves relative to fixed heat source and scattering volume



a) Temperature distribution (infrared thermography)
b) Direct measurements taken every 20 seconds

Equivalency Between Direct and QSS Measurements

- **QSS experimental details**
  - Multiple data points were collected for different predetermined distances between heat gun and scattering volume
  - Profile measured for 10 minutes per location
  - Transformation of coordinates to convert to real space
- **Results**: Excellent agreement between the direct real time and QSS measurements
- **Application**: *In-situ* neutron diffraction measurement of transient temperature and stress fields during thermal processing
- **Publication**: Woo *et al.*, *Applied Physics Letter*, **88**, 261903 (2006)