Engineering

4:00–4:30 **Status of Commissioning NRSF2 at HFIR and Construction of VULCAN at SNS**
Camden R. Hubbard and Xun-Li Wang
Oak Ridge National Laboratory, Oak Ridge, TN

4:30-5:00 **In Situ Neutron Diffraction Studies of Mesoscopic Deformation Behavior of Structural Alloys**
Prof. Hahn Choo
Department of Materials Science and Engineering
University of Tennessee, Knoxville, TN

5:00-5:30 **Application of In-situ Neutron Diffraction Tools Towards Fundamental Understanding of Material Behavior During Thermo-Mechanical Processing**
Dr. Suresh Babu
Edison Welding Institute, Columbus, Ohio

5:30-6:00 **Prospects for Neutron Tomography and High-Speed Radiography: Complex Structure Imaging**
Prof. Les Butler
Department of Chemistry, Louisiana State University

Status of Commissioning NRSF2 at HFIR and Construction of VULCAN at SNS

Camden R. Hubbard[1] and Xun-Li Wang[2]

[1] Metals and Ceramics Division

[2]Spallation Neutron Source

Oak Ridge National Laboratory

SNS-HFIR User Group Meeting

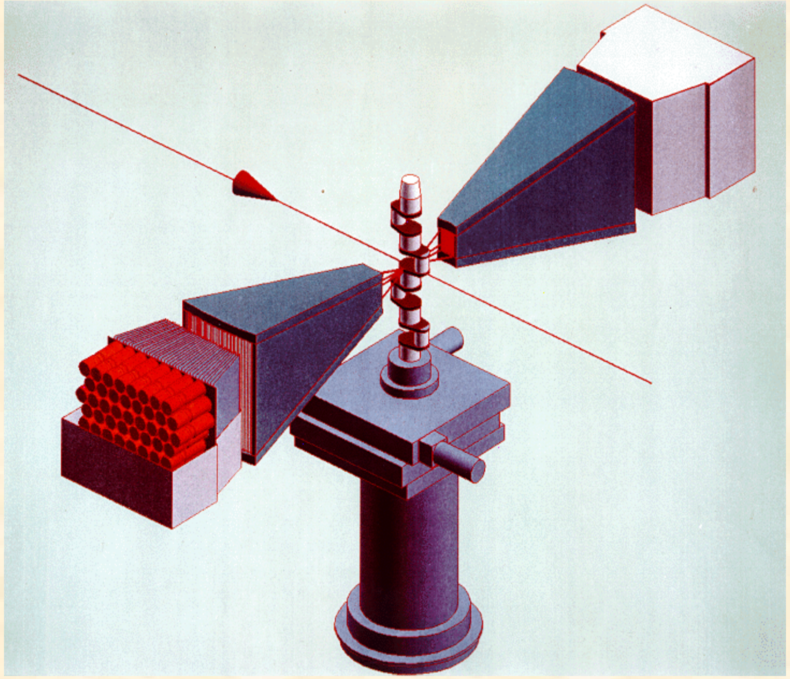
11 October 2005

Outline

- **Neutrons & Engineering Diffraction Instruments**
- **HFIR**
  - **Upgrades**
  - **Neutron Residual Stress mapping Facility (NRSF2)**
  - **2005 & 2006 projected schedule**
- **SNS**
  - **Overview**
  - **VULCAN**

Neutrons provide unique opportunities for stress and texture measurements

- **Applicable to nearly any crystalline material - i.e. structural materials**
- **Neutron diffraction measures:**
  - **Non destructive Bragg diffraction**
  - **Elastic strain for each phase, many hkl**
  - **Strain tensor**
  - **Intergranular (type II) strains**
  - **Texture and defect type & density**
- **Easy to set up ancillary facilities for in-situ loading and thermal cycling**
- **Real-time measurements possible**
  - **At highest flux sources**
  - **Aided by large detector arrays**
- **Through thickness mapping complements x-ray surface stresses**



Neutron strain mapping has evolved rapidly since its demonstration in the 1980's

- 1st Generation (1980's - 1999)
  - Typically built up from existing powder diffraction instruments, shared time,
    - limited detector coverage and sample sizes
    - strain sensitivity to ~ 100 ppm, resolution to ~1 mm
  - By end of 1990's about 25 instruments worldwide
  - Round robins conducted, International standard drafted
- 2nd Generation (2000-   )
  - Dedicated instruments built specifically for strain mapping
  - Optimized delivery of neutrons (source, guides, monochromators, optics)
    - Flux > 10^7 neutrons/cm^2/sec
  - Larger detector coverage with ~10-20x gain in counting rate
  - Larger specimens and ancillary equipment support
  - Sensitivity and reproducibility 50-100 ppm, resolution to ~0.3 mm
  - Text books published & college classes emerge
- 3rd Generation (2004-   ;~10x gain over 2nd generation)
  - Highest flux pulsed sources with unique, advanced neutron optics
  - Very large detector coverage and sample capacity
  - Sensitivity to <50 ppm, resolution to ~0.1 mm in 1-D

A survey of selected reactor based neutron strain mapping facilities shows many new instruments and upgrades

Country	Neutron Source	Strain Mapping Instrument	"Generation" (approx. yr instrument commissioned)
France	Institut Laue-Langevin (ILL)	SALSA	1st (~1995) 2nd (2005)
USA	High Flux Isotope Reactor (HFIR)	NRSF NRSF2	1st (1991) 2nd (2005)
USA	NIST Center for Neutron Research (NCNR)	BT-8	1st (1996?)
USA	Univ. of Missouri Research Reactor	---	1st (1995?)
Canada	Canadian Neutron Beam Center at NRU (CNBC)	E-3, L-3	1st (~1988) 2nd (~1998)
Australia	Bragg Institute, ANSTO	Strain Scanner	?? (2005)
Germany	FRM-II	Stress-Spec	2nd (2005)
Korea	Hi-flux Advanced Neutron Application Reactor	RSI	?? (2004)

Strain mapping instruments at spallation based neutron sources are also being built rapidly

Country	Neutron Source	Strain Mapping Instrument	"Generation"
USA	IPNS	(flux too low for mapping)	--
Switzerland	SINQ	POLDI	?
USA	LANSCE	SMARTS	2nd (2003)
UK	ISIS	ENGIN ENGIN-X	2nd (1996?) 3rd (2004)
USA	SNS	VULCAN	3rd (2008)
Japan	JPARC	?	3rd (2009?)

NRSF, First Strain Mapping Facility at HFIR

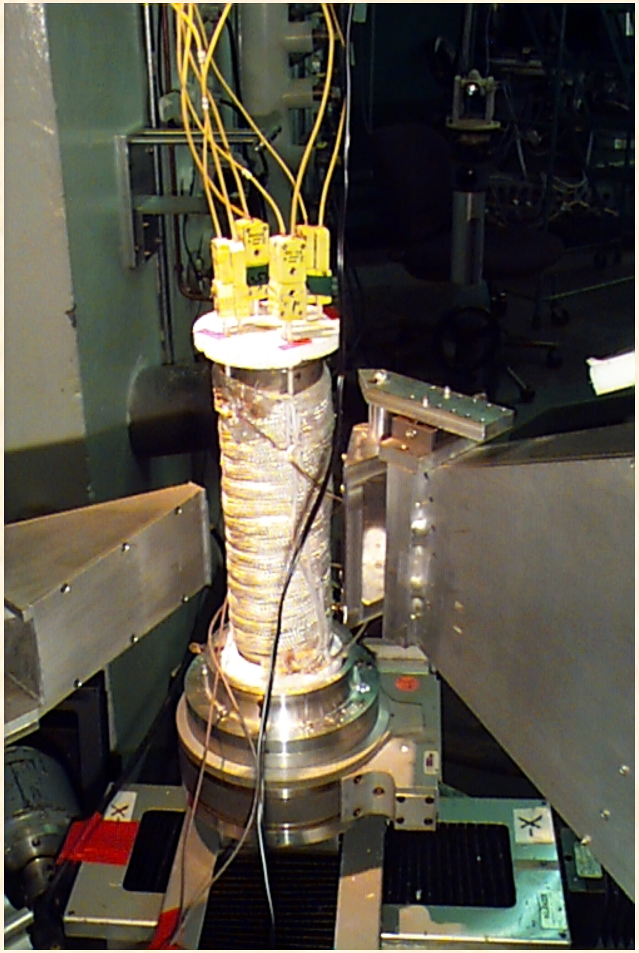
ORNL's first generation neutron residual stress facility shared use of a 3-axis spectrometer at HB-2

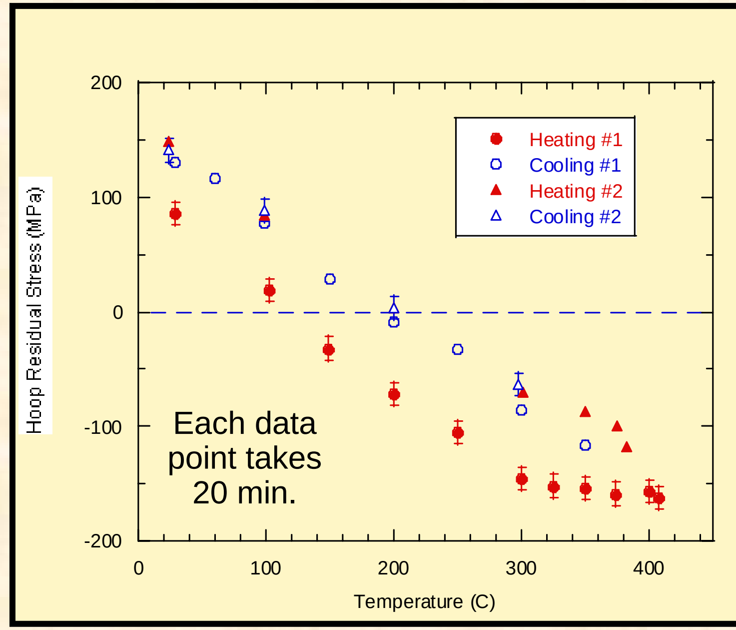
Large samples could be studied such as the strains about a girth weld in 24" diameter SS pipe

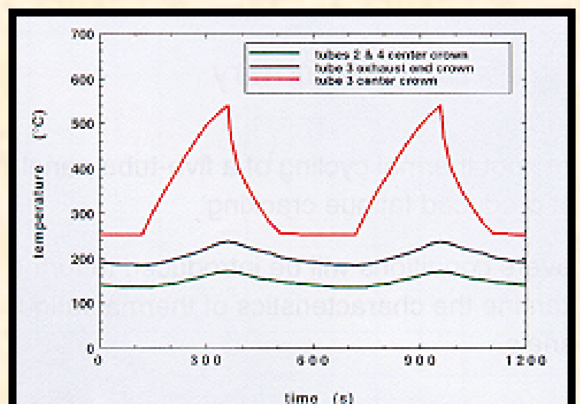
XYZ translation stages enabled mapping in 3-D

Primary focus was on engineering studies

Measurement speed was limited - only simple process simulation studies could be done

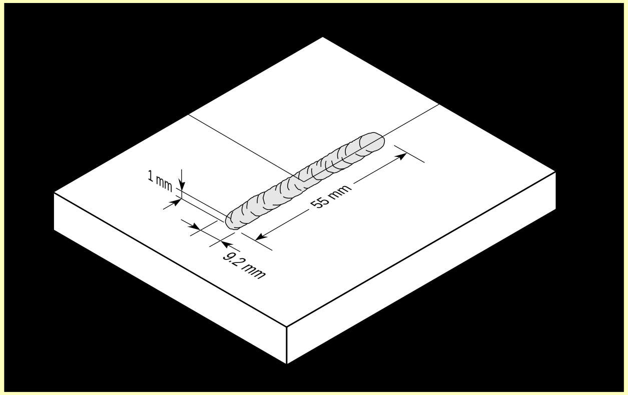




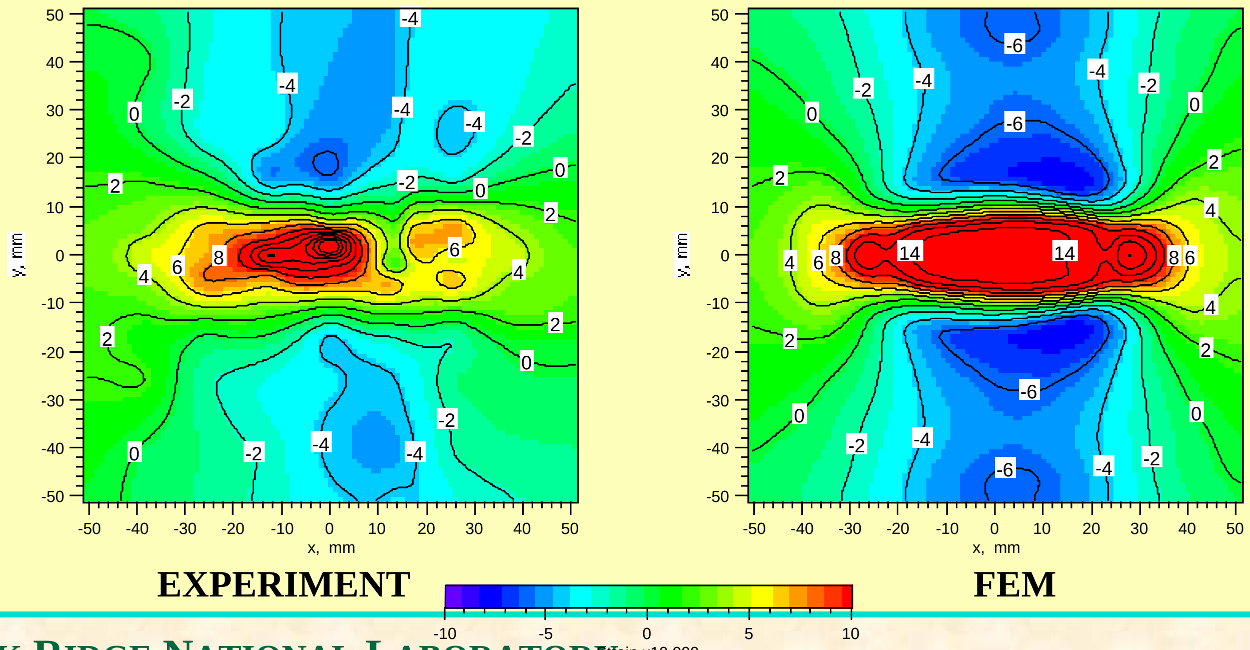


Stress in 304L cladding of CS boiler tubes as function of temperature, Wang et al. APL, 1999

Neutron strain mapping enabled quantitative calibration of finite element models



Yet, parametric studies on the first generation instruments were too time consuming for many proposals



EXPERIMENT

FEM

Fe-Al Weld Overlay

On CS, Feng et al.

ASME PVP,1996.

Single depth below weld bead was mapped

First generation neutron residual stress mapping instruments had limitations

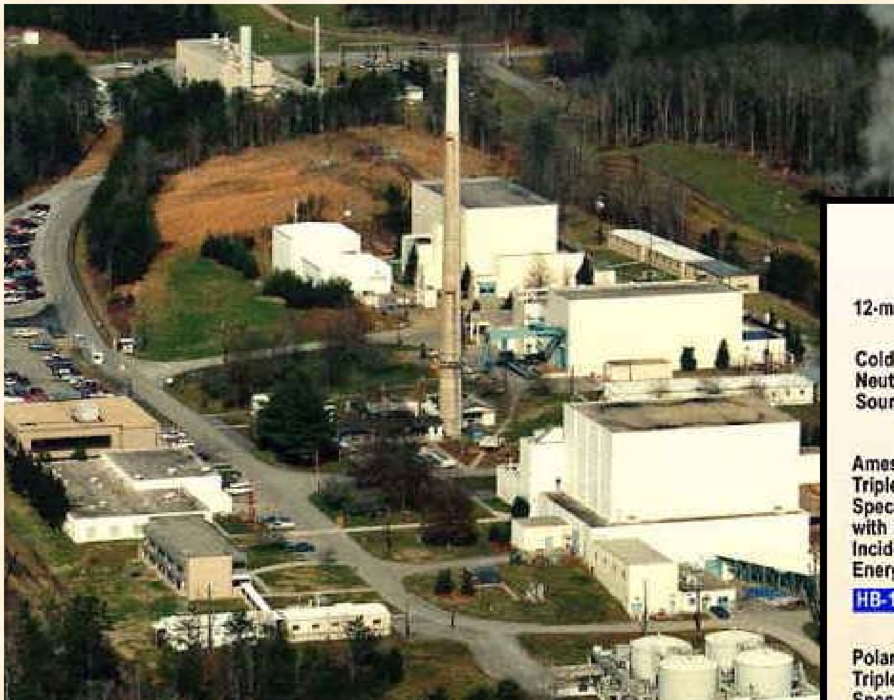
- Often shared time on existing scattering instruments
- Instrument not necessarily optimized for strain measurement
- 2-D measurements took many days or weeks
  - Parametric studies were often not feasible
  - Yet measurements with varying conditions are needed for model validation
- Inadequate for “real-time” in-situ stress measurement
  - For understanding the kinetics of dynamic stress relaxation processes
  - Critical to model development efforts
- Couldn’t capture microstructure and strain simultaneously
- Multiple hkl measurements were difficult
- Large grains (>50 μm) cause “spotty” peaks
  - Required oscillation of sample where possible
  - Need to measure strain in individual grains in some cases
- Nearer surface stress mapping often needed to complete picture

The Second Generation Neutron Residual Stress Mapping Facility

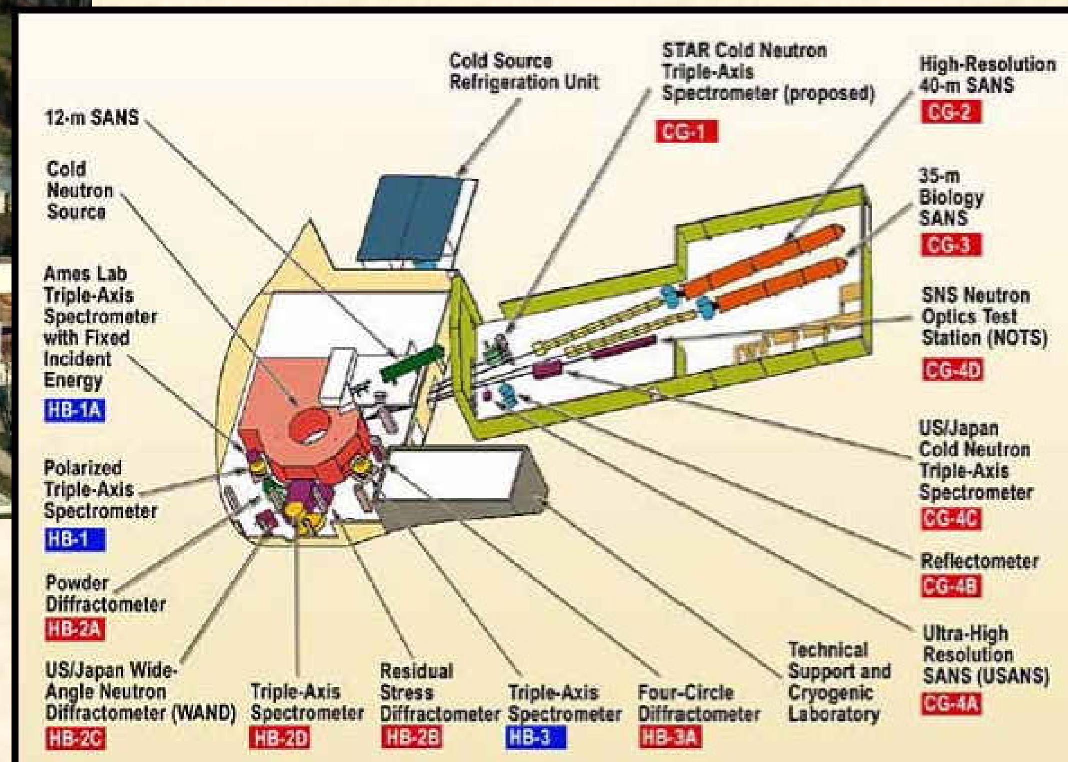
(NRSF2)

installation begun May 2004
commissioning April - July, 2005

High Flux Isotope Reactor, one of the two highest flux reactor facilities worldwide, is undergoing major upgrades in scattering facilities



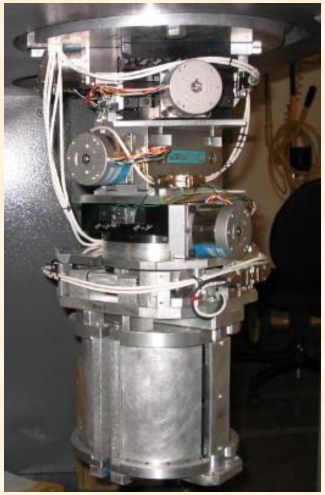
New reflector and beam tubes in 2000-2002. New scattering instruments are to be “world class”, with upgrades complete in 2008.



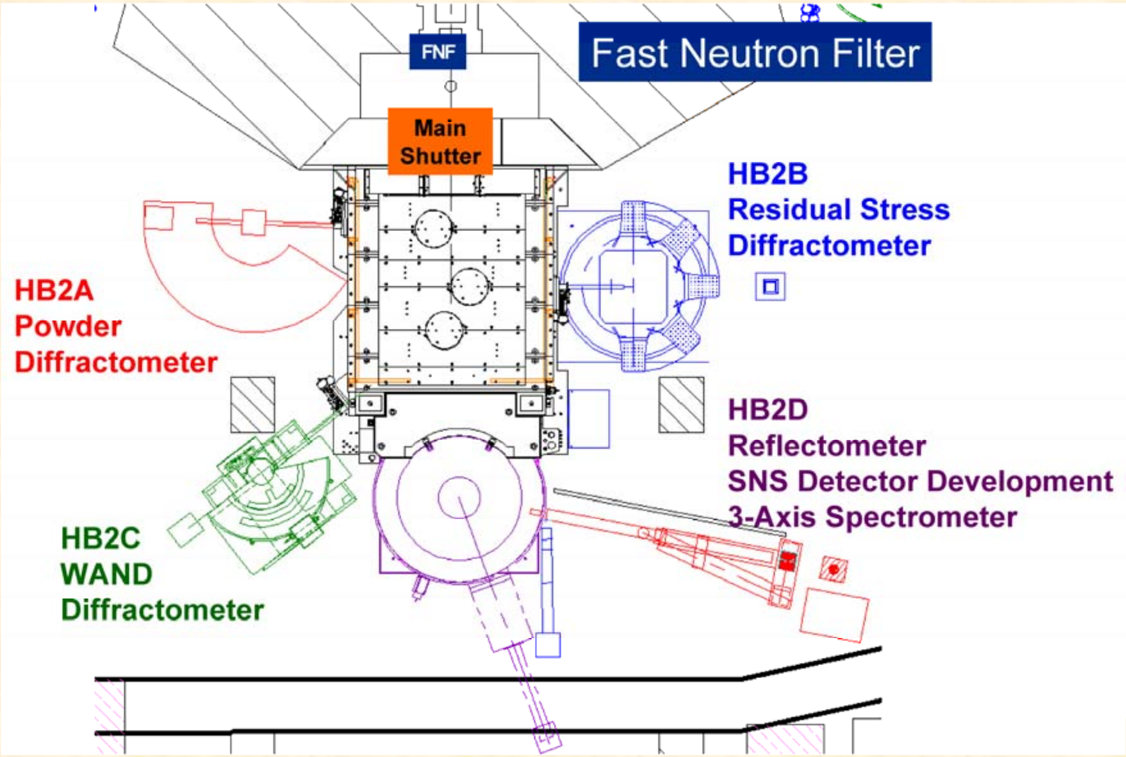
Built in the 1960’s primarily for isotope production and irradiation. Has only 4 beam tubes

NRSF2 instrument design goals were based on science needs and aim to overcome limitations

- **Capacity to measure wide range of specimens**
  - **Small research specimens (e.g. crack tips, rivets, interfaces)**
  - **Large industrial specimens (e.g. engines, castings, blades, tubes and panels)**
  - **Single crystals and those with "large" individual grains**
- **Studies of various materials (Fe, SS, Al, Zr, Be, ZrO_2, ...)**
  - **Multiple wavelengths or pulse sources**
  - **Measurement of intergranular strains (type II)**
- **Moderate FWHM resolution (~0.3°)**
  - **Working 2θ range of 70° to 110° or greater**
  - **Precision of measurement to 0.002° 2θ or better**
- **Faster measurement with higher accuracy**
  - **Higher flux in small area (> 10^7 n/cm^2/sec)**
  - **Cubic and match stick sampling volumes from ~0.5 to 100 mm^3**
  - **Dynamic measurements (e.g. load, fatigue, temperature, ….)**



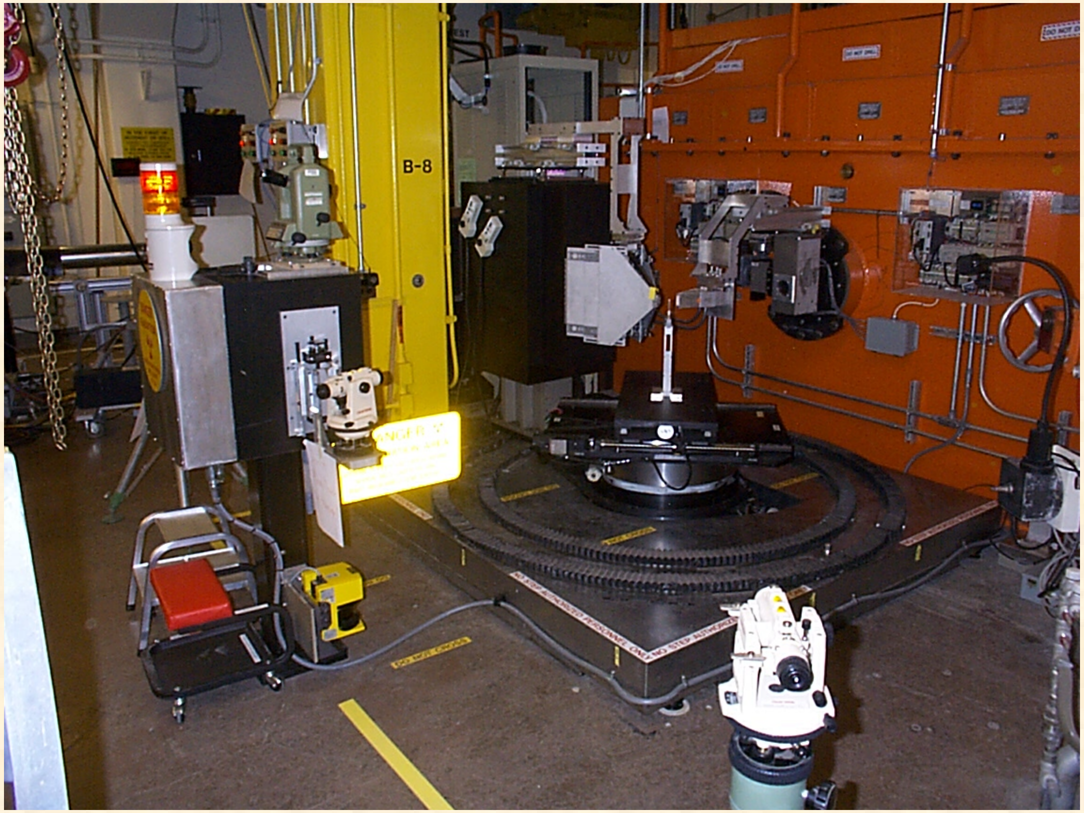
NRSF2 is one of four new instruments to be installed around HFIR's HB-2 tunnel - fixed take off angle imposed



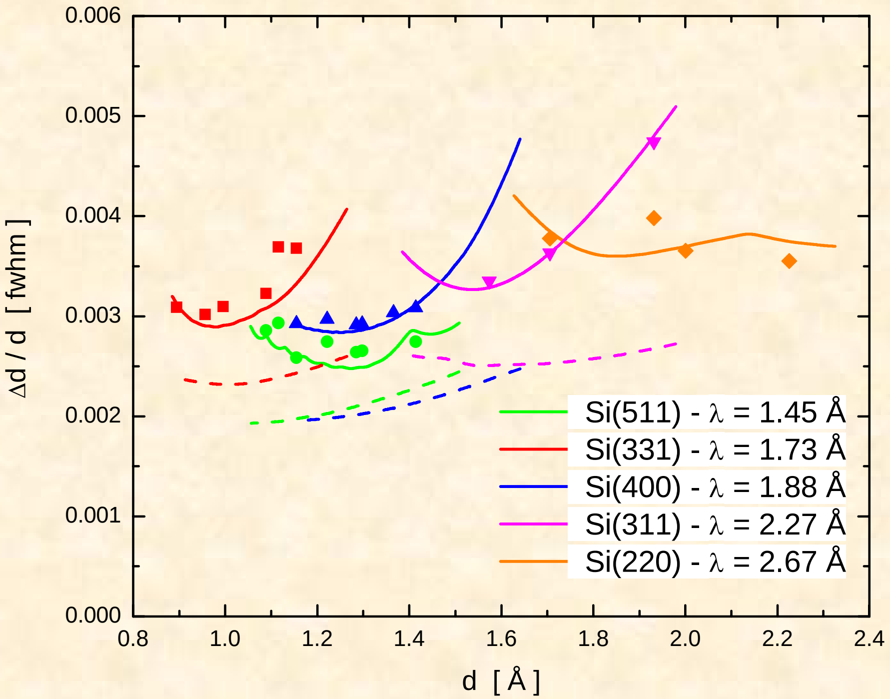
- ✓ New double crystal, doubly focusing Si wafer monochromator
  - higher flux at specimen
  - Six selectable wavelengths
- ✓ Dedicated location - 100% of time to materials science and engineering
- ✓ Two interchangeable large specimen goniometers
  - XYZ and Kappa
- ✓ 7- PSD detector array
- ✓ Load frame for materials behavior studies

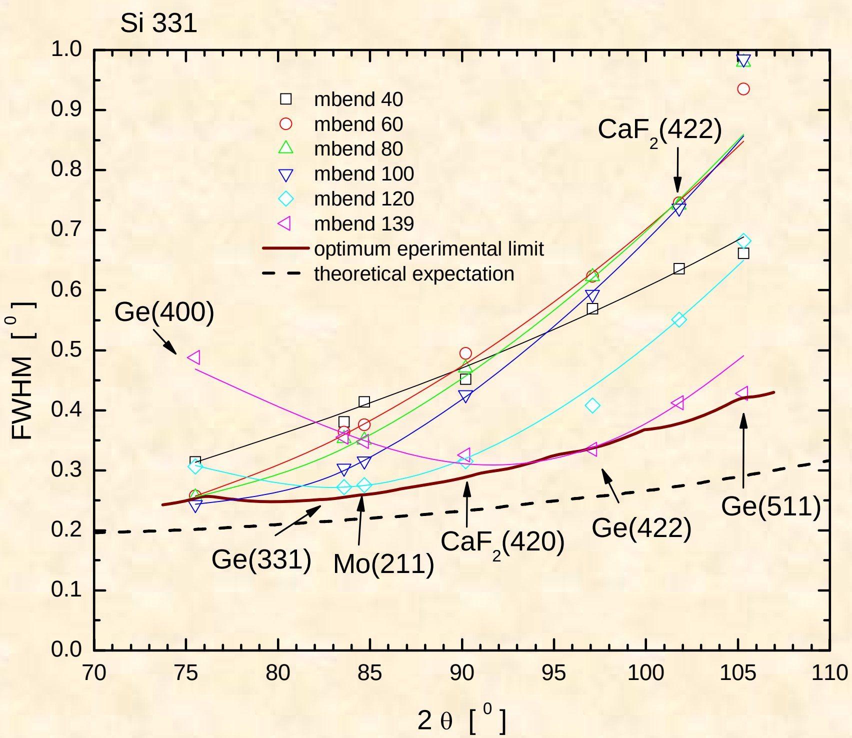
Large 2θ/ Ω goniometer with XYZ stages improves accuracy and extends capabilities

- **Mechanically rigid, highly reproducible positioning**
- **2θ accuracy ~ 0.001°**
  Ω accuracy ~ 0.003°
- **X & Z-stages = 400 mm**
  Y-stage = 200 mm
  precision = 0.01 mm
- **200 kg load directly on XY**
- **50 kg load capacity on Z**
- **Large platform to allow use of accessories**
  - **load frames**
  - **chi-phi orienteer**
  - **bottom loading z-stage**
- **Increased distance between beam height and XY stage**



The “Missouri” Si wafer, double crystal, doubly focusing monochromator provides NRSF2 choices of λ from 1.45 to 2.67 Å. FWHM optimized by changing the bending radius



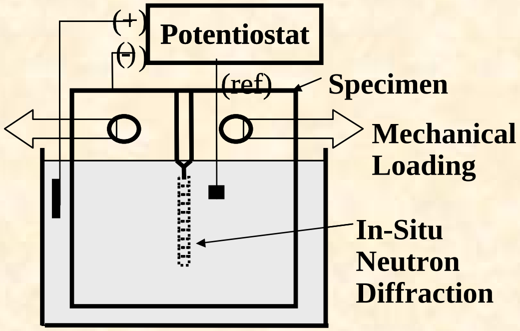


Load frame & environmental cells added for

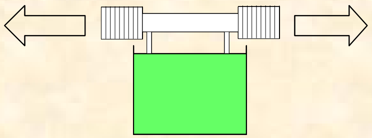
systematic in-situ deformation and materials behavior studies

- An automated load frame **has been developed to study specimens under large uniaxial loads for in-situ deformation studies.**
- **A wet-chemical, environmental cell is being developed for:**
  - hydrogen charging
  - **cathodic protection**
  - **corrosion studies**
- **Key features include:**
  - **5000 lbf loading capacity**
  - Tension/compression **(static)**
  - Low cycle fatigue **(dynamic)**
  - **Automated load control and macro-strain recording**
- **Operational test began in November 2004**

NRSF2's load frame on cart ready to be placed onto XYZ unit

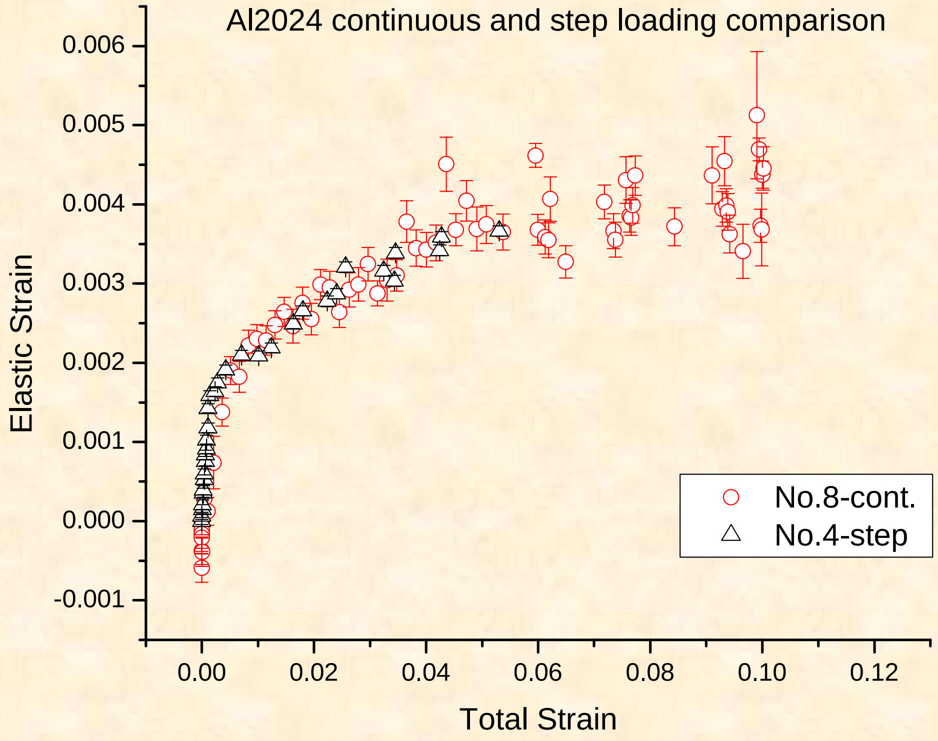


Environmental cell for electrochemical charging



2nd sample geometry

Continuous Loading Versus Step Loading Yields Similar Results and is >50% Faster



Neutron data collection time:

- 2 min/point step loading required **60 minutes**
- 15 sec/point for continuous loading required **25 minutes** and extended to greater total strain

> ➢ *Continuous loading enables neutron diffraction materials behavior studies more comparable to conventional mechanical property testing*

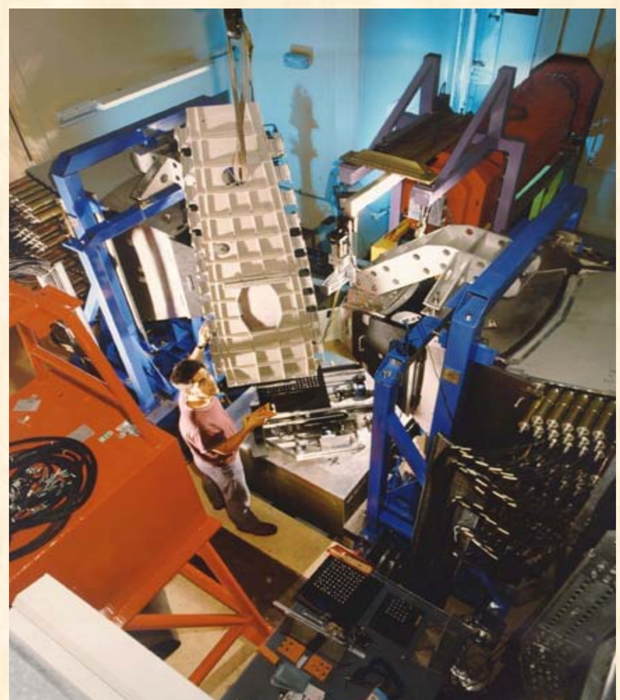
Spallation Neutron Sources

2^{nd} and 3^{rd} Generation Neutron Sources for Strain Mapping and In-situ Materials Behavior Studies

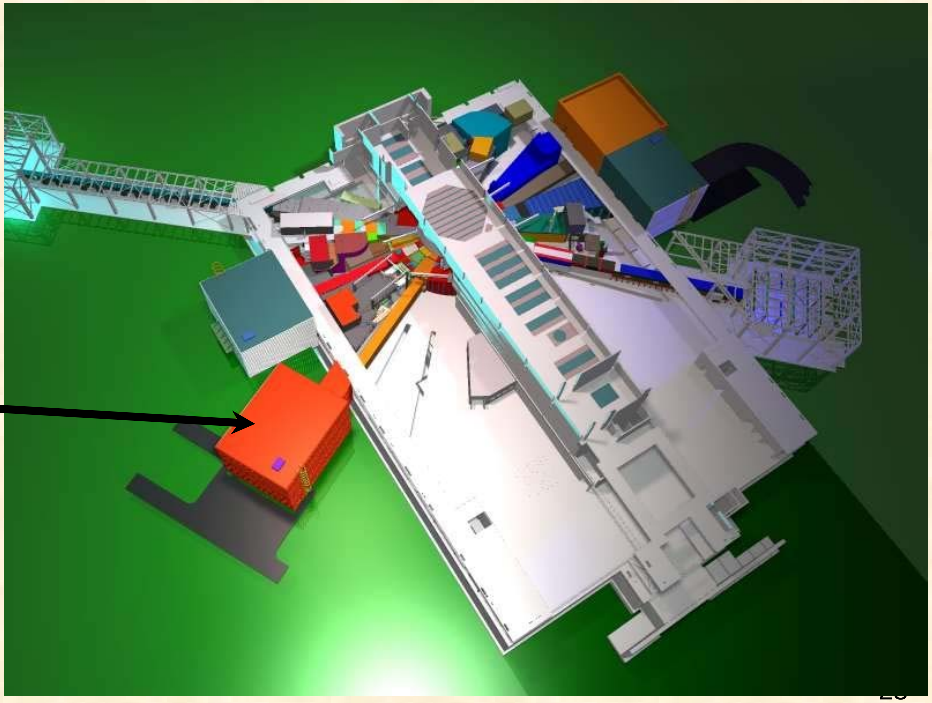
Large gains (~15x) in count rate achieved with upgrade from ENGIN to ENGIN-X at ISIS

Experiment type	ENGINŠX count time	ENGIN count time
Strain scanning, Al, 2x2x2 mm gauge, 50mm path length, per point	5 minutes	1.3 hours
Strain scanning, Fe, 2x2x2 mm gauge, 14mm path length, per point	5 minutes	1.5 Š 2 hrs
Strain scanning, Fe, 2x2x2 mm gauge, 30mm path length, per point	20 minutes	5 hours
Strain scanning, Fe, 4x4x4 mm gauge, 60mm path length,per point	1 hour	Impossible
In situ loading, 4x8x4 mm gauge, Fe, per measurement	1.5 minutes	1 hour
In situ loading, 4x8x4 mm gauge, Ti, per measurement	7 minutes	4.5 hours

Wing spar specimen being set up for strain mapping at ENGIN-X

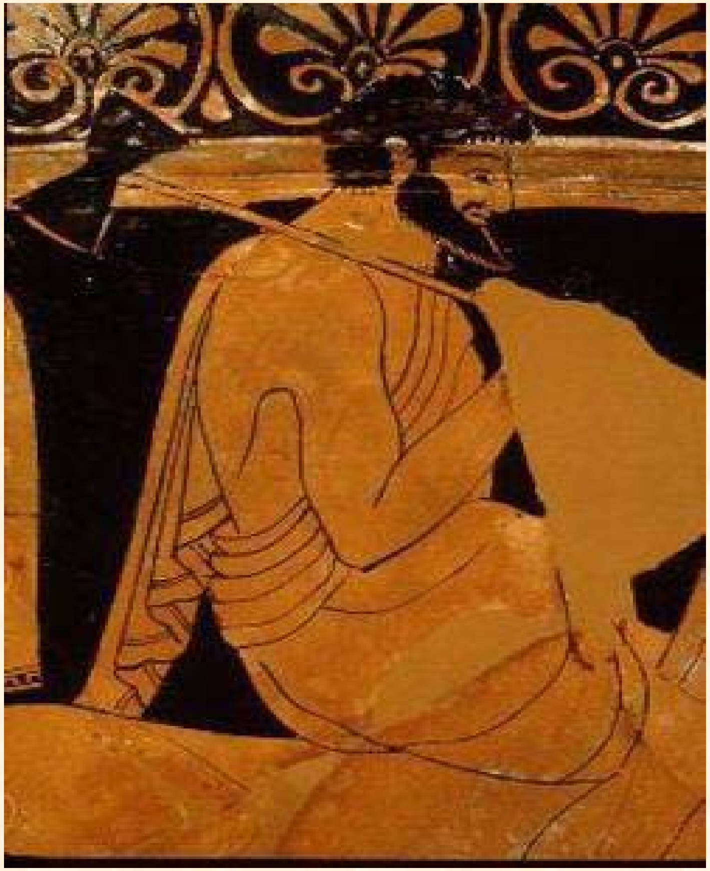


VULCAN is the next (3rd) generation engineering instrument *- SNS is Over 90% complete and first neutrons are scheduled for mid 2006 with full power by 2008*



VULCAN - Engineering and Materials Science

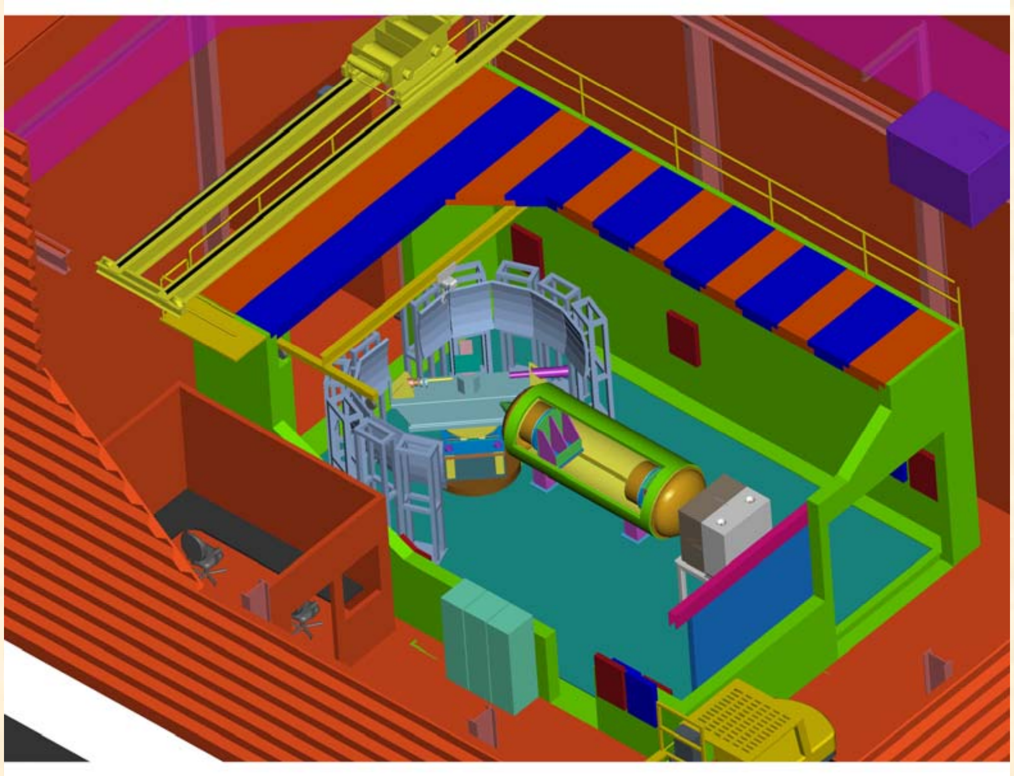
VULCAN is named after the Roman God of fire and metal working



A son of Jupiter and Juno, VULCAN was the craftsman that forged the armor of the gods, their drinking vessels, and many of their objects of ornamentation. Vulcan was also the god of fire. Venus was his wife.

VULCAN is designed to tackle a broad range of problems in materials science and engineering

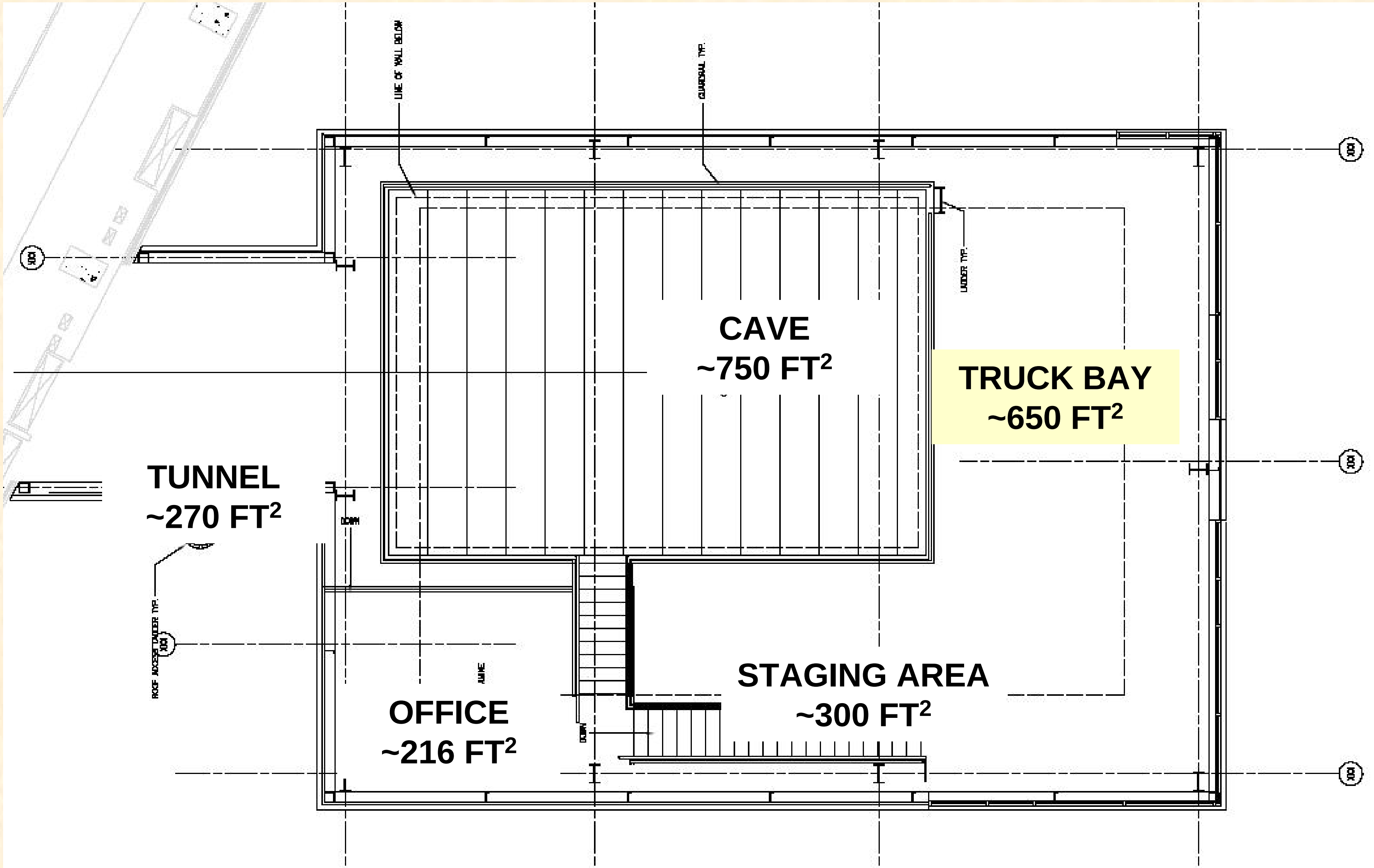
~ 10 times the flux as ISIS



Ancillary equipment such as furnaces and load frames will be integrated parts of the instrument

- Rapid volumetric (3D) mapping with a sampling volume of 1 mm^3 and a measurement time of minutes
- Very high spatial resolution (~0.1 mm) in one direction with a measurement time of minutes
- ~20 well defined reflections for in-situ loading studies
- Ability to study kinetic behaviors in sub second
- Simultaneous SANS measurements

Architectural Drawing – Plan Section



Projection of count time for VULCAN compared to that obtained at ENGIN-X is ~ 10x faster for same sampling volume

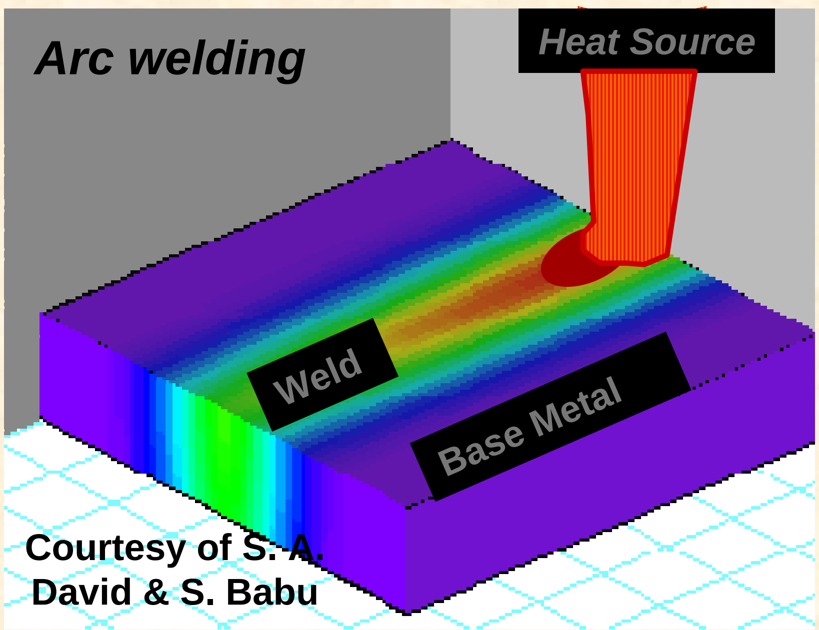
Experiment type	ENGINŠX count time	ENGIN count time
Strain scanning, Al, 2x2x2 mm gauge, 50mm path length, per point	5 minutes	1.3 hours
Strain scanning, Fe, 2x2x2 mm gauge, 14mm path length, per point	5 minutes	1.5 Š 2 hrs
Strain scanning, Fe, 2x2x2 mm gauge, 30mm path length, per point	20 minutes	5 hours
Strain scanning, Fe, 4x4x4 mm gauge, 60mm path length,per point	1 hour	Impossible
In situ loading, 4x8x4 mm gauge, Fe, per measurement	1.5 minutes	1 hour
In situ loading, 4x8x4 mm gauge, Ti, per measurement	7 minutes	4.5 hours

Experiment type	VULCAN count time
Strain scanning, Al, 1x1x1 mm gauge, 50mm path length, per point	4 minutes
Strain scanning, Fe, 1x1x1 mm gauge, 14mm path length, per point	4 minutes
Strain scanning, Fe, 2x2x2 mm gauge, 30mm path length, per point	2 minutes
Strain scanning, Fe, 4x4x4 mm gauge, 60mm path length, per point	6 minutes
In situ loading, 4x8x4 mm gauge, Fe, per measurement	9 seconds
In situ loading, 4x8x4 mm gauge, Ti, per measurement	1 minute

Dynamic study of metallurgical changes in welds (*L* ~ cm, *t* ~ sec) becomes possible at SNS

Welding introduces
- **Compositional gradients**
- **Microstructure gradients**
- **Stress gradients**

Complex issues in welding arise from the coupled interactions of temperature, time, atmosphere, stress, and phase stability

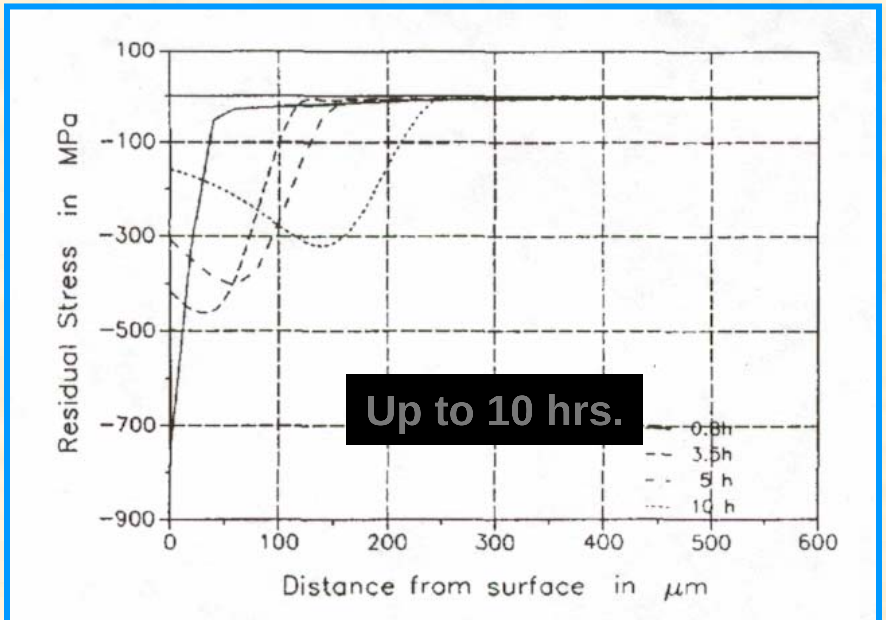


Many unanswered questions
- **the effect of phase transformation**
- **influence of precipitation**
- **effect of recrystallization and recovery**
- **development of microvoids and cavities**
- **joining of dissimilar material**

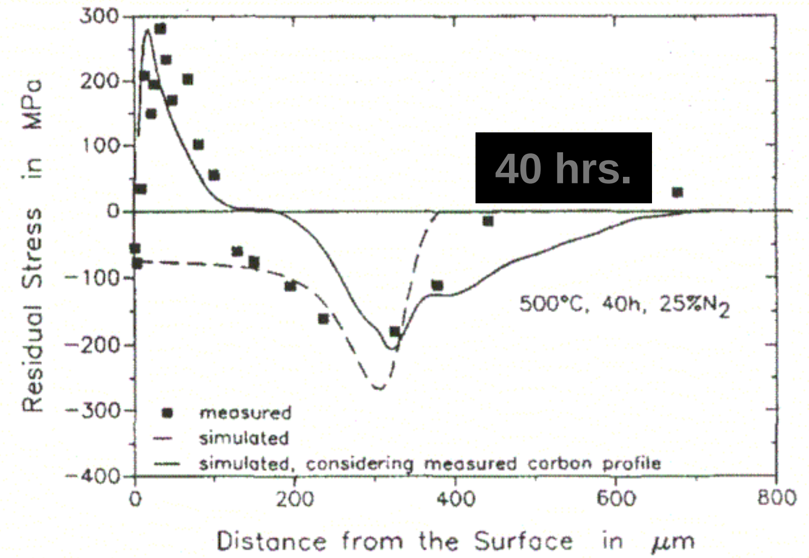
Challenge has been to describe these interactions comprehensively

Spatial resolution to ~0.1 mm for in-situ study of surface engineering processes to be achieved by Bragg Mirror optics (Stoica, et al)

Stress development in nitriding steel



P. Buchhagen and T. Bell, Comp. Mat. Sci. (1996)



Volume misfits & the increasing volume fraction of nitrides cause a compressive residual stress in the surface layer

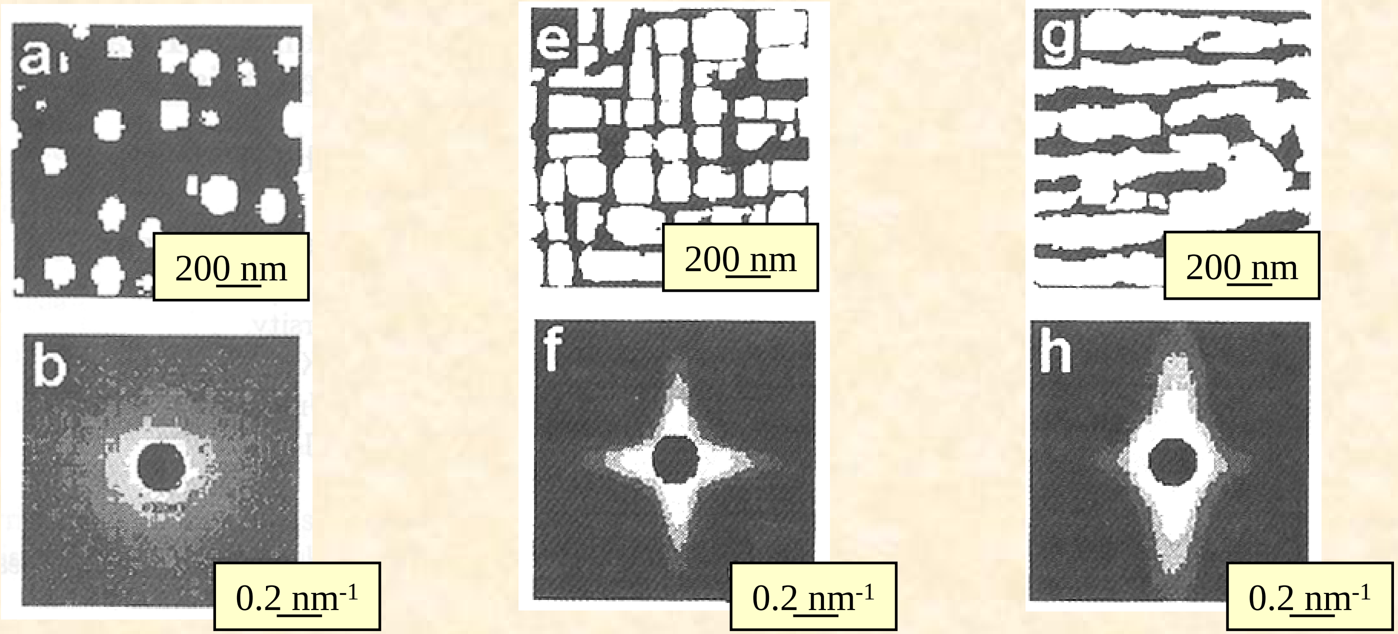
Long nitriding time leads to dissolution of carbides which changes stress in the surface layer from compression to tension

Simultaneous SANS and WAND will elucidate the development of nanostructure during real-time fatigue studies

T=1048 K (0 MPa)
Misfit = 0%

T=1048 K (0 MPa)
Misfit = -0.5 %

T=1048 K (130 MPa)
Misfit = -0.5 %



Lattice misfits can be measured by diffraction

Nanostructure can be mapped by SANS

Study of Ni-Al-Mo alloy, P. Fratzel et al., Solid-Solid Phase Transformations, 1999

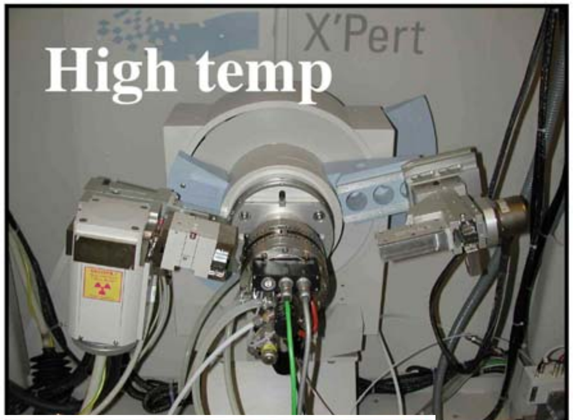
VULCAN will be best-in-class, NRSF2 also excellent

Comparison of best powder diffractometers (source for D20, GEM, DRACULA- Alan Hewat)

	D20 (ILL)	GEM (ISIS)	DRACULAR (ILL)	VULCAN (SNS)	NRSF2 (HFIR)
Flux 10^7 n/s/cm^2	5	0.2	5	12.9	2 - 8 λ dependent
Detector Coverage (ster.)	0.5	3.5	3.0	~3	---
Highest Resolution (%)	0.5	0.4	0.5	0.2	0.3

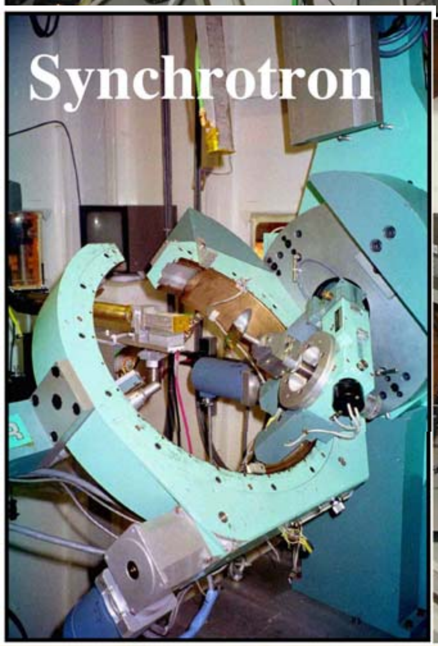
Residual Stress User Center provides unique X-ray, synchrotron, & neutron facilities for stress mapping

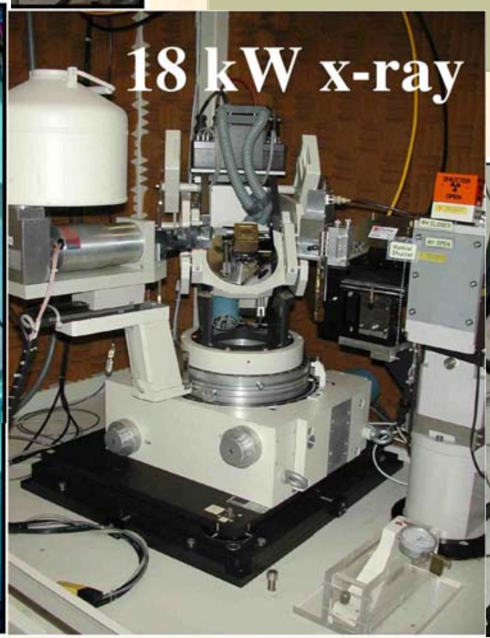
Determining stress at difficult locations in large and small specimens at ambient or at temperature or under load

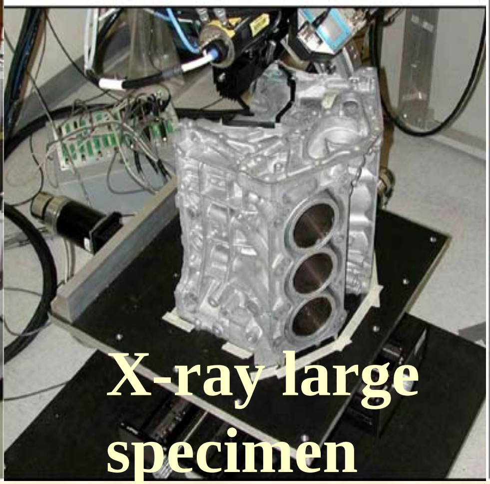


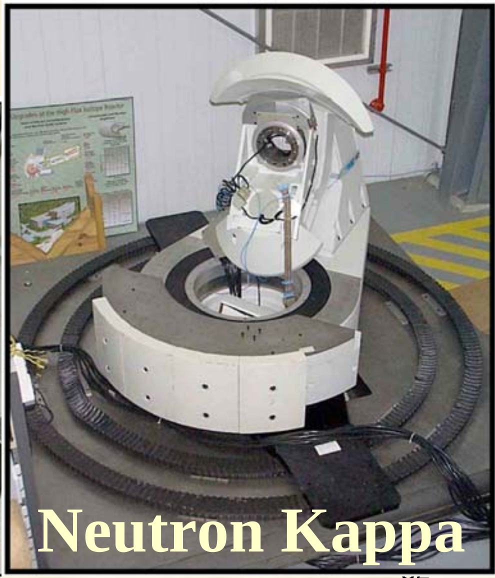
- Large or small specimens
- Flat or curved
- Thin films or bulk
- Ambient or elevated temperature or load

Contact: Camden Hubbard
hubbardcr@ornl.gov
http://html.ornl.gov









Thanks and Remember the Posters Session after the Reception

- The effect of welding process on the residual stress distribution in welded cruciform parts
- Effect of intensive quenching on part residual stress conditions
- Residual strains under torsion using hollow cylinder steel specimens and neutron diffraction
- Changes in lattice strain profiles around a fatigue crack through the retardation period after overloading
- In-situ neutron measurement and modeling of phase stress evolution during deformation and fracture of Al matrix composites
- Study of granular materials in compression using NRSF2
- Incoherent neutron scattering measurements of hydrogen-charged Zircalloy-4
- Comparison between in-situ time-resolved neutron diffraction measurements based on quasi-steady state phenomenon and direct real-time experiment

Engineering

4:00–4:30 **Status of Commissioning NRSF2 at HFIR and Construction of VULCAN at SNS**
Camden R. Hubbard and Xun-Li Wang
Oak Ridge National Laboratory, Oak Ridge, TN

4:30-5:00 **In Situ Neutron Diffraction Studies of Mesoscopic Deformation Behavior of Structural Alloys**
Prof. Hahn Choo
Department of Materials Science and Engineering
University of Tennessee, Knoxville, TN

5:00-5:30 **Application of In-situ Neutron Diffraction Tools Towards Fundamental Understanding of Material Behavior During Thermo-Mechanical Processing**
Dr. Suresh Babu
Edison Welding Institute, Columbus, Ohio

5:30-6:00 **Prospects for Neutron Tomography and High-Speed Radiography: Complex Structure Imaging**
Prof. Les Butler
Department of Chemistry, Louisiana State University