



NRC-CNRC

From **Discovery**
to **Innovation...**

Engineering and Industrial Applications

Neutron scattering at Chalk River Laboratories

John Root
NRC - Canadian Neutron Beam Centre

ASM International Educational Symposium

Neutrons for Materials Science and Engineering Oak Ridge, April 18, 2007



National Research
Council Canada

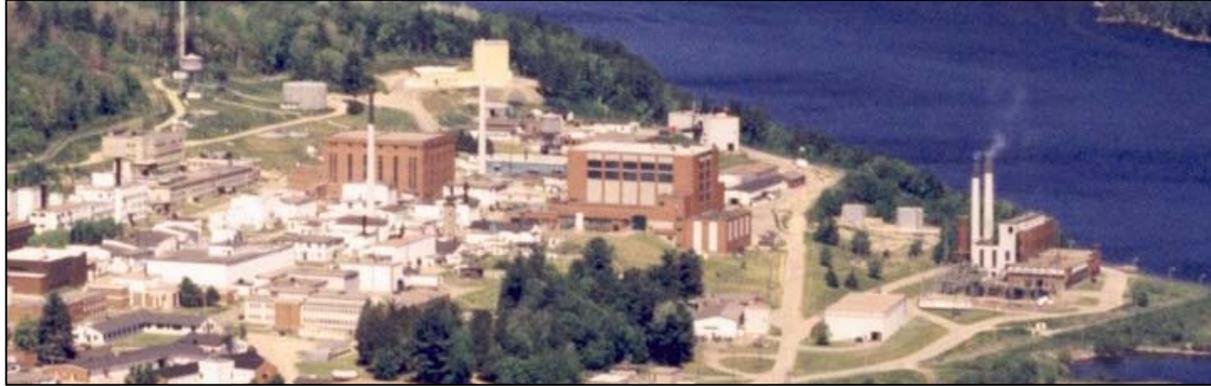
Conseil national
de recherches Canada

Canada

NRC-CNRC

From *Discovery*
to *Innovation...*

Innovation system



Government laboratories



Universities



Industries



Applied Neutron Diffraction for Industry

ANDI

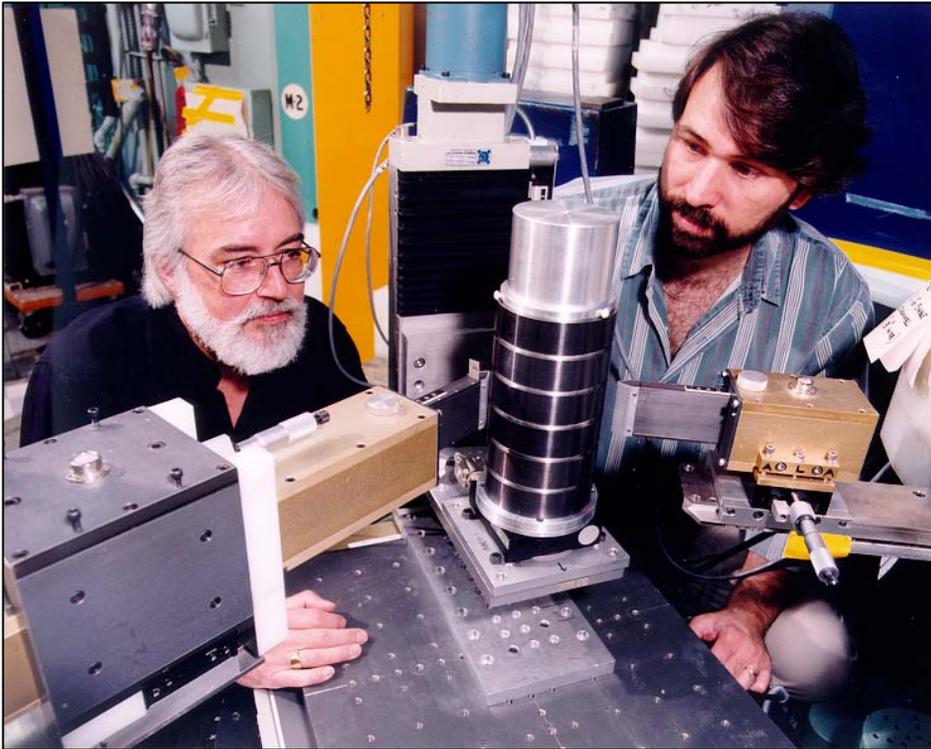
- Beginning in mid-1980's, the Chalk River neutron beam group (government laboratory) undertook to apply neutron scattering methods to problems of industry.
- First demonstrations of residual stress mapping [1,2] and intergranular stress effects [3] in engineering materials and components

Nuclear sector

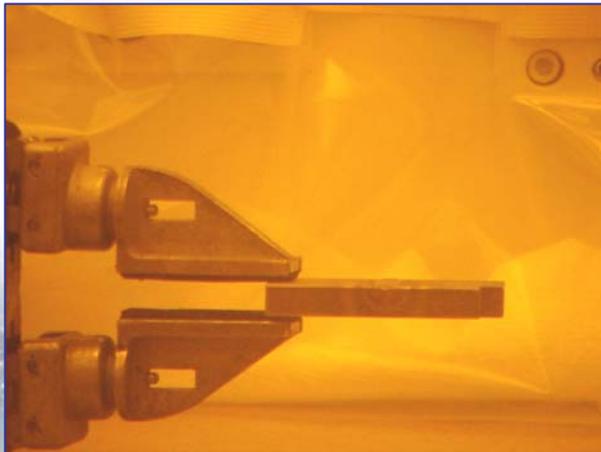
Qualifying a new supplier

Would we be compromising reliability of our product if we switch to components made by a different supplier?

- Homogeneity
- Crystallographic texture
- Residual stresses
- Minority phases



Neutron diffraction surveys bulk material rapidly and gives detailed crystallographic information vs. circumferential position in six specimens.



Handling an active specimen



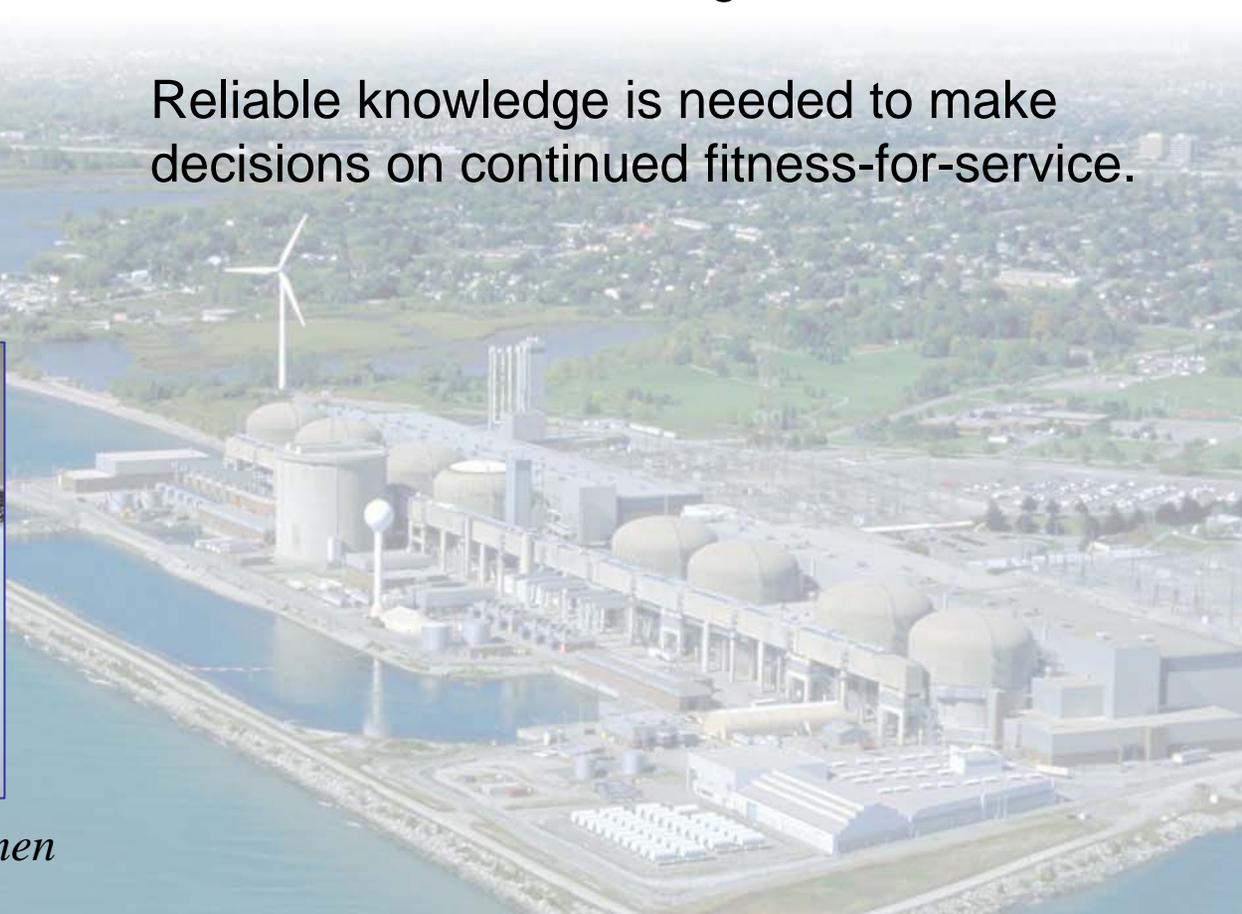
Stress-mapping in 200Sv/h specimen

Nuclear sector

Plant life extension

After fast-neutron irradiation, weld stresses may be relieved (?), so the probability of stress-corrosion cracking is lower...

Reliable knowledge is needed to make decisions on continued fitness-for-service.



Oil & Gas sector

Avoiding plant outages



If this heat exchange tubing leaks, I will have to shut down my oil refinery. We'll lose \$100Ks per day, so I better relieve the residual stresses left over from making this twisted tube.... but is my stress-relieving heat treatment really doing any good?

Hey, maybe the high operating temperature of the plant is enough to relieve stresses anyway!

Neutron mapping of residual stress around the “circumference” of this twisted tube can be made before and after heat treatment to evaluate how big are the initial stresses, where they are biggest, and whether or not a heat treatment actually relieves the stresses.

Oil & Gas sector

Cost, reliability, safety

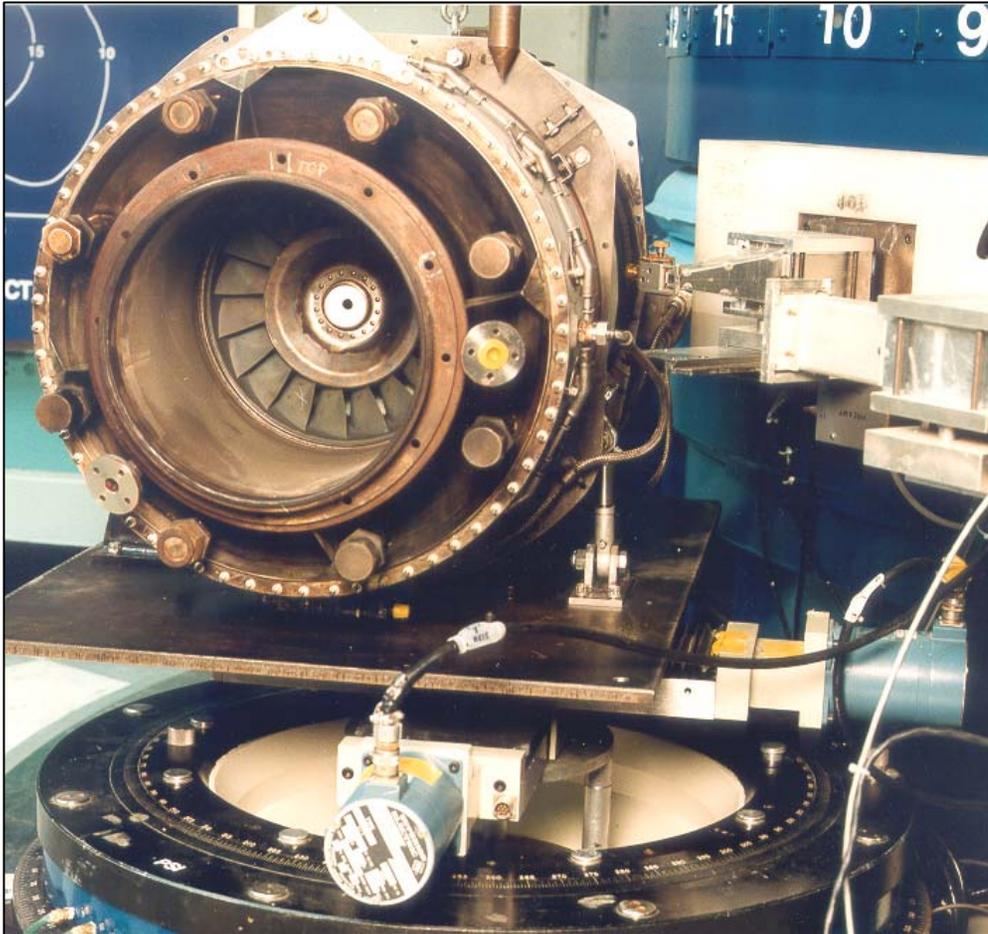


If we join gas pipelines together with a sleeve junction (2 welds instead of 1 butt weld), would we be just amplifying the probability of a rupture, gas leak or explosion due to stress-related effects near the weld? How bad are those stresses anyway? Should sleeve welds be a new standard practice?

Neutron stress mapping characterizes the welds in this 1000 lb “specimen” of gas pipeline.

Aerospace sector

Knowledge-intensive, regulated industry



Failure of components can lead to loss of life.

Validating computer models of operating conditions.

Evaluating real components:

- Engines, discs, blades
- Landing gear
- Wing structures
- Rivets, welds
- Skins

Ref. [4]

Manufacturing sector

Opening new markets

Light-weight pressure cylinder for natural gas.

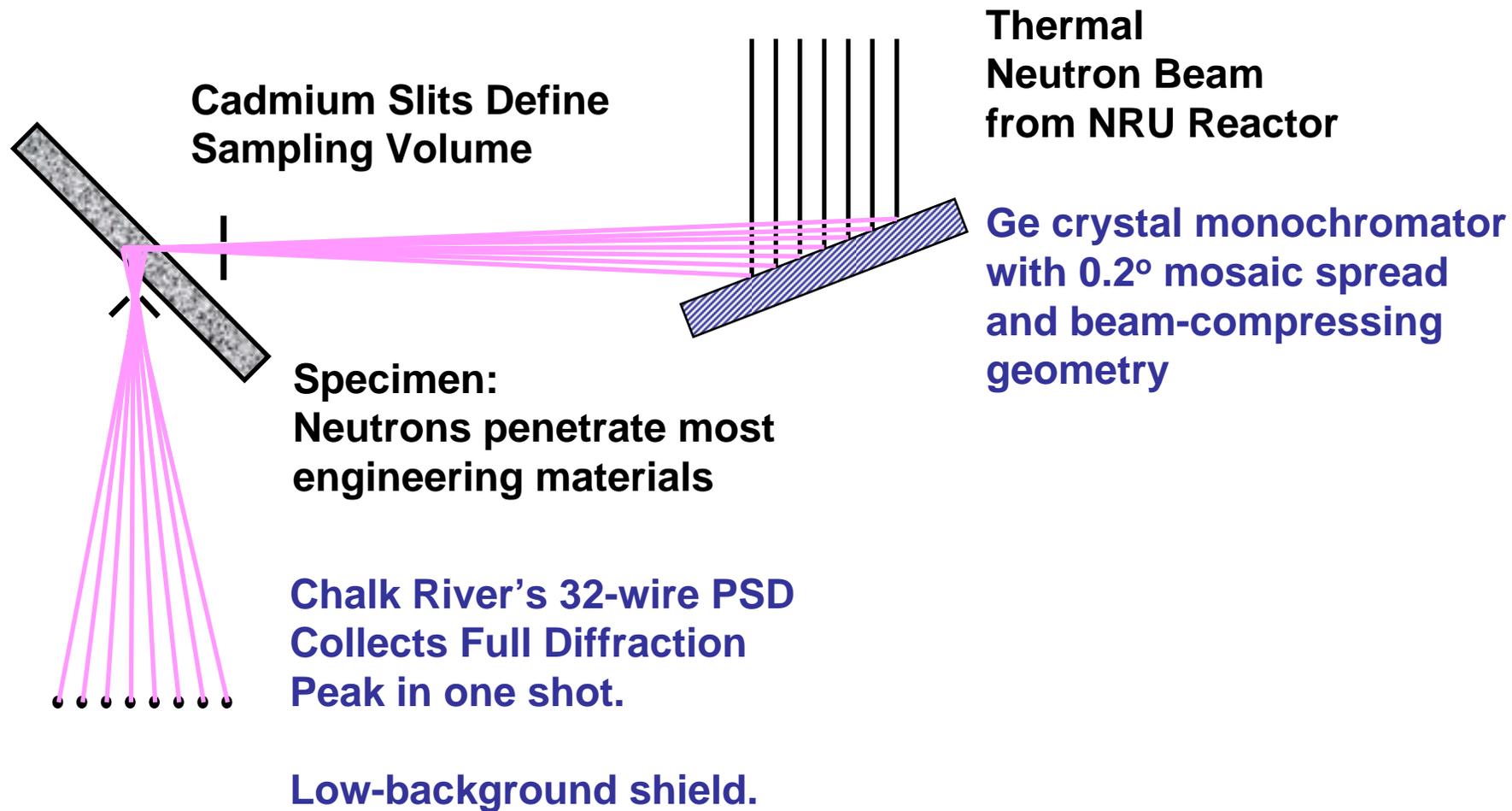


Tougher bolts
(TRIP steel)



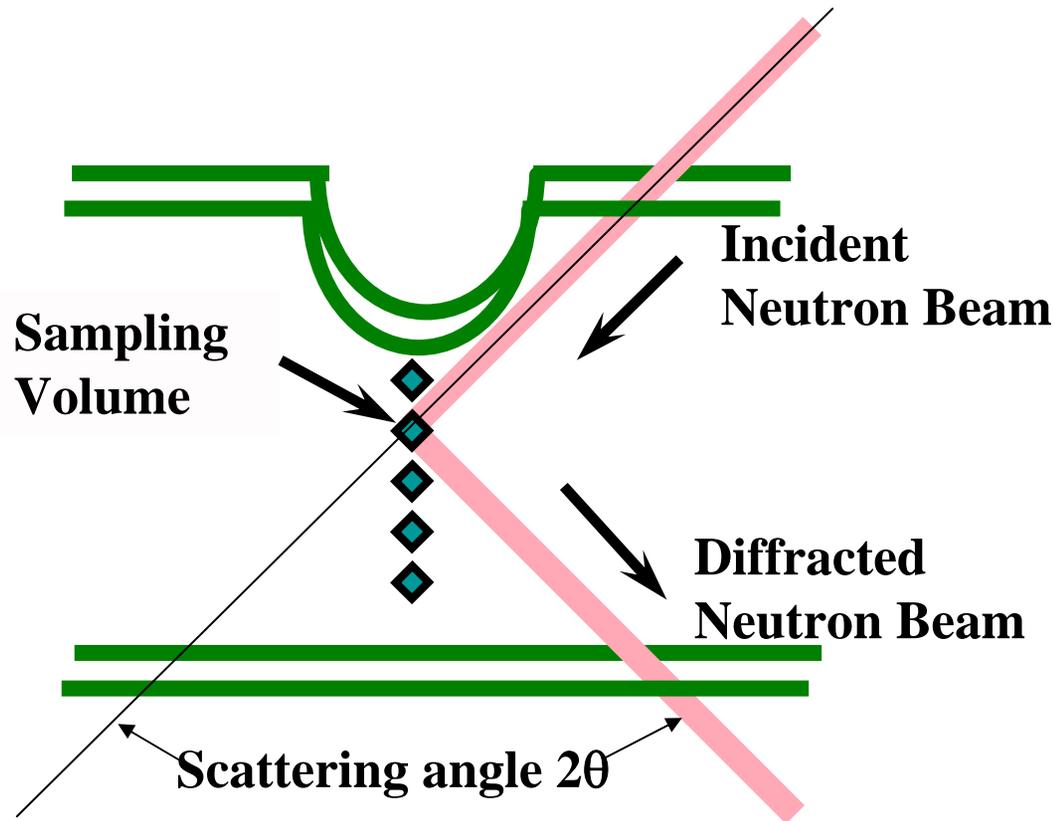
“Based on this evidence, the Steel Structures Subcommittee of the Canadian Standards Association recommended temper-levelled plate be approved for use.”

Diffraction-based scanning



Non-destructive scans

Sampling volume / pixel



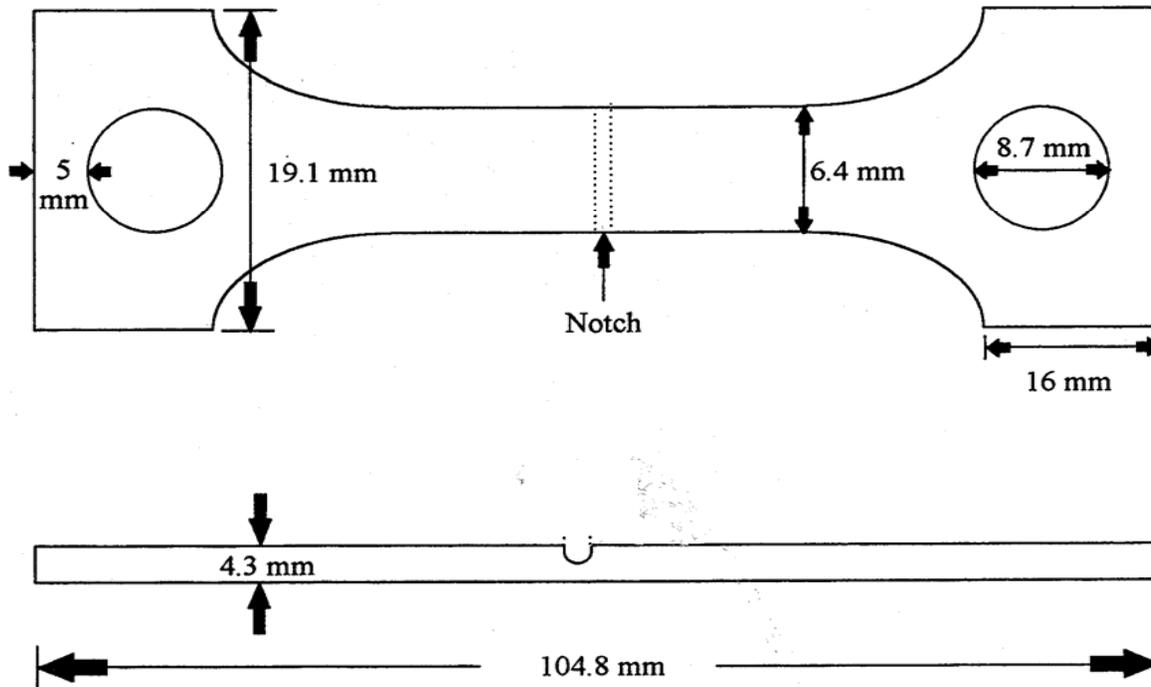
Spatial resolution $\sim 1 \text{ mm}^3$
is the intensity limit at a
medium-flux neutron
source, for untextured,
fine-grained material.

Time-averaged
monochromatic flux at
specimen $\sim 2 \times 10^6 / \text{cm}^2/\text{s}$

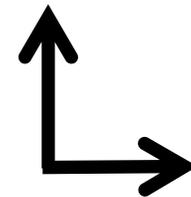
Crystal lattice strain
 $\sin(\theta_{\text{ref}}) / \sin(\theta) - 1$

Fundamental knowledge To inform fitness-for-service guidelines

Investigating possible stress-relaxation near a fretting flaw in a Zr-2.5Nb pressure tube. Aspects suitable for publication. Costs shared.



Tube axis

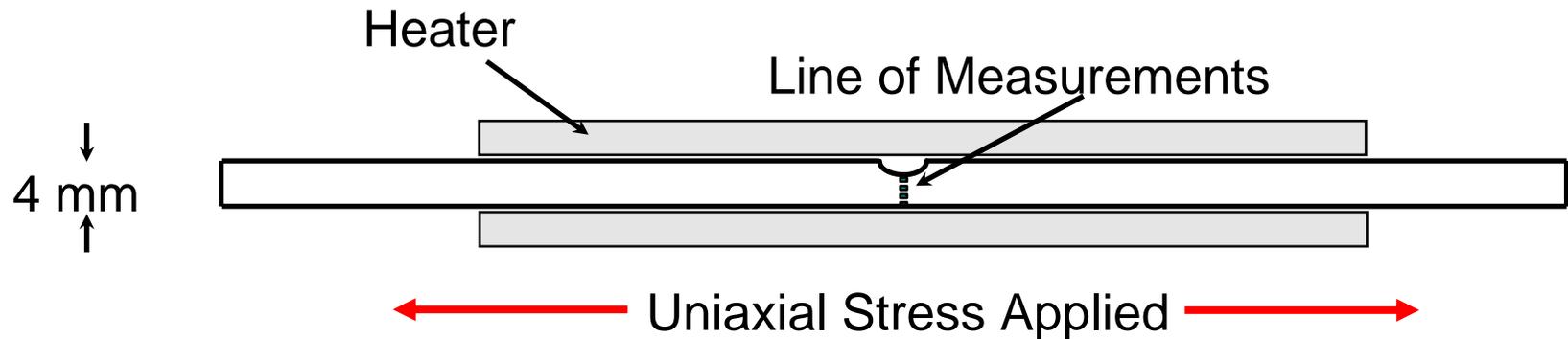


Tube circumference

“Longitudinal” direction
of tensile specimen

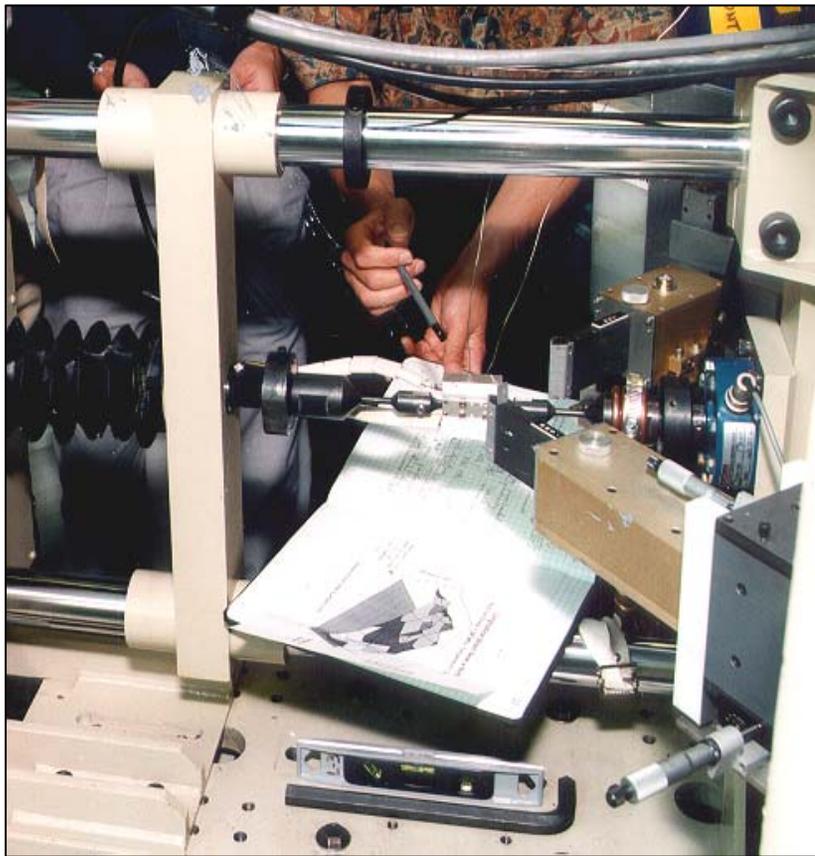
Representative conditions

Applied load, temperature, flaw



- Material = Zr-2.5Nb, width 6 mm.
- Temperature = 250 °C.
- Semicircular groove, radius 1 mm.
- Sampling volume 0.5 x 0.5 x 5 mm³.

Challenge



How it really looks

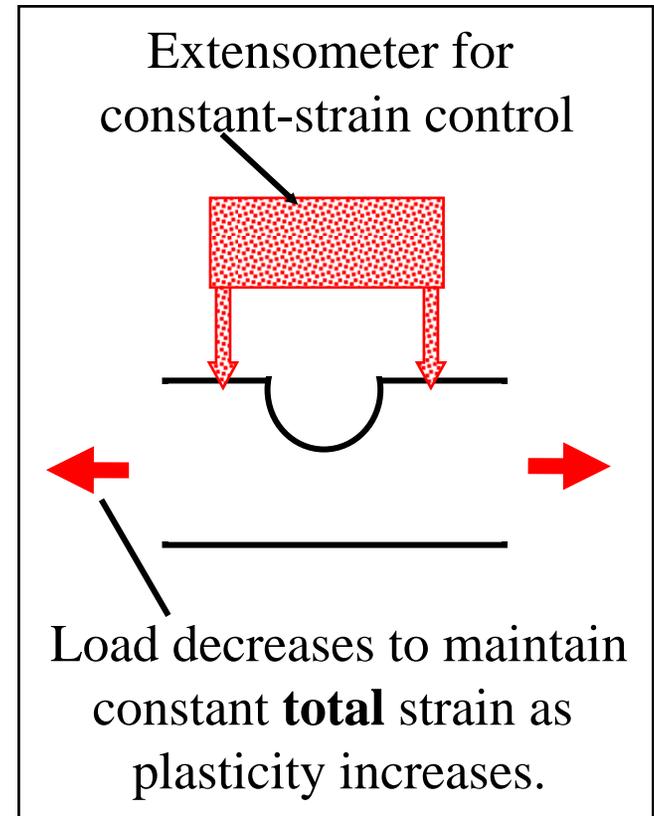
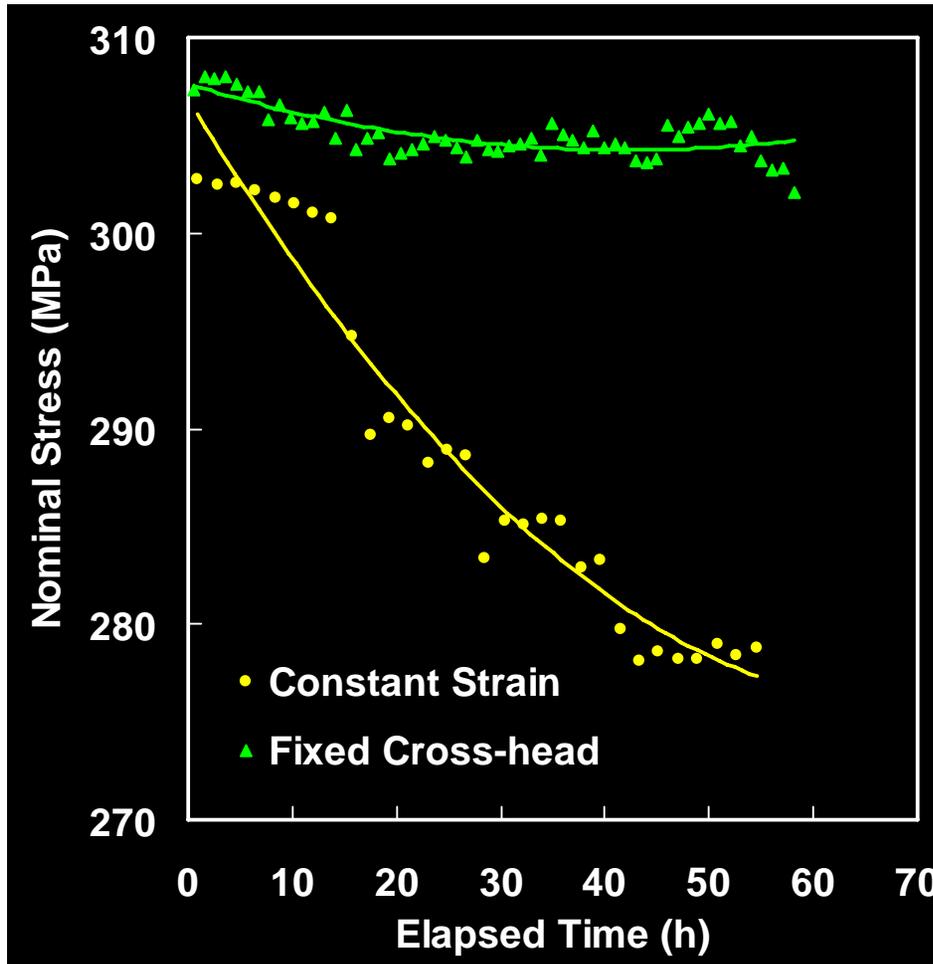
Load frame is screw-driven, has a max. load of 50 kN and operates in various modes:

- Fixed cross-head
- Fixed load (active loop)
- Fixed strain (active loop)

Temperature is monitored by a thermocouple imbedded in sample, and set by cartridge heaters.

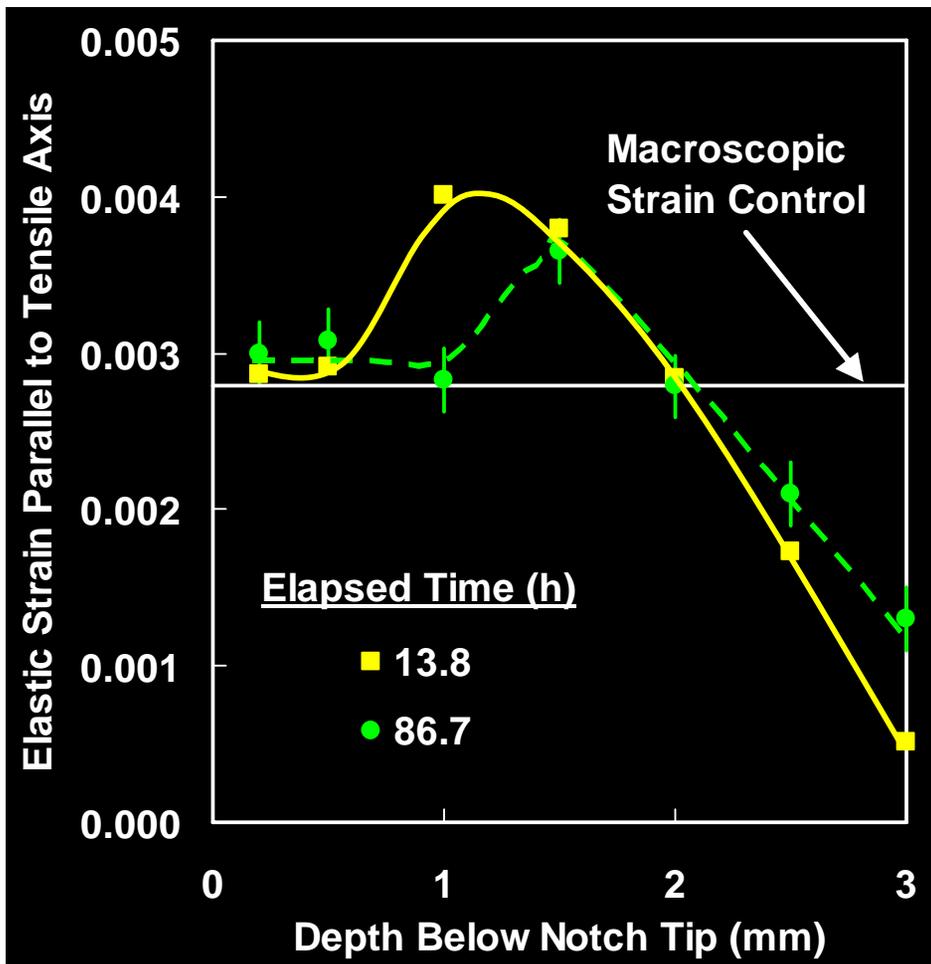
Test machine mounted on XY translation system.

Constant total strain Relaxation by plastic deformation



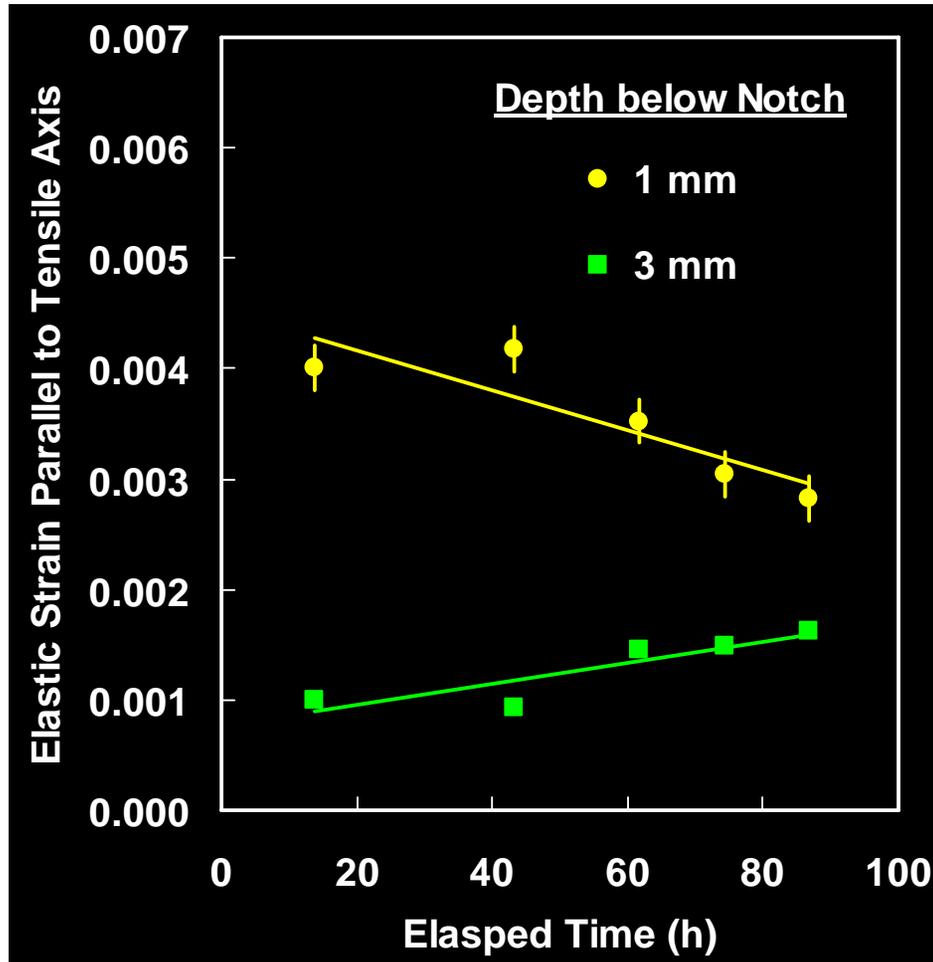
Relaxation of elastic strain

Applied load, temperature, flaw



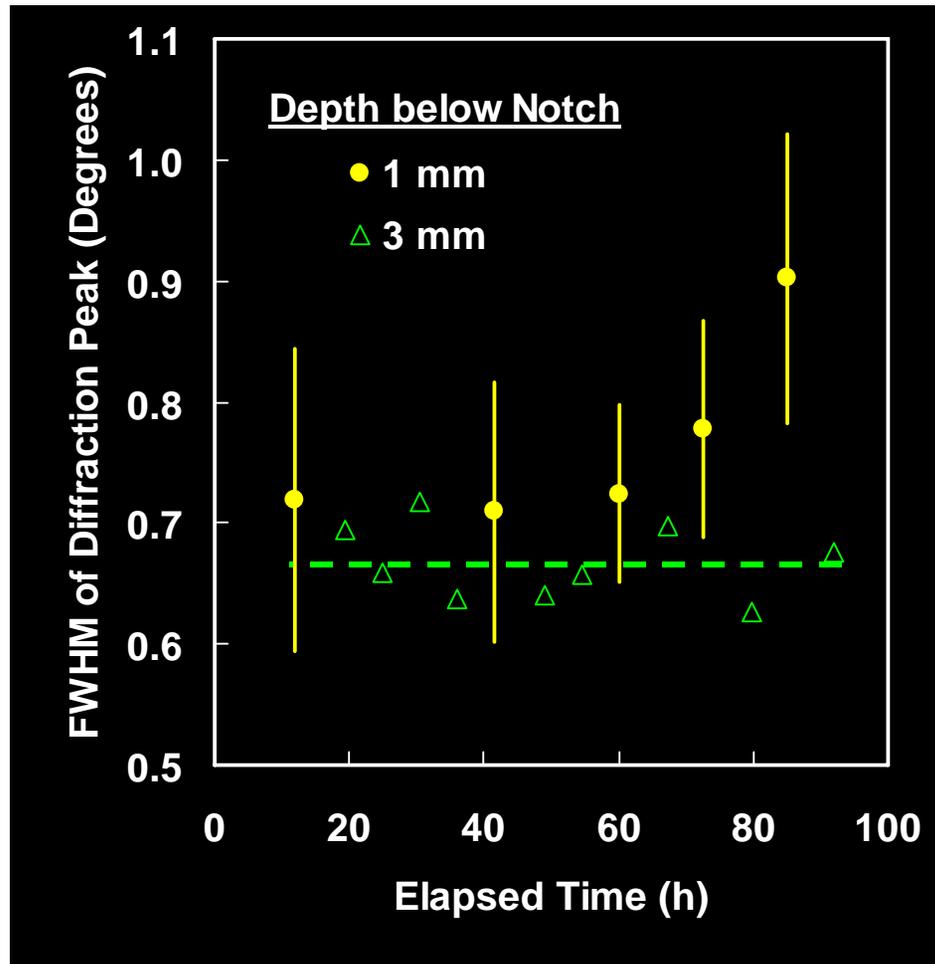
- 20 min. per strain reading
- Spatial resolution suitable to characterize strain gradients
- Measuring (0002) peak and the transverse (hoop) strain component boosts intensity via crystallographic texture.
- The “action” seems to be happening close to the notch...

Evolution with Time



Since the **total** (macroscopic) strain is constant, a reduced **elastic** strain corresponds to an increase in **plastic** strain near the notch.

Indication of plasticity



Plasticity generates:

- Intergranular stresses
- Crystal lattice defects

Both phenomena cause the diffraction peak width (FWHM) to increase.

Results on stress near notch

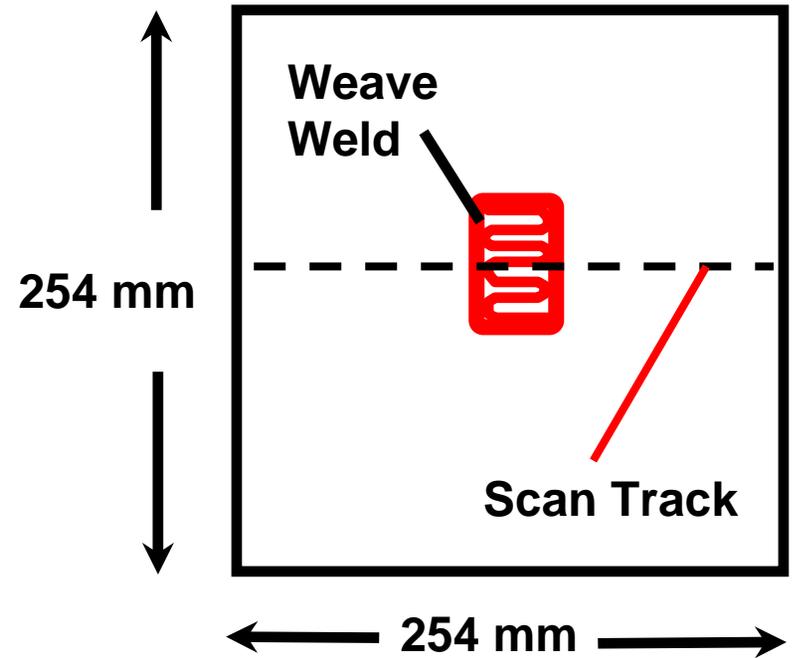
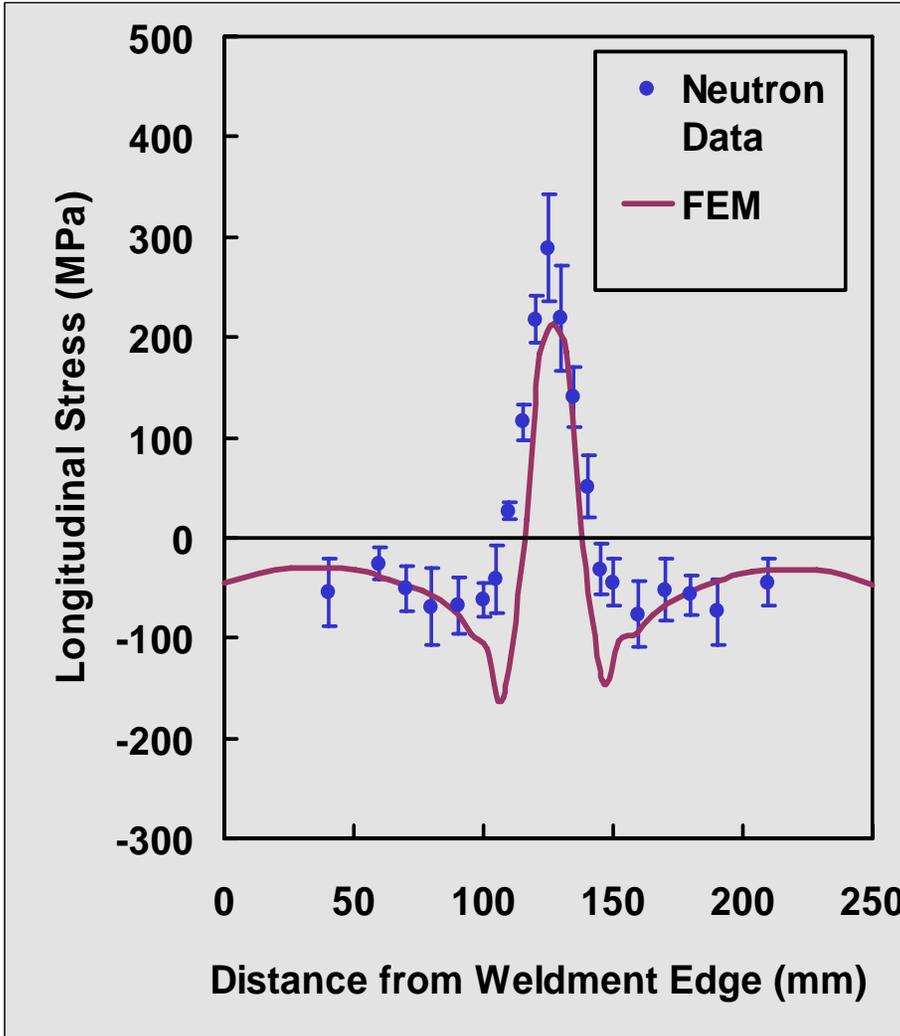
- It may be feasible, with neutron diffraction, to study the growth of the plastic zone near a stress-concentrating defect, as a function of time at conditions of importance for pressurized systems.
- Knowledge may, in future, aid with dispositioning of flaws that appear during operation, and affect fitness-for-service decisions.

Ref. [5]

Partially supported by CANDU Owners Group,
WPIR 3214, Working Party 32.

Welding

Ubiquitous in industry – rich in challenges

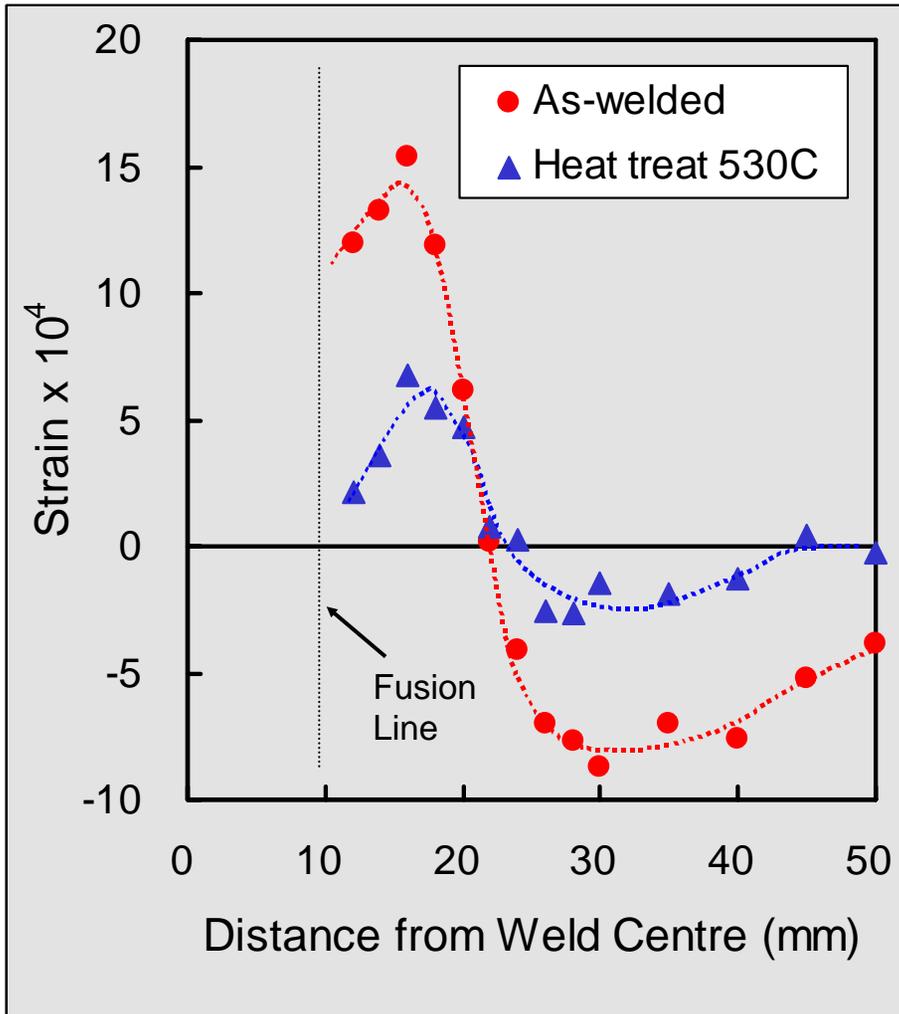


Modelling, practical worries about stress-corrosion cracking.

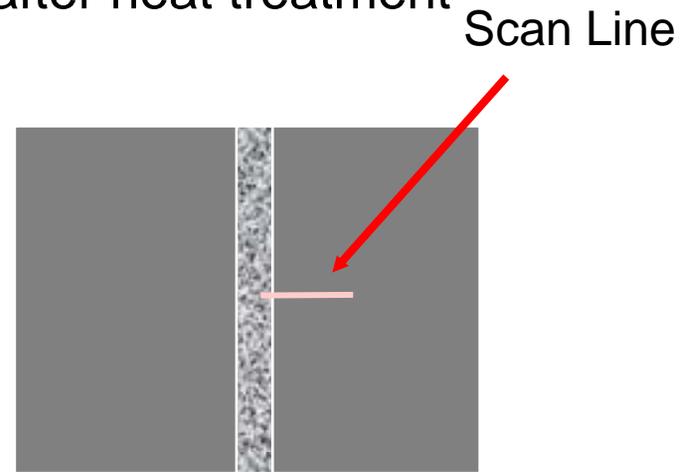
Ref. [6]

Evaluating Stress-Relief

Is treatment effective? **YES**



Measurement of strains parallel to a weld, before and after heat treatment

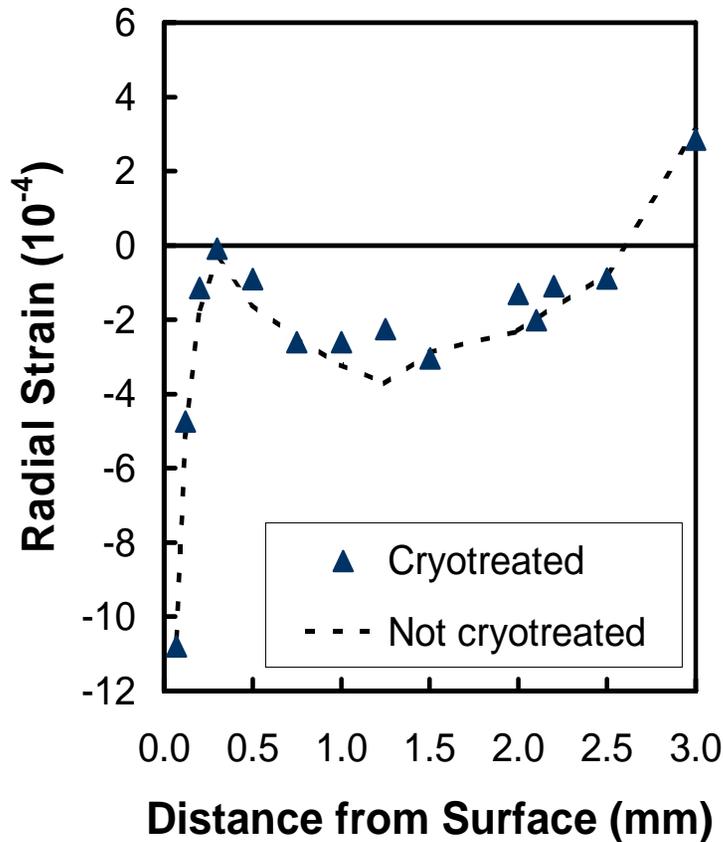
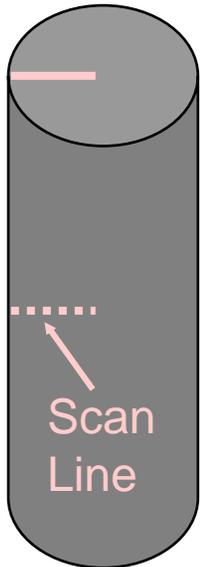


Heat treatment at 530 °C for 1 h reduces residual strain by 60%

Ref. [7,8]

Evaluating Stress-Relief

Is treatment effective? **NO**

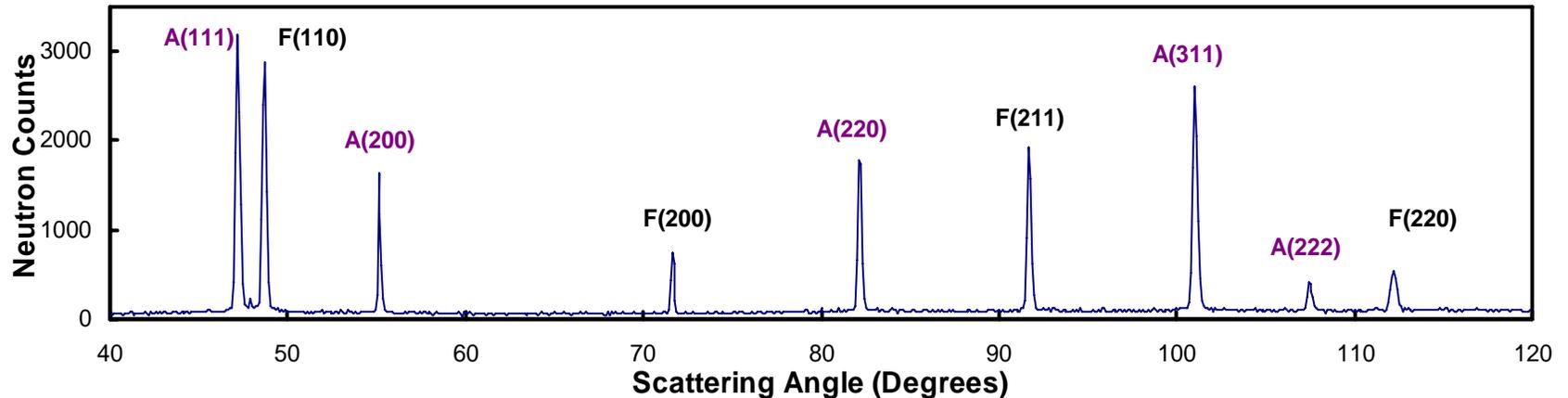


Steel rod contains residual stresses that vary sharply near the surface.

Treatment with cryogenic temperatures was claimed to relieve the stresses, but the measured strain distributions are identical within the precision of strain measurement.

Other Diffraction Apps.

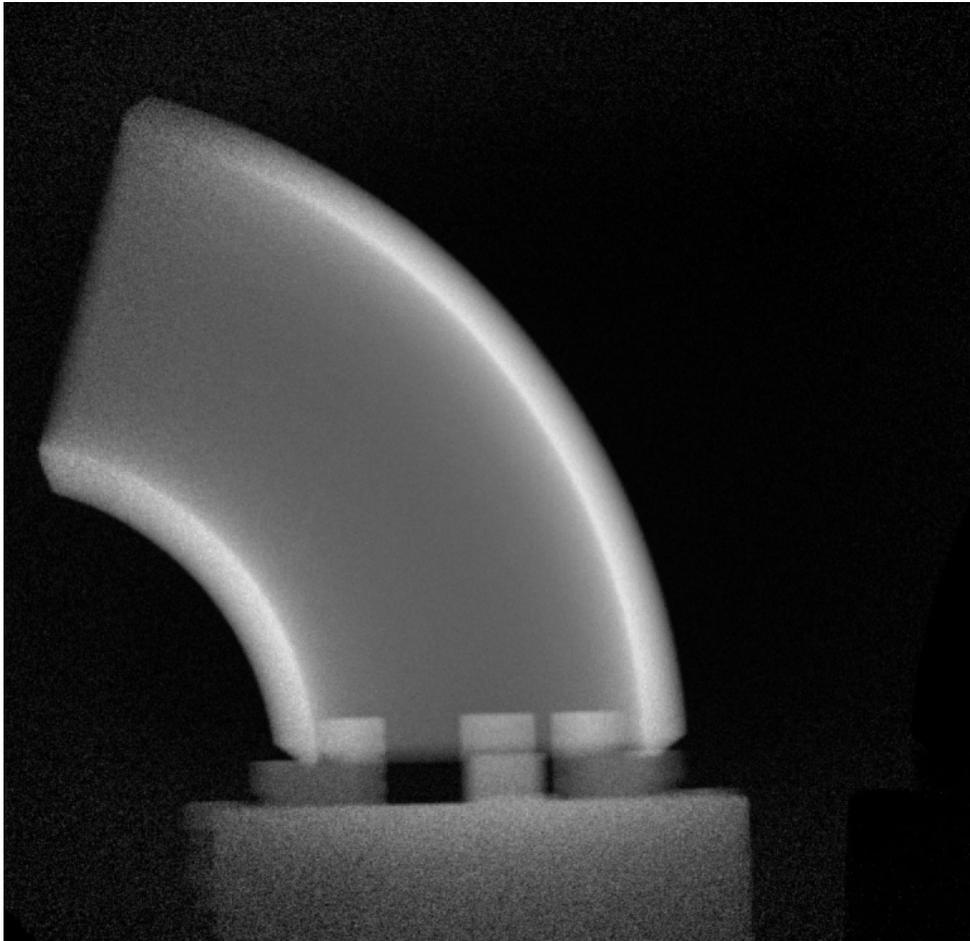
Mixed austenite - ferrite



- Phase analysis by powder diffraction
- Texture (preferred orientations of crystallites)
- Grain size analysis
- Analysis of material homogeneity
- Intergranular / interphase stress analysis
- Non-invasive thermometry
- Kinetics of precipitation / dissolution
- Lifetime assessment from peak broadening (damage)
- ...

Tomography

Combining many radiographs



Radiographs collapse absorption information onto a single plane.

By combining many radiographs, with different orientations, one can obtain three-dimensional images of internal structures.

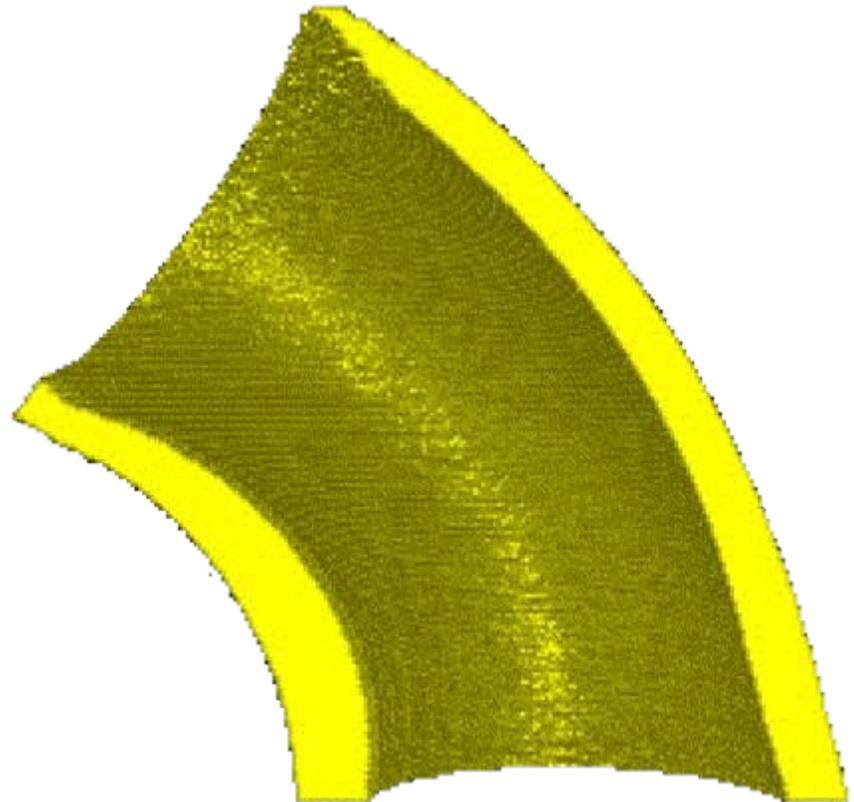
Rogge, Huang, using neutron-sensitive Mar345 image plate detector

Neutron Tomography

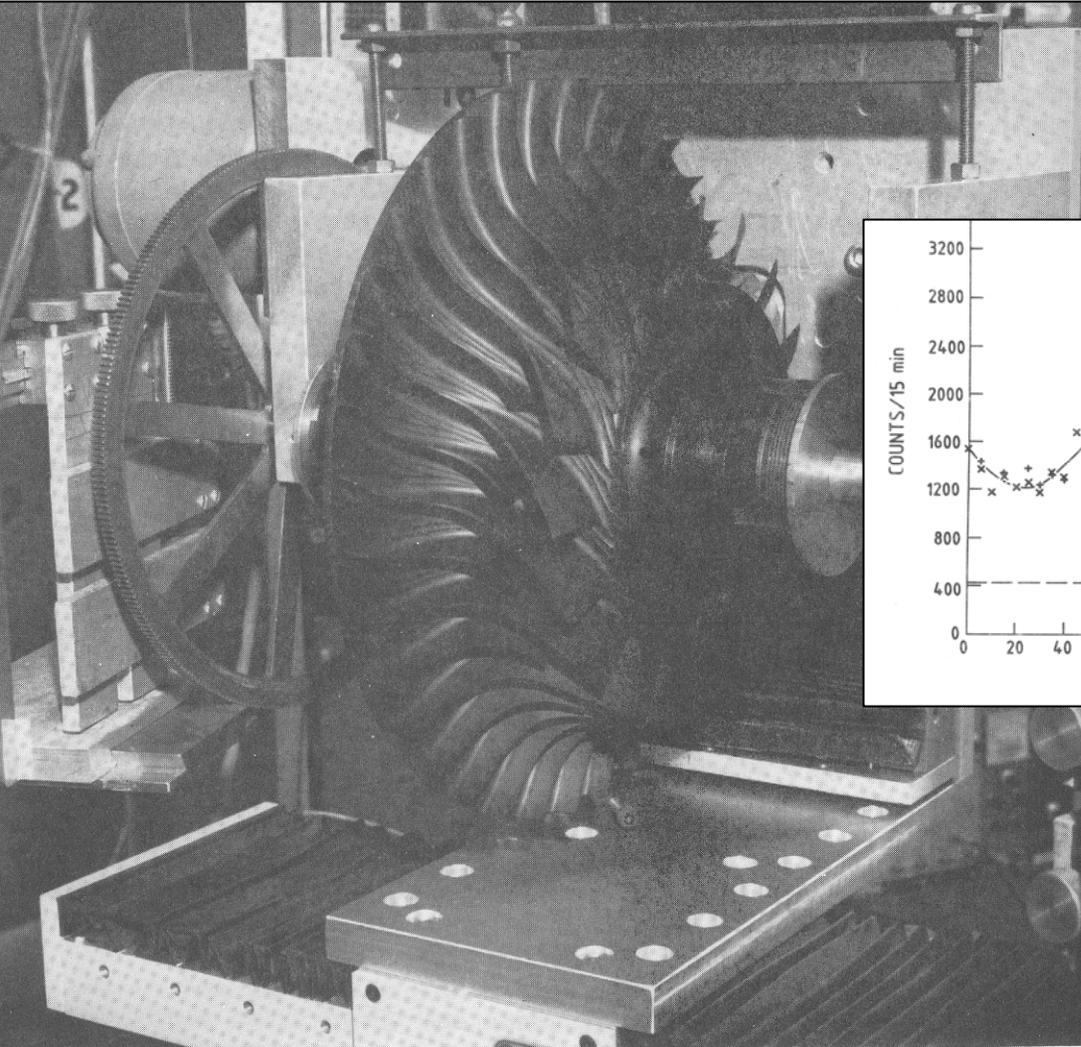
Distortion due to bending a pipe

After the radiographs have been recorded numerically, the underlying three-dimensional structure can be reconstructed and slices made to extract features of interest.

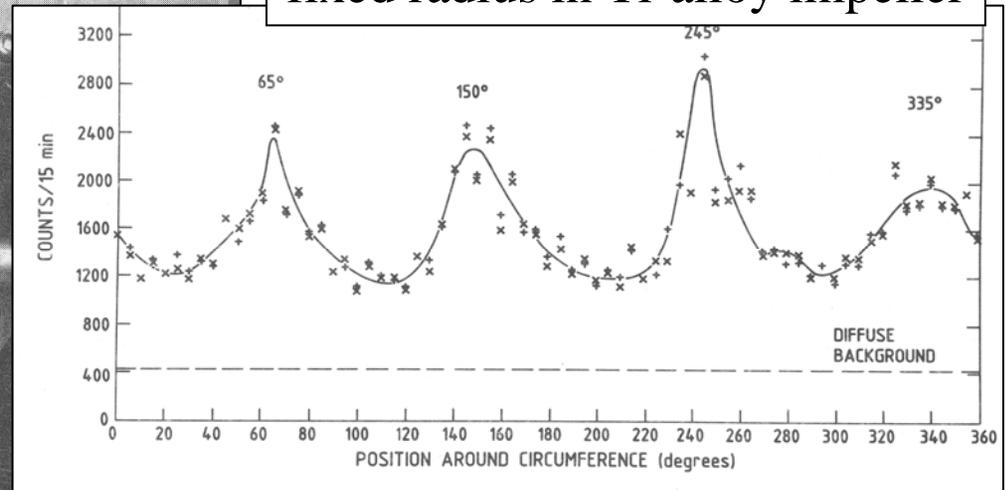
Rogge, Huang



Homogeneity



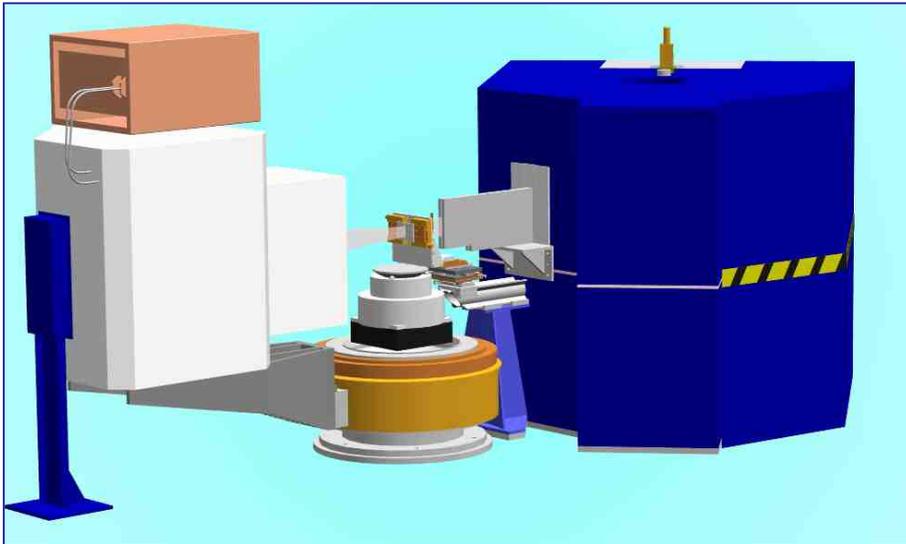
Neutron (0002) intensity vs. circumferential position at fixed radius in Ti-alloy impeller



Forging process of original billet, resulted in 4-fold symmetry of enhanced crystallite orientations in finished component.

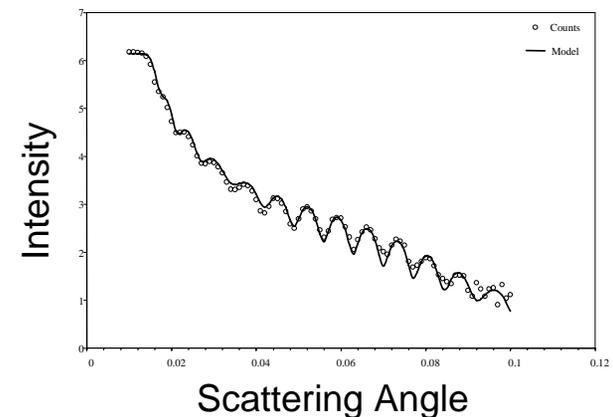
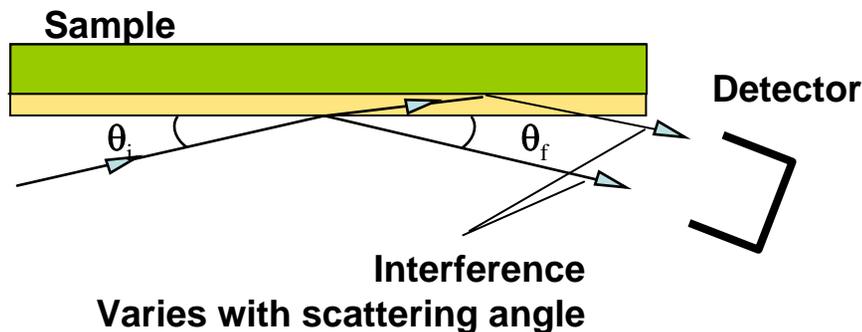
Neutron Reflectometry

...to characterize thin films, interfaces

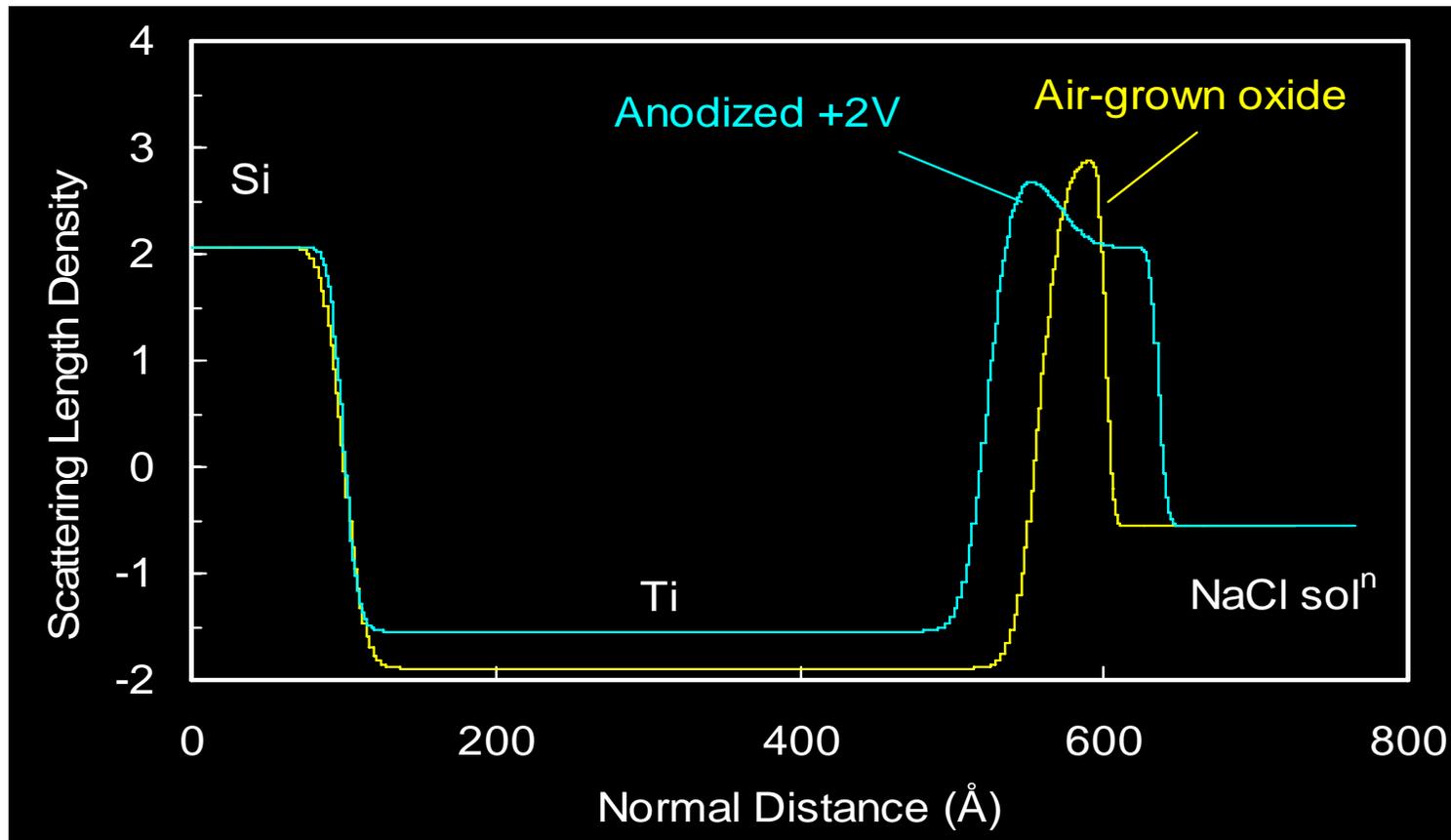


A new beam line at Chalk River. Project led by UWO, supported by 12 universities, and managed by NRC.

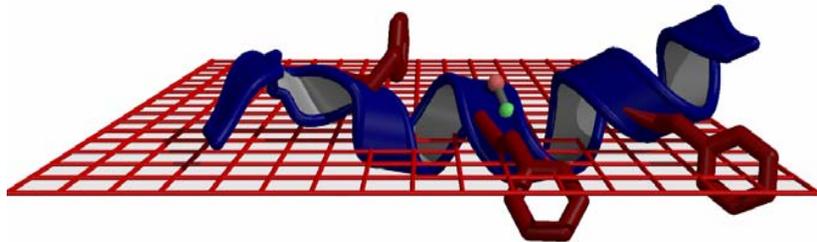
- polymer coatings
- electroplating
- hydrogen storage media
- information storage media
- biomimetic membranes



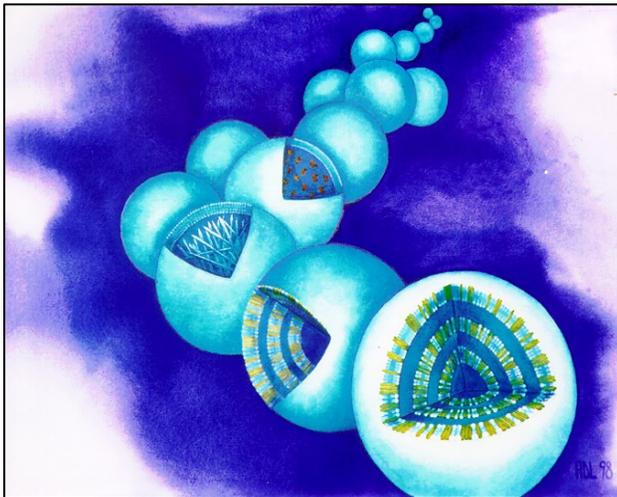
How oxides grow ...on a thin film of titanium



Future applications for soft materials, nanostructures



Ref. [10]



Life-science industries:

- SIV viral fusion peptide on membrane
- Cholesterol in membranes
- Unilamellar / multilamellar Vesicles
- Lipopolysaccharides, antibiotic-resistant bacteria
- Polymer coatings for medical implants

Communications, information industries:

- Organic electronics
- Magnetic multilayers: spintronics

Advanced materials industries:

- Carbon nanotubes
- Mesoporous media

References

1. A.J. Allen, M.T. Hutchings, C.G. Windsor and C. Andreani, *Adv. in Physics* 34 (1985), 445.
2. T.M. Holden, R.A. Holt, G. Dolling, B.M. Powell and J.E. Winegar, *Met. Trans.* 19A (1988) 2207.
3. S.R. MacEwen, C. Tome and J. Faber, *Acta. metall.* 37 (1989), 979.
4. J.H. Root, T.M. Holden, D.C. Tennant and D. Leggett, “Temperature: Its Measurement and Control in Science and Industry”, J.F. Schooley ed., *AIP* 6 (1992) 1231.
5. J.H. Root, J. Katsaras, J.F. Porter and B.W. Leitch, *J. Press. Vess. Tech.* 124 (2002), 366.
6. A.S. Oddy, J.M.J. McDill, J.E.M. Braid, J.H. Root and F. Marsiglio, *Proc. 5th Int. Conf. On Trends in Welding Research* (1999), 931, ASM International.
7. C.E. Coleman, G.L. Doubt, R.W.L. Fong, J.H. Root, J.W. Bowden, S. Sagat and R.T. Webster, *ASTM STP* 1245 (1994), 264.
8. J.H. Root, C.E. Coleman, J.W. Bowden, M. Hayashi, *J. Press. Vess. Tech.* 119 (1997), 137.
9. Z. Tun, J.J. Noël, and D.W. Shoesmith, *J. of Electrochem. Soc.*, 146 (1999), 988.
10. J.P. Bradshaw, M.J.M. Darkes, T.A. Harroun, J. Katsaras and R.M. Epand, *Biochemistry* 39 (2000) 6581.

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