Applications of neutron diffraction to engineering problems

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Reference material

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- "Neutron Data Booklet" Eds. A. –J. Dianoux and G.H. Lander (Institut Laue Langevin: Grenoble) 2003
- "Neutrons and Synchrotron Radiation in Engineering Materials". Proc. of the First Hamburg Fall School, eds.A. Schreyer, H. Clemens, W. Riemers and A.R. Pyzalla, 2008.

Concept

The interplanar spacing, d_{hkl} , constitutes an intrinsic strain gauge for the material with the help of Bragg's law

$$\lambda=2d_{hkl}\sin\theta_{hkl}$$

The Miller indices (*hkl*) describe the atomic planes, λ is the neutron wavelength and $2\theta_{hkl}$ is the angle of diffraction through which the neutrons are turned

An accuracy of $\pm 0.01^\circ$ in 2θ at 90° leads to a precision in strain

$$\Delta d/d=\cot\theta d\theta =1\times 50\times 10^{-4}/57=1\times 10^{-4}$$

The key factor is the high penetration of neutrons through most industrial materials (8% through 25mm steel). This means you can get stress at depth.

Calculation of the elastic strain in a given direction from the lattice spacing

$$\varepsilon^{hkl} = (d^{hkl} - d^{hkl}_0) / d^{hkl}_0$$

- Here d^{hkl}_0 is the the interplanar spacing of the crystal lattice for {hkl} planes in the absence of a macroscopic stress and d^{hkl} is the spacing of the intact sample. This is the step where one can make serious systematic errors
- Remember that diffraction does not measure the plastic strain only the elastic strain!

Strain tensor to stress tensor as an analog of Hooke's law

General form

$$\sigma_{ij} = \frac{E^{hkl}}{(1+\nu^{hkl})}\{\varepsilon_{ij}^{hkl} + \frac{\nu^{hkl}}{1-2\nu^{hkl}}(\varepsilon_{11}^{hkl} + \varepsilon_{22}^{hkl} + \varepsilon_{33}^{hkl})\}$$

writing this out for the 11 and 12 coordinates

$$\sigma_{11} = \frac{E^{hkl}}{(1+\nu^{hkl})(1-2\nu^{hkl})}\{\varepsilon_{11}^{hkl}(1-\nu^{hkl}) + \varepsilon_{22}^{hkl}\nu^{hkl} + \varepsilon_{33}^{hkl}\nu^{hkl}\}$$

$$\sigma_{12} = \frac{E^{hkl}}{1+\nu^{hkl}}\varepsilon_{12}^{hkl}$$

The E^{hkl} and ν^{hkl} are "diffraction elastic constants". They are linear calibration constants which relate the macroscopic stress in the sample to the lattice strains for a given crystallographic [*hkl*] direction. The coordinate set is quite arbitrary. The thought process is that we 'stick' a coordinate system onto the sample and then work everything out in terms of that coordinate system.

Examples of the economic impact of residual stresses

- Over-rolled CANDU pressure tube. Power reactor shut-downs and loss of revenue
- Stress corrosion cracking in bent steam generator tubing. This reduces the efficiency of the steam generator since cracked tubes have to be blocked off.
- Welds in 1960’s vintage nuclear power stations. These have run for 40-50 years and there are 168 of them in the USA. Can the licensing be extended to 60 years? A major problem is stress corrosion cracking in these welds, but what are the stresses in these old manual welds? Replacement costs are gigantic!
- Weld modeling to improve quality and therefore lifetime. We ought to be able to calculate weld stresses from the input parameters. But, the models have to be benchmarked against experiment otherwise it is “garbage in = garbage out”

Topics of high impact from the "Residual Stress Summit 2010"

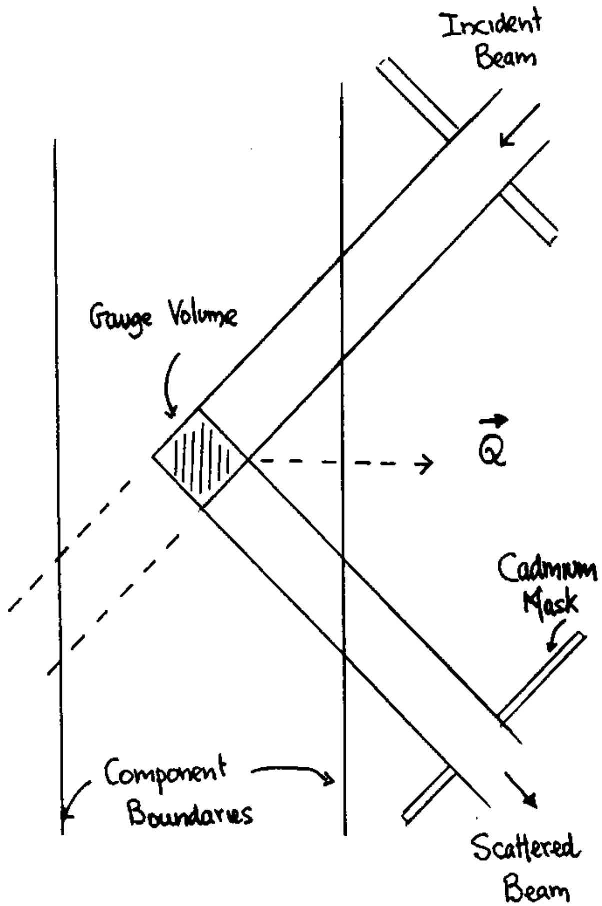
- Welding residual stress measurement
  - Mitigation of PWSCC – Currently dissimilar metal welds
  - Validation of numerical analyses
  - The uncertainty in measurement and experiment

- Material Characterization of Irradiated Materials
  - Reactor pressure vessel material
  - Effect of embrittlement in the material in the presence of stress

- Stresses in forged aero-engine components such as turbine discs that are subsequently machined to size. If the part distorts during machining due the stresses it must be scrapped.

Pressurizer nozzle 7.5in diam and 1.2in.thick.
Slide courtesy of D.L. Rudland (USNRC)

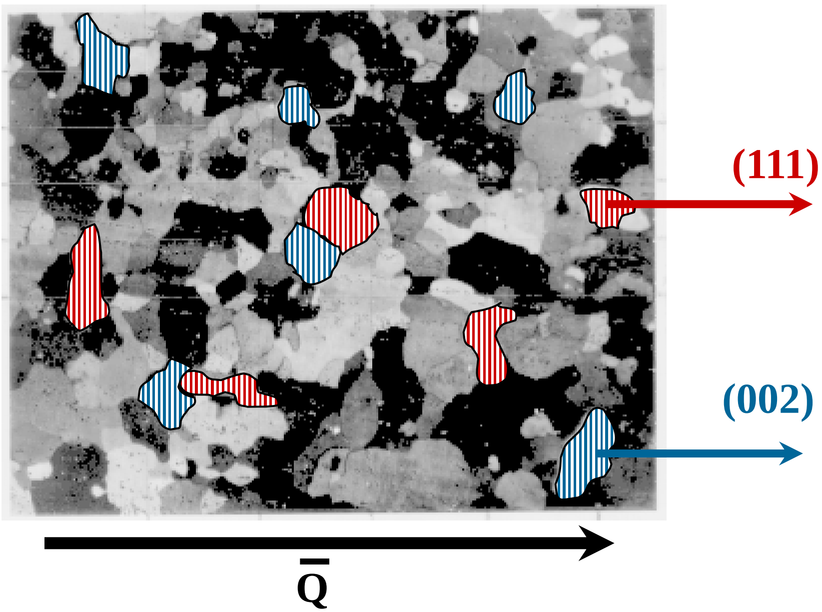
Stress mapping



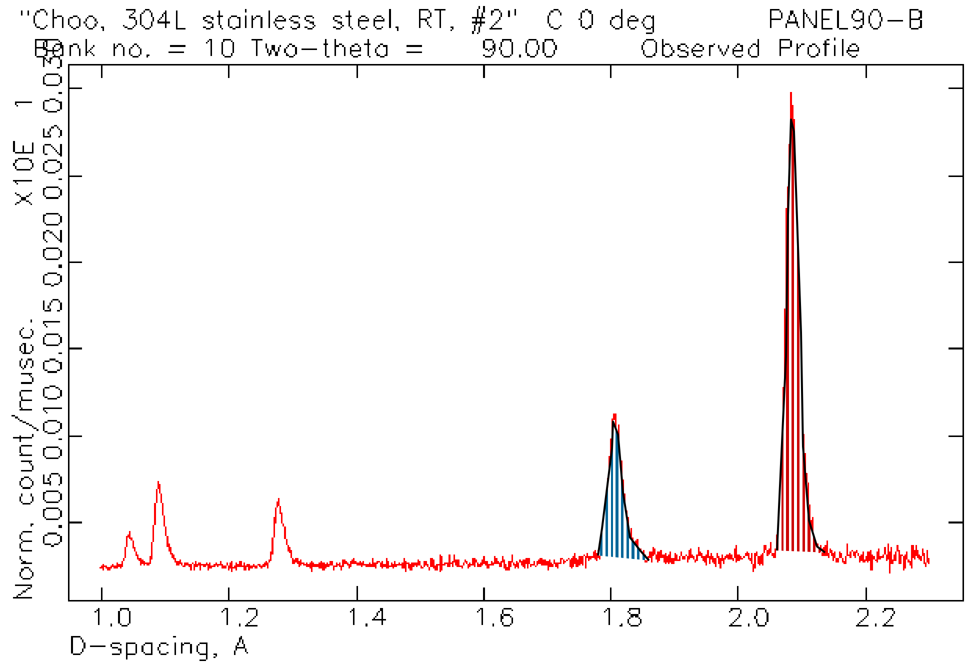
- The incident and diffracted beams are defined by slits in absorbing cadmium. The slits are typically between 0.5 and 5mm wide.
- The gauge volume is defined by the intersection of the incident and diffracted beams.
- The direction of strain measurement is along the bisector of the incident and diffracted beams
- The gauge volume must be entirely within the test sample boundaries

Polycrystal nature of diffraction

Polycrystalline Aggregate



Stainless Steel

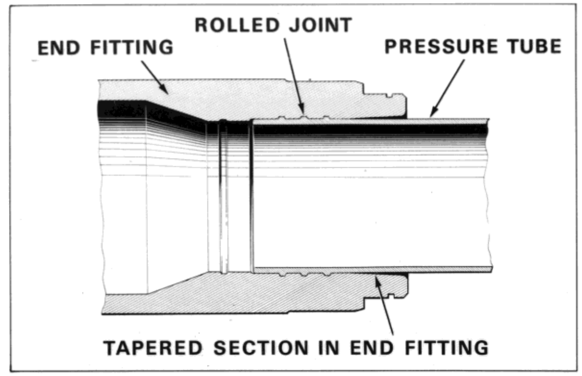


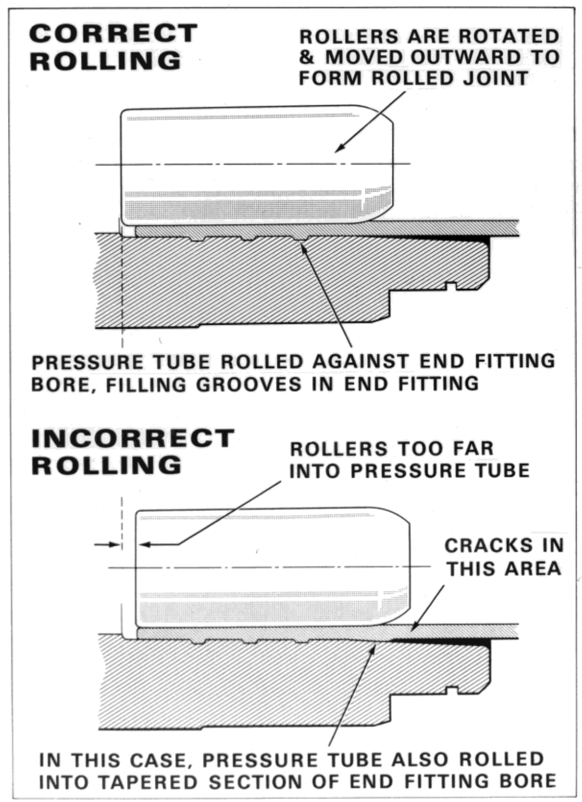
- Grains with plane normals parallel to the diffraction vector, which is defined by the instrument geometry, diffract into a detector.
- Each grain orientation (hkl) contributes to a distinct peak, giving the interplanar spacing for that set of orientations.
- Slide courtesy of Don Brown

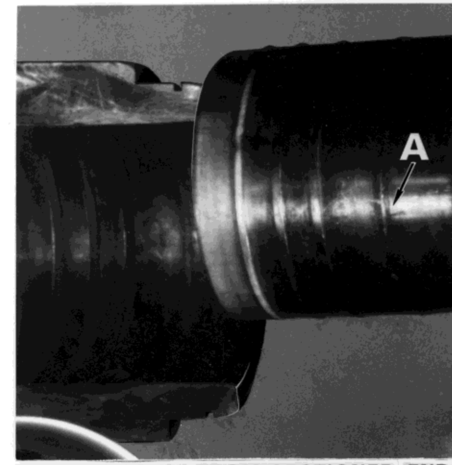
Where do macroscopic residual stresses come from?

- Usually caused by an inhomogeneous distribution of plastic deformation through the sample. The spatial scale of the <u>macroscopic field</u> is of the size of the part.
- For example a bead-on–plate weld. The bead would like to shrink freely as the temperature fell, as determined by the coefficient of thermal expansion, but is constrained by the cooler plate. The bead and nearby plate deform plastically but far from the bead the deformation in the plate is elastic. We get a tensile stress in the weld.
- A beneficial surface compressive stress field is generated by shot-peening. The surface deforms plastically in compression but below the surface the deformation is elastic. After the treatment the surface shows a compressive stress.

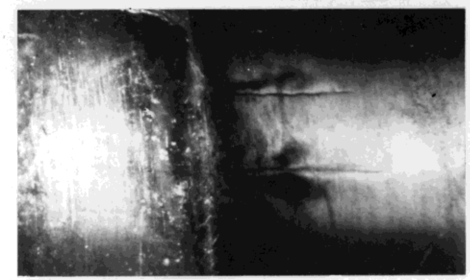
Drawings and photographs
of the rolling procedure for
CANDU pressure tubes.
If the rolling tool is pushed
too far into the end-fitting
The tube is unsupported
and a high hoop stress is
given to the tube. Because
Of the strong texture this
gives a high stress on
(0002) planes. They are pulled
apart and this is where
hydrogen sits in the ZrNb
Lattice. In practice
this gave severe hydride
cracking problems



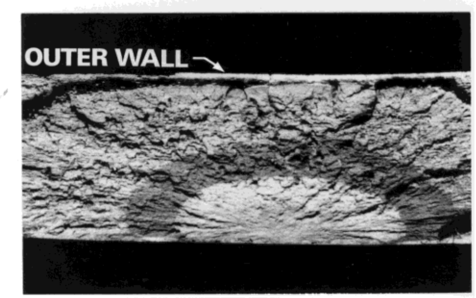




TUBE REMOVED FROM SECTIONED END FITTING (CRACKS IN AREA 'A')



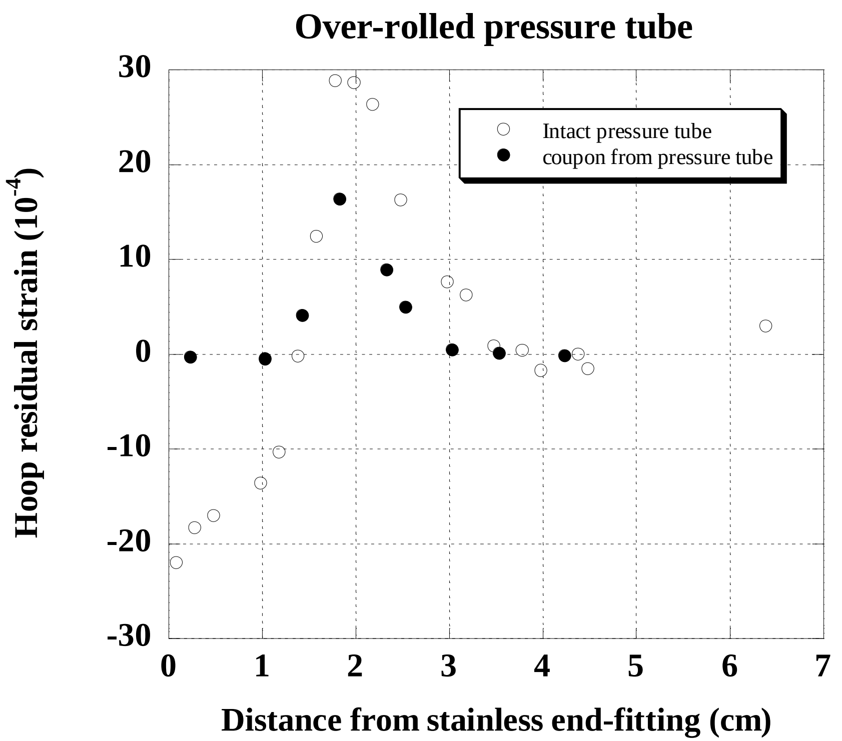
ENLARGED VIEW OF AREA 'A'



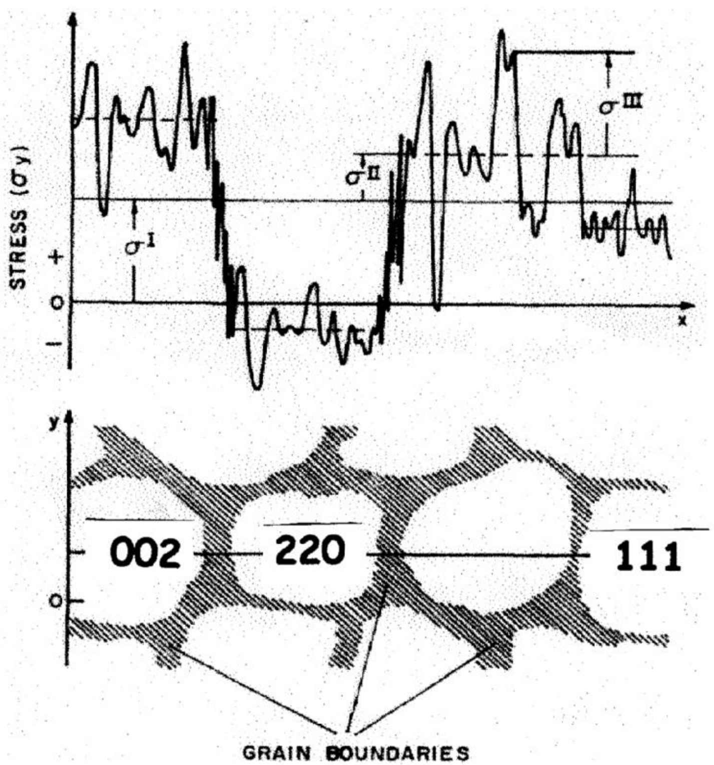
VIEW OF CRACK SURFACE

Over-rolled CANDU pressure tube

- The hoop strain is shown for the intact tube constrained by the end-fitting and for a lengthwise coupon cut from the end-fitting
- Where the tube was constrained by the end-fitting (0-1.4cm) the strain has vanished for the coupon. This is simply an elastic clamping by the end-fitting.
- In the region of the peak strain (around 2cm) the peak has halved in size for the coupon, but not disappeared. The part that vanished corresponds to the macroscopic stress. The part that remained where the plastic deformation had occurred is an intergranular strain on the scale of the grains



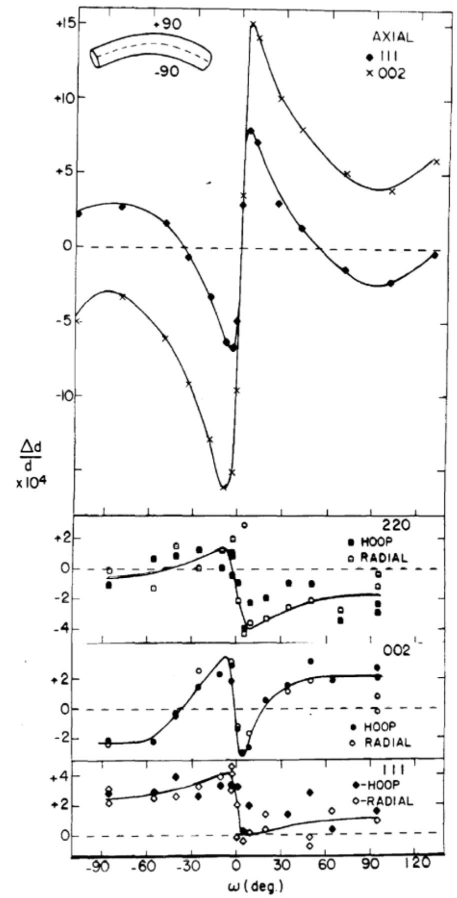
Cartoon representing the stresses on three length scales



- The macroscopic or type-1 stress is by definition the average stress in a small region of the sample and has the same value in every grain. The spatial scale is the size of the part
- The intergranular, grain-to-grain or type-2 stress is the deviation from the average stress in each grain. It varies between cystallographic directions <hkl> because of the anisotropy of slip and elastic response. The scale is the size of the grain
- The intragranular stress varies within the grains, around defects and near grain boundaries.

Example of the interplay between type-1 and type-2 effects; strains in severely bent Incoloy800 steam generator tube

- The (002) strains are much larger than the (111) strains though this can be partly explained by the diffraction elastic constants
- What is very "wrong" is that at the top and bottom of the bend the (002) and (111) strains have opposite signs
- Which one reflects the residual stress field? Which one is right?

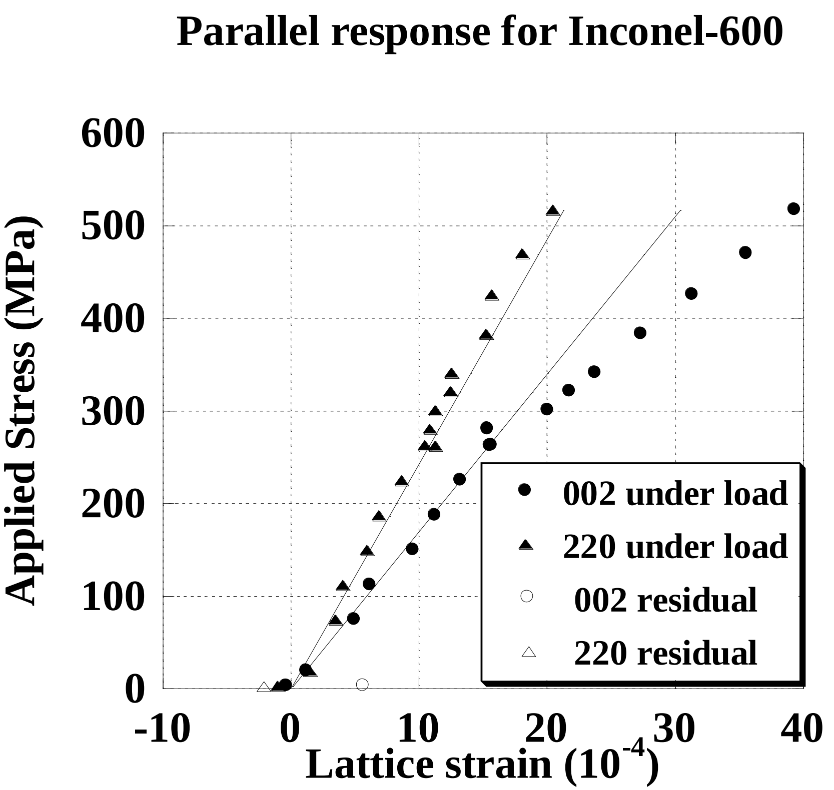


Where do intergranular or type-2 stresses come from?

- These stresses have the spatial scale of the grains. As we move from grain to grain the stress (and the strain) changes
- Grains with different plane normals [*hkl*] directed along the direction of an applied stress deform by different amounts. This is because the elastic response and the plastic response are anisotropic with respect to crystal direction. (For example in fcc metals the dislocations only move in <110> directions in {111} planes.)
- The distribution of plasticity among the grains (this inhomogeneous plastic deformation) that generates the residual stress field <u>always</u> also create intergranular stresses in principle!
- Interestingly, while the macroscopic stress is crucial for engineering, the intergranular stresses hold the interesting materials science, details of the modes of deformation and the elasticity etc

Strain response parallel to the applied stress in Inconel-600 demonstrating the appearance of intergranular strains in a tensile sample

- Suppose we apply a stress so that some crystallites have slipped but others have continued to deform elastically. When the applied stress is removed, the more elastically extended grains compress the less extended ones and are themselves left in tension.

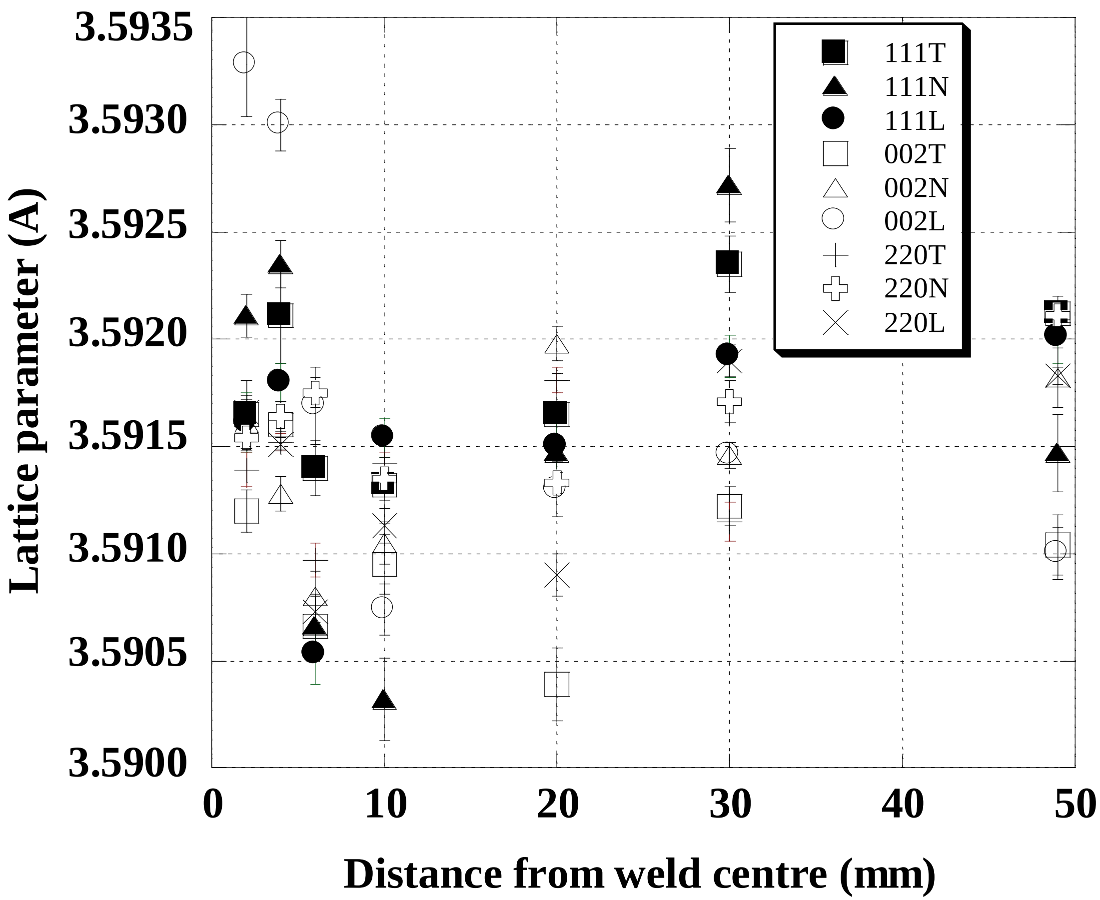


Effects of intergranular or type-2 stresses (2)

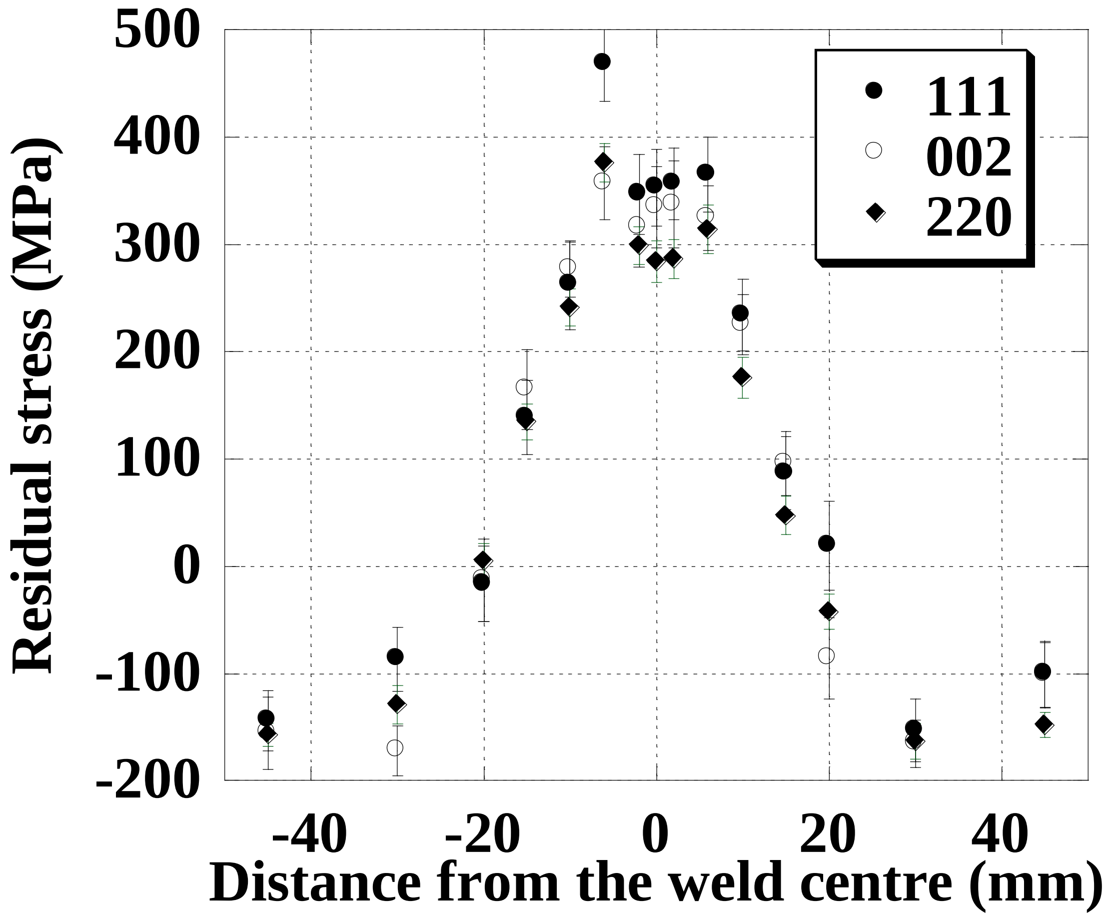
- The intergranular strains bias the measurement of strain. For example, the intergranular strain for the {002} is typically positive while that for the {220} is negative. Measurements intended to measure the macroscopic strain with the {002} reflection have an additional intergranular contribution while measurements with the {220} reflection are reduced by the intergranular contribution.
- The intergranular stresses must balance among the different grain orientations in a small volume in every direction and so are not detected with a mechanical strain-gauge. Engineers have difficulty with stresses that they cannot measure with a strain gauge!

Measuring the stress in a weld while addressing this question of stresses on different scales?

- Reference lattice spacings, d^{hkl}_0. These can be measured on small coupons cut from a companion weld. (Cutting destroys the macroscopic stress field but it leaves intergranular grain-scale stresses/strains and lattice parameter effects due to chemistry unchanged.) We then subtract the coupon spacings from the intact spacings for each <hkl> and each direction
- If the experimental approach to obtaining the reference lattice spacings is correct the computed stress field (macrostress) will be identical for all *{hkl}*.
- Make enough measurements over the whole piece to check stress balance and boundary conditions!
- We expect changes in lattice parameter upon melting so we have to be careful about annealing!



Reference lattice parameters derived from small coupons cut from a 316SS weld. While there is a fair amount of scatter, probably because of grain size, the longitudinal (002) result indicates an intergranular contribution in the weld centre. (A similar ferritic weld shows changes in lattice parameter independent of crystal and sample direction and hence likely chemical in origin)



Longitudinal macroscopic residual stress in the austenitic 316 stainless steel weld computed from longitudinal, transverse and normal strains derived from (111), (002) and (220) reflections. The reference spacings were taken from coupons cut from a companion weld. The appropriate diffraction elastic constants were used. The measurements were made on the RESA diffractometer in Tokai, Japan

Doing science with intergranular stresses: the need for models to simulate the data

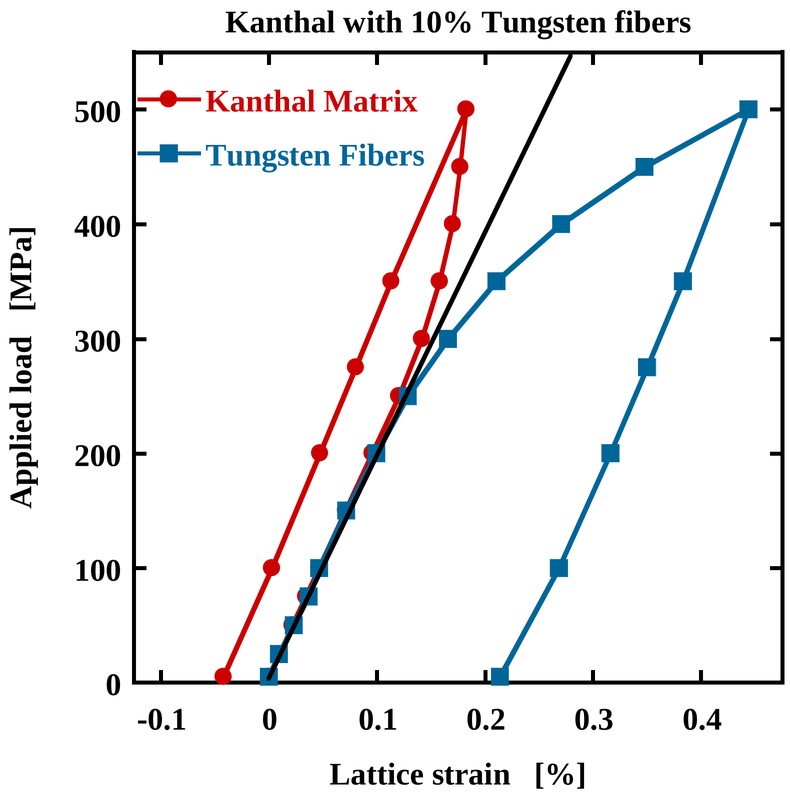
- Intergranular strains were discovered decades ago but there was no way of interpreting the results. The models let you calculate just what is measured in neutron or x-ray diffraction.
- EPSC model simulation: 15000 grains to simulate the texture. Calculate the deformation in each grain in its chosen orientation with its single crystal response and its allowed slip modes in the applied stress field as modified by the average of all the other grains and iterate to self-consistency
- FE model: many thousands of elements to simulate texture. Each element is a one single crystal with a chosen orientation, single crystal elastic constants and slip modes. Calculate the boundary conditions between each element and the applied conditions to obtain the strain tensor in each element,

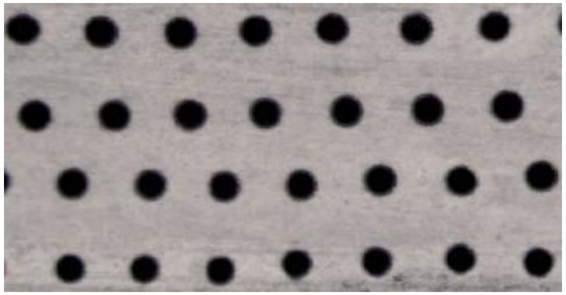
Doing materials science with neutrons: Tungsten-Kanthal composite as a model for a polycrystal

Consider loading the two constituents in parallel. In the elastic region the response is nearly equal. Once the composite reaches macroscopic yield, the elastic strain in the kanthal no longer increases with applied stress unlike the W. The Kanthal has yielded and the W has taken a greater share of the load.

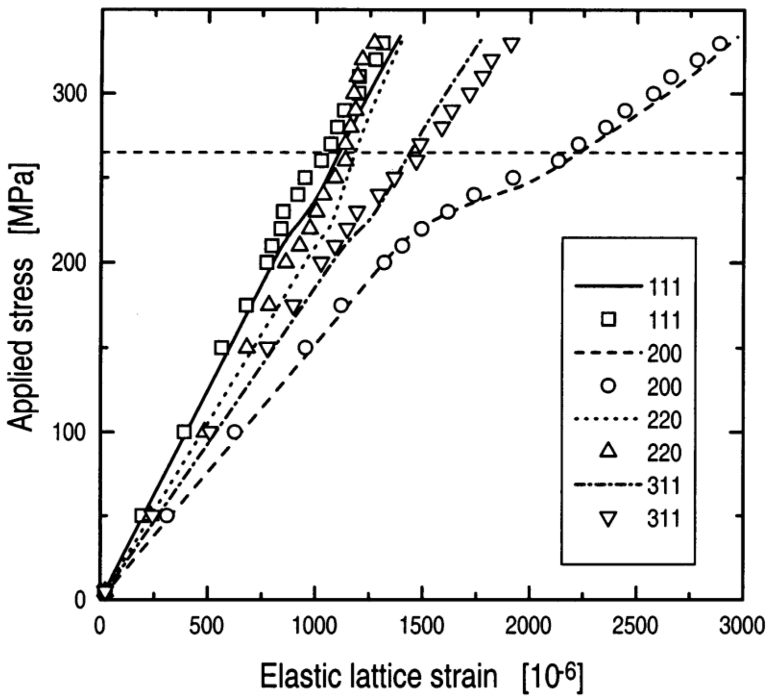
A polycrystalline metal can be thought Of as a composite with every grain Orientation a different constituent.

Slide courtesy of D.W. Brown

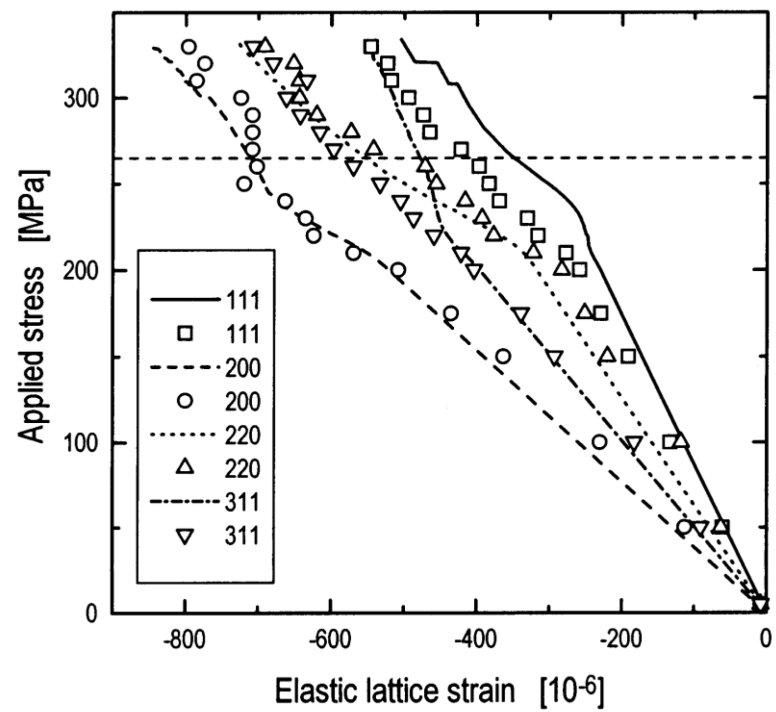




Modelling the response of an FeNiCr stainless steel. (Clausen et al)



(a) Parallel (||) to the tensile axis.



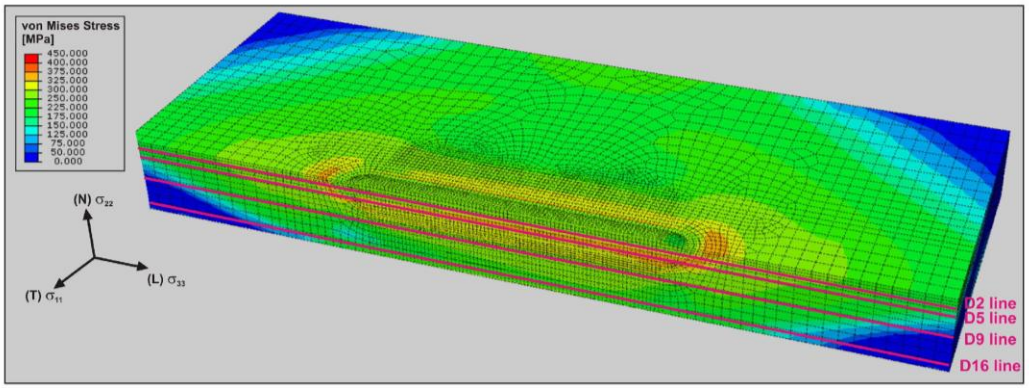
(c) Perpendicular ($\perp$) to the tensile axis.

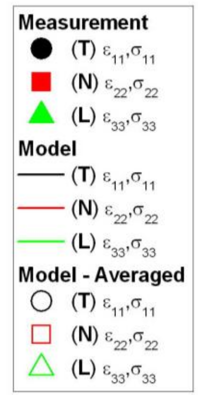
What will the intense new sources such as VULCAN at SNS or TAKUMI at J-PARC do for engineering?

- Detailed stress-mapping to discover the size and location of stresses that adversely affect function and to compare with FEM which is the engineer's primary tool. For example…….
- (a) stresses in critical parts such as landing gear, pressure tubes, rivets, nuclear welds, turbine discs etc.
- (b) well-characterised welds to drive intelligent weld process design
- (c) thicker sections up to 50mm in Ni alloys and 60mm for Fe alloys. However, there are limits……
- (e) measurement of all elements of the stress tensor

Simulations vs. Neutron Measurement

D2, D5, D9, D16 lines





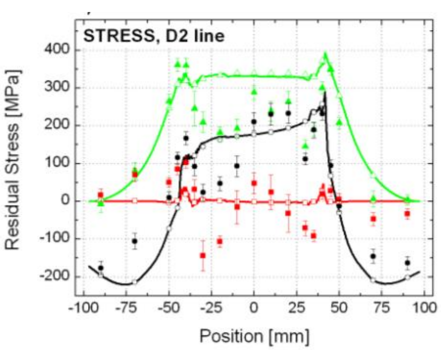
D2 line: weld d0: [-40 : 40]

D5 line: weld d0 [-40 : 40]

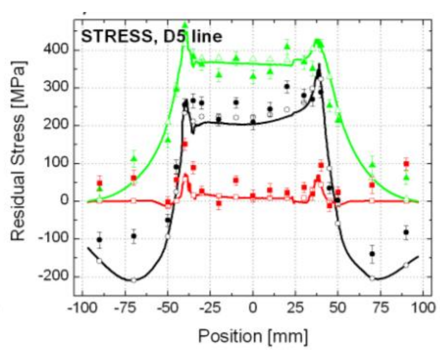
D9 line: parent d0 only

D16 line: parent d0 only

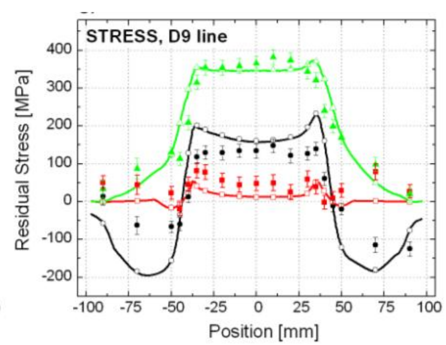
D2 line



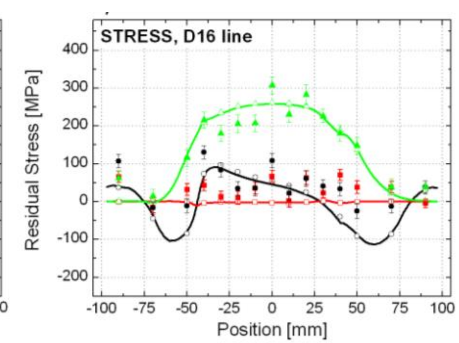
D5 line



D9 line



D16 line



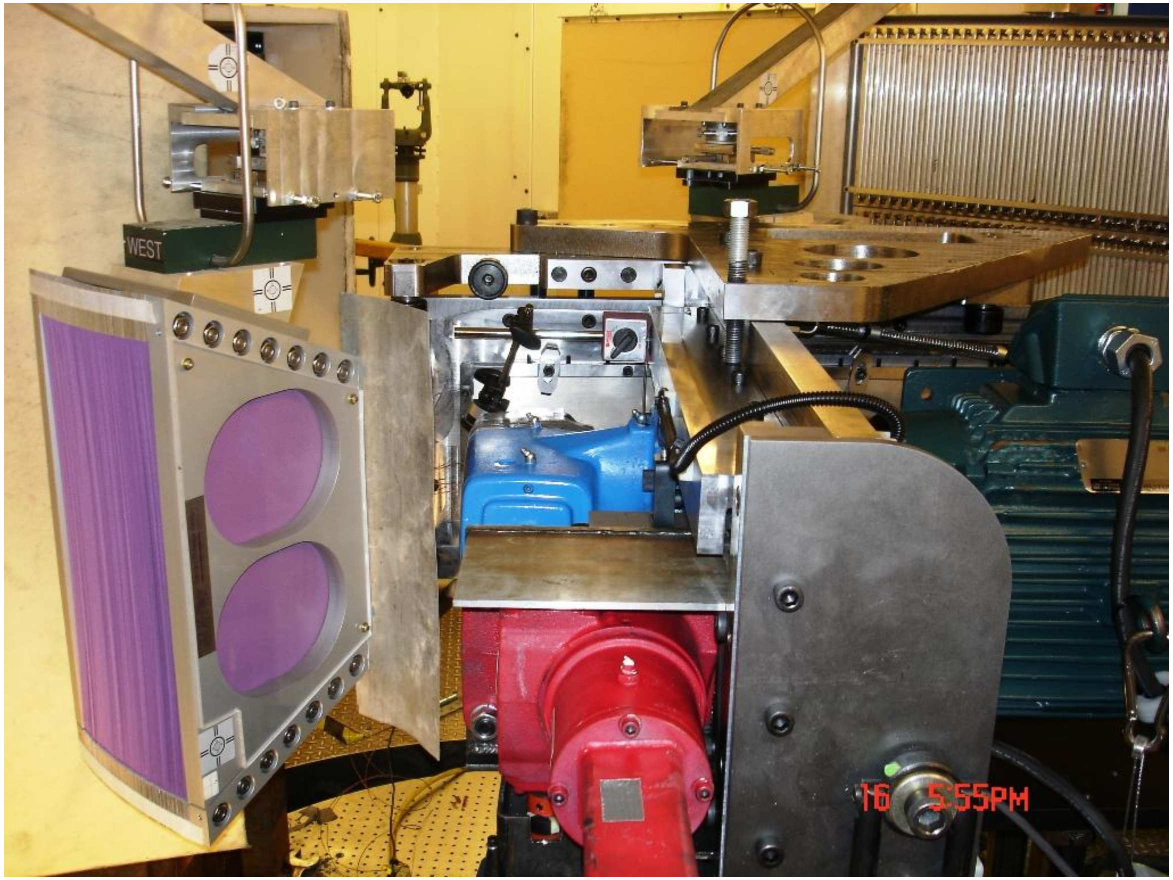
Measurements and calculations of Muransky, Luzin, Kirstein, Holden, Bendeich and Edwards

Extensions of present science making use of intergranular strains

- Determining constitutive models of aggregate polycrystal behaviour ; finding the slip systems or other modes of deformation, such as twinning, in complex materials such as Be or Ti or U or Zr-2, or two-phase Zr2.5%Nb via the intergranular stresses
- Phase changes under load (Co-WC) or "smart" materials such as $PbMnNbO_2$ which are sensitive to electric or magnetic fields
- Optimising thermo-mechanical processing and function of composites ex-situ

New science possible because of the high data collection rate

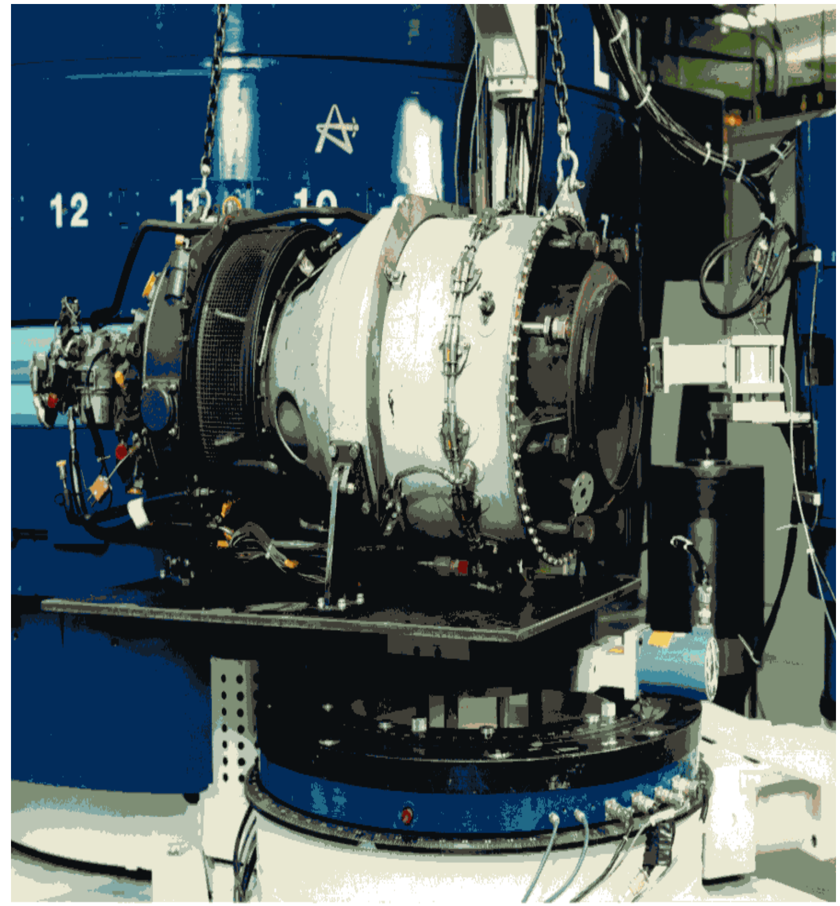
- Stresses in operating machinery; contact stresses in gears, running engines, ball-races
- Time and space dependence of chemical reactions including intermediate species
- Time dependence of recrystallisation
- In-situ processing; annealing ,welding, casting, powder processing
- Transient response to applied loads



Friction stir weld equipment installed at the SMARTS Diffractometer at Los Alamos for in-situ measurements. Tests have also been done on a spiral weld at Chalk River.

Strains in rotating machinery

- Strains in a running gas-turbine engine
- Strains in gears and bearings
- These kinds of tests can readily be done in safety within the massive hutches used to protect personnel from radiation



Conclusions

- Neutrons can have a major impact on important engineering topics because of the ability to measure stress at depth although there are limits.
- Note the polycrystalline nature of engineering components and the impact of intergranular effects.
- Reference lattice parameters require great care. Look for and try to explain chemistry and intergranular effects
- Be careful with the coverage of points in the sample so that one can check the stress balance and boundary conditions with the measured stresses
- Can do some great science with the intergranular effects

Finale

- Best of luck in your careers as young scientists. There are lots of opportunities worldwide
- If you are an experimentalist be absolutely sure that your experiments are correct. In engineering a mistake can be the difference between life and death!!
- Remember that neutrons are "not the only game in town" with the advent of intense short-wavelength synchrotron x-ray beams