Applications of neutron diffraction to engineering problems

T.M. Holden
Northern Stress Technologies
Deep River, Ontario, Canada

Oak Ridge National Laboratory, 26th June 2014

Reference material

- “Introduction to the characterization of residual stress by neutron diffraction”. M.T. Hutchings, P.J. Withers, T.M. Holden and T. Lorentzen, (Taylor and Francis: Boca Raton) 2005
- “Introduction to diffraction in Materials Science and Engineering”, A.D. Krawitz (John Wiley and Sons ,Inc.: New York) 2001
- “Non-destructive testing-Standard Test Method for determining residual stress by neutron diffraction”, ISO/TS 21432: 2005
- “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.
- “Practical Residual stress measurement methods”. Ed. G.S.Schajer, (Wiley:Chichester) 2013.
- “Residual Stress: Measurement by Diffraction and Interpretation”, I.C. Noyan and J.B. Cohen in “Materials Research and Engineering”, eds. B.I. Ischner and N.J. Grant, Springer-Verlag, New York, 1967.

Concept

The interplanar spacing, d_{hkl} constitutes an intrinsic strain gauge for all diffraction measurements in crystalline materials. It can be measured 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.

A precision of $\pm 0.01°$ in $2\theta_{hkl}°$ around 90° leads to a precision in strain $\Delta d/d=\cot\theta d\theta= \pm 1\times10^{-4}$ as does a precision in time of 3μsec in a total time-of-flight of 30,000μsec.

The key factor for neutrons is the high penetration of neutrons through most industrial materials (8% through 25mm steel). This permits measurements 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

General form

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

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 above is therefore an analog of Hooke's Law. 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. We can then transform coordinates later if needs be.

Examples of the economic impact of residual stresses

- 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!

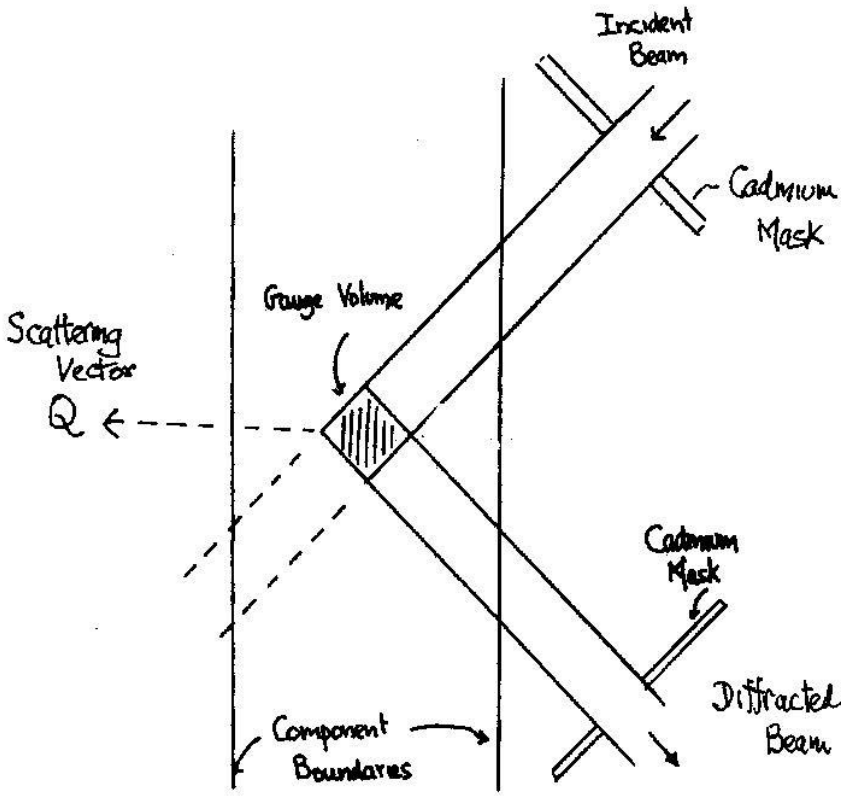
- New methods of welding have been developed to minimise stress. Is the stress minimised in fact? Can we in fact model the weld stresses accurately including the effect of sold state phase transitions and then benchmark these against experiment?

Topics of high impact (slide courtesy of D.L. Rudland, USA Nuclear Regulatory Commission)

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

- Material Characterization of Irradiated Materials
  - Reactor pressure vessel material
  - High fluence measurement (does the fluence ruin the toughness?)
  - Direct toughness measurement

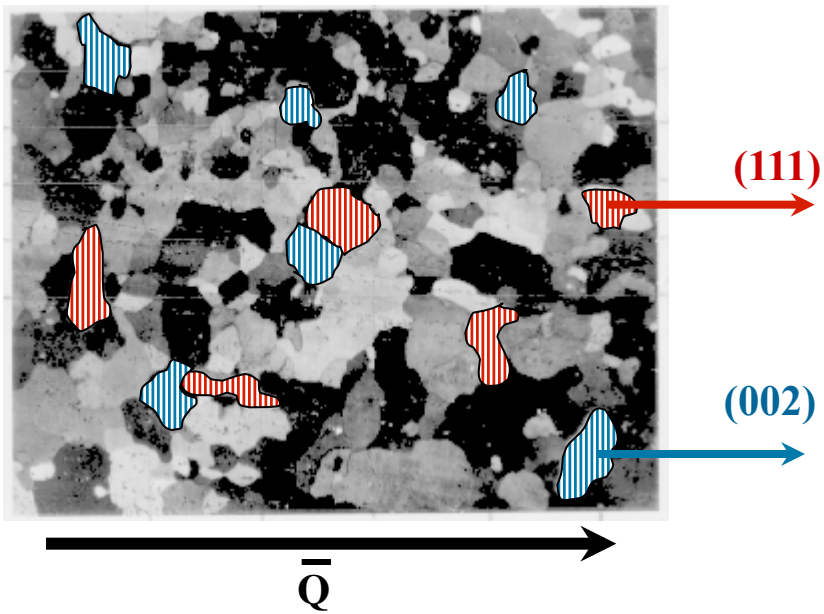
Stress mapping



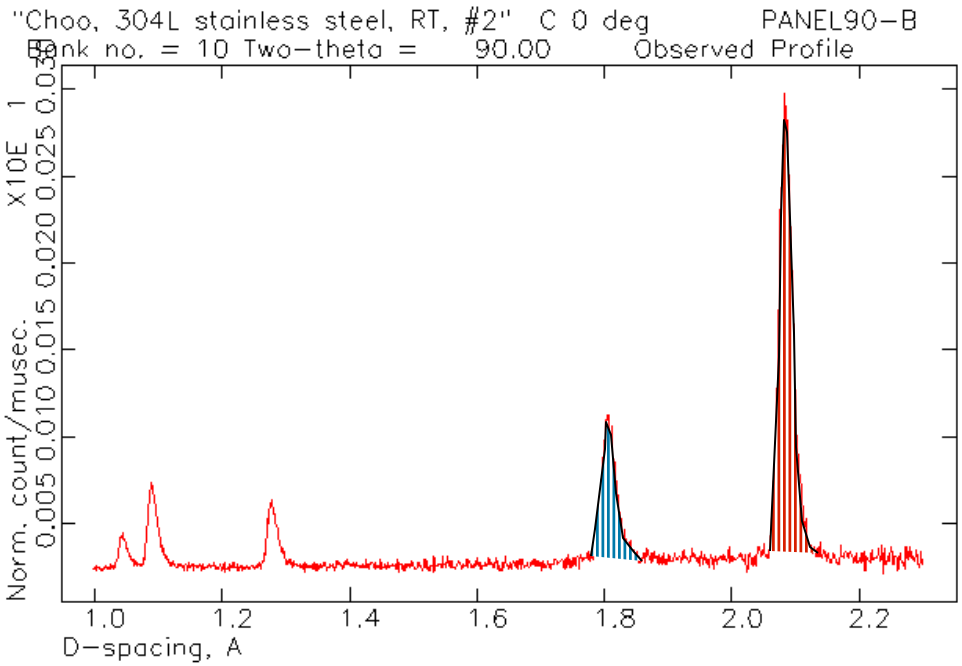
- The incident and diffracted beams are defined by slits in absorbing cadmium. The slits are typically between 0.5 and 5mm wide.
- The gauge volume is defined by the intersection of the incident and diffracted beams.
- The direction of strain measurement is along the bisector of the incident and diffracted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

Response of Grain Orientations in a polycrystal

Polycrystalline Aggregate



Stainless Steel

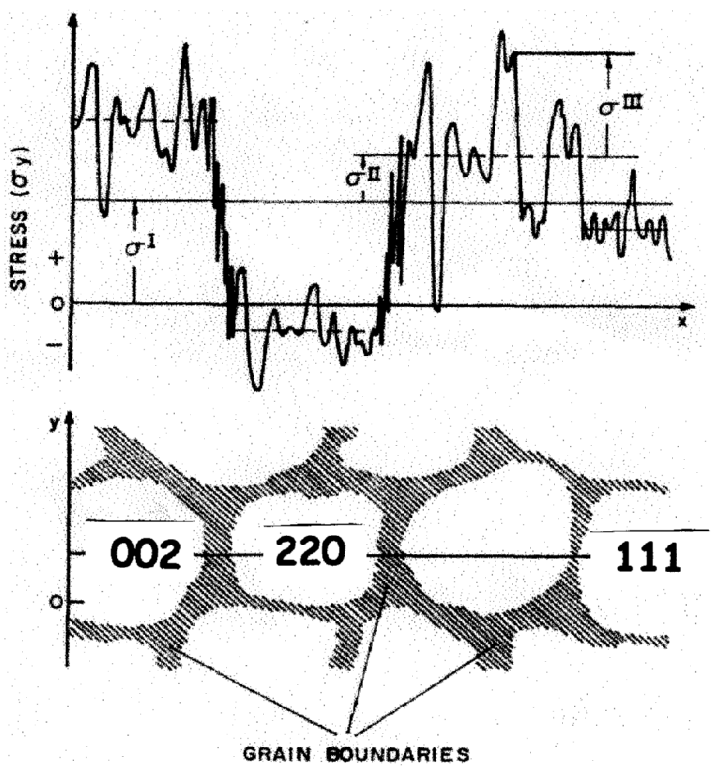


- Grains with plane normals parallel to the diffraction vector, which is defined by the instrument geometry, diffract into a detector.
- Each grain orientation (hkl) contributes to a distinct peak, given by the interplanar spacing.
- Slide courtesy of Don Brown

Origin of macroscopic residual stress

- It is usually caused by an inhomogeneous distribution of plastic deformation through the sample. The spatial scale of the macroscopic field is of the size of the part.
- For example a bead-on–plate weld. The bead would normally shrink freely as the temperature fell, as determined by the coefficient of thermal expansion, but is constrained by the cooler plate. The bead deforms plastically (and so may the plate near the bead) but far from the bead the deformation in the plate is elastic. In this case 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
and the surface then shows a compressive stress.

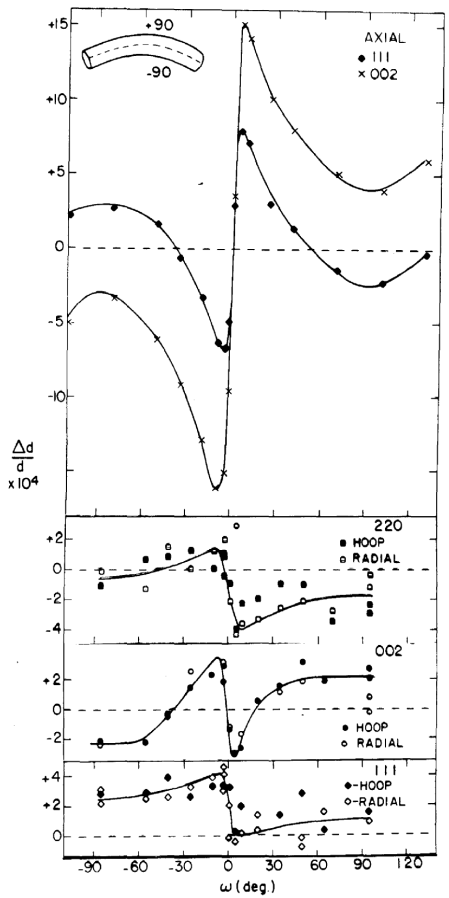
Schematic of the stresses on three length scales



- The macroscopic or type-1 stress is the same in every grain. In fact it is the stress averaged over all grain orientations [hkl] at a particular location. The spatial scale is on the size of the part
- The intergranular, grain-to-grain or type-2 stress is different in different crystallographic directions because of the anisotropy of slip and elastic response. In fact it is the deviation from the average stress in a given grain. The scale is the size of the grain
- The intragranular stress varies <u>within the grain</u>, around defects or 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?



Origin of intergranular or type-2 stresses. A polycrystal is a composite!

- The type-2 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. The elastic response is anisotropic with respect to crystal direction. The plastic response is also anisotropic; for example in fcc metals the dislocations only move in <110> directions in {111} planes.
- Suppose we apply a stress which exceeds the yield point so some crystallites have yielded and others have not. When the applied stress is removed, the elastically extended grains compress the less extended grains and are themselves left in tension. Examples of this behaviour are shown for Inconel-600.
- This occurs for any stress field such as that generated in making a weld. The effects that generate the residual stress field always also create intergranular stresses in principle!

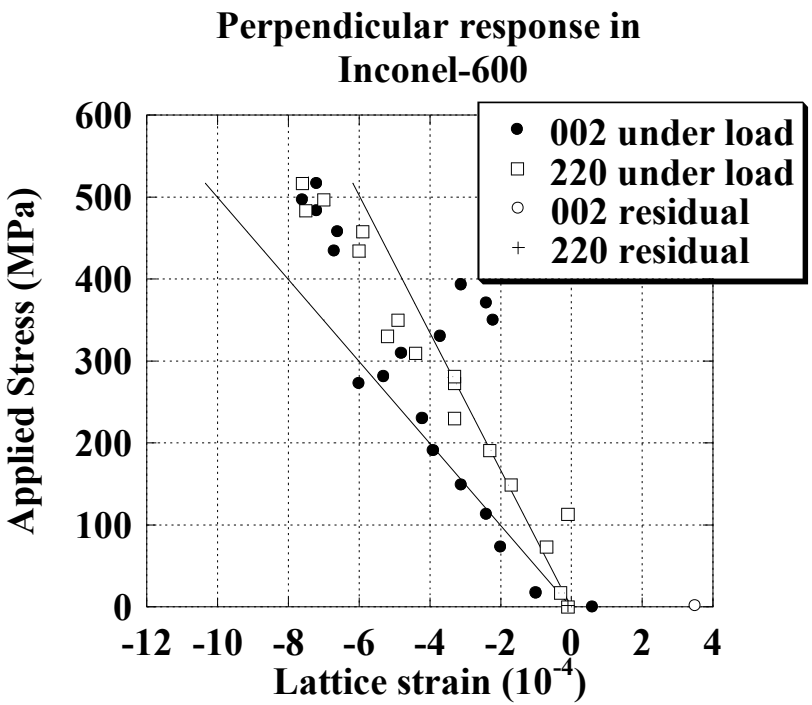
Effects of intergranular or type-2 stresses (2)

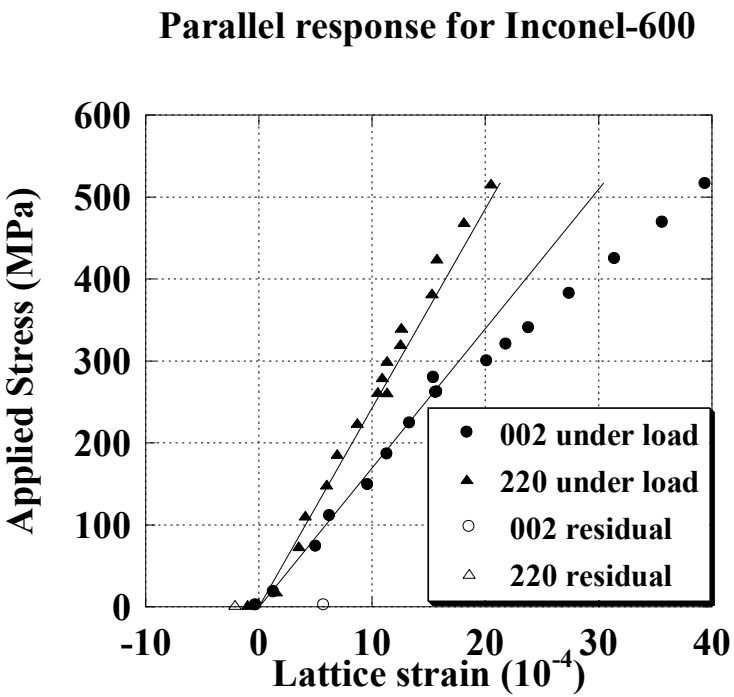
- The intergranular stresses must balance among the different grain orientations in a small volume in every direction and so are not detected with a mechanical strain-gauge which gives the average.

- The intergranular stresses cause the deviations from the $\sin^2\psi$ rule used for X-ray diffraction since they do not follow a simple tensor behaviour.

- The intergranular strains bias the measurement of strain by diffraction. For example, the intergranular strain found for {002} planes in fcc materials is typically positive while that for {220} planes 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.

Strain response parallel and perpendicular to the applied stress in Inconel-600 generating intergranular strains



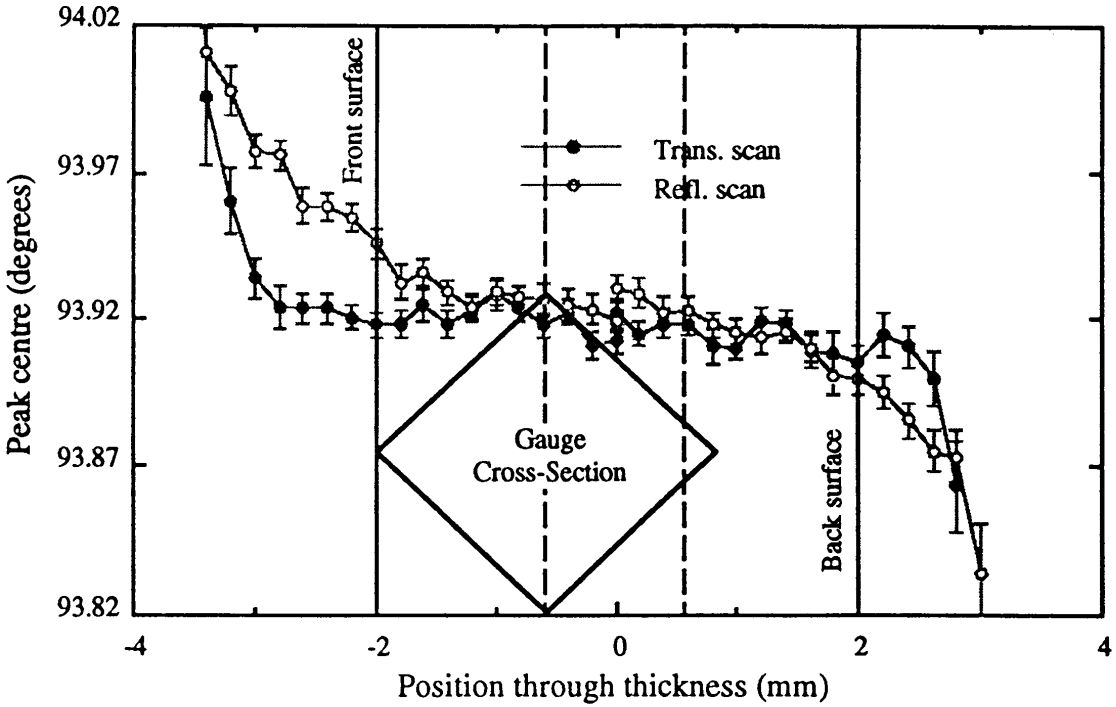


Engineering or materials science
Macroscopic or intergranular

- The engineer is nearly always interested in the type-1 or macroscopic stress. Since the demonstration experiment for the school is a residual stress measurement of a bead-on-plate weld the pitfalls and how one goes about the test will be examined for the case of the TG4 weld-in-slot measurement. (O. Muransky et al. Int. J. of Solids and Structures, 49, (2012) 1045-62.) First the pitfalls.

- The material scientist is usually interested in the type-2 or intergranular stress. In this case the nature of the modes of deformation and how the microstructure affects the interpretation are of interest. In this case the problem of load-sharing between the matrix and added minor phases for the superalloy In 718 will be examined. The problem is unsolved though the elements of a solution are clear.

Partly filled gauge volume: an Fe powder

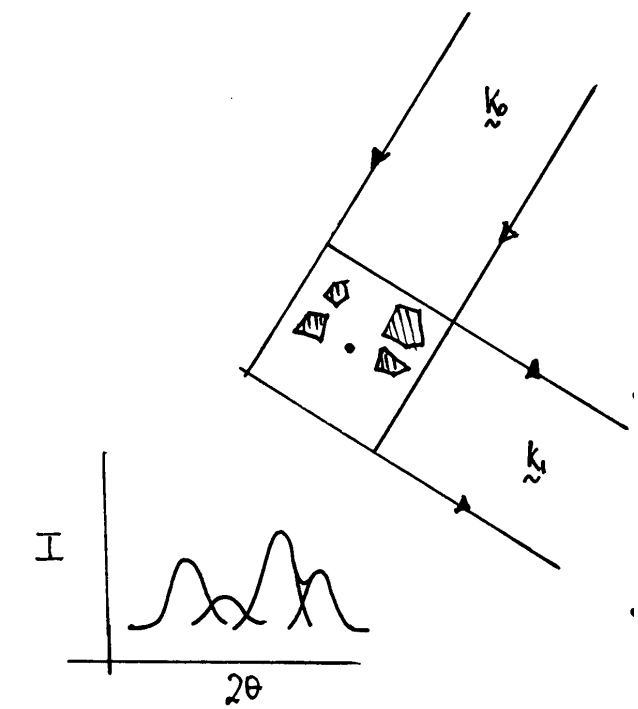


Huge spurious shifts occur when the gauge volume is only partly filled at a surface.
The same systematic error will occur at an internal surface such as in a dissimilar metal weld or at severe texture gradients.

Big grain errors

Big grains may be statistically offset randomly from centre of the gauge volume and sometimes only two or three grains contribute to the intensity. This gives an error in d-spacing which shows up as a large scatter in lattice spacing from point to point well outside the assigned uncertainty. The instrument is calibrated with a small-grain standard powder where the average position of all the small grains is the centre of the gauge volume

Large fluctuations in the intensity of the peak from point to point accompany the scatter in lattice spacing. Not a good sampling of the behaviour in any case. To alleviate rotate or rock the sample a few degrees to increase number of grains sampled.



The essentials in measuring weld stresses

- Setting up the sample carefully, ideally making use of the SSCANS software since the sample distorts on welding. Make sure that the gauge volume is always completely filled otherwise serious systematic errors occur.

- Reference lattice spacings, d^{hkl}_0. These can be measured on small coupons cut from a companion weld. (This is actually less than ideal since the weld line can be randomly wavy and the companion different from the actual.) Cutting destroys the stress field but it leaves intergranular grain-scale stresses/strains, or effects due to chemistry changes on the lattice parameter, unchanged. Remember lattice parameters can change on melting

- There has to be an FEM calculation of the stresses and therefore a benchmarking exercise associated with the work!

- Make enough measurements over the whole piece to check stress balance and boundary conditions! Check your results over and over again!

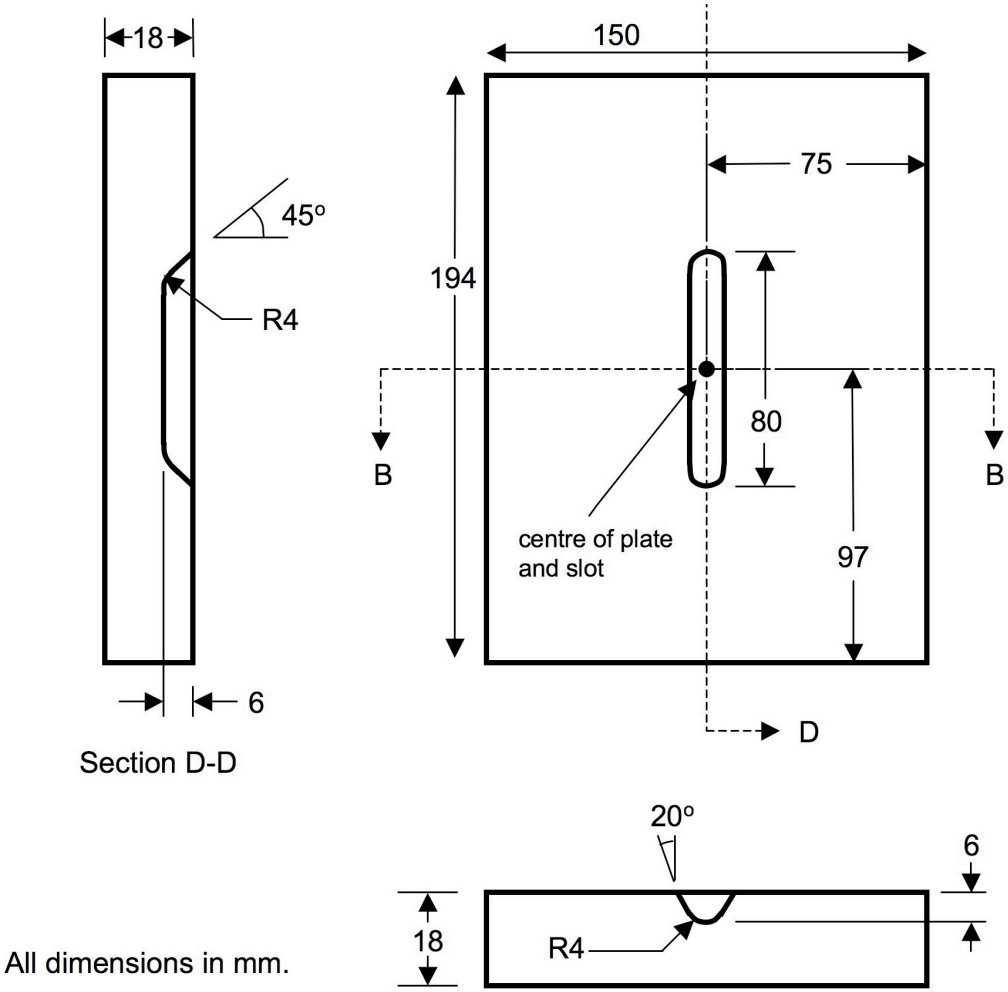
Net TG4 – three pass slot-welded specimen

AISI 316L plate containing three superimposed TIG beads laid into a slot

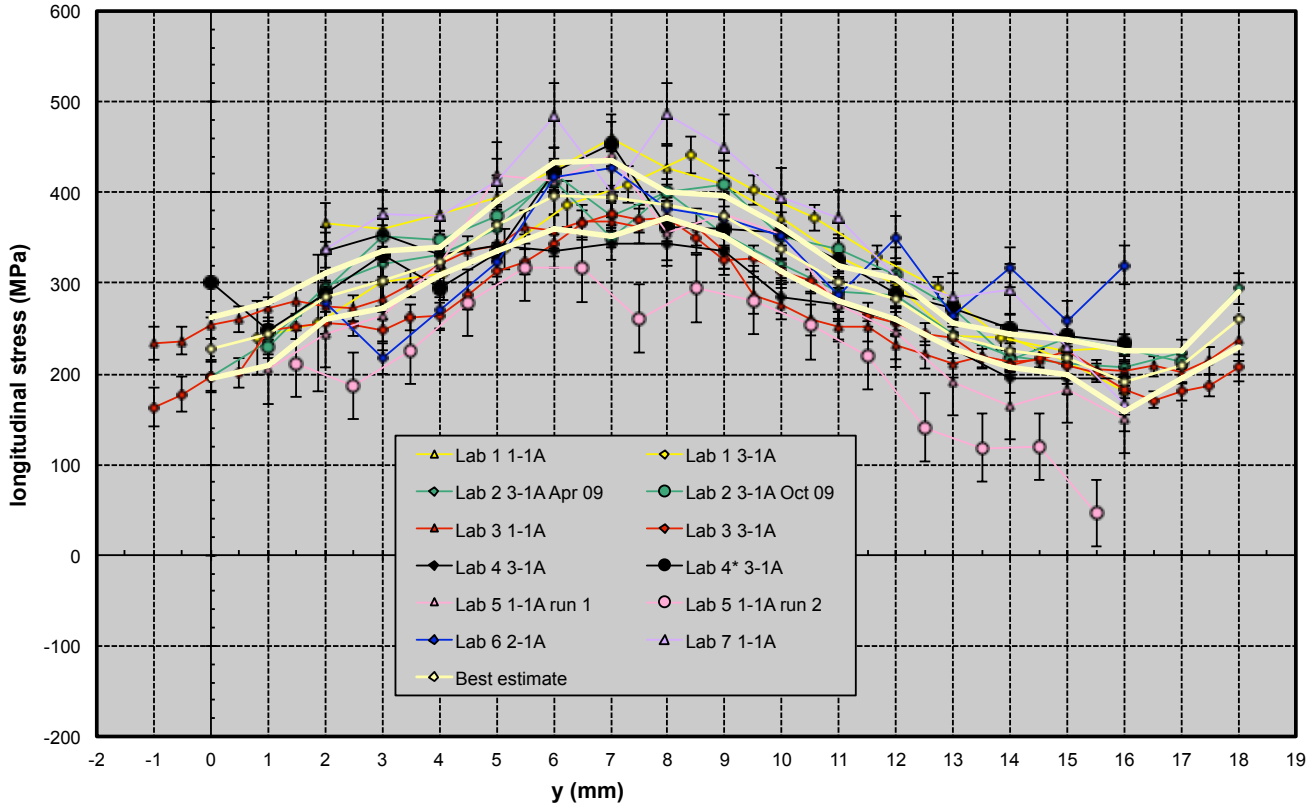
Increase in complexity over NeT TG1
- Multi-pass weld
- Significant volume of weld metal

Specimen remains compact enough for straightforward neutron and synchrotron diffraction measurements

Moving heat source finite element predictions remain feasible



Slides courtesy of Prof. Mike Smith

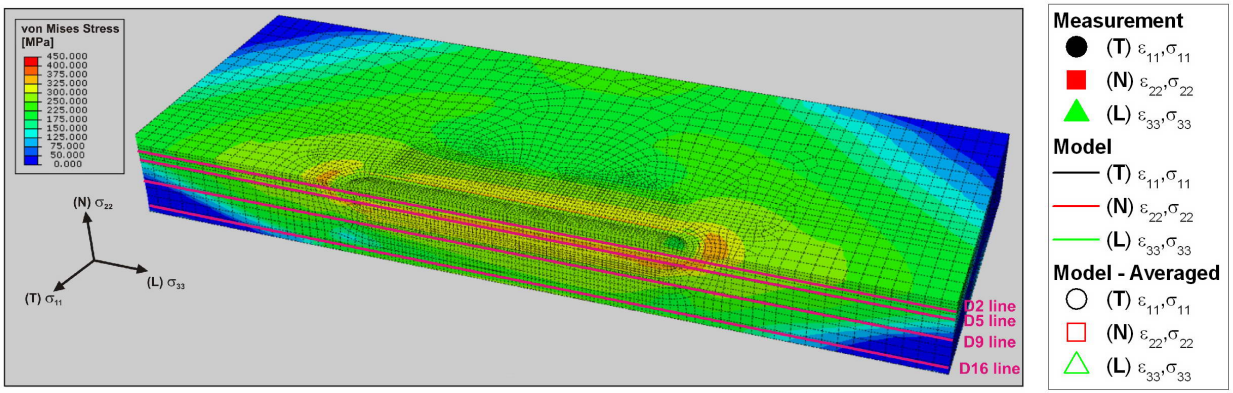


A cautionary Tale! The results are a compendium of work from 7 labs. The variation in the values of longitudnal stress between labs is far outside the errors. The typical uncertainty of measurements at a single instrument is much higher than the fitting error normally quoted Only a minority of labs reported according to the protocol. We do not yet know who fully followed best practice. The unwelded control was mostly ignored. Handling of stress-free. reference samples proved a major issue. Not clear that measurement recommendations always followed. Slide courtesy of Prof. Mike Smith

The setup of the TG4 sample at ANSTO to measure the transverse and normal strains
Showing the sample and the incident and diffracted beams

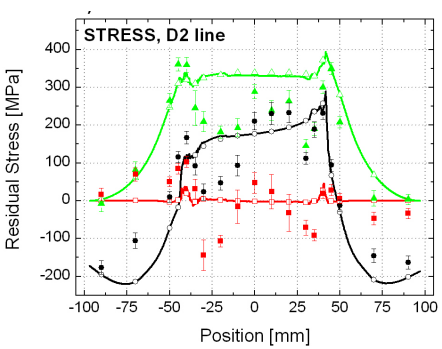
Simulations vs. Neutron Measurement

D2, D5, D9, D16 lines

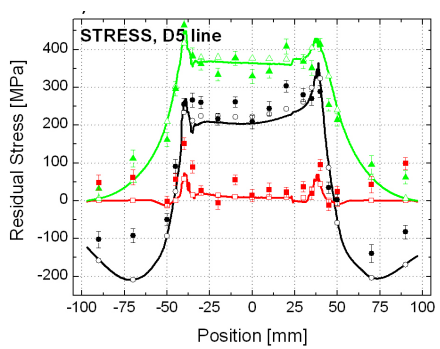


D2 line: weld d0: [-40 : 40]
D5 line: weld d0 [-40 : 40]
D9 line: parent d0 only
D16 line: parent d0 only

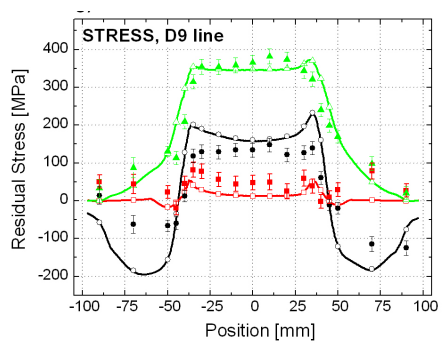
D2 line



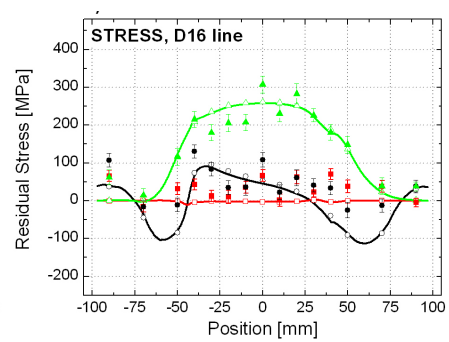
D5 line



D9 line



D16 line



Measurements and calculations of Muransky, Luzin, Kirstein, Holden, Bendeich and Edwards.
The D lines run parallel to the weld direction at depths of 2,5mm etc

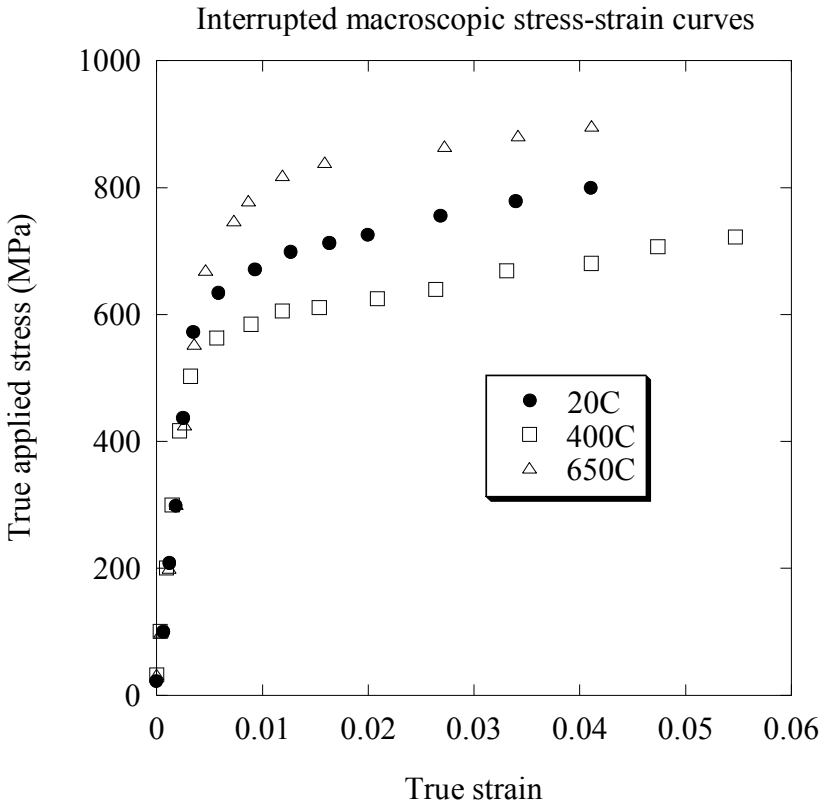
What are the many assumptions that go into the calculation? (garbage in=garbage out!)

- Finite element calculation
- Temperature/time response of the weldment at many points with thermocouples
- Weld pool shape as it moves across the part
- Information about the welding speed and the energy supplied
- Thermal modelling to simulate the temperature excursions and then (uncoupled) mechanical modelling to get the stress and strain
- Hardening models to represent cyclic thermo-mechanical loading of the weld cycles

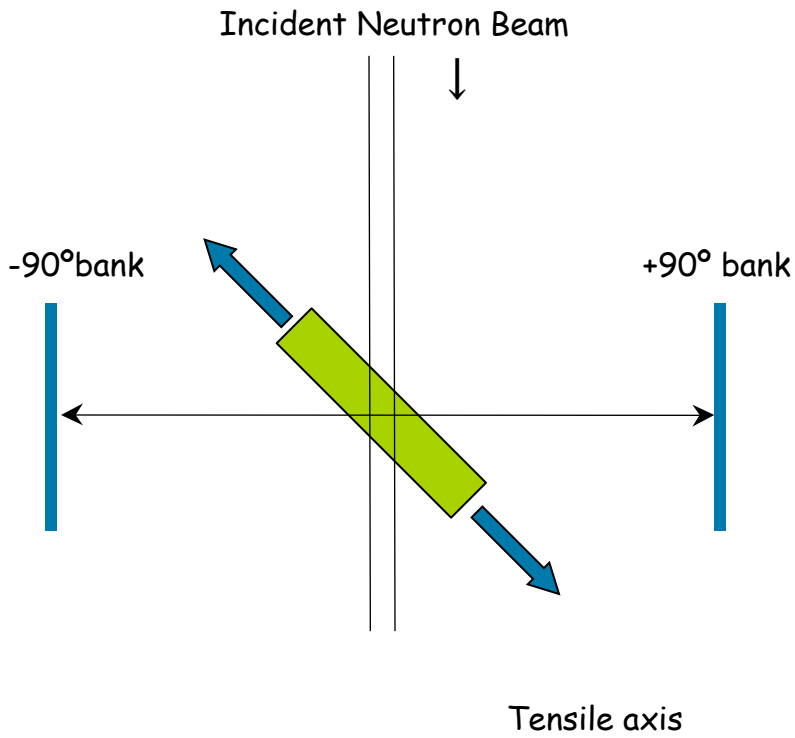
Inconel 718

- A superalloy used for turbine discs with good strength at high temperatures. The γ-phase matrix is usually strengthened with precipitates of the tetragonal γ”-phase Ni_3Nb, the γ’ -phase Ni_3Al and the orthorhombic δ-phase Ni_3Nb.
- The γ” and γ’ -phases are unstable with respect to the δ-phase on ageing and in service and the mechanical properties of the alloy such as the stress-strain curve can become degraded.
- The aim of the experiment was to find the load sharing between the matrix and the strengthening δ-phase under applied stress at 20, 400 and 650°C.
- The difficulty of the experiment comes from the low (2%) concentration of the δ-phase. A two-phase Rietveld fitting procedure was used to find the lattice parameters of the matrix and the δ-phase in the time-of-flight experiment at the SMARTS diffractometer at Los Alamos.

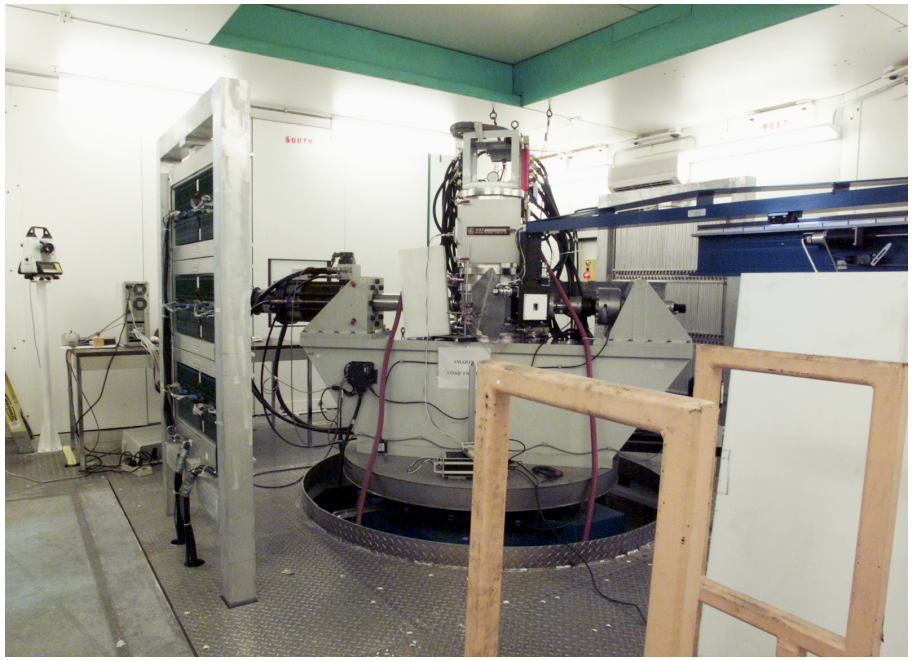
Interrupted macroscopic stress-strain curves

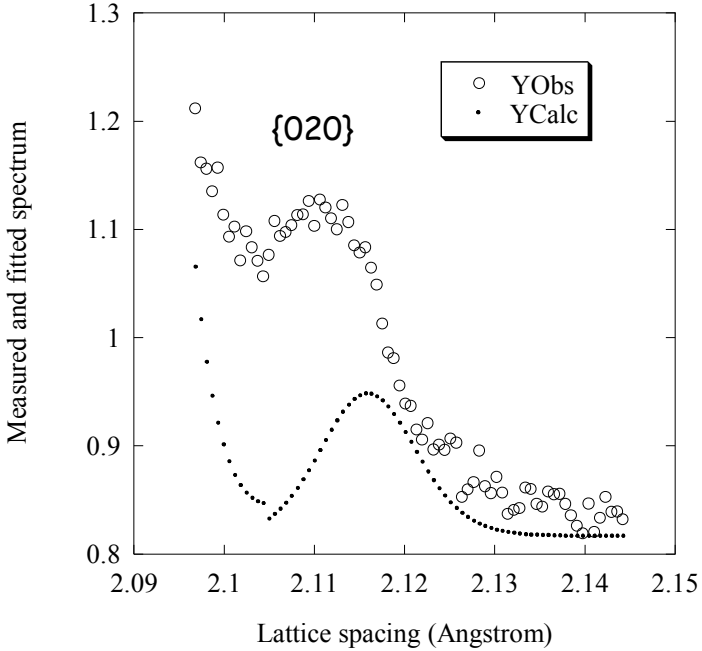


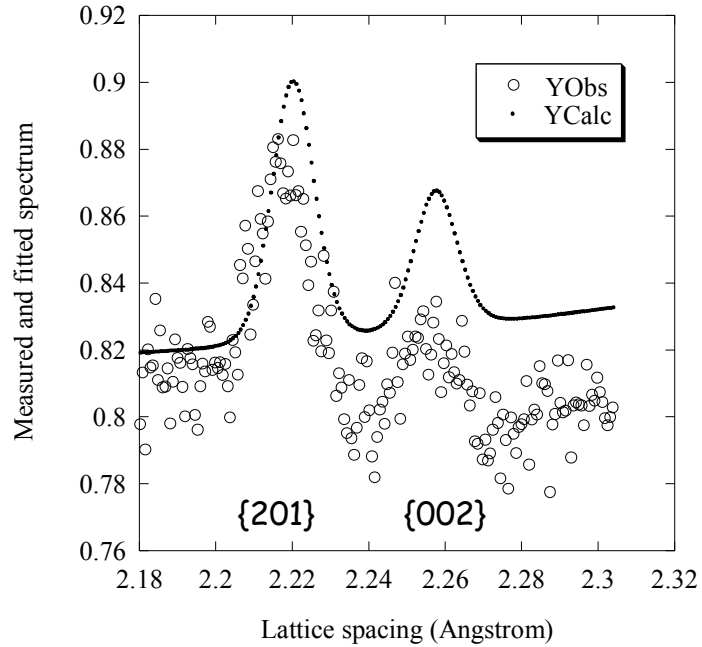
Odd temperature dependence. The first thought is “Did we mess up?”

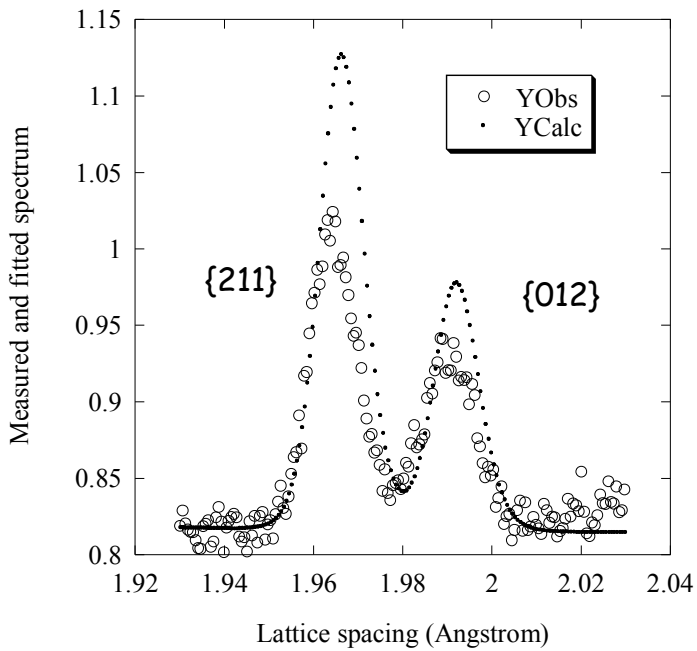


SMARTS diffractometer
at Los Alamos National Laboratory
showing load-frame
Slide courtesy of Don Brown









Five δ-phase peaks were identified in the time-of-flight spectrum and they were comparable with the background. They were about 50 times weaker than the γ-phase peaks

Microstructure and alignment of the δ-phase within the γ-matrix or know your material

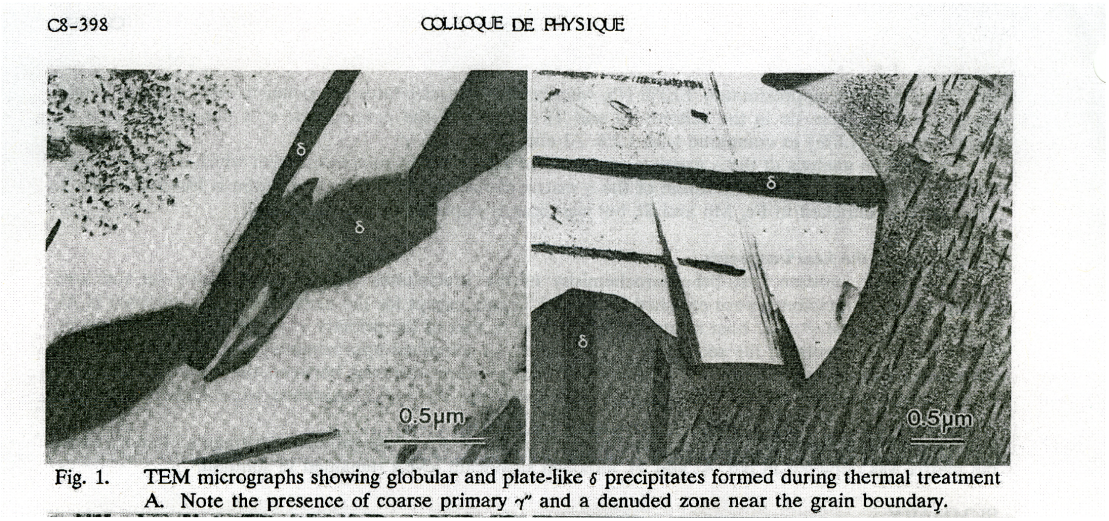


Fig. 1. TEM micrographs showing globular and plate-like δ precipitates formed during thermal treatment A. Note the presence of coarse primary γ″ and a denuded zone near the grain boundary.

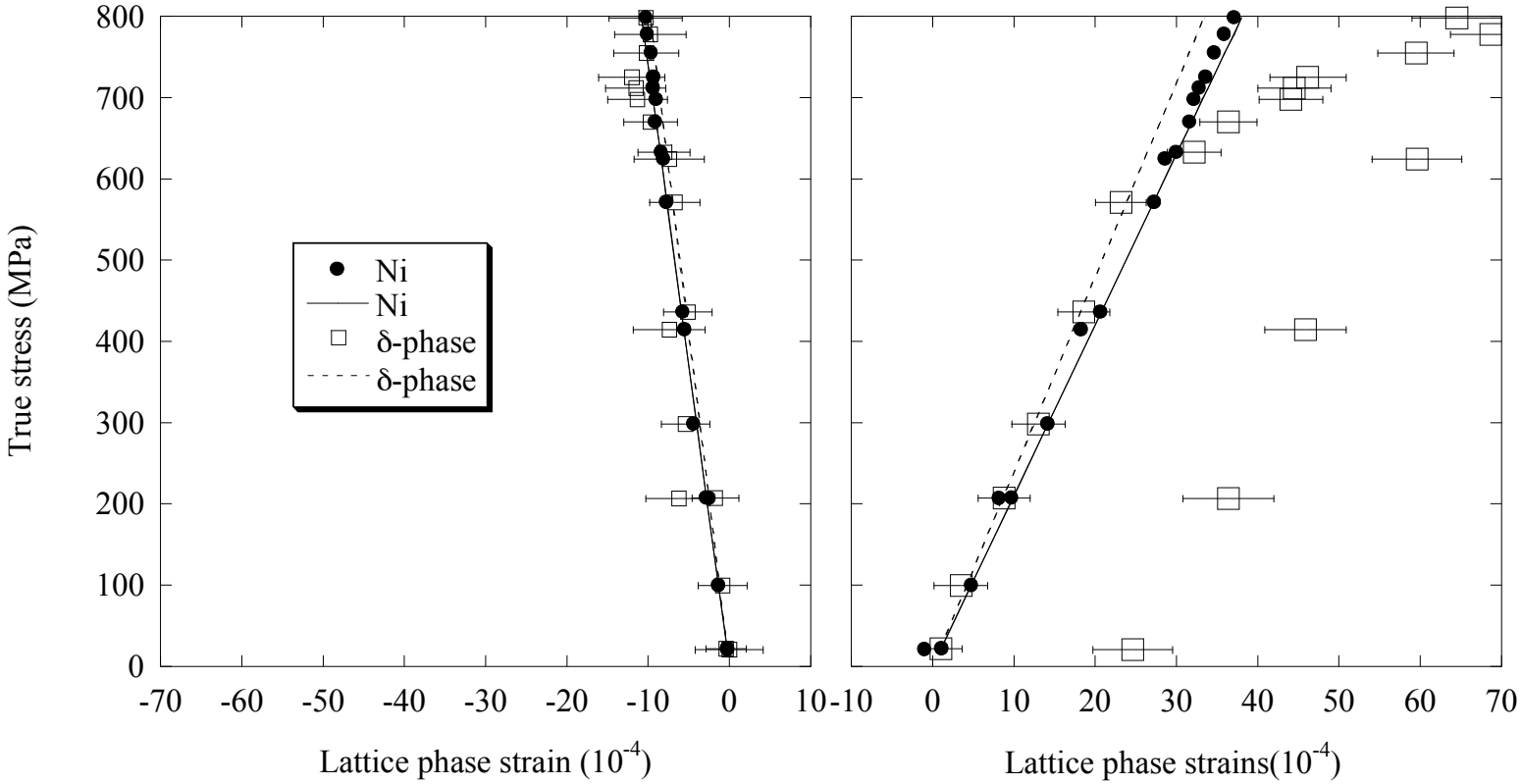
According to M.G. Burke and M.K. Miller, Colloque de Physique C8, Suppl. Au No. 11, Tome 50, 1989, who found the microstructure, the alignment between the two phases is given by

$$< 010 >_\delta = < 111 >_\gamma \qquad < 001 >_\delta = < \bar{1}\bar{1}2 >_\gamma \qquad < 100 >_\delta = < 1\bar{1}0 >_\gamma$$

The strains

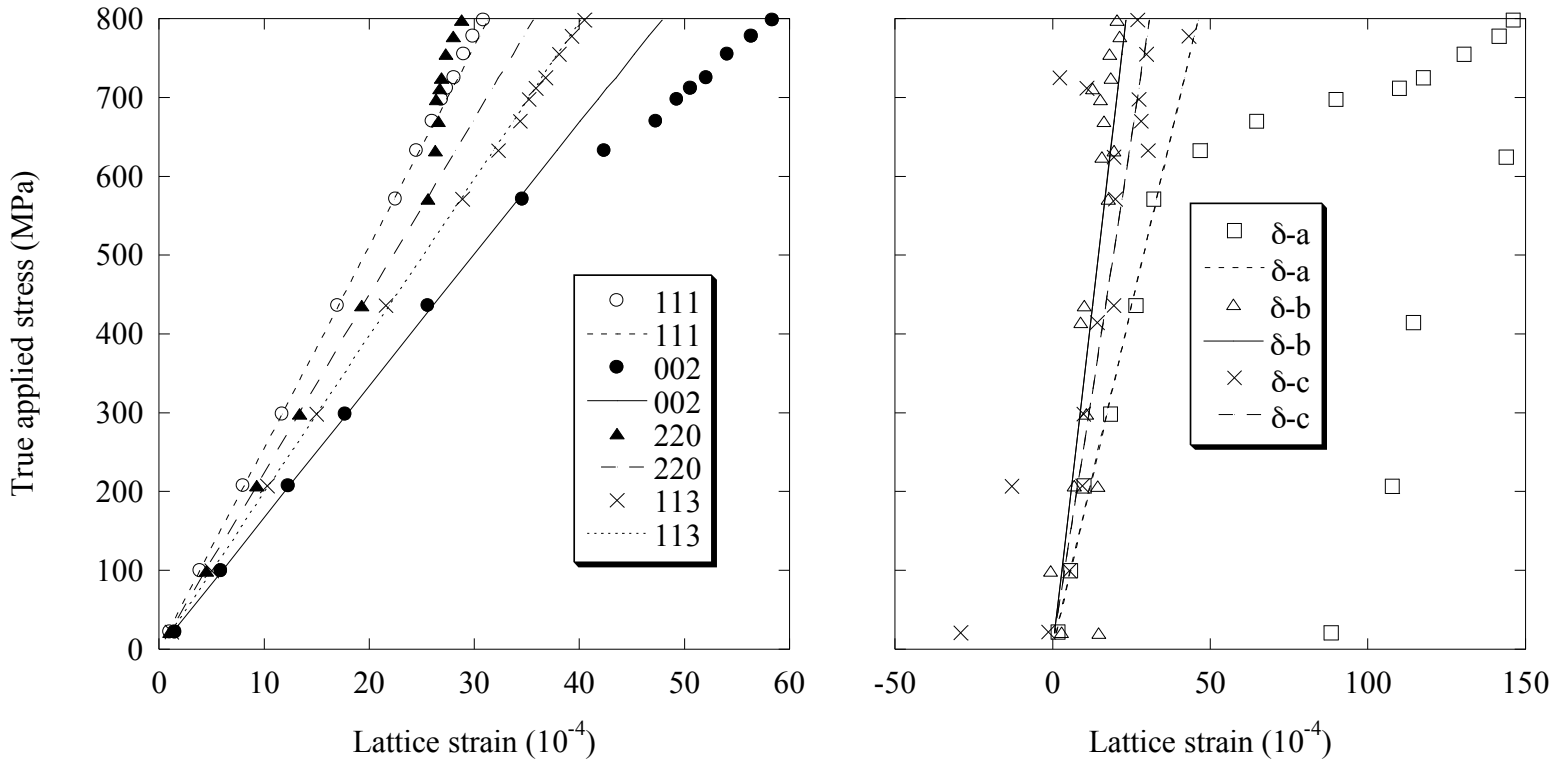
- There are probably two sets of strains. The first is between grains of the matrix which are adjacent to one another in the microstructure. These are the intergranular strains which exist in the matrix of a regular Ni alloy.
- The second is between specific grains in the matrix and the δ-phase. These are interphase strains. There is a one to one correspondence between directions in the two phases such as $(020)_\delta$ and $\{111\}_\gamma$ and $(002)_\delta$ and $(-2,-2,4\}_\gamma$. The microstructure is such that the grains of the δ-phase are not adjacent to each other. The analog is "currants in a bun".

Phase strains parallel and perpendicular to the applied stress in the γ-matrix (just the average "a") and the δ-phase (average strain) to get the big picture



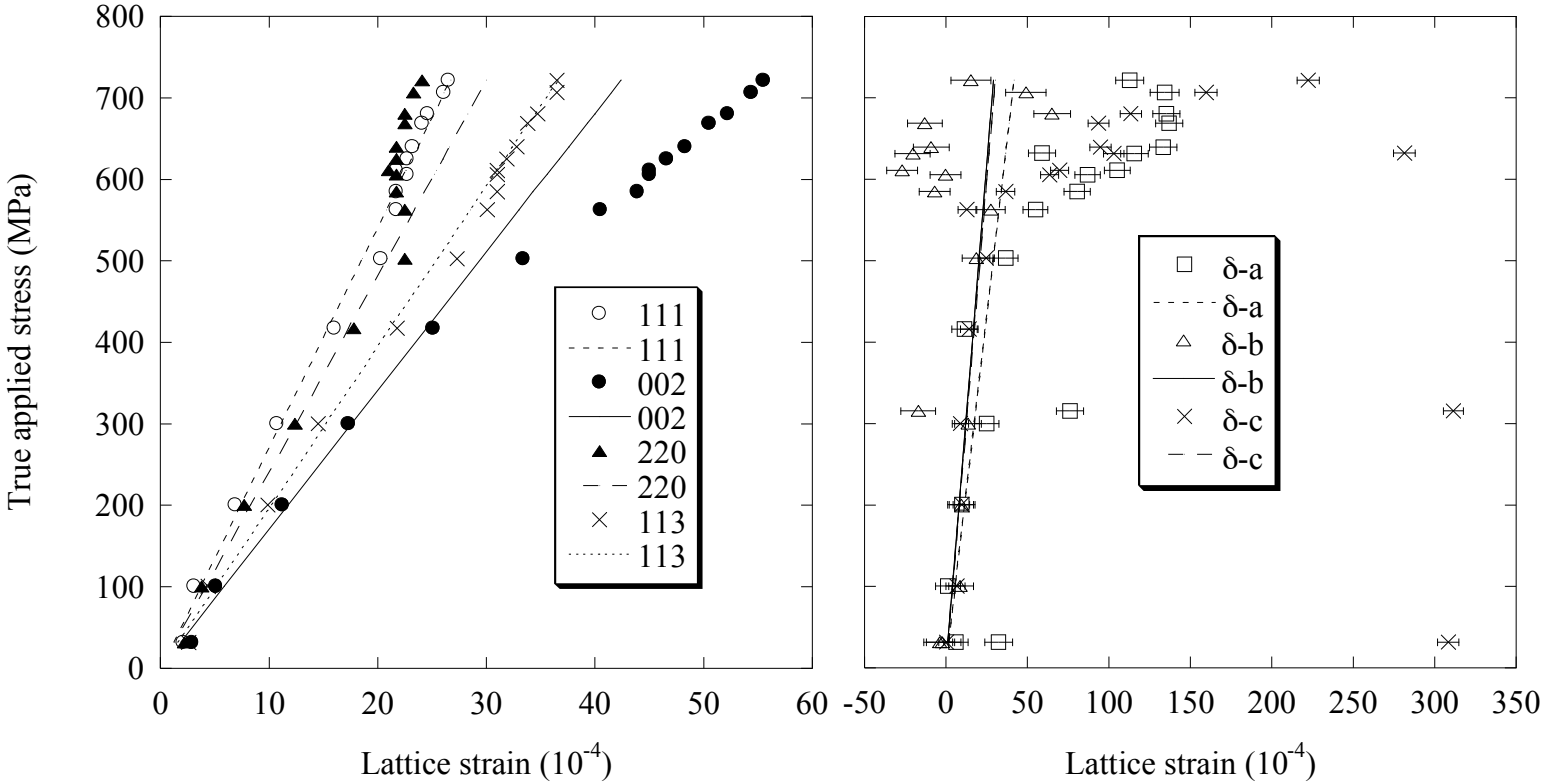
The lines are fits to the initial linear response. The δ-phase loads up with respect to the γ-phase as if the two were loaded in parallel with the δ-phase stiffer plastically. Note the huge magnitude of the strains in the δ-phase. The residual phase strain in the δ-phase is tensile and compressive the γ-matrix.

Parallel grain response of the γ-matrix and the three orthorhombic strains associated with the δ-phase at 20°C



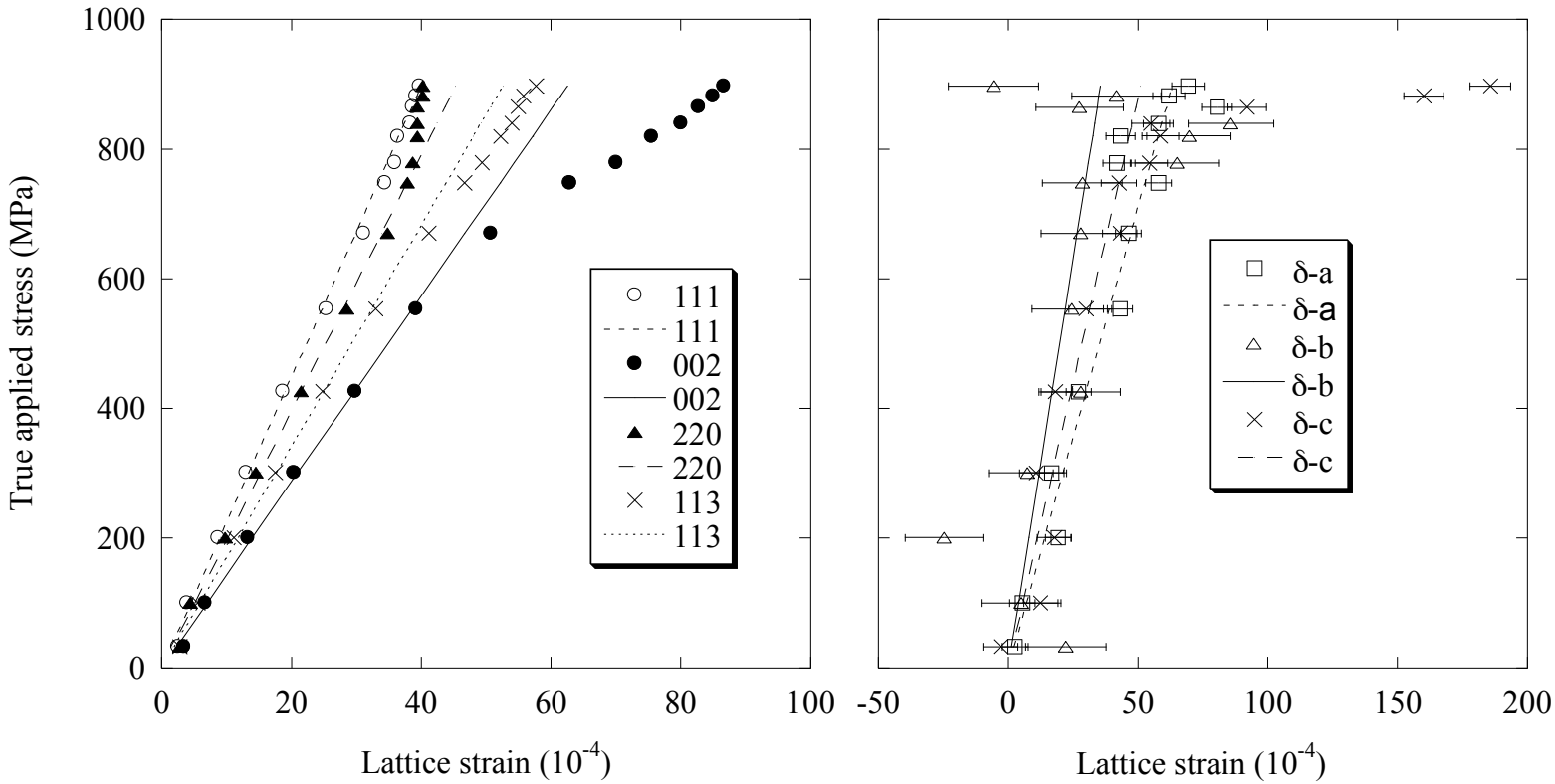
For the Ni matrix the {220} indicates slip for the matrix whereas the {002} loads up in the usual way for Ni. The a-strains of the δ-phase load up but the c-strains indicate slip. Departures from initial linearity occurs in both phases at nearly the same applied stress although the yield point must be slightly lower for the γ-phase. Note the far greater strains in the δ-phase

Parallel grain response of the matrix and orthorhombic strains in the δ-phase at 400°C



At 400°C the a-axis of the δ-phase initially takes on load but then appears to slip at 650MPa. The c-axis does not slip in this case but loads up and dominates the behaviour. This may indicate a change in the nature of the dislocations at 400°C so let us look at the slip systems.

Parallel grain response of the γ-matrix and the δ –phase at 650°C



At 650°C only the c-axis of the δ-phase takes on load. The other directions remain linear to within the assigned uncertainty. This is different to both lower temperatures. The response of the γ-matrix remains much the same at all temperatures as regards which directions indicate slip and which accumulate stress.

Slip systems in the γ- and δ-phases

- The slip system in the γ-phase is {111}<1-10>. From the orientation relationships one would expect that this would correspond to (010)[100] and in fact the [100] distance in the δ-phase and the [110] distance in the γ-phase are identical.
- Fortunately, Hagihara et al. have found all the slip systems in pure Ni_3Nb and so this gives us clues about the δ-phase. The four slip systems are
- (001)[100], (010)[100], (010)[001] and {201}<10-2>
- The critical resolved shear stresses for these show a strong temperature dependence

Slip systems in Ni3Nb(Hagihara et al.)

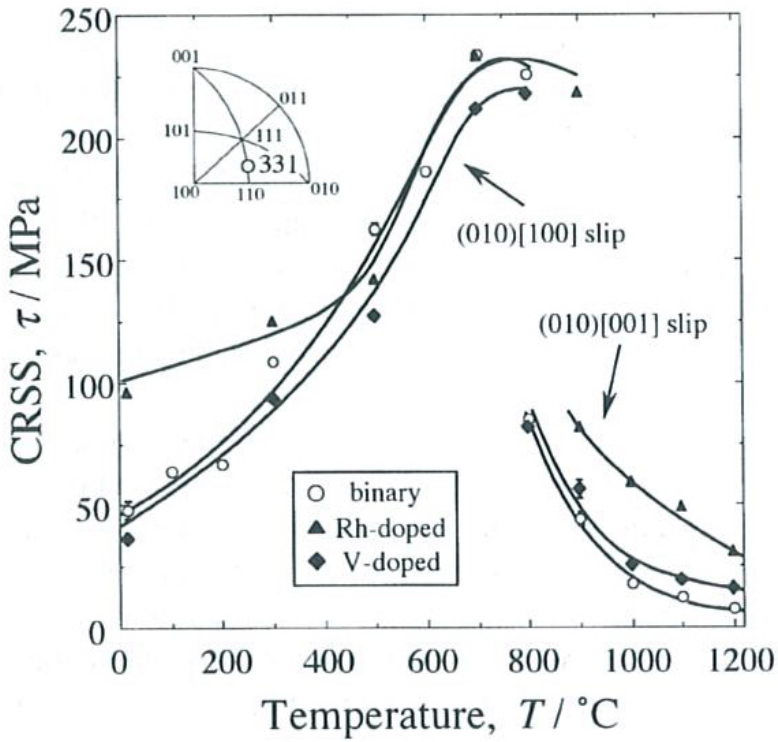
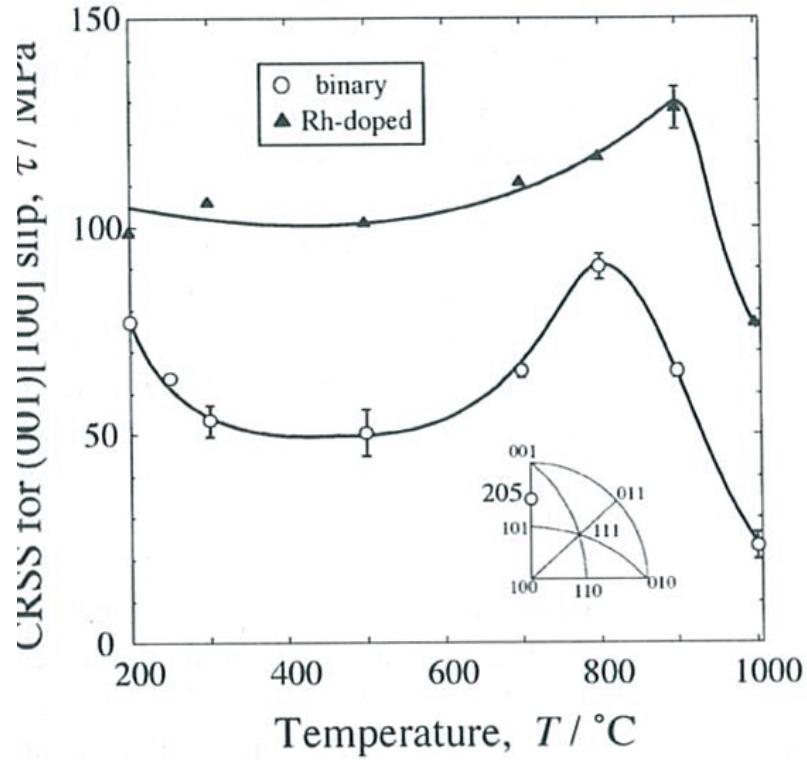


Fig. 6 Temperature dependence of CRSS for slip on (010)[100] and for slip on (010)[001] in V-doped, Rh-doped ternary and binary Ni_3Nb single crystals deformed in the [331]* orientation in compression.



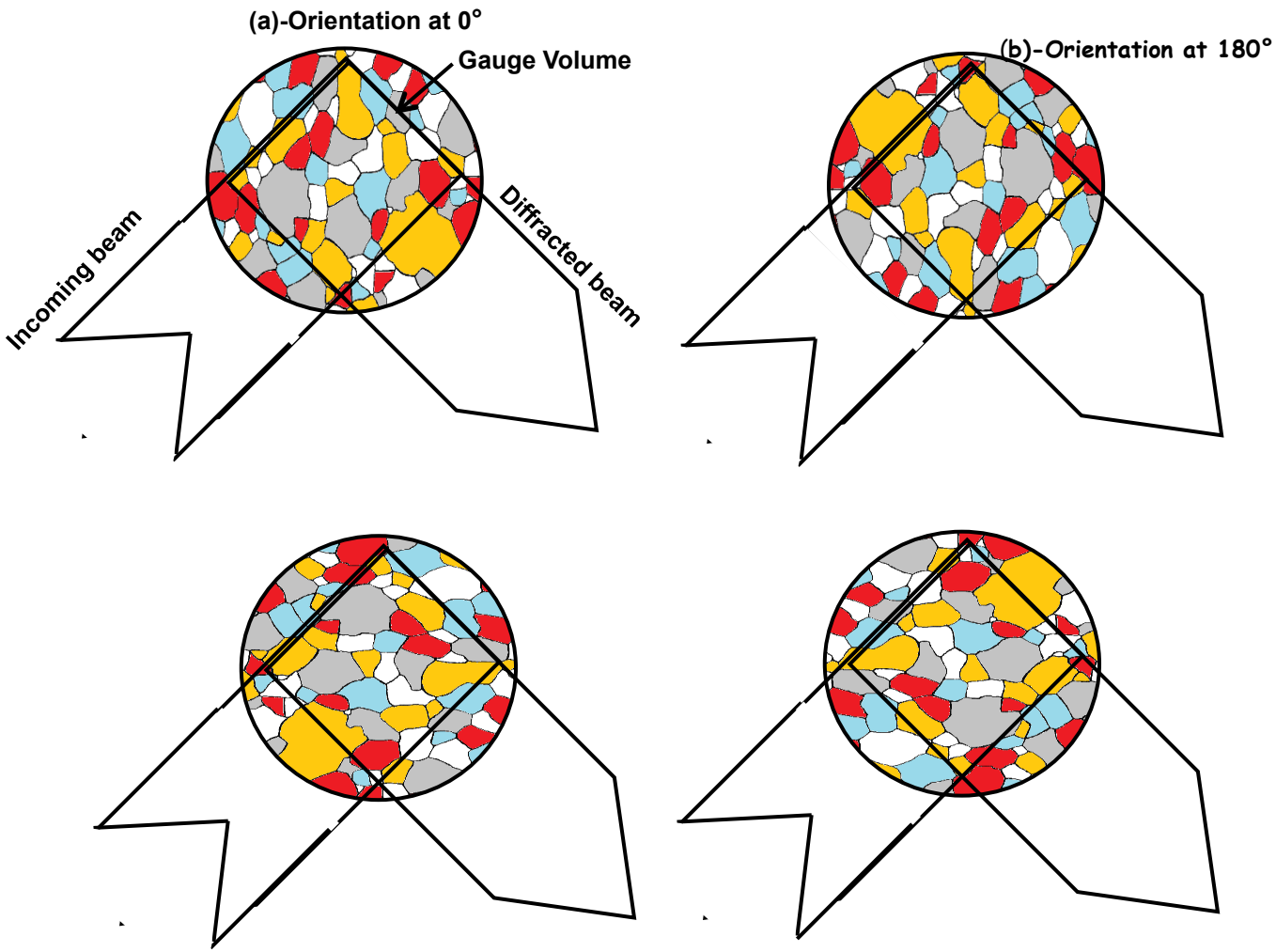
3 Temperature dependence of CRSS for slip on (001)[100] in -doped ternary and binary Ni_3Nb single crystals deformed in the [205]* entation in tension.

(010)[100] dominates at RT, (010)[001] dominates above 800C and (001)[100] can undercut (010)[100] at temperatures like 400°C. The fact that the critical resolved shear stress goes up with temperature is called Keir-Wilsdorf locking

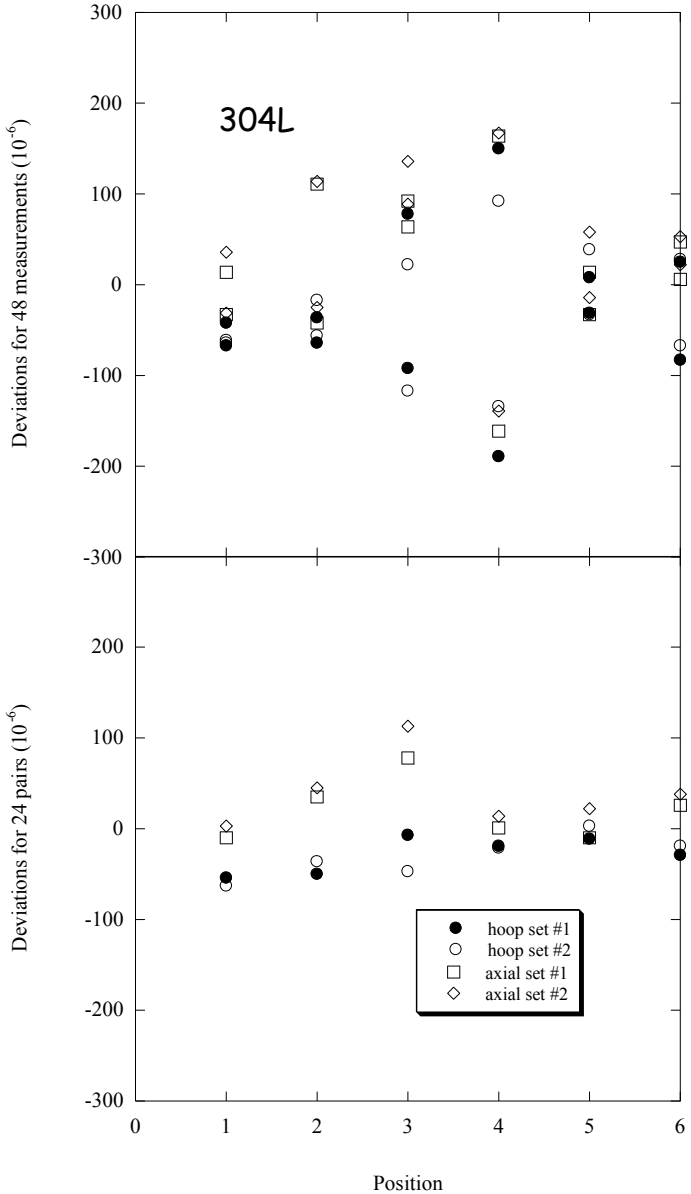
What have we found out about load-sharing in this composite?

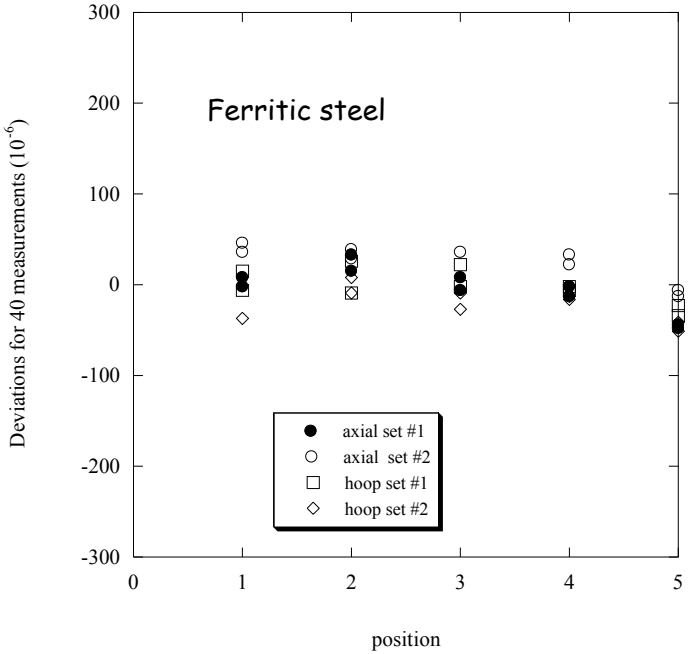
- The δ-phase loads up with respect to the matrix at all temperatures.
- Slip occurs both the matrix and the δ-phase as shown by the intergranular and orthorhombic strains.
- The orthorhombic strains in the δ-phase behave differently at the three temperatures possibly indicating a change in the nature of the dominant dislocation operating in the δ-phase
- The grain response of the Ni matrix stays much the same as to which grains appear to slip and which grains accumulate load as the temperature is raised although the stress level varies

Effect of 180° rotation on grain position in the gauge volume



Slide courtesy of Yeli Traore





Deviations from the average lattice parameter for 48 measurements and 24 pairs for 304L stainless steel. The average lattice parameter was 3.59500 Å and the standard deviations, expressed as a strain, were 84 and 42×10-6 respectively. The scatter and the standard deviation fall by about 50% on taking 180° pairs. Analogous data for small-grained ferrite

Conclusions

- Neutrons do have a major impact on important engineering topics because of the ability to measure stress at depth.
- Take very great care with reference lattice parameters. 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
- Be aware of the polycrystalline nature of engineering components and the impact of intergranular effects.
- Note the texture: It may give important clues as to the interpretation

Finale

- Best of luck in your careers as young scientists and engineers. 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!!
- Neutrons are “not the only game in town” with the advent of intense short-wavelength synchrotron x-ray beams and the other mechanical methods of measuring residual stress such as the contour method