Applications of neutron diffraction to engineering problems

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Concept

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

 $\lambda = 2d_{hk}/\sin\theta_{hk}$

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 ±0.01° in 20 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

 $\epsilon^{hk/}=(d^{hk/}-d^{hk/}_{0})/d^{hk/}_{0}$

- 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

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

writing this out for the 11 and 12 coordinates

$$\varepsilon_{11}^{hkl} = (\sigma_{11} - \nu^{hkl}\sigma_{22} - \nu^{hkl}\sigma_{33}) / E^{hkl}$$

$$\varepsilon_{12}^{hkl} = (1 + v^{hkl})\sigma_{12} / E^{hkl}$$

The E^{hkl} and v^{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

- 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 modelling. Surely we ought to be able to calculate weld stresses. But, the models have to be benchmarked against experiment otherwise it is "garbage in=garbage out"

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 tube texture this gave a high strain in {0002} planes which is where hydrogen Prefers to sit in the lattice. In practice this gave severe hydride cracking problems





FITTING (CRACKS IN AREA 'A')



ENLARGED VIEW OF AREA 'A'



VIEW OF CRACK SURFACE

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
 - Direct toughness measurement



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

Response of Grain Orientations

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 <u>macroscopic field</u> 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.

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 texture this gave a high (0002) strain which is where hydrogen sits in the Lattice. In practice this gave severe hydride cracking problems



Over-rolled CANDU pressure tube

- The hoop strain is shown for the intact tube constrained by the end-fitting as well as 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 constraint caused 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 is an intergranular strain on the scale of the grains where the plastic deformation had occurred



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 average stress 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. The scale is the size of the grain
- The intragranular stress varies within the grain, around defects or near grain boundaries

Examples 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 (1)

- These are stresses which 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 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
- The intergranular stresses also 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 actually bias the measurement of strains. 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.

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



So how do we tackle a problem like 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 stress field but it leaves intergranular grain-scale stresses/strains, or effects due to chemistry changes on the lattice parameter, unchanged.)
- Check for changes in lattice parameter upon melting!
- 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!



Distance from weld centre (mm)

Reference lattice parameters derived from small coupons cut from the 31655 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

Hexagonal close-packed Zr-4 weld joining two plates

- Fairly isotropic intrinsic elastic constants
- Anisotropic plastic deformation (prism, basal, and pyramidal slip and tensile twinning)
- Coefficients of thermal expansion quite different in the *a*- and *c*-directions so expect thermal intergranular strains
- Strong parent rolling texture. Strong texture gradient through the weld.
- Small grain size.
- Small reference coupons cut from a companion weld
- Measurements made on the SMARTS diffractometer at Los Alamos of all (hkil) reflections permitted by the rolling texture.







Reference lattice spacings for {10.1} planes from small coupons cut from a companion weld. The results are presented as the fractional change with respect to material far from the weld. Different behaviour is seen in the three sample directions. The results are not consistent with chemistry changes but with intergranular strains from cooling a weld sample with strong rolling texture in the parent and weak solidification texture in the weld as shown by the EPSC result



There are two ways of proceeding. 1 Assume single values for the normal, transverse and longitudinal stress at each location and fit to all the measured strains at each location (about 60 in all three directions together) using the appropriate elastic constants. The uncertainty is derived from the fitting process and the errors in the strains. 2 Take those reflections which can be measured for all three sample directions at a particular location and solve for the stresses. 2. Do the two

methods of getting the stress agree? Yes! Doing science with the intergranular strains .In-situ response perpendicular to the rod axis for rod-textured Zr-2 in compression and tension

In <u>tension</u> the <10-10> grains show PR slip and <0002> grains accumulate large tensile strains which are eventually limited by PY slip.

- In <u>compression</u> <10-10> grains show PR slip at a higher applied stress than tension because of the thermal strains. Initially <0002> accumulates residual strain. At -340MPa tensile twinning initiates and limits the <0002> strain dramatically
- The results allow us to get estimates of the critical resolved shear stresses of the slip and twinning systems



What does the future hold with new sources of unprecedented intensity such as VULCAN at SNS or TAKUMI at J-PARC?

- 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 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
- (d) biomaterials
- (e) measurement of all elements of the stress tensor

Simulations vs. Neutron Measurement D2, D5, D9, D16 lines



D2 line









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

Extensions of present science

- 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 materials such as PbMnNbO₂ 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



In-situ investigation of chill casting

- The region of interest is the "mushy zone" near the interface between solid and liquid
- (Dan Meier and Chad Sinclair UBC)





Conclusions

- Neutrons can have a major impact on important engineering topics because of the ability to measure stress at depth.
- Be aware of the polycrystalline nature of engineering components and the impact of intergranular effects.
- 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
- Note the texture: It may give important clues as to the interpretation
- Good luck in your careers as young scientists!

Choice of reference lattice spacings, d_{hkl}^{0} , part1

- This is where things can really go wrong before you even begin!
- Cut a small coupon from a companion sample. Cutting destroys the stress field and hence the associated strains. However, because of their spatial scale the intergranular strains are not relieved.
- Note that the reference coupon will probably not be strictly strain/stress free. The lattice spacing $d_{hk/}^0$ will probably be different in the three directions. Thus $d_{hk/}^0$ in direction 11 should be compared with the $d_{hk/}$ in direction 11 in the intact sample, etc.
- In a weld one should always cut coupons from the whole width of the weld, since welding changes the microstructure and can bring solute atoms in and out of solution. The chemical variation of d_{hkl}^0 will generally be more significant than that due to strain. Aaron Krawitz was the first to point this out.

Choice of reference lattice spacings, d_{hkl}⁰, part2

- If one cannot cut the sample it is probably best to do the d_{hkl} measurement at a location where the stress is expected to be small or zero.
- One can use stress balance to derive d_{hkl}^{0} . However, it is better to have a value of d_{hkl}^{0} and check stress balance when you have the stress field!
- Choose reflections for which the type-2 strains are expected to be small, say 111 and 113 for fcc, 110 or 112 for bcc. This approach is not rigourous but only empirical since it can depend on the texture. For hexagonal close-packed metals there are no such reflections.
 - Be careful about annealing. This can easily change the microstructure and lead to chemistry changes in solute elements.

Big grains: causes and effects



- Philosophical: not a good
 sampling of grains from the
 distribution
- Big grains are offset from the centre of the gauge volume
- Indicator: strong intensity changes from point to point or with sample angle proportional to \sqrt{N}
- Large scatter in the strain data