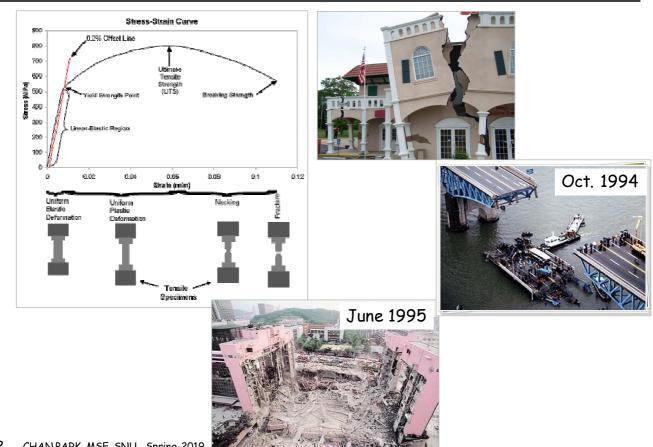
residual stress analysis using XRD $(sin^2\psi \text{ method})$

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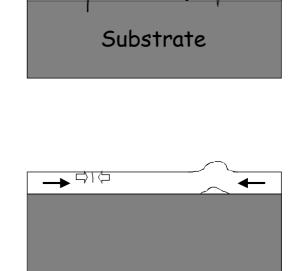
Stress (Engineering)

> Tensile (+) stress

✓ Leads to cracking and crack growth

- > Compressive (-) stress
 - ✓ Good, can close cracks

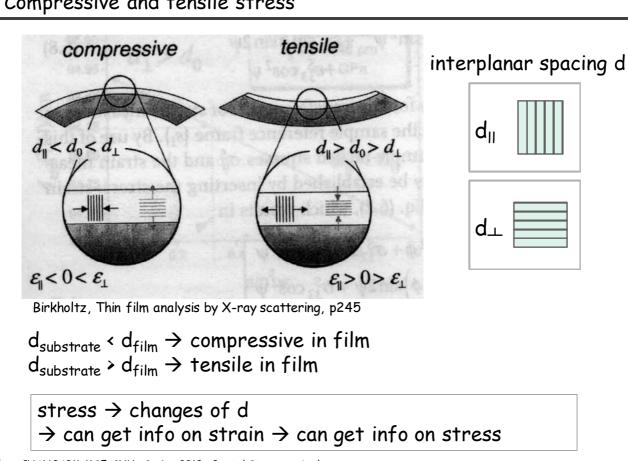
 \checkmark Too high \rightarrow buckling



coating

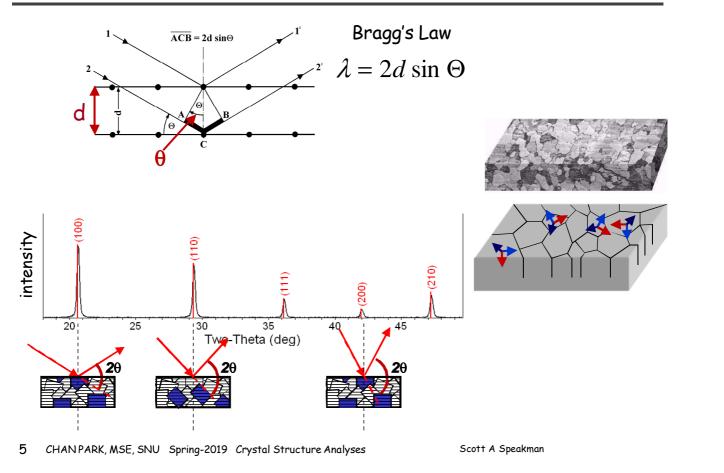
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Panalytical

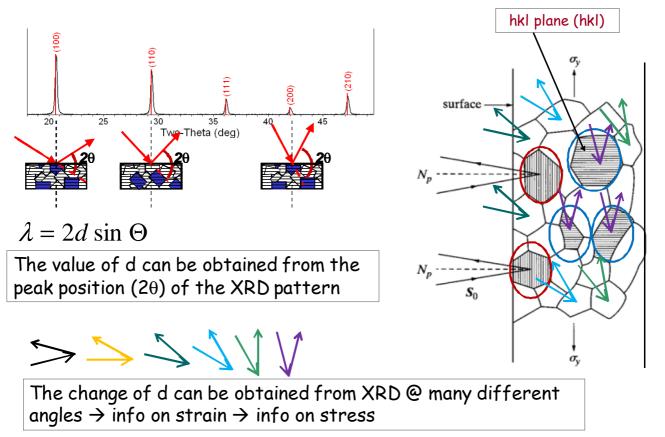


Compressive and tensile stress

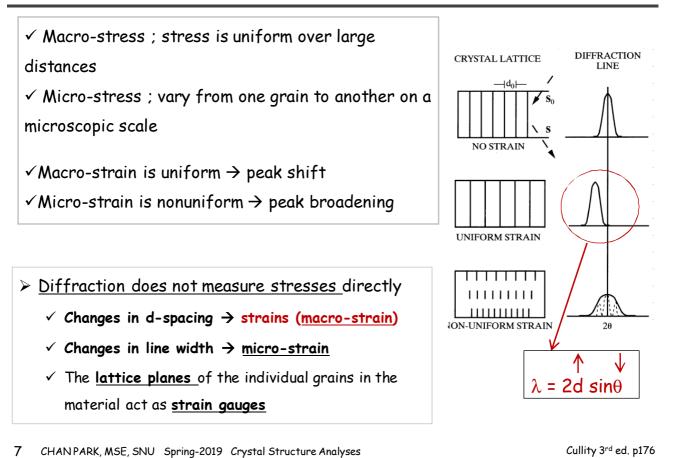
X-ray diffraction



X-ray diffraction



Macro-stress & Micro-stress



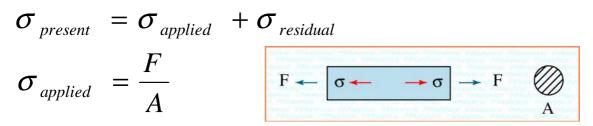
Applied stress & Residual stress

- Stress = applied stress + residual stress
- > Applied stress ; any externally applied load

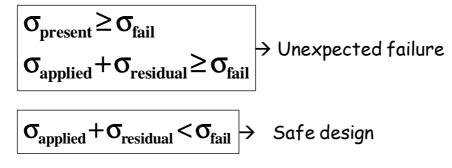
Residual stress ;

- ✓ stress existing in a solid body in the absence of applied force
 - Typically caused by forming or heating (mechanical working, differential thermal expansion)
 - Especially welding, casting, forging, rolling, machining, cooling, etc.
 - Important in Fatigue Life, Corrosion Resistance, Dimensional Stability, Brittle Fracture, Distortion
 - Can be found in metals, ceramics, biological materials, composites, films everything
- ✓ can affect material performance
- ✓ can be beneficial or detrimental
 - Residual Surface Stress (e.g. in toughened glass)
 - Stress corrosion cracking
- We can't measure stress directly, only strains

tension or compression which exists in the bulk of a material without application of an external load

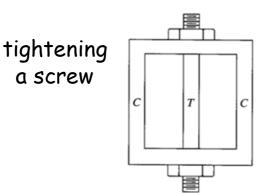


When F = 0 (no external force), $\sigma_{\it present} = \sigma_{\it residual}$

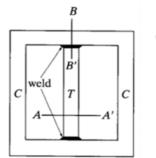


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Residual stress

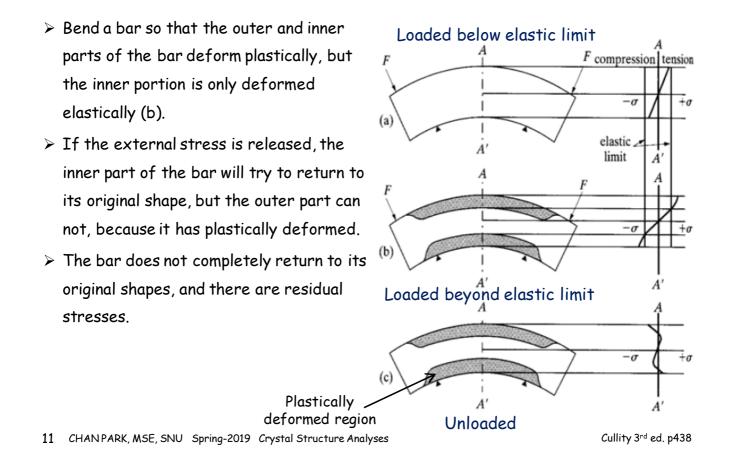


- When the nuts on the central bar are tightened, the bar is put into tension and the outer frame into compression
- There is no external load but the components are stressed

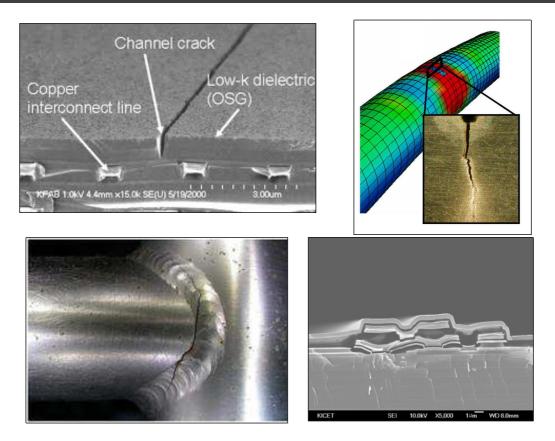


welding

- During welding the central bar undergoes thermal expansion
- On cooing, this leaves the bar under tension and the outer frame under compression



Residual stress - no external forces



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>Intrinsic stress

- ✓ Stress developed during film deposition
- ✓ Misfit strain
- ✓ Microstructural change (e.g. grain growth)
- ✓ Phase transition (due to differences in density)

> Extrinsic stress

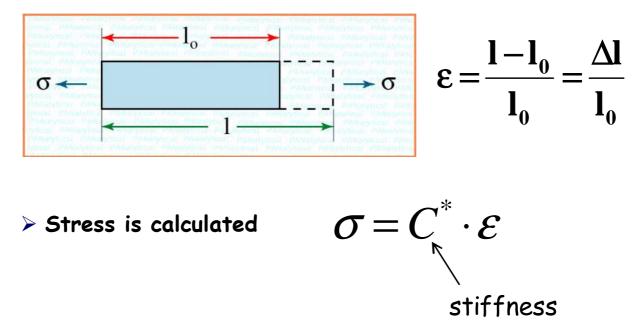
✓ Thermal stress (due to difference of CTE b/w film and substrate)

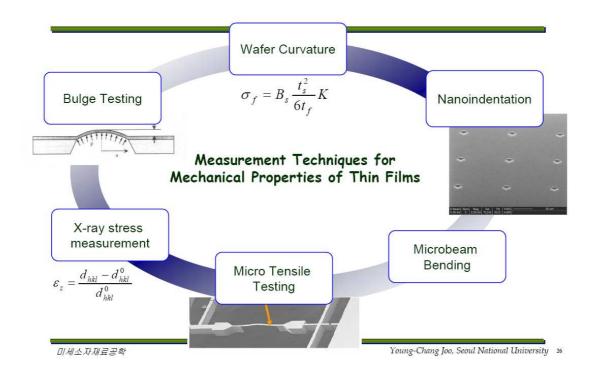
CTE; coefficient of thermal expansion

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How to measure stress ?

> Only strain can be measured



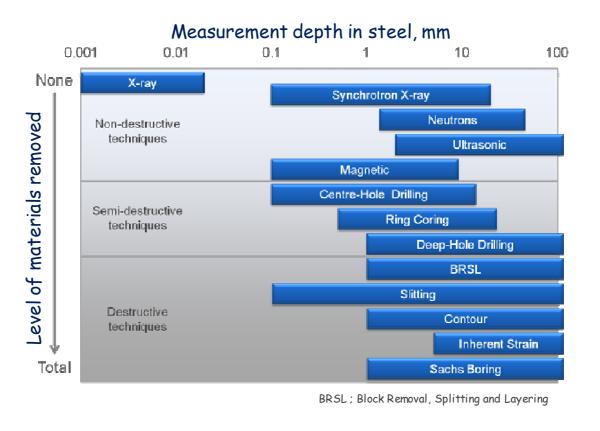


Residual Stress Measurement

- > Mechanical methods
 - ✓ Hole-drilling technique
 - ✓ Deep hole
 - ✓ Sectioning
 - ✓ Contour
 - ✓ Excision, Splitting, Curvature, Layer removal, Slitting, etc.
- > Diffraction methods
 - ✓ <u>X-ray diffraction</u>
 - ✓ Synchrotron X-ray diffraction
 - \checkmark Neutron diffraction

Non-destructive methods have an advantage ← measurements can be repeated at will and further data can be collected

- > Magnetic Barkhausen noise method
- Ultrasonic method
- > Thermoelastic, Photoelastic (birefringent), Indentation



www.veqter.co.uk/residual-stress-measurement/overview

RS measurement techniques

Technique	Advantage	Disadvantage	
X-ray diffraction	Versatile, Widely available, Portable, Wide range of materials, Macro and Micro RS	Lab-based systems, Small components, surface stress measurement	
Synchrotron XRD	Improved penetration & resolution of X-rays, Depth profiling, Fast, Macro and micro RS	Special facility needed, Lab-based systems	
Neutron Diffraction	Optimal penetration & resolution, 3D maps, Macro and Micro RS	Special facility needed, Lab-based system	
Hole Drilling	Fast, Easy use, Widely available, Hand-held. Wide range of materials, Deep hole drilling for thick section components	Destructive, Interpretation of data, Limited strain sensitivity and resolution	
Sectioning	Wide range of material, Economy and speed Hand-held	Destructive, Interpretation of data, Limited strain resolution	
Contour	High-resolution maps of the stress normal to the cut surface, Hand-held, Wide range of material, Larger components	Destructive, Interpretation of data, Impossible to make successive slices close together	
Barkhausen Noise	Very fast, Hand-held, Sensitive to microstructure effects especially in welds	Only ferromagnetic materials, Need to divide the microstructure signal from that due to stress	
Ultrasonic	Widely available, Very fast, Low cost, Hand- held	Limited resolution, Bulk measurements over whole volume	
Raman/Fluor escence	High resolution, Portable systems	Surface measurements, Interpretation, Calibration, Limited range of materials	

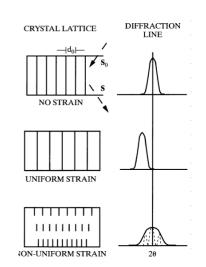
- Diffraction methods offer a <u>nondestructive</u> method for evaluating stress and residual stress in a material
 - ✓ Understanding residual stress is important as it is not just the external stress that determines when a material will fail
- > Alternative methods are destructive
- Diffraction can be used to examine stresses in multiphase materials and how they are partitioned between phases
 - ✓ Useful in composites to understand e.g. how a fiber reinforcement is performing
- ➤ Modern X-ray methods allow measurements on a micron length scale → stress distributions can be mapped out

Stress measurement by diffraction

Diffraction techniques do not measure stresses

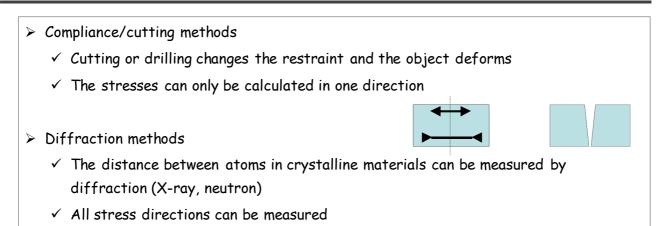
in materials directly

- \checkmark Changes in d-spacing \rightarrow strains
- \checkmark Changes in line width \rightarrow microstrain
- The lattice planes of the individual grains in the material act as strain gauges



To get an estimate of the stress in a part of the diffraction, measurement must be calibrated or a calculation must be performed

RS Measurement > cutting vs. diffraction, diffraction vs. strain gauge



- > Diffraction methods
 - ✓ Measured lattice strains are "absolute quantities" relative to a zero-strain data
 - ✓ Allows RS as well as applied stress to be measured
- > Strain gauge
 - ✓ Can only measure the <u>strain difference</u> between the initial condition when the gauge was attached and some subsequent condition
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RS Measurement > XRD

> Advantages

- ✓ <u>Non-destructive</u>; <u>Widely available</u>; Macro and micro stresses can be measured;
- ✓ Laboratory or "on-site" measurements; <u>Bi-axial residual stress</u> measurements;
- \checkmark Small gauge volume \rightarrow great for measuring <u>surface stress gradients</u>;
- ✓ High magnitude residual stresses are measured <u>accurately;</u>
- ✓ Complex shapes can be measured providing rotation of the measuring head is not restricted;
- ✓ Very <u>quick and easy</u> to apply the process, and therefore <u>cheap</u>

Disadvantages

- ✓ Measurement depths of only 10-20µm as standard,
 - when coupled with electro-polishing, surface removal depths of up to 1-1.5mm are achievable;
- ✓ Only applicable to <u>polycrystalline</u> materials;
- ✓ Accuracy seriously affected by grain size and texture;
- ✓ A good component <u>surface finish</u> is essential, so may need delicate <u>preparation</u>.

www.veqter.co.uk/residual-stress-measurement/x-ray-diffraction

- Conventional XRD
 - ✓ Penetration depth ~ 10s of um
 - → <u>surface stress measurement</u>
 - → irradiated volume can be considered to be in a state of plane stress (biaxial stress)
 - ✓ <u>Simple</u> stress-strain <u>equation</u>, <u>no need</u> for precise determination of <u>stress-</u> <u>free lattice plane dimension</u>
- > Synchrotron XRD
 - ✓ Penetration depth ~ 100s of mm
 - \rightarrow irradiated volume can not be considered to be in a state of plane stress.
 - \rightarrow full 3-Dim stress condition must be considered.
 - → <u>need</u> to have precise value of <u>stress-free lattice plane dimension (major</u> source of error)

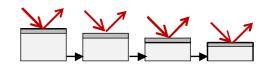
Neutrons vs. X-rays

- > Absorption is not such a big issue for neutrons.
- > You can make measurements inside components.

	neutron		X-ray	
Ζ	μ <mark>(cm⁻¹)</mark>	† _{50%} (mm)	μ <mark>(cm⁻¹)</mark>	t _{50%} (micron)
Al	0.10	69.3	131	52.9
Ti	0.45	15.4	938	7.39
Fe	1.12	6.19	2424	2.86
Ni	1.86	3.73	407	17.0
W	1.05	6.60	3311	2.09

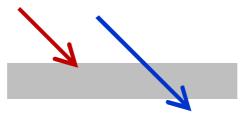
RS Measurement > X-ray & Neutron diffraction

- X-ray strain measurement provides information on the surface of a material.
 - \checkmark Surface information is important as failure often starts at the surface.
 - ✓ Info from the inside can be obtained.



 Removing the surface layer can destroy the specimen, and the relaxation can change the residual stress.

> Neutron diffraction can be used to make measurements inside a part.

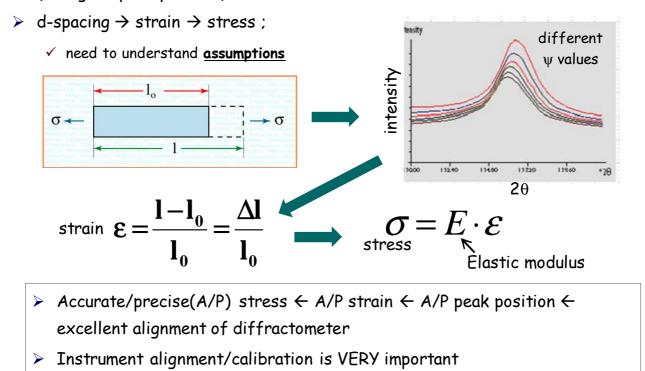


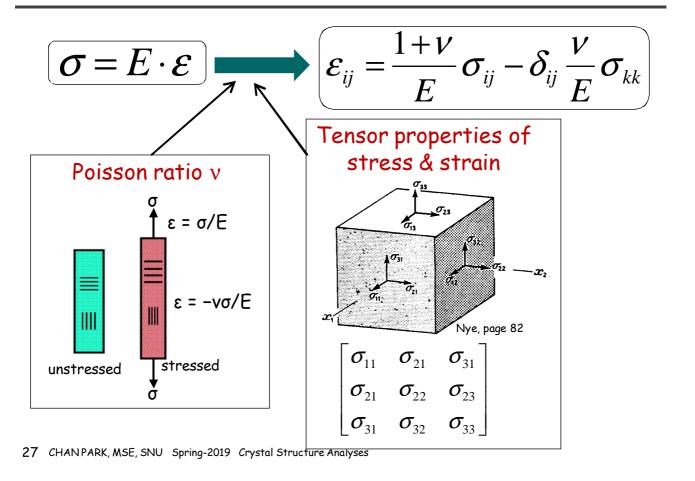
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Krawitz

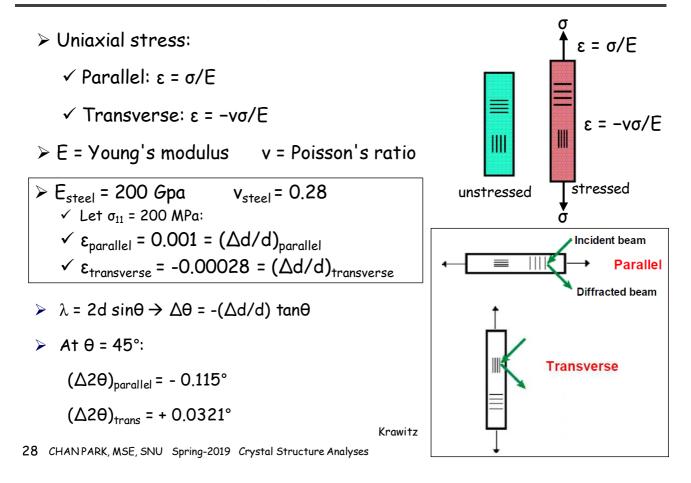
How to measure stress using XRD?

 Diffraction does not measure stress or strain → gives the changes in d-spacing (change in peak position)

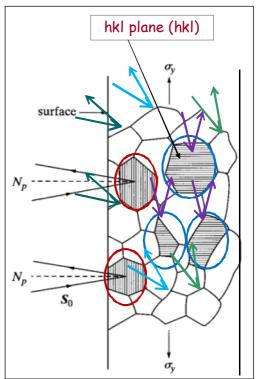


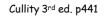


Uniaxial stress on a bar specimen

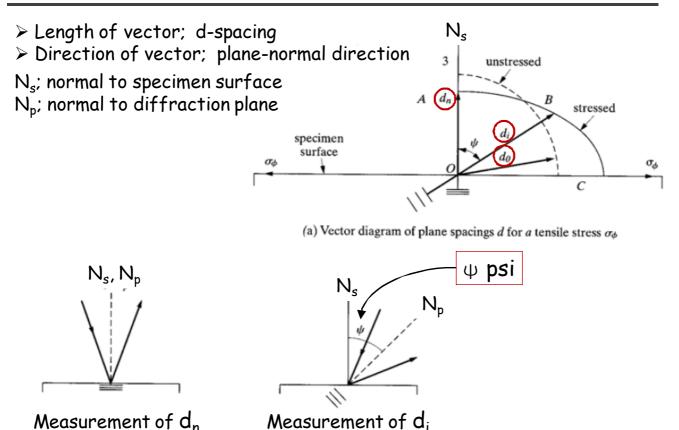


- When the d-spacing of a reflection is measured, <u>only grains with the planes</u> <u>oriented in a given direction</u> contribute to diffraction.
- If we <u>change the orientation of the</u> <u>specimen and re-measure the d-spacing</u>, we are looking at a different population of grains and we get a <u>different d-spacing due</u> <u>to different stress levels</u>.



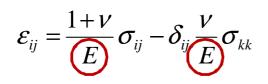


Orientation dependence of d-spacing

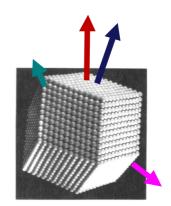


Elastic constants

- > The elastic constants (E in $\sigma = E \cdot \varepsilon$) used in the stress calculation should be obtained from diffraction measurements on reference materials or by using values mechanically measured in different directions on single crystal specimens.
 - ✓ Individual crystallites are not elastically isotropic.
 - ✓ Young's modulus and Poisson's ratio for the (111) reflection will not in general be the same as those e.g. for the (110) reflection.



 E can also change when the stress state changes



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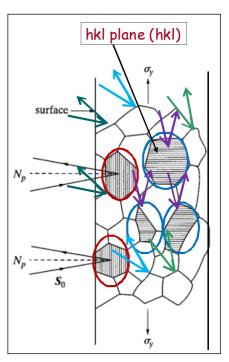
Krawitz

Stress free reference

> Strain is obtained from a diffraction measurement

using $\epsilon = (d_{\psi\phi} - d_0)/d_0$

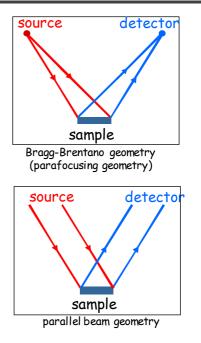
- \checkmark d_{wo} ; measured d-spacing in some direction $\psi \phi$
- \checkmark d₀ ; d-spacing for the stress free material
 - It is very difficult to get d₀ directly ← to prepare
 a stress free piece of material with exactly the
 same composition is very difficult.
 - Using a similar piece of material may not be good enough as we are trying to measure very small changes in d-spacing.
- We can sometimes avoid the need to measure d₀ by making diffraction measurements at several angles (ψ).



Optics & sample preparation

- Using a divergent beam is not desirable. The instrument needs constant realignment.
- Parallel beam optics are the way to go.

 sample displacement and focusing are not issues.



- Sample preparation
 - ✓ Smooth clean surface
 - The polishing of the surface can change the stresses that you wish to measure !

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Stress as a function of depth

- > To measure residual stress as a +100function of depth using X-rays, you +80may need to carefully remove some +60of the surface. +40**RESIDUAL STRESS (ksi)** +200 2 3 -20DEPTH BELOW SURFACE (10-3 IN.) > The stress relief that occurs will -40Dissection method have to be accounted for. X-ray method -60-80
 - -100 Residual stress in ground steel close to the surface

> Sample preparation

- ✓ Smooth clean surface
- ✓ However, if you try to polish the surface, you will change the stresses that you wish to measure !!!
- Portable instrument
 - ✓ In case one needs to measure surface residual stress in large components, it is possible to buy small mobile diffractometers that can be moved to the specimen and mounted on the specimen surface.

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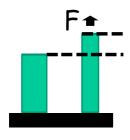
Practical problems

> Stress measurement uses positions of powder diffraction lines (d-spacing).

- If the sample contains very large grains, you do not really have powder lines, just single crystal spots. It can be very difficult to accurately estimate the <u>position of a powder line</u> under these conditions.
- > <u>Texture</u> can lead to very low intensity for some sample orientations.
- ➤ Highly textured bodies may not be elastically isotropic → some of the assumptions (isotropic, biaxial stress) that go into the basic theory for converting the strain measurements to stress tensor components in sin²ψ method, <u>fail</u>.

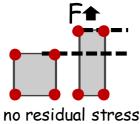
Krawitz

Residual stress



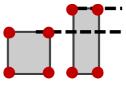
Deformation of a rod induced by external force

deformation \rightarrow strain \rightarrow displacement in the body relative to a reference length



Deformation of simple cubic induced by external force deformation \rightarrow strain \rightarrow displacement relative to

inter-atomic length

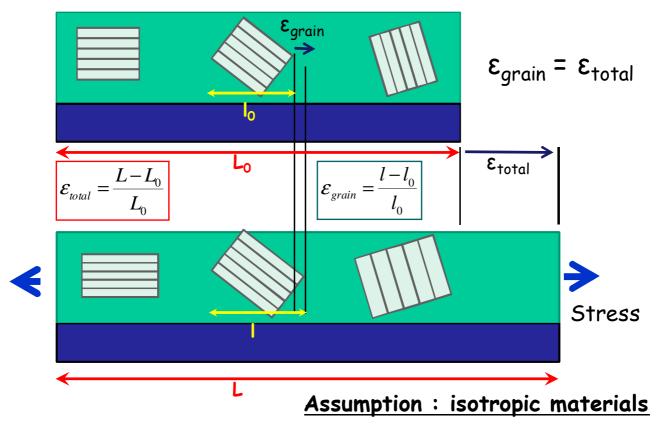


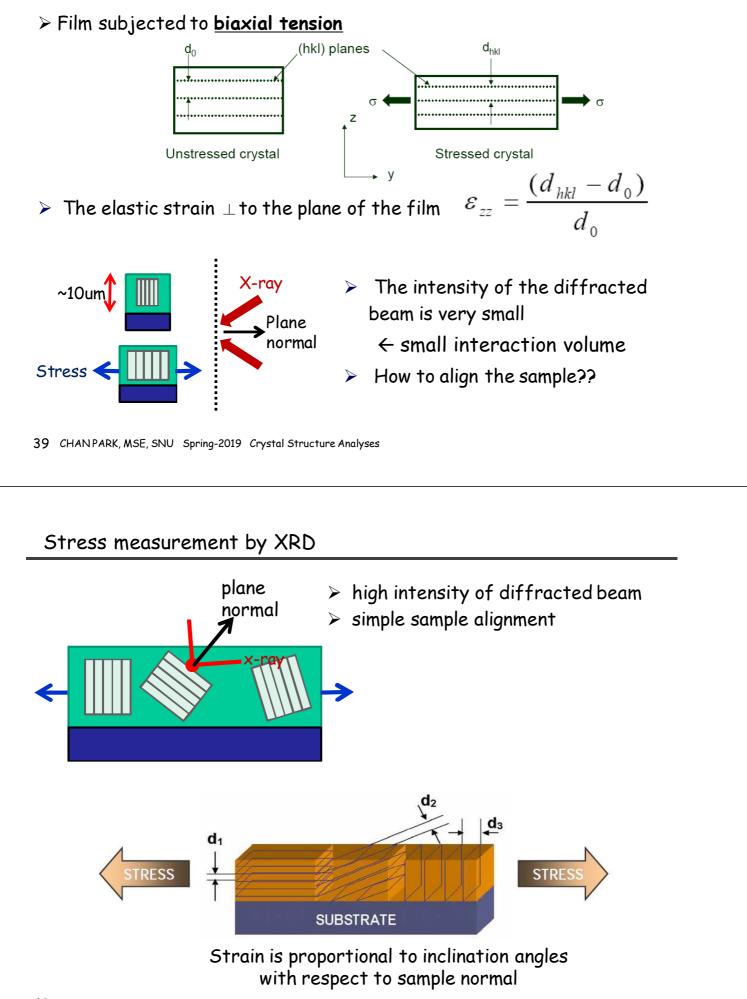
Deformation of simple cubic induced by residual stress Residual stress can be calculated from displacement Displacement of atoms → change of lattice parameter

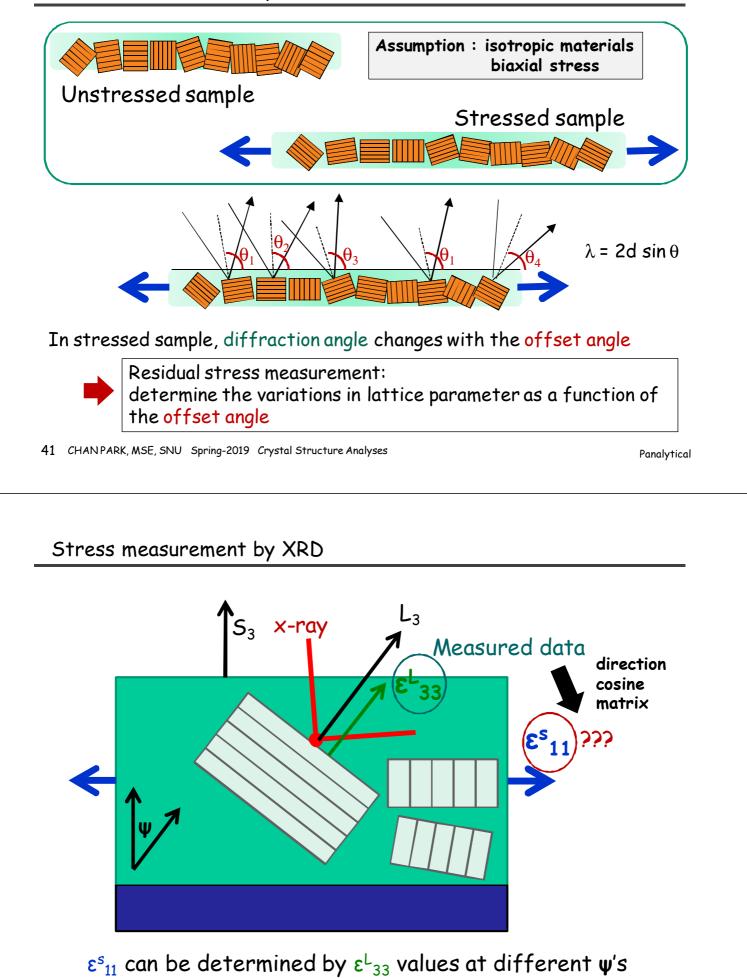
no applied stress

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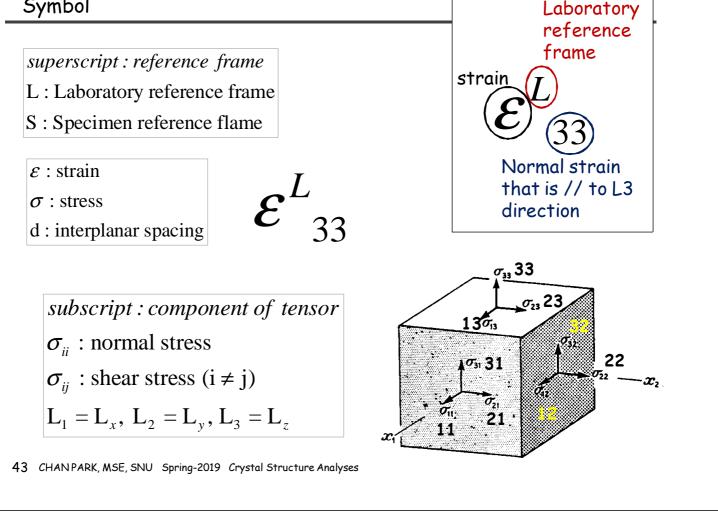
Residual stress measurement



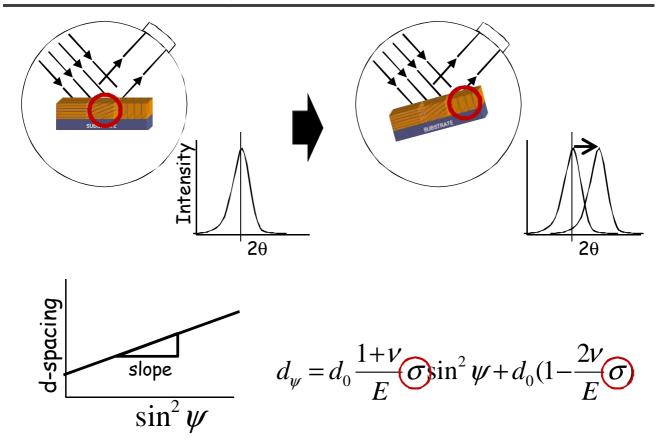




Symbol



Stress measurement by XRD



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- 2-theta (20) The Bragg angle, the angle between the incident (transmitted) and diffracted X-ray beams
- Omega (ω) The angle b/w the incident X-ray beam and the sample surface. Both ω and 2θ lie in the same plane
- > Phi (ϕ) The angle of rotation of the sample about its surface normal
- > Chi (χ) Chi rotates in the plane normal to that containing ω and 2 θ . This angle is also sometimes (confusingly)referred as ψ

> Psi (ψ)

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Angles

> 2-theta (2θ), Omega (ω), Phi (φ), Chi (χ)

► Psi (ψ) - Angle through which the sample is rotated, in the sin² ψ method. We start at ψ = 0, where ω is half of 2 θ and add (or subtract) successive ψ offsets. For example, 10, 20, 30 and 40°.

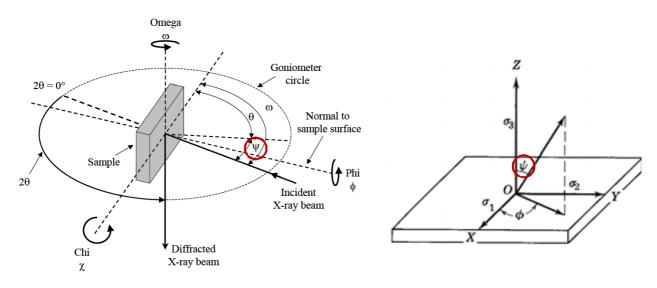
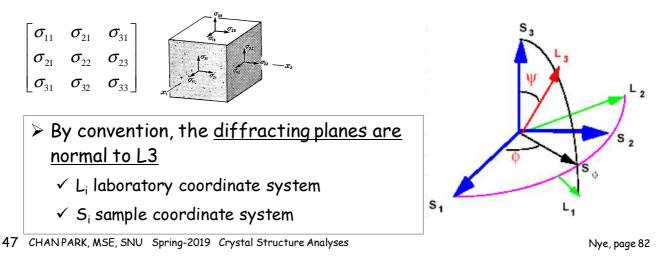
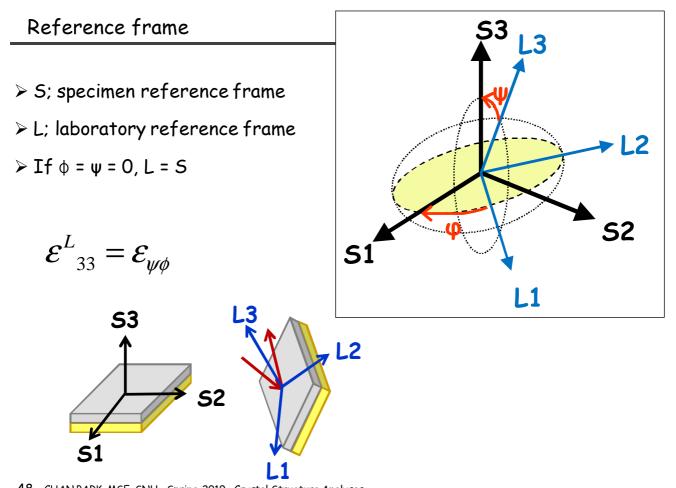


Figure 6.3 Angles and rotations used in residual stress measurement (For a horizontal system with a positive psi offset.)

➤ possible states

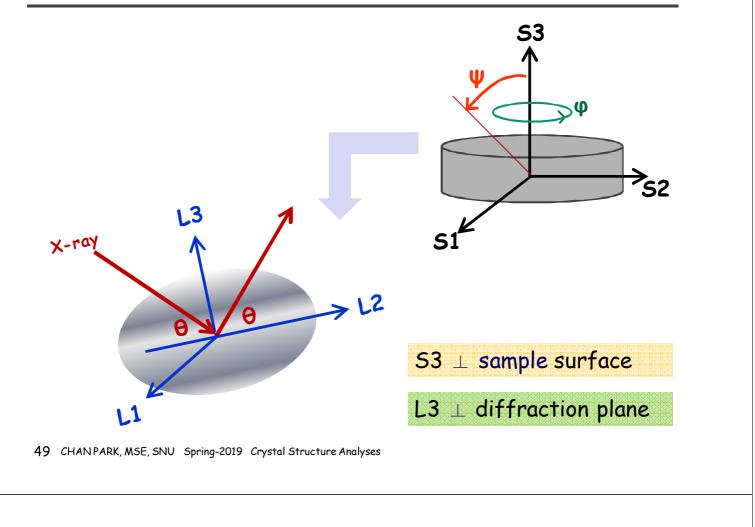
- ✓ 3 unequal principal stresses (σ_1 , σ_2 , σ_3) → Triaxial state of stress
- ✓ 2 out of 3 principal stresses are equal (say σ_1 , $\sigma_2 = \sigma_3$) → Cylindrical state of stress
- ✓ All 3 are equal (say $\sigma_1 = \sigma_2 = \sigma_3$) → Hydrostatic/spherical state of stress
- ✓ 1 of 3 is zero (say $\sigma_1, \sigma_2, \sigma_3 = 0$) → Biaxial/2D state of stress
- ✓ 2 of 3 is zero (say $\sigma_1, \sigma_2 = \sigma_3 = 0$) → Uniaxial state of stress



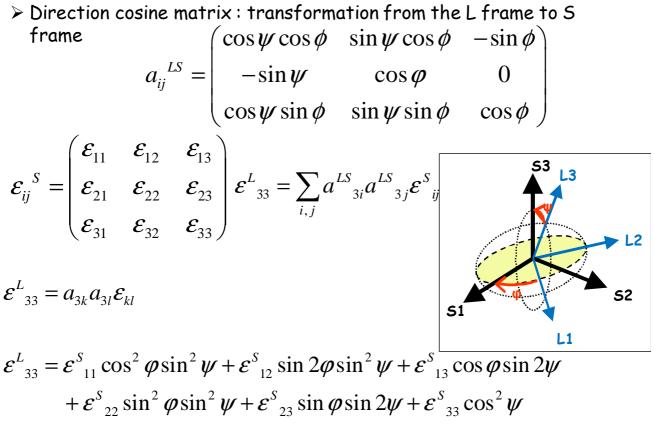


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Reference frame



Translation $L \rightarrow S$

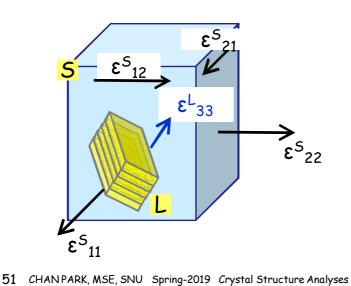


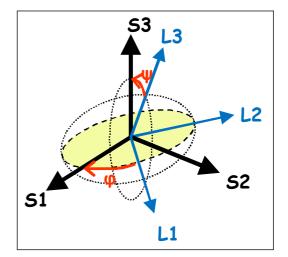
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Translation $L \rightarrow S$

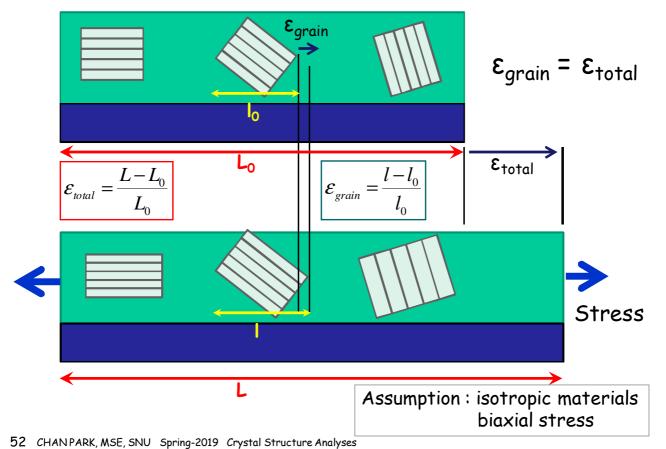
$$\varepsilon^{L}{}_{33} = \varepsilon^{s}{}_{11}\cos^{2}\phi\sin^{2}\psi + \varepsilon^{s}{}_{12}\sin 2\phi\sin^{2}\psi + \varepsilon^{s}{}_{13}\cos\phi\sin 2\psi + \varepsilon^{s}{}_{22}\sin^{2}\phi\sin^{2}\psi + \varepsilon^{s}{}_{23}\sin\phi\sin 2\psi + \varepsilon^{s}{}_{33}\cos^{2}\psi$$
(1)

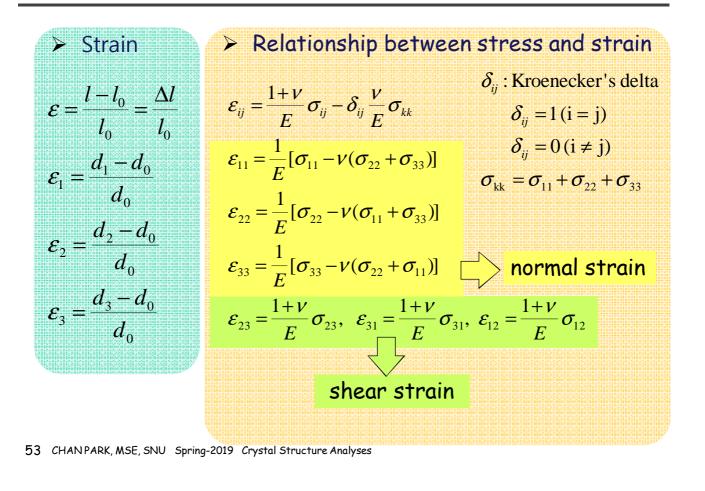
strains measured in L frame (diffraction plane) \rightarrow strains in the S frame (sample)





Residual stress measurement





Hooke's Law

$$\varepsilon^{L}{}_{33} = \varepsilon^{s}{}_{11}\cos^{2}\phi\sin^{2}\psi + \varepsilon^{s}{}_{12}\sin 2\phi\sin^{2}\psi + \varepsilon^{s}{}_{13}\cos\phi\sin 2\psi + \varepsilon^{s}{}_{13}\cos\phi\sin 2\psi + \varepsilon^{s}{}_{22}\sin^{2}\phi\sin^{2}\psi + \varepsilon^{s}{}_{23}\sin\phi\sin 2\psi + \varepsilon^{s}{}_{33}\cos^{2}\psi \qquad (1)$$

$$\varepsilon_{ij} = \frac{1+\nu}{E}\sigma_{ij} - \delta_{ij}\frac{\nu}{E}\sigma_{kk} \quad \text{Hooke's Law}$$

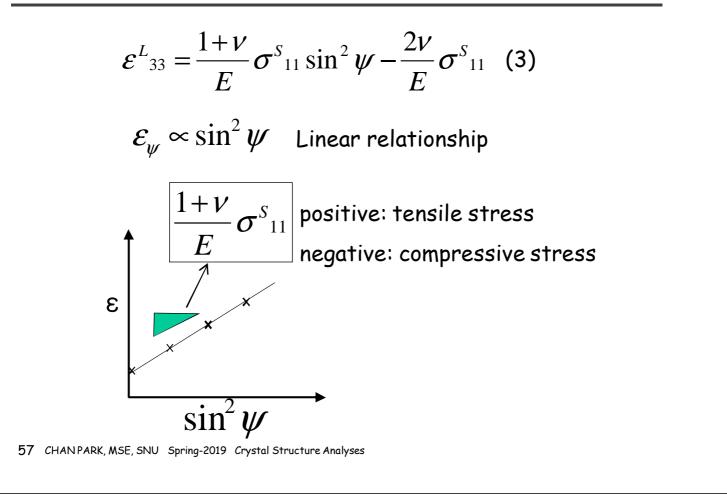
$$\varepsilon^{L}{}_{33} = \frac{1+\nu}{E}\{\sigma^{s}{}_{11}\cos^{2}\phi + \sigma^{s}{}_{12}\sin 2\phi + \sigma^{s}{}_{22}\sin^{2}\phi - \sigma^{s}{}_{33}\}\sin^{2}\psi + \frac{1+\nu}{E}\sigma^{s}{}_{33} - \frac{\nu}{E}(\sigma^{s}{}_{11} + \sigma^{s}{}_{22} + \sigma^{s}{}_{33}) + \frac{1+\nu}{E}\{\sigma^{s}{}_{13}\cos\phi - \sigma^{s}{}_{23}\sin\phi\}\sin 2\psi \qquad (2)$$

Biaxial stress

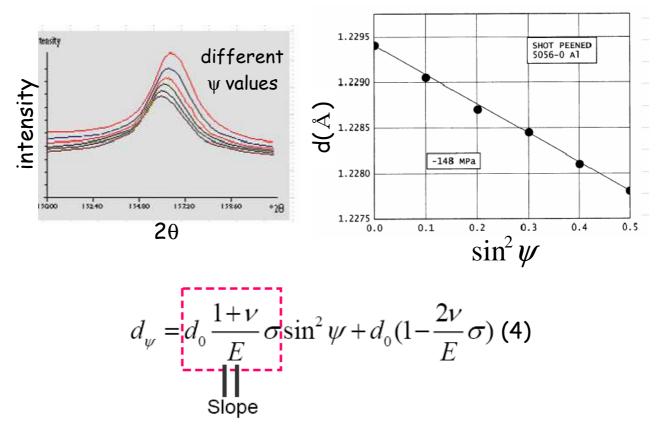
$$\varepsilon^{L_{33}} = \frac{1+\nu}{E} \{\sigma^{s_{11}} \cos^{2} \phi + \sigma^{s_{12}} \sin 2\phi + \sigma^{s_{22}} \sin^{2} \phi - \sigma^{s_{33}} \} \sin^{2} \psi + \frac{1+\nu}{E} \sigma^{s_{33}} \\ -\frac{\nu}{E} (\sigma^{s_{11}} + \sigma^{s_{22}} + \sigma^{s_{33}}) + \frac{1+\nu}{E} \{\sigma^{s_{13}} \cos \phi - \sigma^{s_{23}} \sin \phi \} \sin 2\psi \\ -\frac{\nu}{E} (\sigma^{s_{11}} + \sigma^{s_{22}} + \sigma^{s_{12}} \sin 2\phi + \sigma^{s_{22}} \sin^{2} \phi) - \frac{\sigma^{s_{33}}}{e^{s_{33}}} \} \sin^{2} \psi + \frac{1+\nu}{E} \sigma^{s_{33}} \\ -\frac{\nu}{E} (\sigma^{s_{11}} + \sigma^{s_{22}} + \sigma^{s_{23}}) + \frac{1+\nu}{E} \{\sigma^{s_{13}} \cos \phi - \sigma^{s_{23}} \sin \phi \} \sin^{2} \psi + \frac{1+\nu}{E} \sigma^{s_{33}} \\ -\frac{\nu}{E} (\sigma^{s_{11}} + \sigma^{s_{22}} + \sigma^{s_{23}}) + \frac{1+\nu}{E} \{\sigma^{s_{13}} \cos \phi - \sigma^{s_{23}} \sin \phi \} \sin^{2} \psi \\ \varepsilon^{L_{33}} = \frac{1+\nu}{E} \sigma^{s_{0}} \sin^{2} \psi - \frac{\nu}{E} (\sigma^{s_{11}} + \sigma^{s_{22}}) \\ \varepsilon^{s_{0}} = \sigma^{s_{11}} \cos^{2} \phi + \sigma^{s_{12}} \sin 2\phi + \sigma^{s_{22}} \sin^{2} \phi \\ = \sigma^{s_{11}} (\text{Biaxial Stress}) \\ \varepsilon^{L_{33}} = \frac{1+\nu}{E} \sigma^{s_{11}} \sin^{2} \psi - \frac{2\nu}{E} (\sigma^{s_{11}})$$
(3)
$$\varepsilon^{s_{0}} = \sigma^{s_{11}} \cos^{2} \phi + \sigma^{s_{12}} \sin 2\phi + \sigma^{s_{22}} \sin^{2} \phi \\ = \sigma^{s_{11}} (\text{Biaxial Stress}) \\ \varepsilon^{s_{0}} = \sigma^{s_{0}} (\sigma^{s_{0}} (\sigma^{s_{0}} (\sigma^{s_{0}} (\sigma^{s_{0}} (\sigma^{s_{0}} (\sigma^{s_{0}} (\sigma^{s_{0}}$$

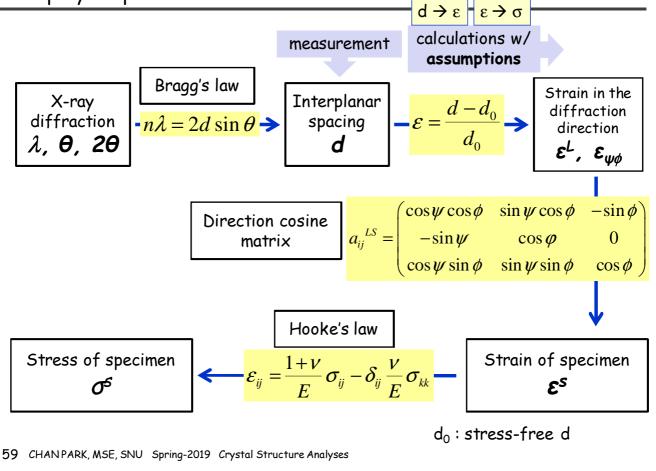
$$b \leftarrow 3$$

$$\begin{split} \widehat{\mathcal{E}}_{33}^{L} &= \frac{1+\nu}{E} \sigma_{11}^{S} \sin^{2} \psi - \frac{2\nu}{E} \sigma_{11}^{S} \end{split} \tag{3} \\ &= \frac{d_{\psi} - d_{0}}{d_{0}} = \frac{1+\nu}{E} \sigma_{11}^{S} \sin^{2} \psi - \frac{2\nu}{E} \sigma_{11}^{S} \\ &d_{\psi} = d_{0} \frac{1+\nu}{E} \sigma_{11}^{S} \sin^{2} \psi + d_{0} (1 - \frac{2\nu}{E} \sigma_{11}^{S}) \end{aligned} \\ &\left(d_{\psi} = d_{0} \frac{1+\nu}{E} \sigma_{11}^{S} \frac{1+\nu}{E} \sigma_{11}^{S} \psi + d_{0} (1 - \frac{2\nu}{E} \sigma_{11}^{S}) \end{aligned} \right) \end{split}$$

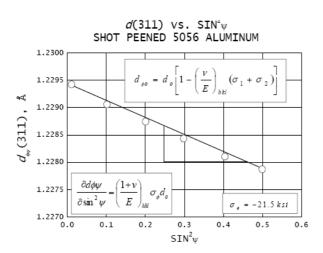


$sin^{2}\Psi$ method





 $sin^2\psi$ method



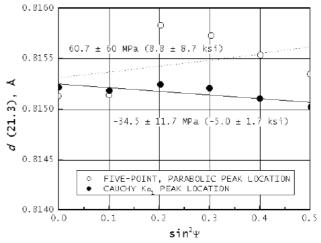
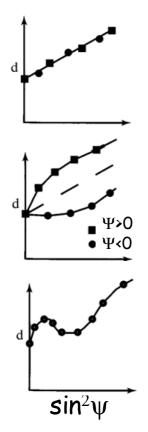


Fig. 3 - A d(311) versus $\sin^2 \psi$ plot for a shot peened 5056-O aluminum alloy having a surface stress of -148 MPa (-21.5 ksi)

Fig. 6 Comparison of d (21.3) versus $\sin^2 \psi$ data taken 0.176 mm (0.0069 in.) below the surface for a ground Ti-6Al-4V sample using two diffraction peak location methods



Biaxial or uniaxial stress gives linear sin² plots

<u>Triaxial stress</u> (all principle components of stress tensor are none zero) does not give a straight line → <u>psi-splitting</u>

Oscillatory - significant levels of <u>texture</u> are present (inhomogeneous stress/strain state within the materials) → the material is no longer elastically isotropic

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Residual stress measurement using XRD

- > Understanding of the assumptions
- > Is the sample homogeneous or heterogeneous?
- > Texture?
- The relationship between the beam size & grain size? Sampling statistics?
- > What components of the stress tensors are considered to be zero?

- > G. S. Schajer, Practical Residual Stress Measurement Methods, Wiley, 2013
- I. C. Noyan and J. B. Cohen, Residual Stress-Measurement by Diffraction and Interpretation, Springer-Verlag, 1987
- V. Hauk, Structural and Residual Stress Analysis by Nondestructive Methods, Elsevier, 1997
- > M. Birkholz, Thin Film Analysis by X-ray Scattering, Wiley, 2006
- B. D. Cullity, S. R. Stock, and S. Stock, Elements of X-ray Diffraction,
 Prentice Hall, 2001
- A. D. Krawitz, Introduction to Diffraction in Materials Science and Engineering, Wiley, 2001