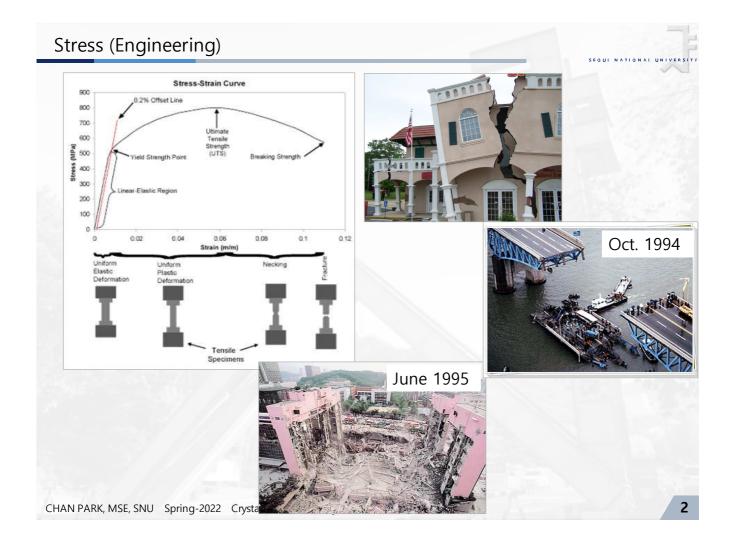
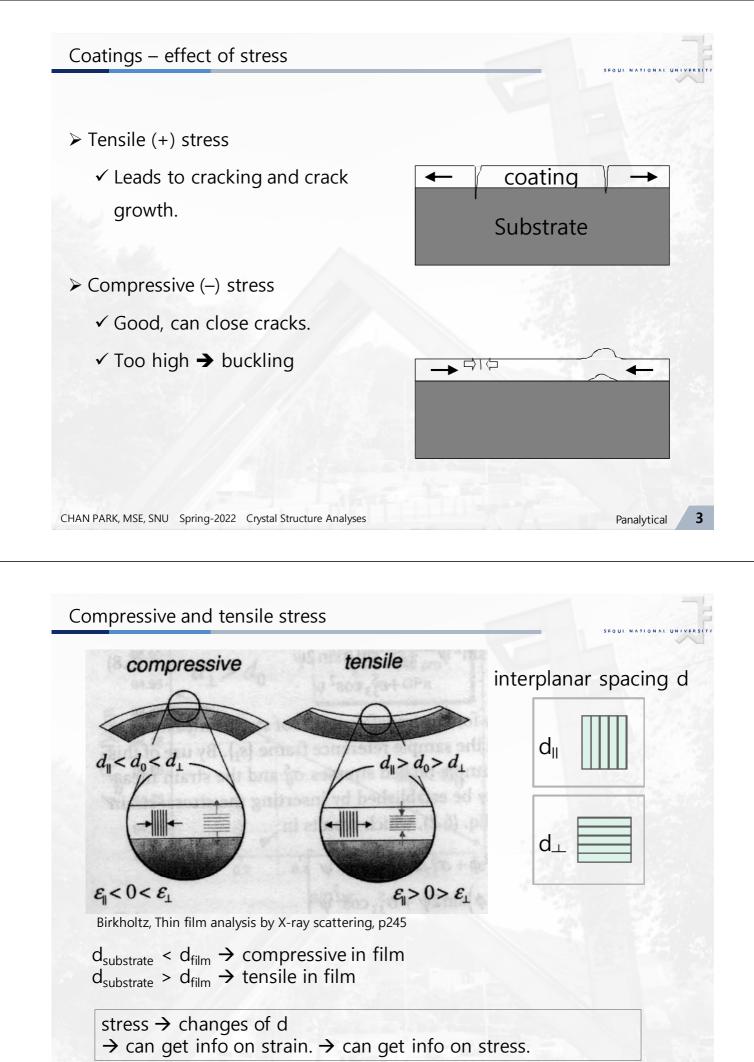
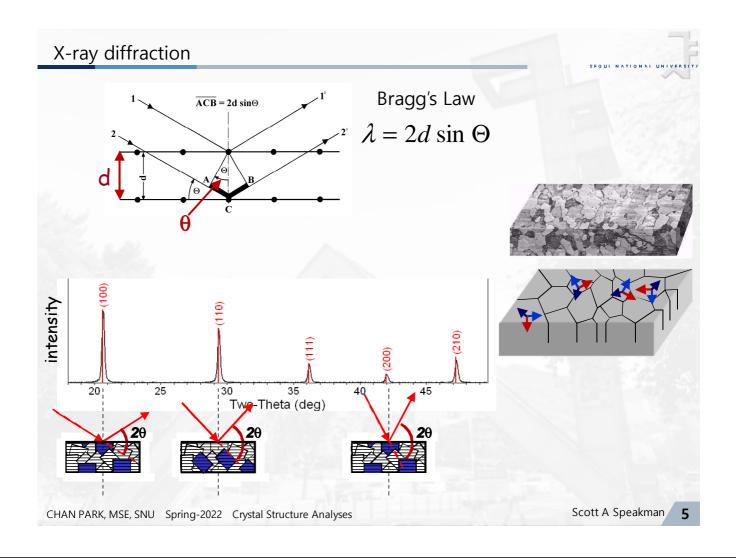
Residual stress analysis using XRD (sin²ψ method)

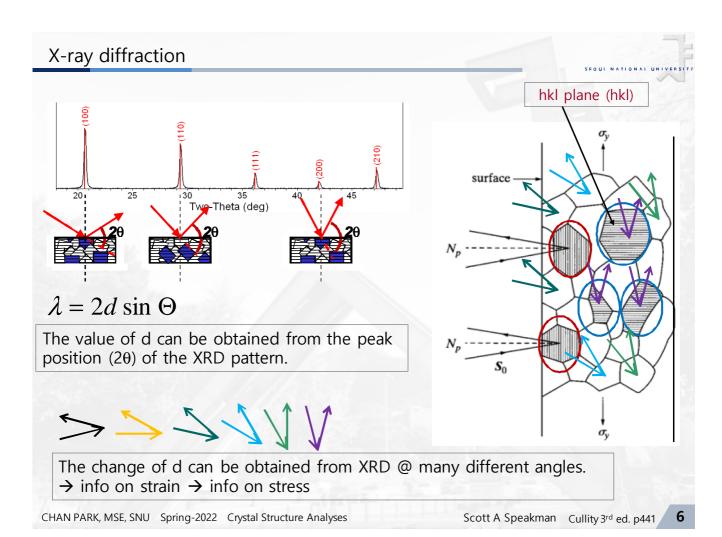
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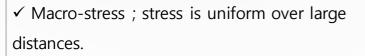
1











✓ Micro-stress ; vary from one grain to another on a microscopic scale.

✓ Macro-strain is uniform. → peak shift

✓ Micro-strain is nonuniform. \rightarrow peak broadening

- Diffraction does not measure stresses directly.
 - ✓ Changes in d-spacing → strains (macro-strain)
 - ✓ Changes in line width → micro-strain
 - ✓ The <u>lattice planes</u> of the individual grains in the material act as <u>strain gauges.</u>

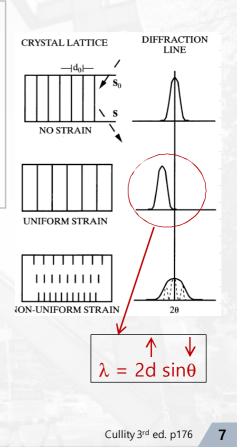
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Applied stress & Residual stress

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

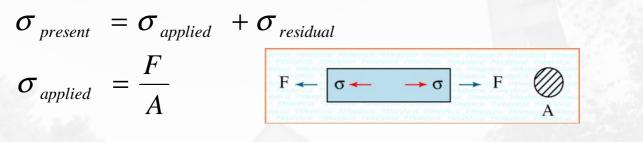
➤Residual stress ;

- ✓ 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.

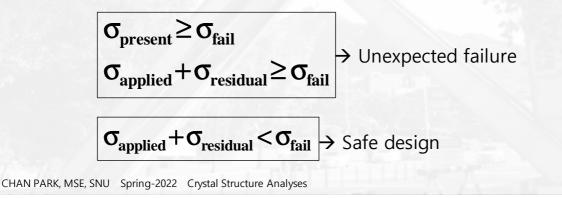


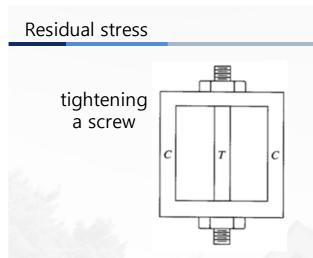
Residual stress

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

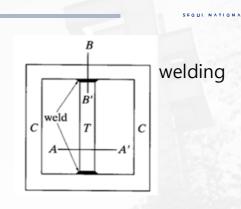


When F = 0 (no external force), $\sigma_{present} = \sigma_{residual}$





- 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.

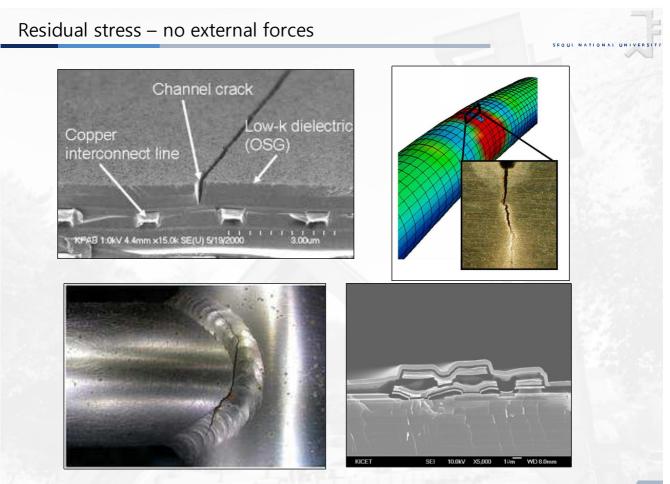


- During welding the central bar undergoes thermal expansion.
- On cooing, this leaves the bar under tension and the outer frame under compression.

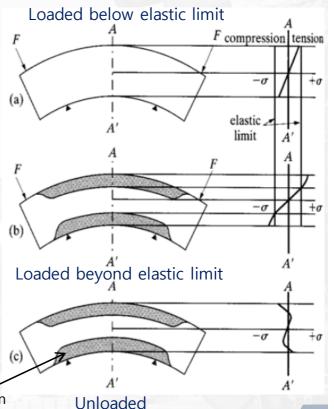
Residual stress

- > Bend a bar so that the outer and inner parts of the bar deform plastically, but the inner portion is only deformed elastically (b).
- > If the external stress is released, the inner part of the bar will try to return to its original shape, but the outer part can not, because it has plastically deformed.
- > The bar does not completely return to its original shapes, and there are residual stresses.

Plastically deformed region CHAN PARK, MSE, SNU Spring-2022 Crystal Structure Analyses



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Cullity 3rd ed. p438

➤Intrinsic stress

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

➤ Extrinsic stress

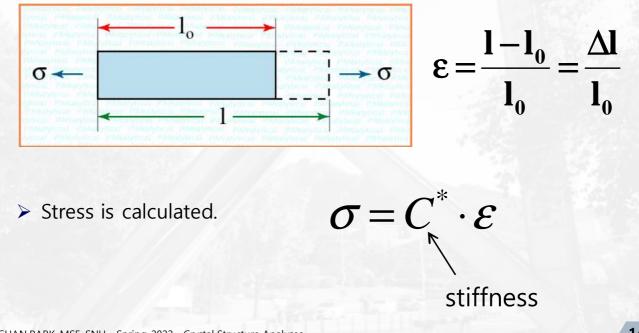
✓ Thermal stress (due to difference of CTE b/w film and substrate)

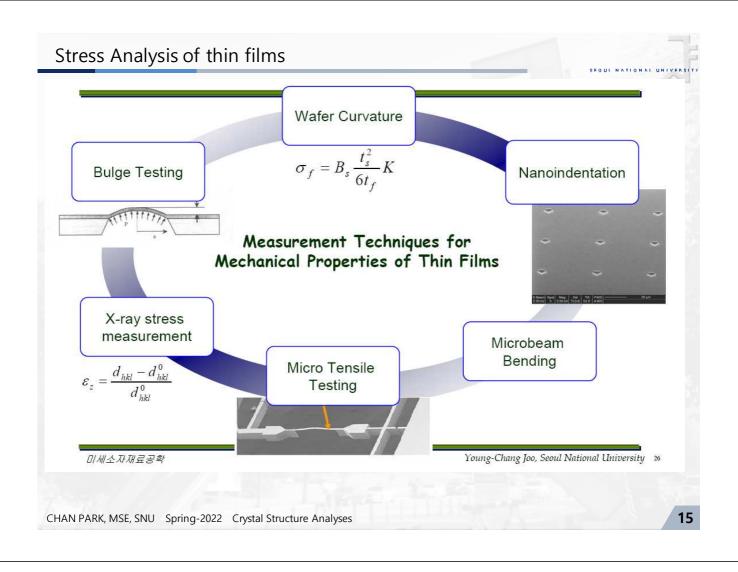
CTE; coefficient of thermal expansion

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How to measure stress ?

> Only strain can be measured.

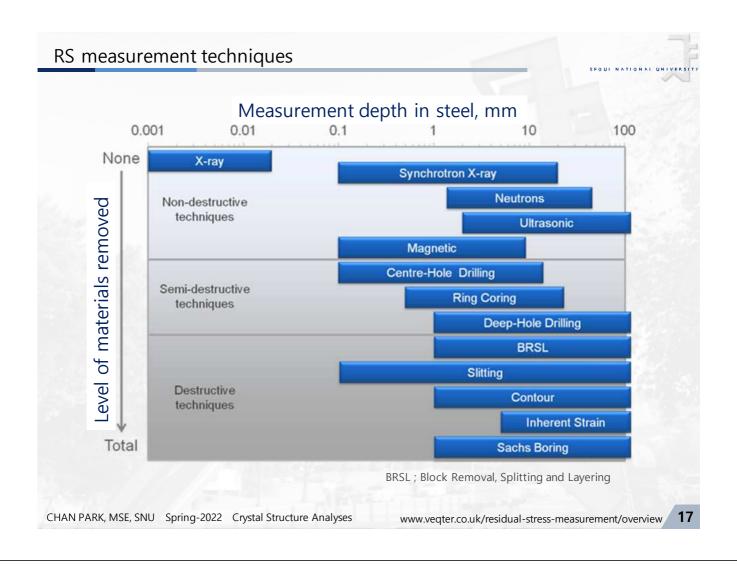




Residual Stress Measurement

- > 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
 - ✓ 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



RS measurement techniques

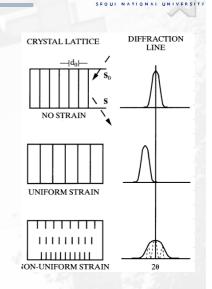
Technique	Advantage	Disadvantage	
X-ray diffraction	Versatile, Widely available, Portable, Wide range of materials, Macro and Micro RS	Lab-based systems, Small components, surface stress measurement	
Synchrotron XRD	Improved penetration & resolution of X-rays, Depth profiling, Fast, Macro and micro RS	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	

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



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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

Compliance/cutting methods

- ✓ Cutting or drilling changes the restraint and the object deforms.
- \checkmark The stresses can only be calculated in one direction.
- Diffraction methods
 - ✓ The distance between atoms in crystalline materials can be measured by diffraction (X-ray, neutron).
 - ✓ All stress directions can be measured.
- > Diffraction methods
 - ✓ Measured lattice strains are "<u>absolute quantities</u>" 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;
- ✓ Small gauge volume \rightarrow great for measuring <u>surface stress gradients</u>;
- ✓ High magnitude residual stresses are measured accurately;
- 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

RS Measurement > XRD > conventional vs. synchrotron

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-free</u> <u>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).

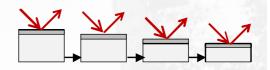
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Neutrons vs. X-rays

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

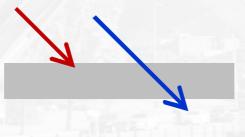
	neutron		X-ray	
Z	µ _l (cm⁻¹)	t _{50%} (mm)	µ _l (cm⁻¹)	t _{50%} (micron)
Al	0.10	69.3	131	52.9
Ti	0.45	15.4	938	7.39
Fe	1.12	6.19	2424	2.86
Ni	1.86	3.73	407	17.0
W	1.05	6.60	3311	2.09

- X-ray strain measurement provides information on the surface of a material.
 - ✓ 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.

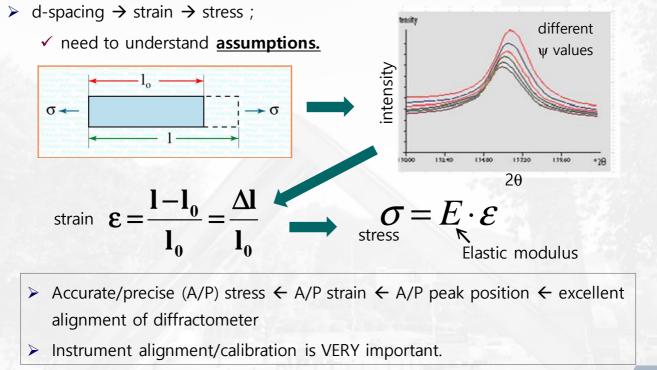
Neutron diffraction can be used to make measurements inside a part.



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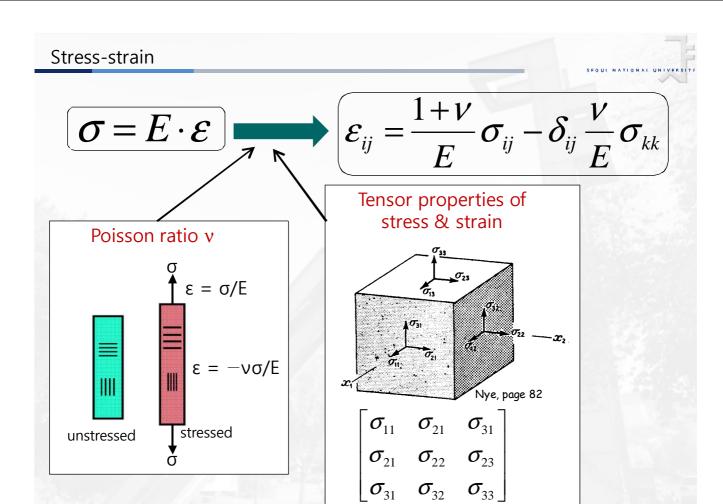
How to measure stress using XRD ?

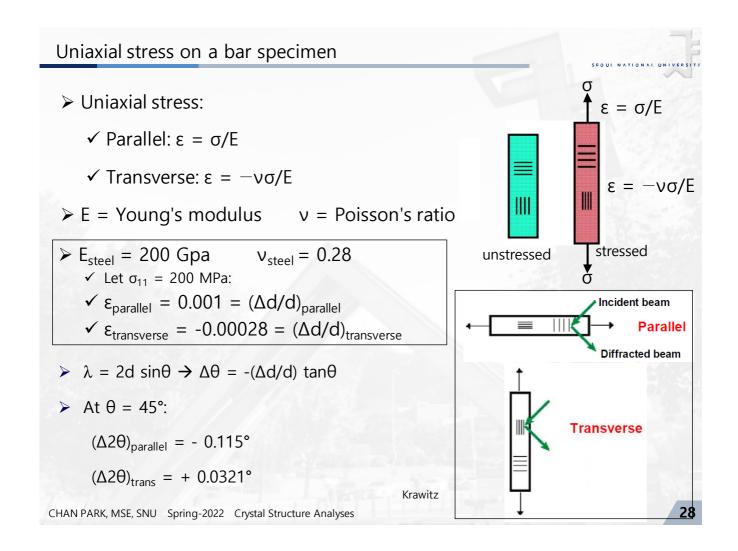
➤ Diffraction does not measure stress or strain. → gives the changes in d-spacing (change in peak position).



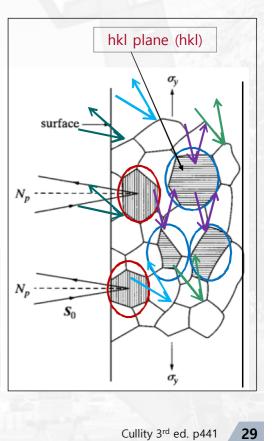
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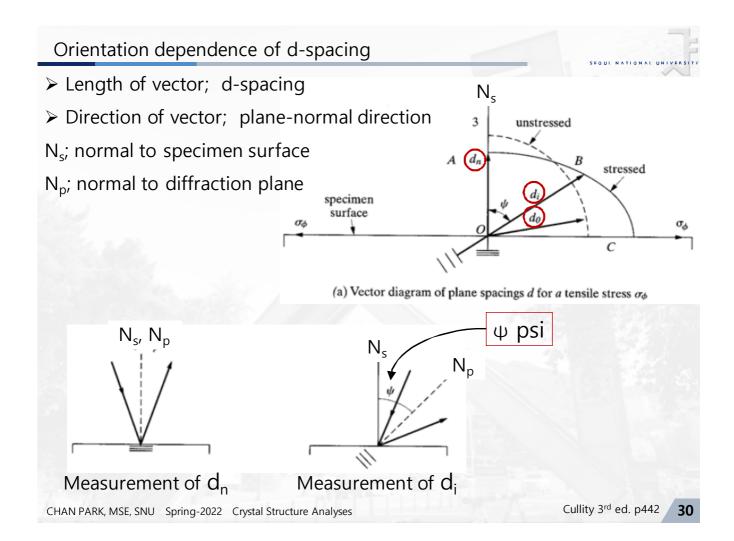
Krawitz



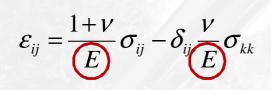


- When the d-spacing of a reflection is measured, <u>only grains with the planes</u> <u>oriented in a given direction contribute to</u> diffraction.
- If we change the orientation of the specimen and re-measure the d-spacing, we are looking at a different population of grains and we get a <u>different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

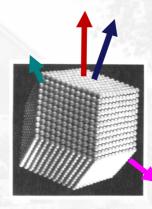




- > The elastic constants (E in $\sigma = E \cdot \varepsilon$) used in the stress calculation should be obtained from diffraction measurements on reference materials or by using values mechanically measured in different directions on single crystal specimens.
 - ✓ Individual crystallites are not elastically 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.



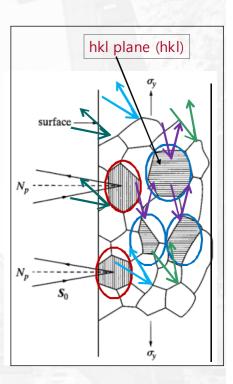
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Stress free reference

Strain is obtained from a diffraction measurement using

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

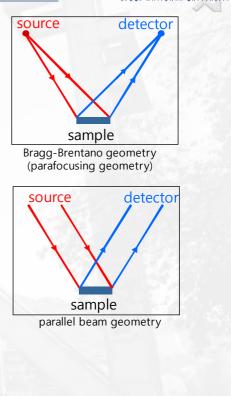
- $\checkmark~d_{\psi\phi}$; measured d-spacing in some direction $\psi\phi$
- ✓ d₀ ; d-spacing for the stress free material
 - It is very difficult to get d₀ directly ← to prepare a stress free piece of material with exactly the same composition is very difficult.
 - Using a similar piece of material 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 d_0 by making diffraction measurements at several angles (ψ).



Krawitz

- 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.



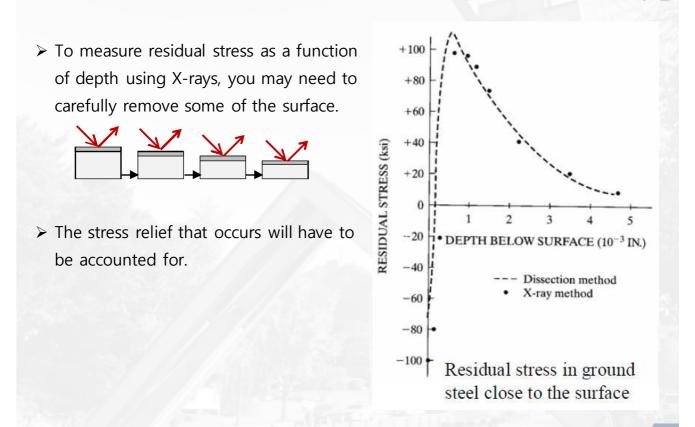
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➤ Sample preparation

- ✓ 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



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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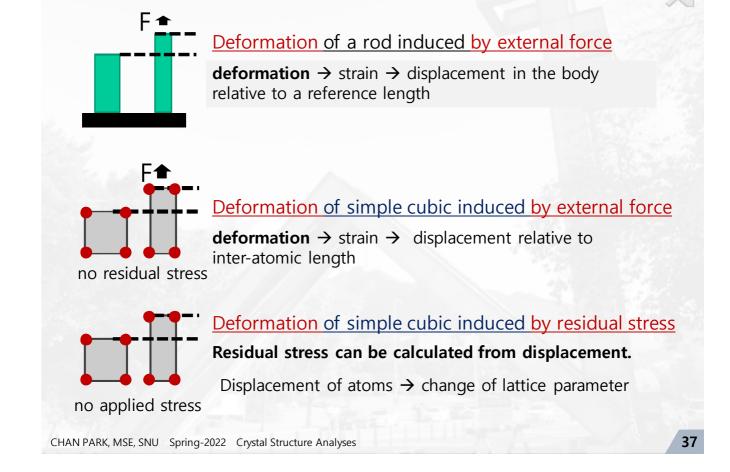
Practical problems

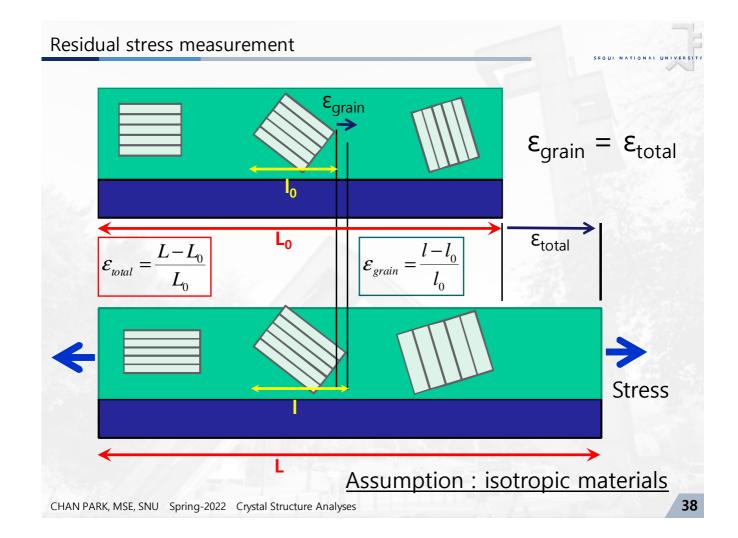
> Stress measurement uses positions of powder diffraction lines (d-spacing).

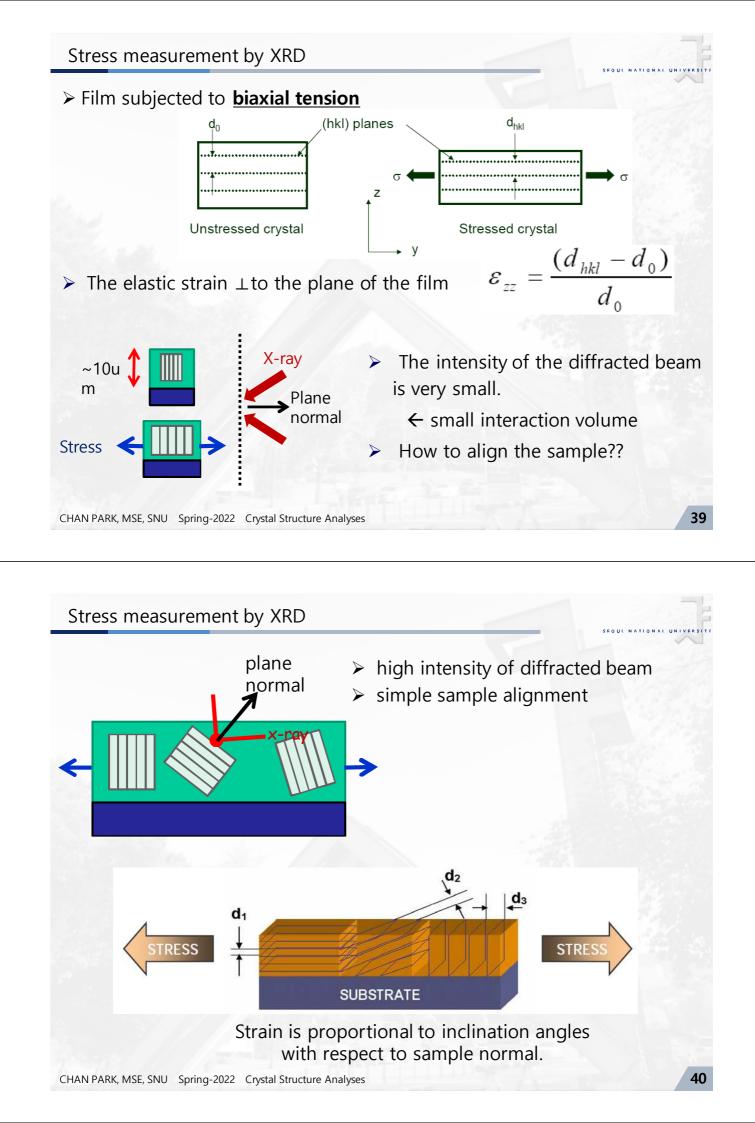
- If the sample contains very large grains, you do not really have powder lines, just single crystal spots. It can be very difficult to accurately estimate the <u>position of a powder line</u> under these conditions.
- > <u>Texture</u> can lead to very low intensity for some sample orientations.
- ➤ 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>.

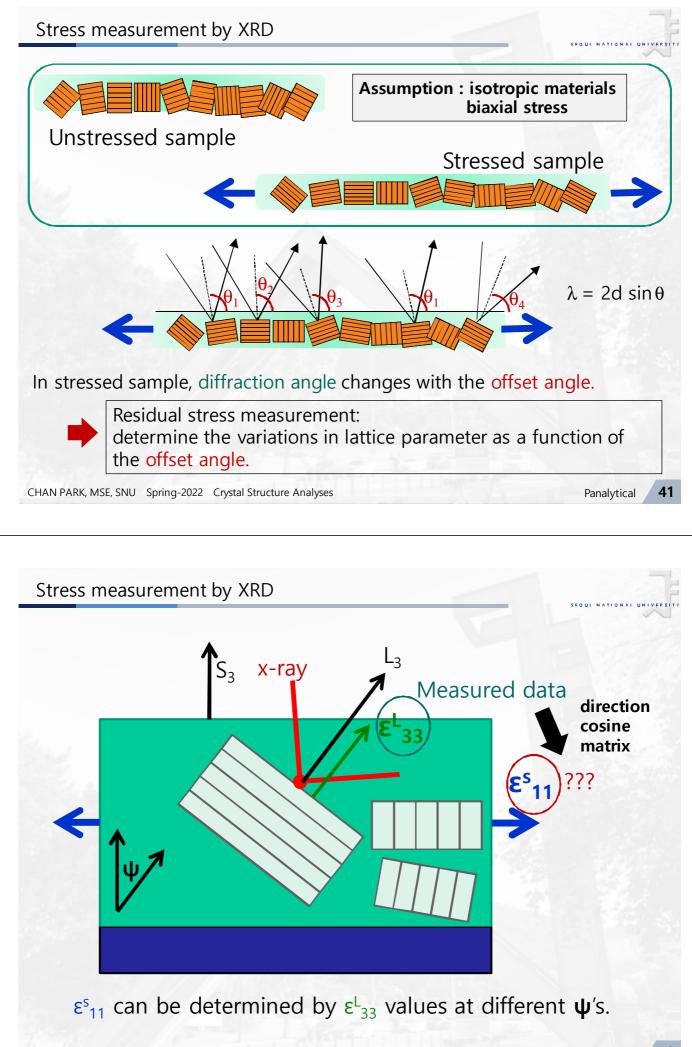
Krawitz

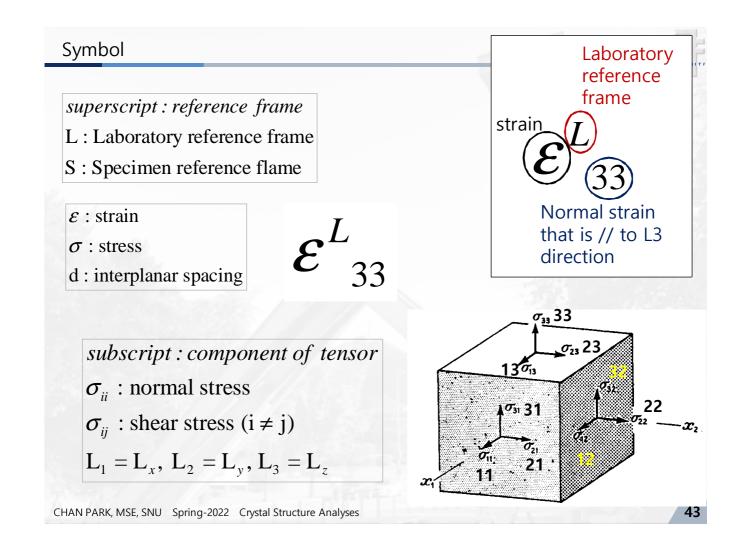
Residual stress

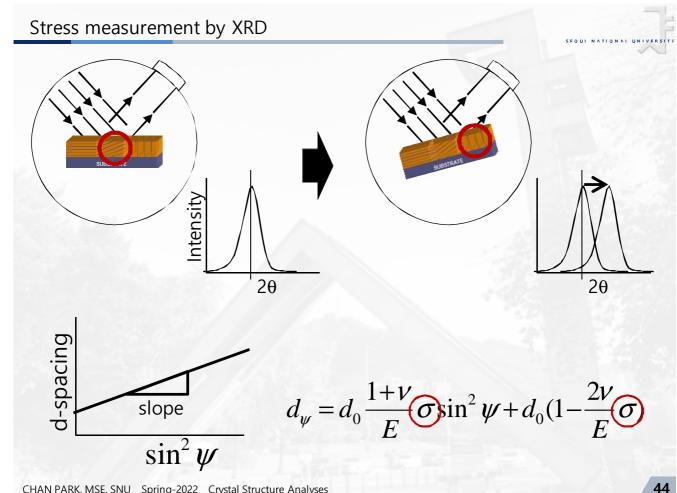










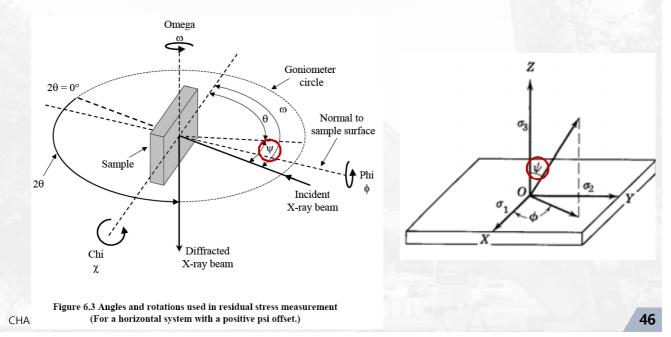


- > 2-theta (2θ) The Bragg angle, the angle between the incident (transmitted) and diffracted X-ray beams
- > Omega (ω) The angle b/w the incident X-ray beam and the sample surface. Both ω and 2 θ lie in the same plane.
- > Phi (ϕ) The angle of rotation of the sample about its surface normal
- > Chi (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)

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 $\psi = 0$, where ω is half of 2 θ and add (or subtract) successive ψ offsets. For example, 10, 20, 30 and 40°.



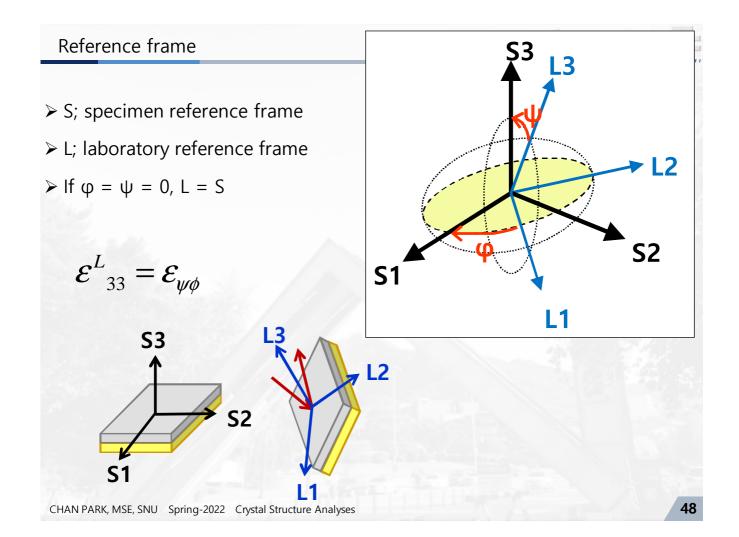
States of stress > possible states ✓ 3 unequal principal stresses (σ₁, σ₂, σ₃) → Triaxial state of stress ✓ 2 out of 3 principal stresses are equal (say σ₁, σ₂ = σ₃). → Cylindrical state of stress ✓ All 3 are equal (say σ₁ = σ₂ = σ₃). → Hydrostatic/spherical state of stress ✓ 1 of 3 is zero (say σ₁, σ₂, σ₃ = 0). → Biaxial/2D state of stress ✓ 2 of 3 is zero (say σ₁, σ₂ = σ₃ = 0). → Uniaxial state of stress ✓ 2 of 3 is zero (say σ₁, σ₂ = σ₃ = 0). → Uniaxial state of stress ✓ 2 of 3 is zero (say σ₁, σ₂ = σ₃ = 0). → Uniaxial state of stress ✓ 2 of 3 is zero (say σ₁, σ₂ = σ₃ = 0). → Uniaxial state of stress ✓ 2 of 3 is zero (say σ₁, σ₂ = σ₃ = 0). → Uniaxial state of stress ✓ 2 of 3 is zero (say σ₁, σ₂ = σ₃ = 0). → Uniaxial state of stress ✓ 2 of 3 is zero (say σ₁, σ₂ = σ₃ = 0). → Uniaxial state of stress ✓ 2 of 3 is zero (say σ₁, σ₂ = σ₃ = 0). → Uniaxial state of stress ✓ 2 of 3 is zero (say σ₁, σ₂ = σ₃ = 0). → Uniaxial state of stress ✓ 2 of 3 is zero (say σ₁, σ₂ = σ₃ = 0). → Uniaxial state of stress ✓ 2 of 3 is zero (say σ₁ = σ₂ = σ₃ = 0). → Uniaxial state of stress ✓ 3 unequal to L3. ✓ 8 unequal to L3.

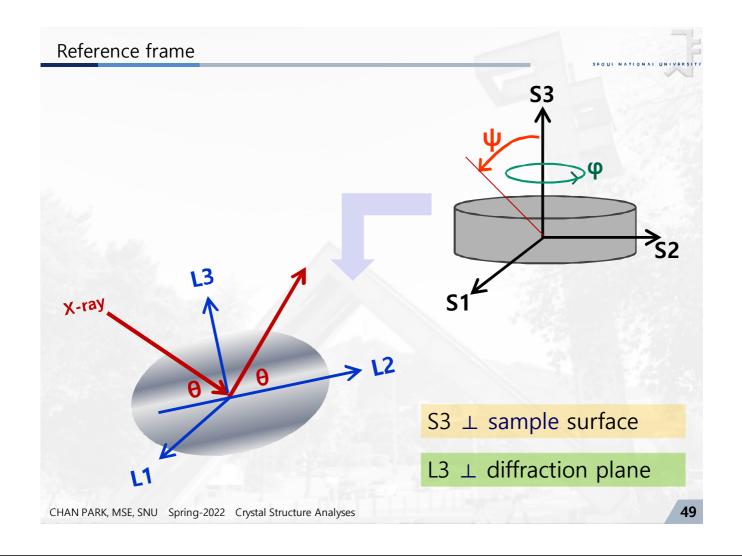
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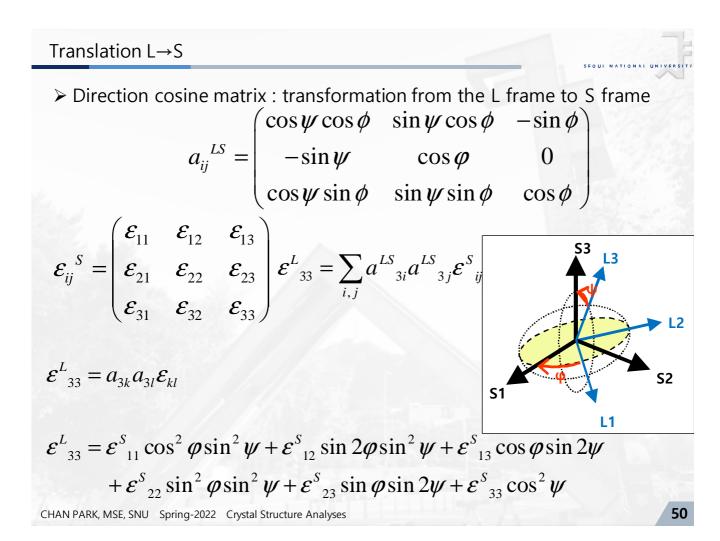
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Nye, page 82

- ✓ L_i laboratory coordinate system
- ✓ S_i sample coordinate system



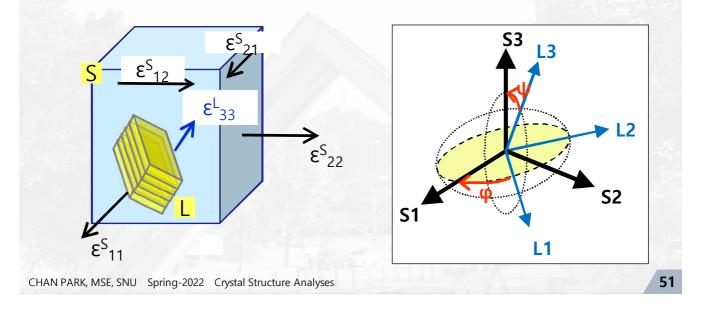


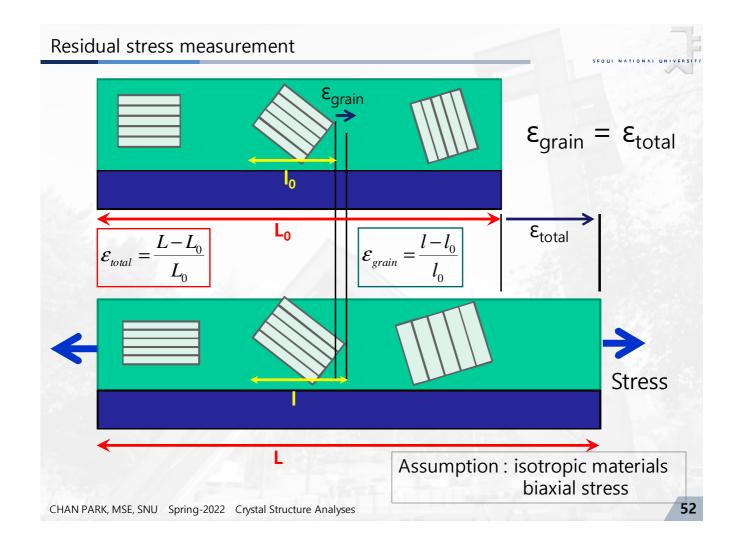


Translation $L \rightarrow S$

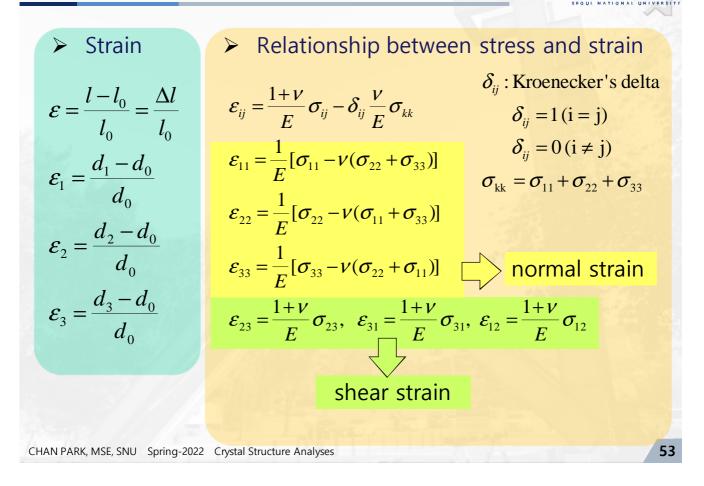
$\varepsilon^{L}{}_{33} = \varepsilon^{S}{}_{11}\cos^{2}\phi\sin^{2}\psi + \varepsilon^{S}{}_{12}\sin 2\phi\sin^{2}\psi + \varepsilon^{S}{}_{13}\cos\phi\sin 2\psi + \varepsilon^{S}{}_{13}\cos\phi\sin 2\psi + \varepsilon^{S}{}_{22}\sin^{2}\phi\sin^{2}\psi + \varepsilon^{S}{}_{23}\sin\phi\sin 2\psi + \varepsilon^{S}{}_{33}\cos^{2}\psi$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

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





Strain and Stress



Hooke's Law

$$\mathcal{E}^{L_{33}} = \mathcal{E}^{s}_{11} \cos^{2} \phi \sin^{2} \psi + \mathcal{E}^{s}_{12} \sin 2\phi \sin^{2} \psi + \mathcal{E}^{s}_{13} \cos \phi \sin 2\psi + \mathcal{E}^{s}_{23} \sin \phi \sin 2\psi + \mathcal{E}^{s}_{33} \cos^{2} \psi \qquad (1)$$

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

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

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Biaxial stress

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

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

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

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

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

$$= \sigma^{s}{}_{11}\cos^{2}\phi + \sigma^{s}{}_{12}\sin 2\phi + \sigma^{s}{}_{22}\sin^{2}\phi + \sigma^{s}{}_{22}\sin^{2}\phi}$$

$$= \sigma^{s}{}_{11}\cos^{2}\phi + \sigma^{s}{}_{22}\sin^{2}\phi + \sigma^{s}{}_{22}\sin^{2}\phi}$$

$$= \sigma^{s}{}_{11}(\text{Biaxial Stress})$$

$$(3)$$

$$= \frac{\varepsilon^{\mu}}{\varepsilon^{\mu}} \propto \sin^{2}\psi - \frac{2\nu}{E}(\sigma^{s}{}_{11})$$

$$(3)$$

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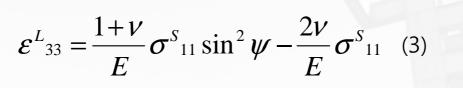
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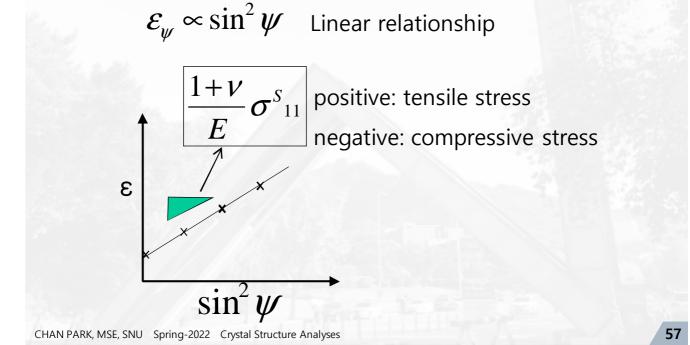
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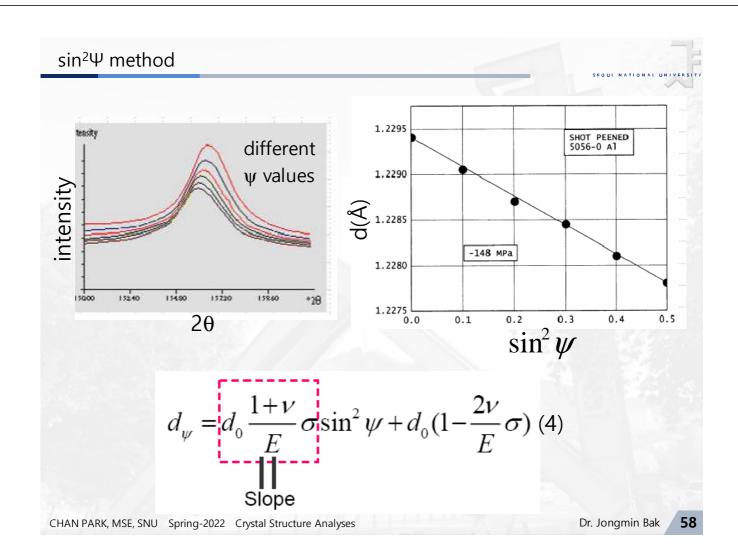
56

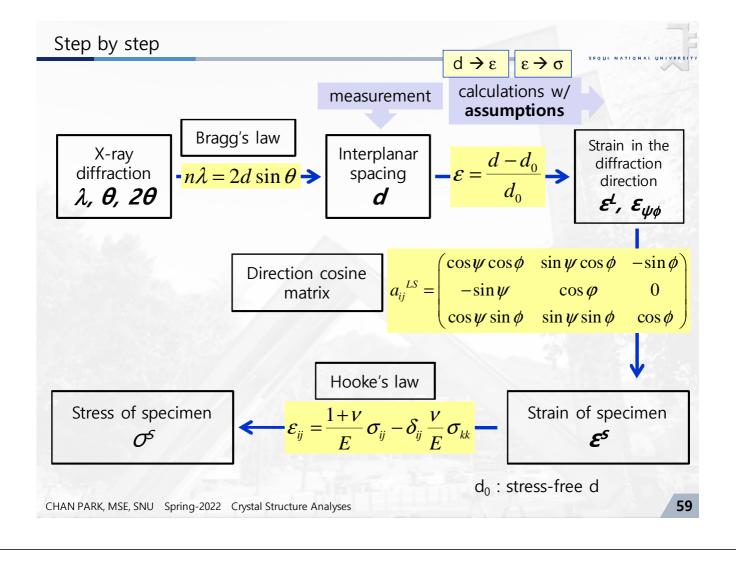
 $\epsilon \rightarrow d$

$$\begin{split} \widehat{\varepsilon}_{33}^{L} &= \frac{1+\nu}{E} \sigma_{11}^{S} \sin^{2} \psi - \frac{2\nu}{E} \sigma_{11}^{S} \end{split} \tag{3} \\ &= \frac{d_{\psi} - d_{0}}{d_{0}} = \frac{1+\nu}{E} \sigma_{11}^{S} \sin^{2} \psi - \frac{2\nu}{E} \sigma_{11}^{S} \\ &= d_{0} \frac{1+\nu}{E} \sigma_{11}^{S} \sin^{2} \psi + d_{0} (1 - \frac{2\nu}{E} \sigma_{11}^{S}) \\ &= d_{0} \frac{1+\nu}{E} \sigma_{11}^{S} \sin^{2} \psi + d_{0} (1 - \frac{2\nu}{E} \sigma_{11}^{S}) \end{aligned} \tag{4} \\ &= \mathcal{E}_{\psi} \propto \sin^{2} \psi \end{split}$$

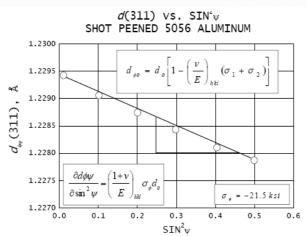








 $sin^2\psi$ method



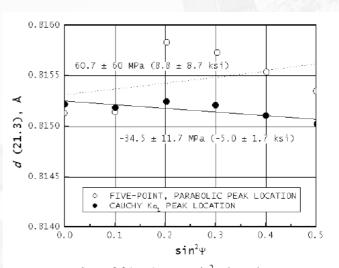
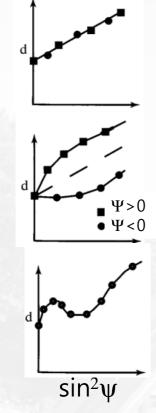


Fig. 6 Comparison of d (21.3) versus $\sin^2\psi$ data taken 0.176 mm (0.0069 in.) below the surface for a ground Ti-6Al-4V sample using two diffraction peak location methods

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)



<u>Biaxial or uniaxial stress</u> gives **linear** $\sin^2\psi$ plots.

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

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

CHAN PARK, MSE, SNU Spring-2022 Crystal Structure Analyses

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?

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