Diffraction Stress Analysis

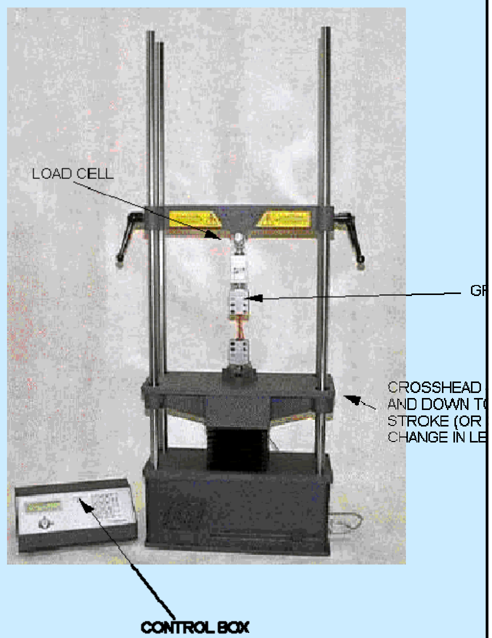
I. C. Noyan
Columbia University
Dept. of Applied Physics and Applied Mathematics

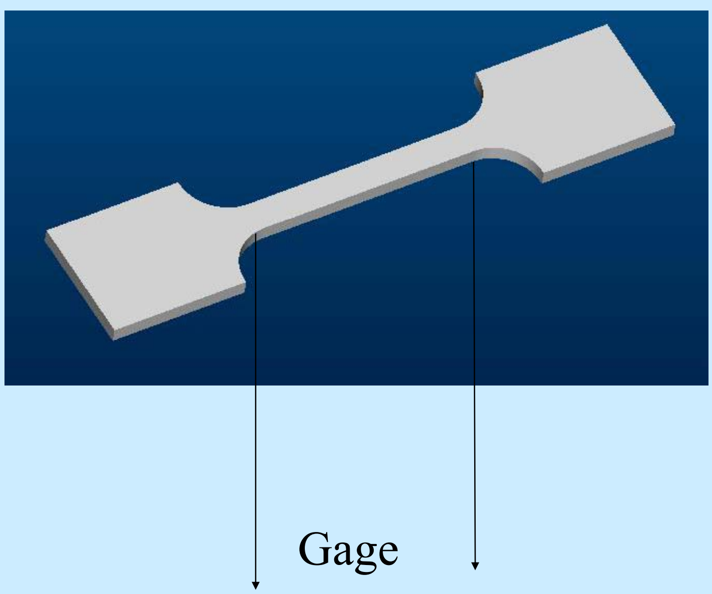
Synopsis

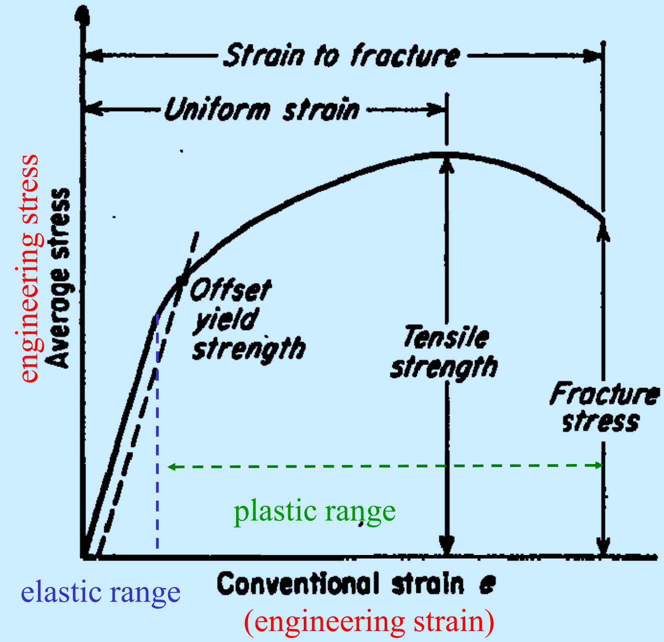
- Stress/strain concepts
- Techniques of stress determination with X-rays
  - Basic Theory
  - Examples
- Suggestions

The Tensile Test

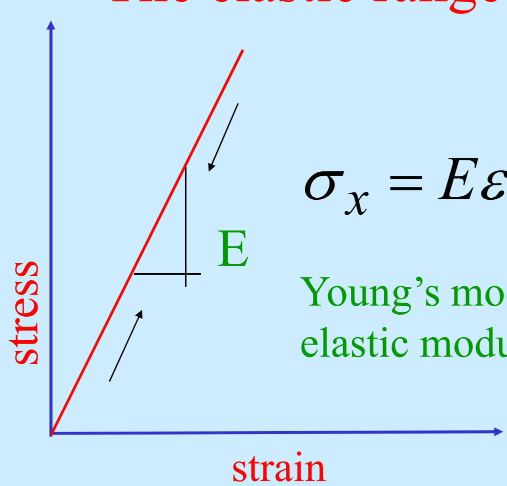
- Specimens are gripped and pulled to failure.
  - The elongation and load are monitored continuously.
  - This data yield the "stress-strain" curve.
  - The strain rate is constant during the test.







The elastic range



$$\sigma_x = E\varepsilon_x$$

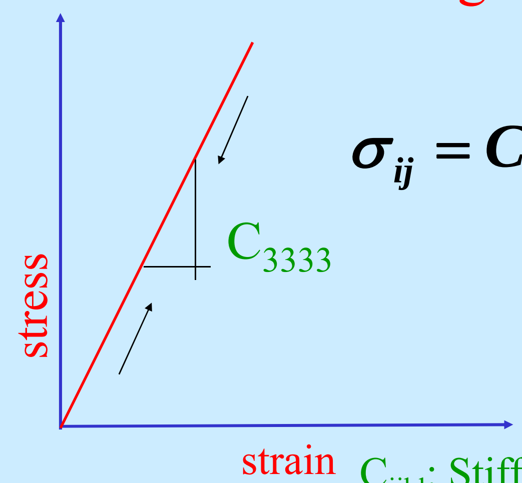
Young's modulus,
elastic modulus.

$$\tau = G\gamma$$

G: shear modulus

$$E = 2G(1+\upsilon)$$

The elastic range



$$\sigma_{ij} = C_{ijkl}\varepsilon_{kl}$$

C_{ijkl}: Stiffness tensor

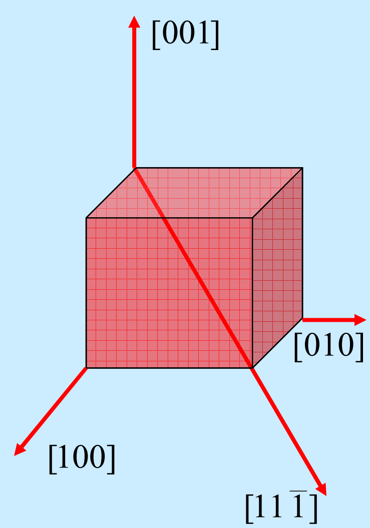
(homogeneous, anisotropic sample).

Definitions

Homogeneous: The property we are measuring is the same along a given direction throughout the entire volume.

– Isotropic: The measured property is independent of direction and location.

– Anisotropic: The measured property is independent of location but changes with direction.



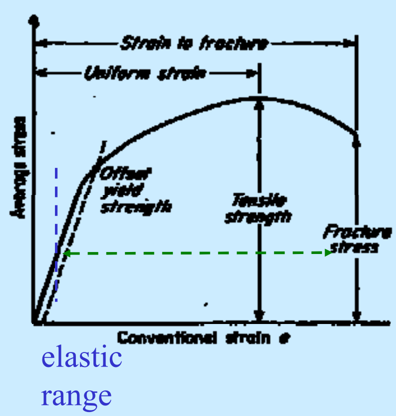
Single crystals are homogeneous for length scales larger than hundreds of unit cube edges.

Polycrystalline aggregates are quasi-homogeneous only for length scales that exceed the representative length $(V_R)^{1/3}$ for the particular aggregate.

Quasi-isotropic

Quasi-anisotropic

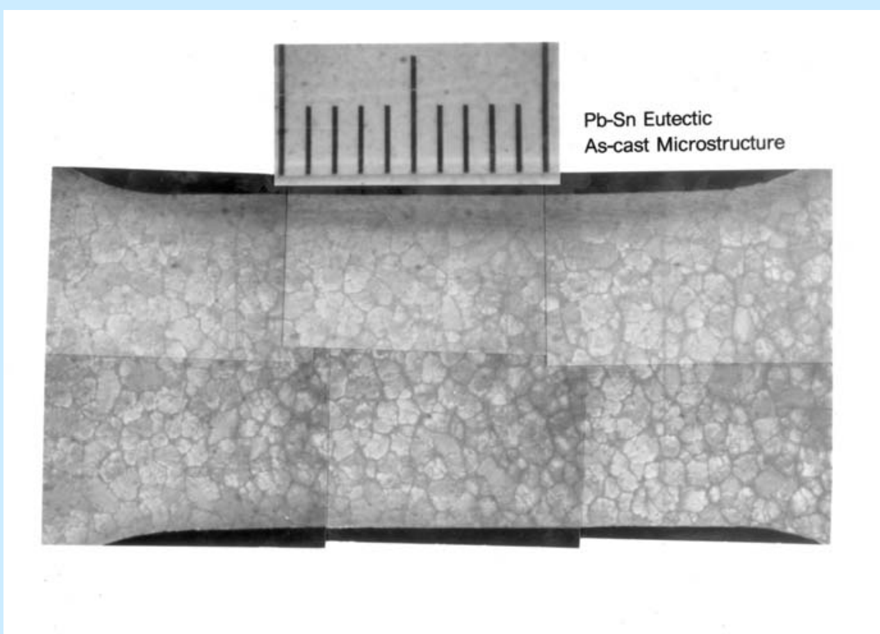
For dimensions smaller than the representative length, the mechanical response of the aggregate is heterogeneous (inhomogeneous).

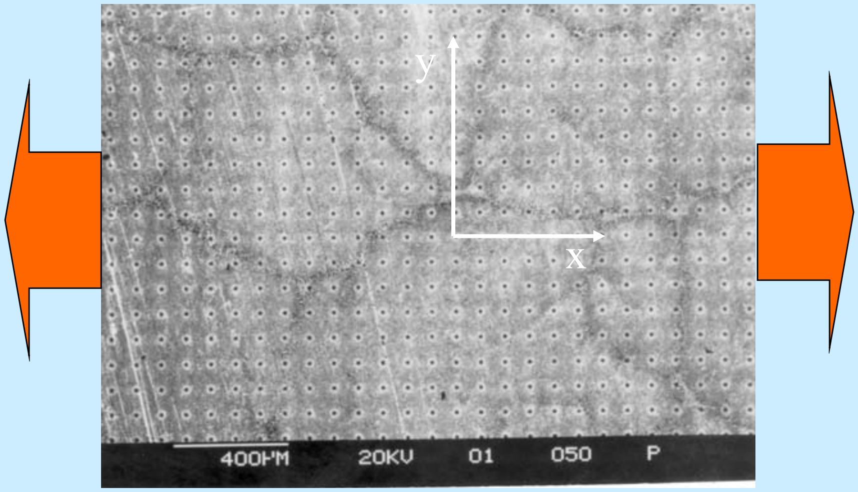


Local values ≠ global averages

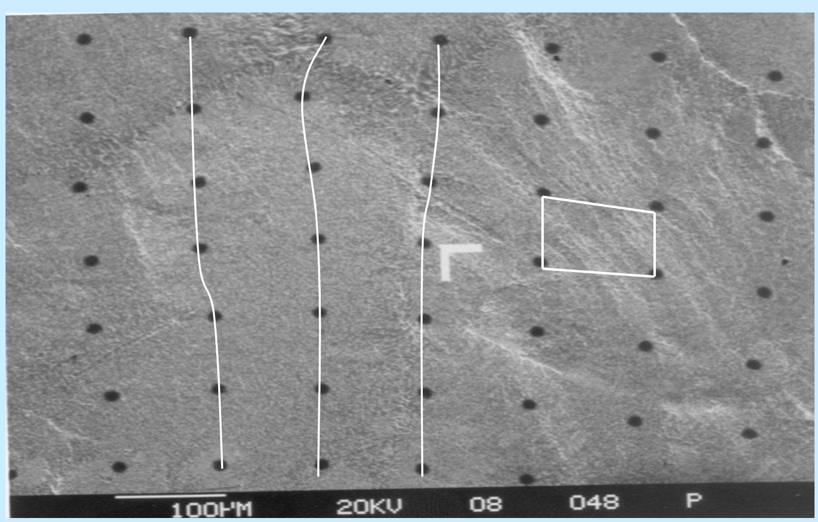
Analysis of local deformation through an evaporated grid

- Pb-Sn dogbone specimens,
- Cast and annealed microstructure, polished and etched:
  - Eutectic (750-800 μm grain size)
  - 98Pb-2Sn (550-600 μm grain size
- Al grid evaporated through a mask on the gage section:
  - 12.5 μm dots on 100 μm centers



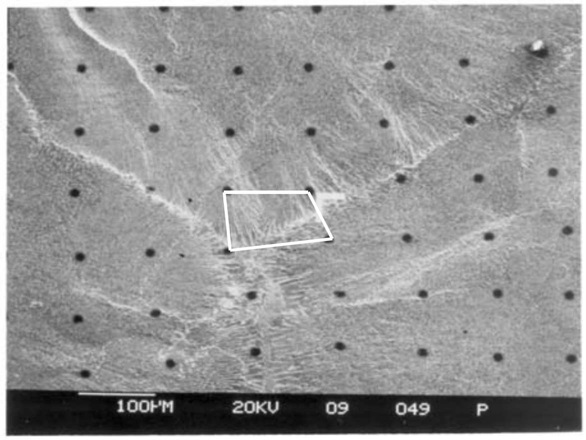


❖ Uniaxial tensile loads were applied to this specimen
❖ We monitored the grid deformation at various plastic strains.



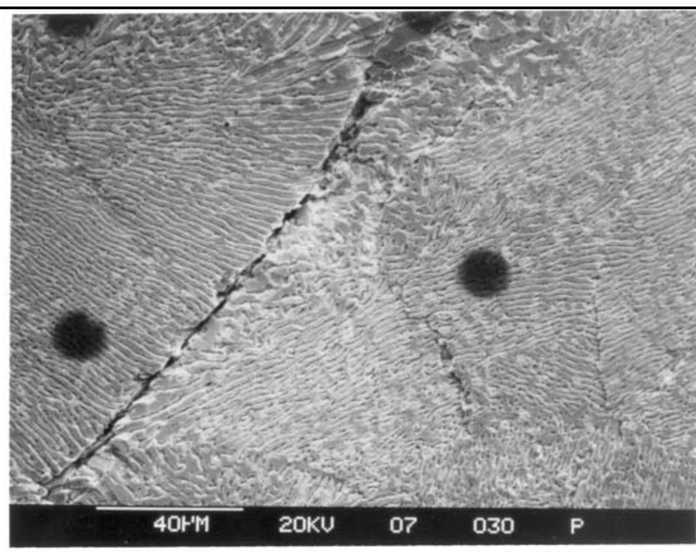
- **Local deformation is not uniaxial**
  - **Rotation, shear**

10 % pl. strain

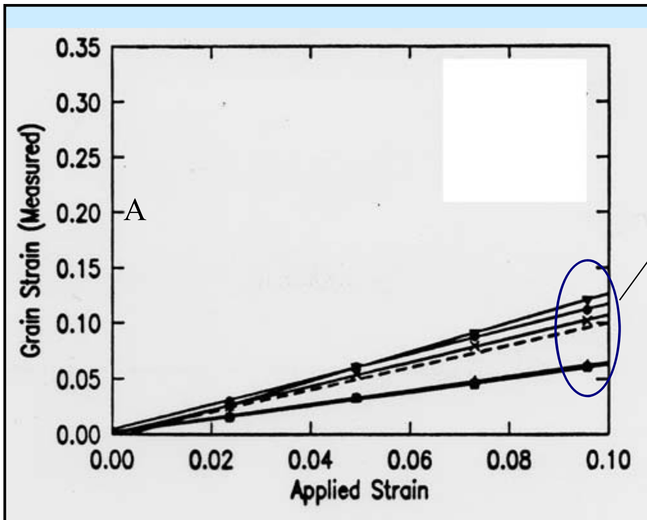


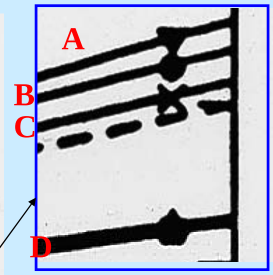
10% pl. strain

Incompatibility is most pronounced @ triple junctions.



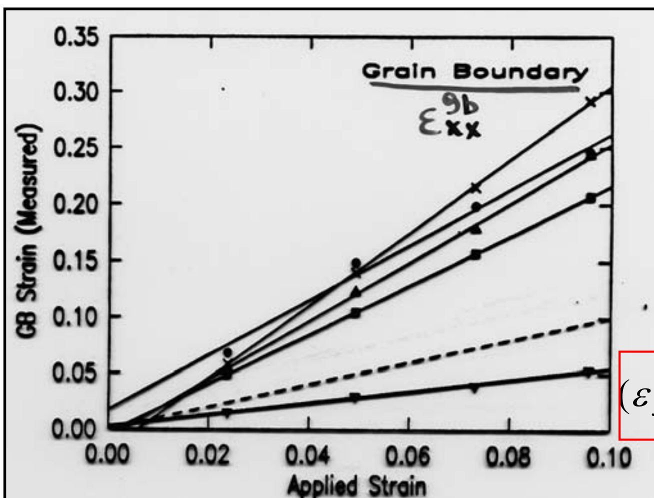
Local crack initiation occurs where the strain localization is highest.





$$(\varepsilon_{xx}^{p})_{i} \neq (\varepsilon_{xx}^{p})_{j}$$

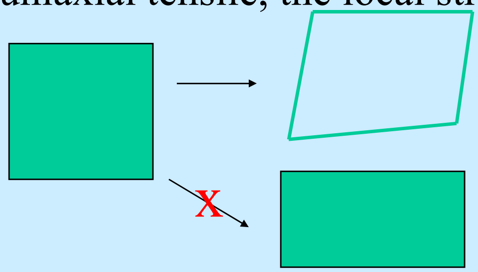
The local plastic strain within each grain (A,B,C.D) changed linearly with applied plastic strain.



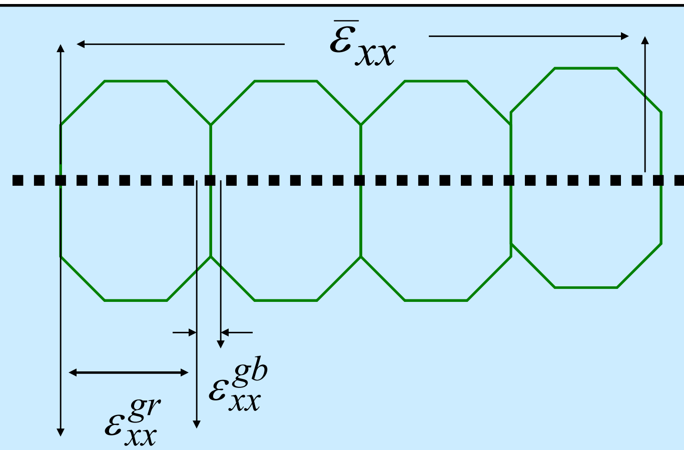
$$\boxed{(\varepsilon_{xx}^{p})_{grain} << (\varepsilon_{xx}^{p})_{gb}}$$

The local plastic strain across each grain boundary also changed linearly with applied plastic strain.

- Thus, even though the far-field strain is uniaxial tensile, the local strain state is not.

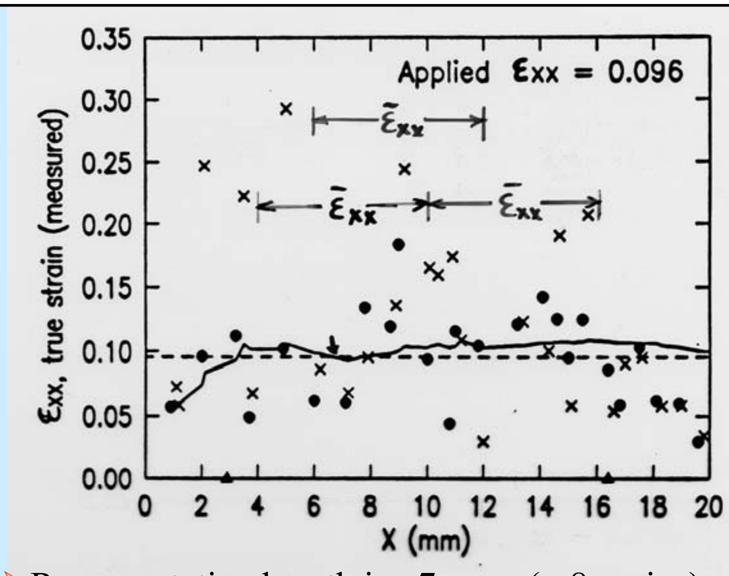


- There is local rotation and shear.
- The strain state is heterogeneous:
  - Strain varies from grain to grain,
  - Grain interior to grain boundary,
  - Across grain boundaries.



Our next task is to find the minimum length scale over which the average strain is equal to the far-field strain.

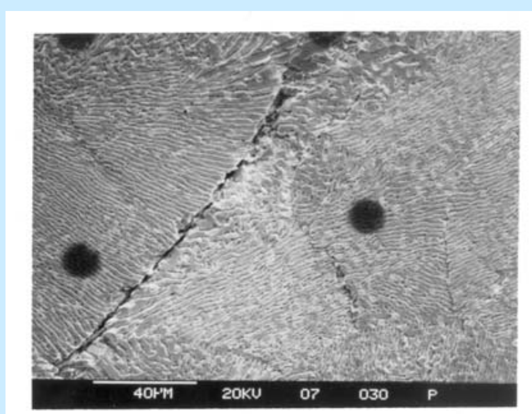
This defines the “representative length”, above which the average strain is invariant with location.



➢Representative length is ~7 mms (~8 grains).

➢The smallest volume is 7mmx7mm x 5 mm (~80 grains).

- The previous analysis used a eutectic structure with “colony” boundaries.



- The results do not change much if we use a single-phase alloy: 98Pb2Sn.

Summary of plastic strain distributions

- Local deformation is non-uniform and can be within .5x to 2x of the applied strain.
- The minimum volume element in which the average strains yield the applied strain is termed the *representative volume*, V_R.
- The average material response for volumes larger than is V_R quasi-homogeneous.

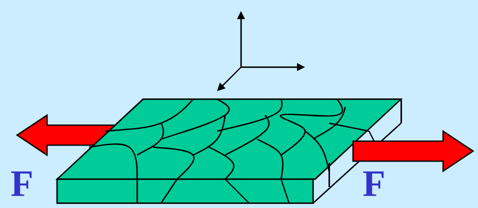
Bonda, N.R. Noyan, I.C. IEEE Trans. Comp. Packag. Manuf. Technol. A , Vol.19, 1996

What about elastic strain distributions?

- Strains are too small; we can not use the same grid technique easily.
- We decided to use a FEM grid to simulate the strain distribution.
  - 400 cubic grains
  - 201 isotropic
  - 199 fully anisotropic
- All randomly placed in a mesh.

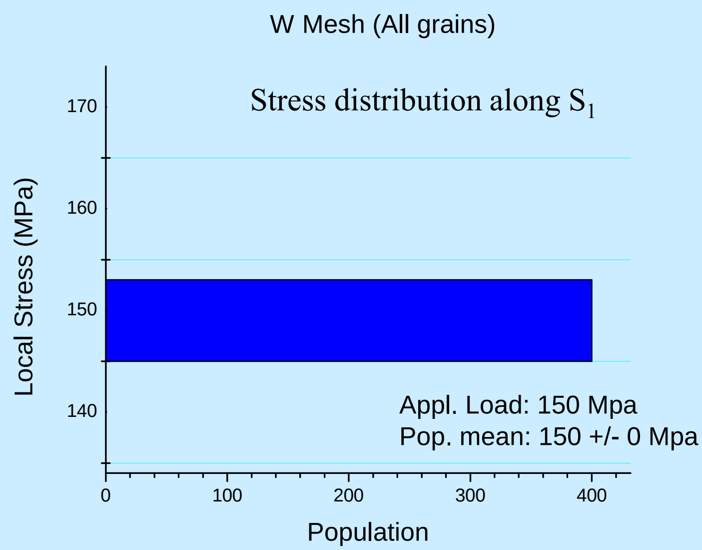
FEM-details

- Abaqus code.
- 8-node C3D8 elements
  - A single layer of cubic grains loaded uniaxially.



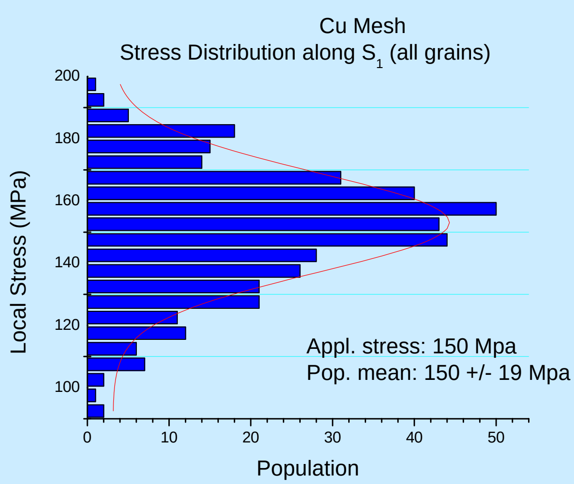
W-mesh

- Tungsten is isotropic.
- Thus, this material served as a test of the mesh.
- We expect all grains to have the same stress state in the sample coordinates independent of orientation.



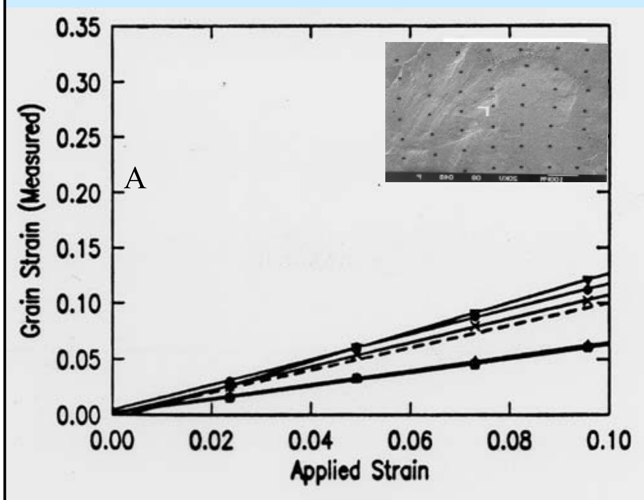
The mesh works, and all grains have the same stress state.

This is not so with Cu.



The mean stress is correct, but the stresses/strains in individual grains can be~ +/- 40% of the mean.

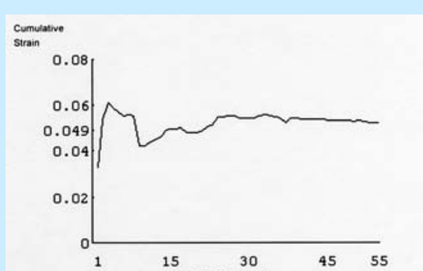
.



The local strain within each grain changes <u>linearly</u> with applied load.

The local strain within any grain can be used to determine the global strain/stress if its proportionality constant is known (applied strain/load measurement).

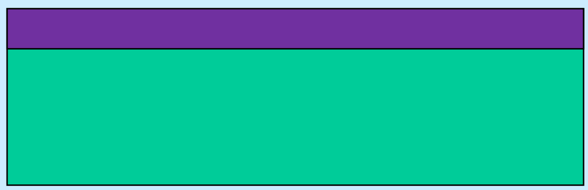
- **Thus, measurement of applied loads is quite straightforward.**
  - **Differential measurement.**
  - **Easy to obtain the correlation parameter vs. applied load.**
- **Measurement of residual stresses/strains is more complicated.**
  - **Each grain may have a different strain value.**
  - **One has to measure enough grains to make sure that local oscillations are averaged out.**

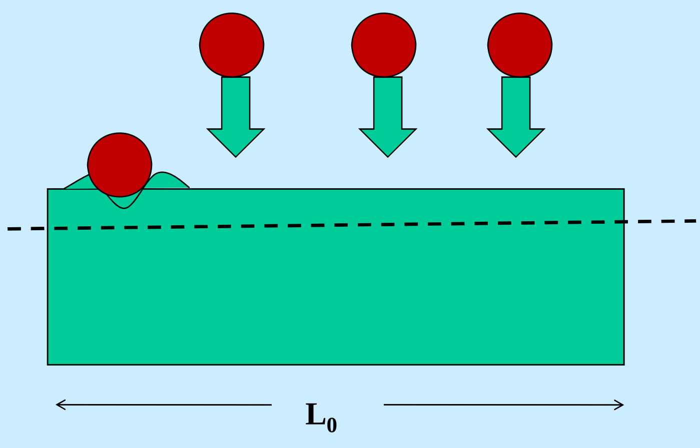


Residual Strain/Stress Definitions

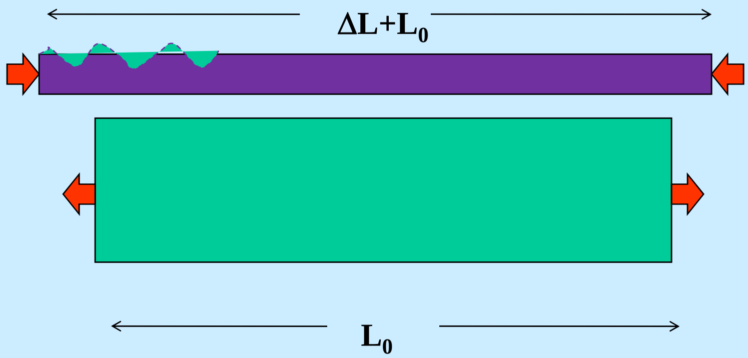
Residual stress: self-equilibrated stresses existing in a free body with no surface tractions.

Stresses in one part of the body balance out the stresses in another part.





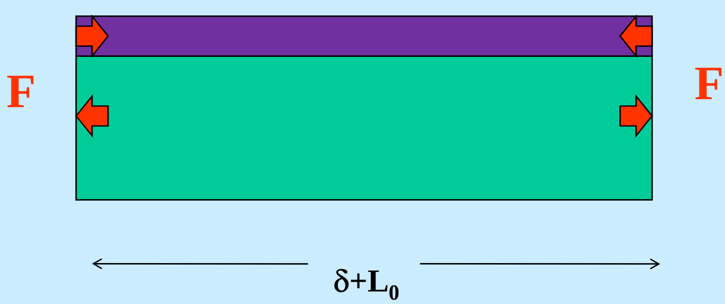
Shot-peening, laser-peening



To match the boundaries across the interface apply external forces to the boundary.
Then glue the interface and relax the external forces.

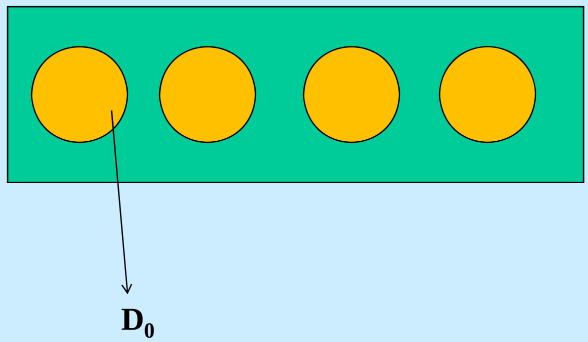
$$\sigma_{surface} = \frac{-F}{t_{surface}}$$

$$\sigma_{bulk} = \frac{F}{t_{bulk}}$$

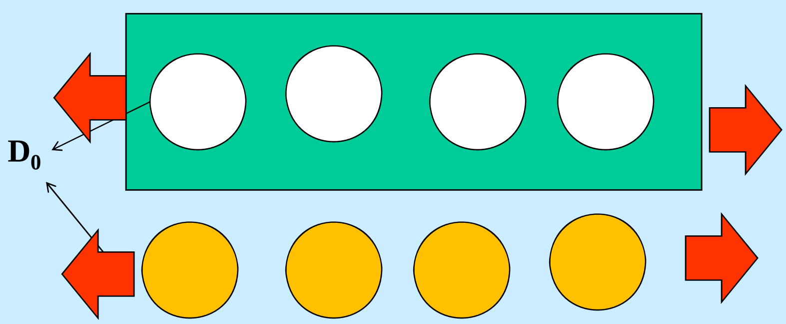


The surface layer is compressed from $\Delta L+L_0$ to $\delta+L_0$ by the bulk (compressive residual stresses).
The bulk layer is pulled to to $\delta+L_0$ from to L_0 by the surface layer (tensile residual stresses). ***MACRO RESIDUAL STRESSES***

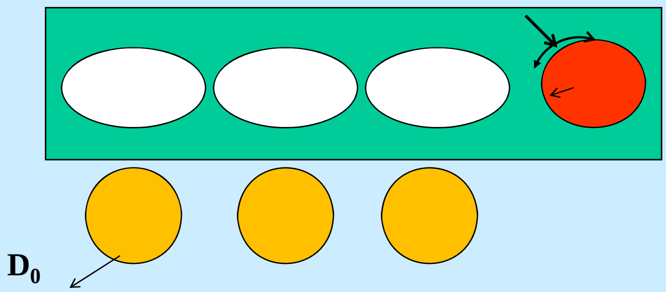
Micro residual stresses



Assume matrix is much softer than the second-phase particles.



Apply a load to the composite that can cause plastic flow in the matrix but not in the precipitates.



The holes in the matrix will no longer have the same shape or diameter as the precipitates.
To fit each precipitate in its hole a complex stress state must be applied. MICRO RESIDUAL STRESSES

In polycrystalline materials we usually measure the sum of macro and micro residual stresses.

The relative magnitudes of these terms depend on the measurement volume.

If you need a "bulk-average" stress, we need to measure a large volume.

If local stresses are needed, than small regions are sampled.

Strain/Stress Determination Techniques with Diffraction

1-Lattice parameter measurements:
-Polycrystals
-Single crystals
2-Curvature measurements
Single crystals
3-X-ray topography measurements
Single crystals

Lattice spacing measurements

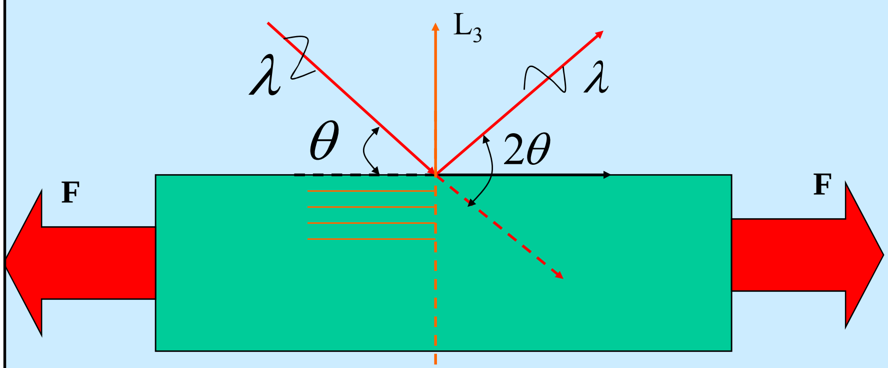
- All techniques start from the measurement of a plane spacing via Bragg's law.

$$\lambda = 2d\sin\theta$$

Once the "d" spacing is determined it can be transformed into a strain:

$$\varepsilon = \frac{d - d_0}{d_0}$$

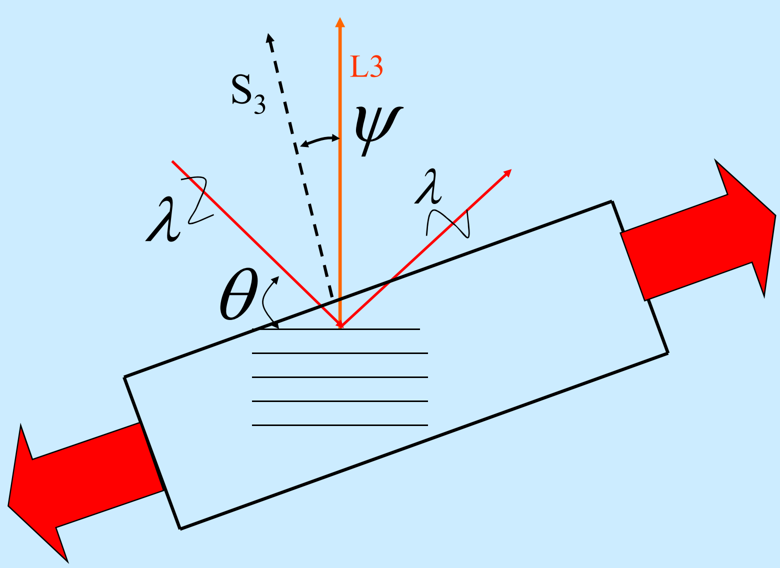
Unstressed plane spacing



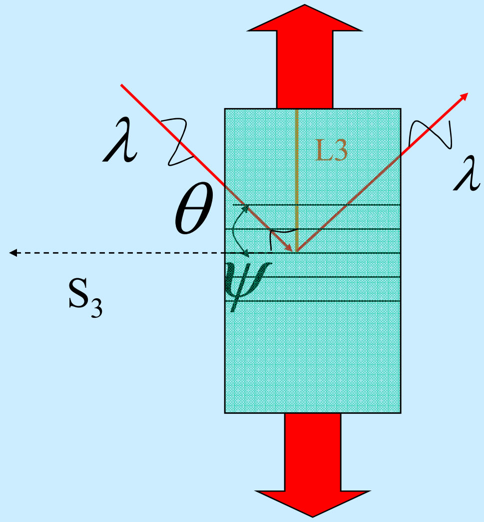
This strain is along the "L_3" direction; the normal to the diffracting planes.

For a tensile applied load,, the strain along the measurement vector @ ψ=0 is the minimum.

$$\frac{d_\psi - d_0}{d_0} = \varepsilon'_{33} = -\nu * \varepsilon_{11}$$

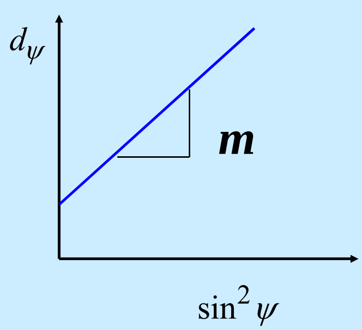


As ψ-tilt increases, the strain L3 increases.



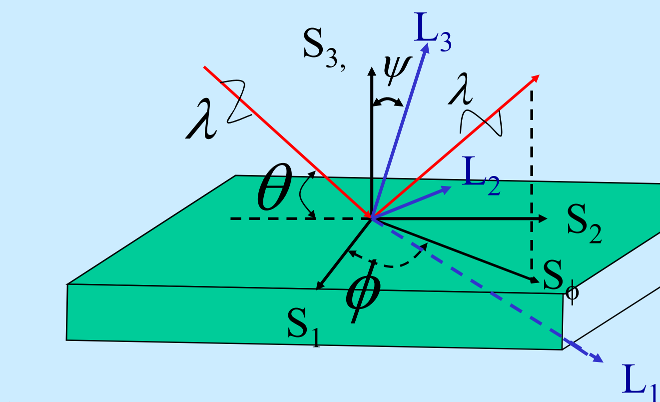
At 90^o ψ-tilt, the strain along the measurement vector is maximum.

- Thus, if the lattice parameter increases with tilt angle (from Poisson contraction to tensile expansion) we have tensile stresses.
- If the lattice parameter decreases with tilt angle (from Poisson expansion to compression) we have compressive stresses.

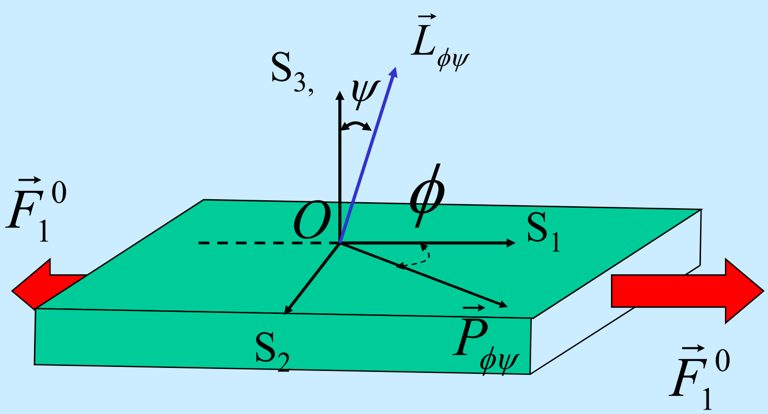


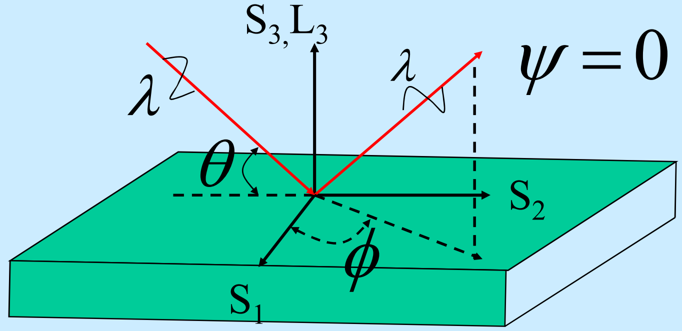
$m > 0$ *tensile stress*

$m < 0$ *compressive stress*



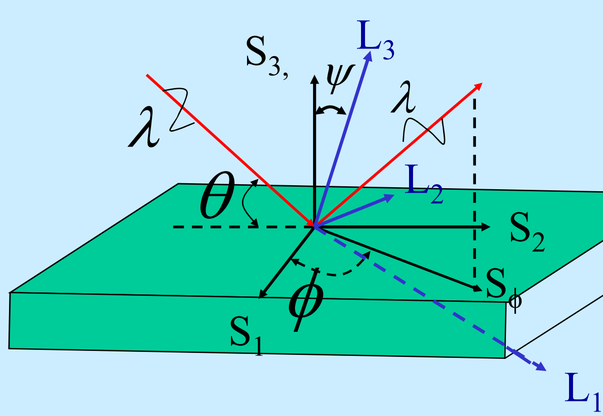
- There are two coordinate systems:
  - **S** defines the sample surface.
  - **L** defines the measurement.
    - Only $\mathbf{L_3}$ is used.
    - For each ψ-tilt, a new **L** system is defined.





The strains along all these different directions are related to the strains in the sample coordinate system by a simple transform.

$$\varepsilon'_{33} = \frac{d_{\phi,\psi} - d_0}{d_0} = \underbrace{a_{3k} a_{3l}}_{\text{direction cosines}} \varepsilon_{kl}$$



$$a_{ij} = \begin{bmatrix} \cos\phi\cos\psi & \sin\phi\cos\psi & -\sin\psi \\ -\sin\phi & \cos\phi & 0 \\ \cos\phi\sin\psi & \sin\phi\sin\psi & \cos\psi \end{bmatrix}$$

The direction cosines linking the two systems (**S** & **L**)

$$\varepsilon'_{33} = \frac{d_{\phi,\psi} - d_0}{d_0} = a_{3k} a_{3l} \varepsilon_{kl} =$$

$$\varepsilon_{11} \cos^2\phi \sin^2\psi + \varepsilon_{22} \sin^2\phi \sin^2\psi + \varepsilon_{12} \sin\phi \cos\phi \sin^2\psi$$

$$+ \varepsilon_{33} \cos^2\psi$$

$$+ \varepsilon_{13} \cos\phi \sin 2\psi + \varepsilon_{23} \sin\phi \sin 2\psi$$

This is ***the*** *fundamental diffraction equation.*

It has six unknown strains.

We can also substitute for the strains in terms of stresses using Hooke's law.

$$\frac{d_{\phi,\psi} - d_0}{d_0} = a_{3k} a_{3l} S_{klmn} \sigma_{mn}$$

This equation is in terms of stresses, but we really determine strains!

For any homogeneous specimen, we can simulate the expected variation of d_ψ with $\sin^2\psi$.

In the case of an isotropic specimen, the above equation becomes:

$$\frac{d_\psi - d_0}{d_0} =$$

$$\boxed{\frac{1+\nu}{E}(\sigma_{11}\cos^2\phi + \sigma_{22}\sin^2\phi + \sigma_{12}\sin\phi\cos\phi - \sigma_{33})\sin^2\psi}$$

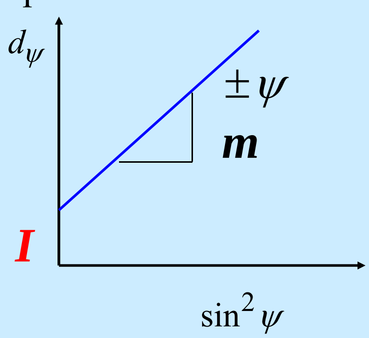
$$+\frac{1+\nu}{E}\sigma_{33} - \frac{\nu}{E}(\sigma_{11}+\sigma_{22}+\sigma_{33})$$

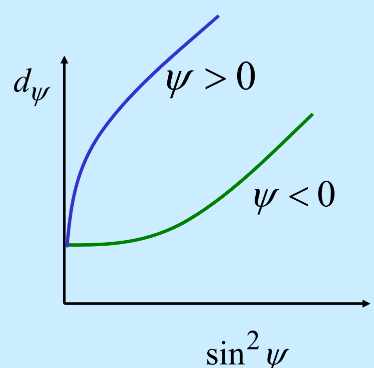
$$\boxed{-\frac{\nu}{E}(\sigma_{13}\cos\phi + \sigma_{23}\sin\phi)\sin 2\psi}$$

This equation has a term that is linear for +/- ψ

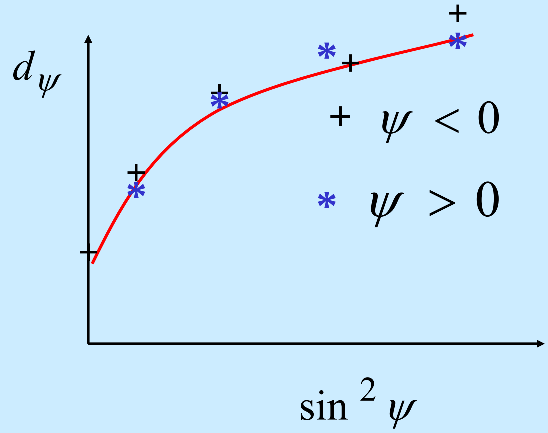
And a term that changes sign with +/- ψ

Thus, we can predict three types of d_ψ vs. $\sin^2\psi$ plots.





$$\begin{pmatrix} \sigma_{11} & \sigma_{12} & 0 \\ & \sigma_{22} & 0 \\ & & 0 \end{pmatrix} \qquad \begin{pmatrix} \sigma_{11} & \sigma_{12} & \sigma_{13} \\ & \sigma_{22} & \sigma_{23} \\ & & \sigma_{33} \end{pmatrix}$$



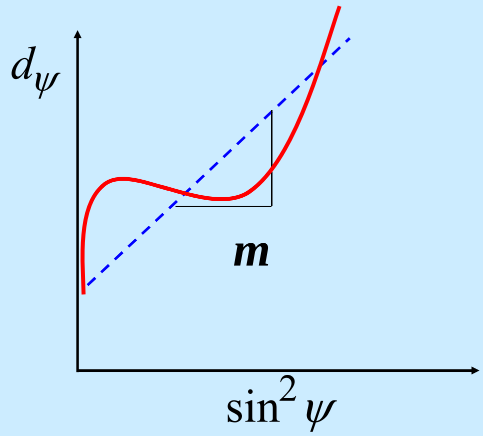
$$\begin{pmatrix} \sigma_{11} & \sigma_{12} & 0 \\ & \sigma_{22} & 0 \\ & & \sigma_{33} \end{pmatrix}$$

Summary of Expected Behaviour

- ❖Linear model: $\sigma_{33} = 0$
  - ❖dψ+ = dψ- for all ψ.
  - ❖All measured d_ψ must fall on a straight line within measurement error.
- ❖Triaxial model
  - ❖dψ+ ≠ dψ- for all ψ.
  - ❖Their sum, and difference, however, must fall on straight lines.

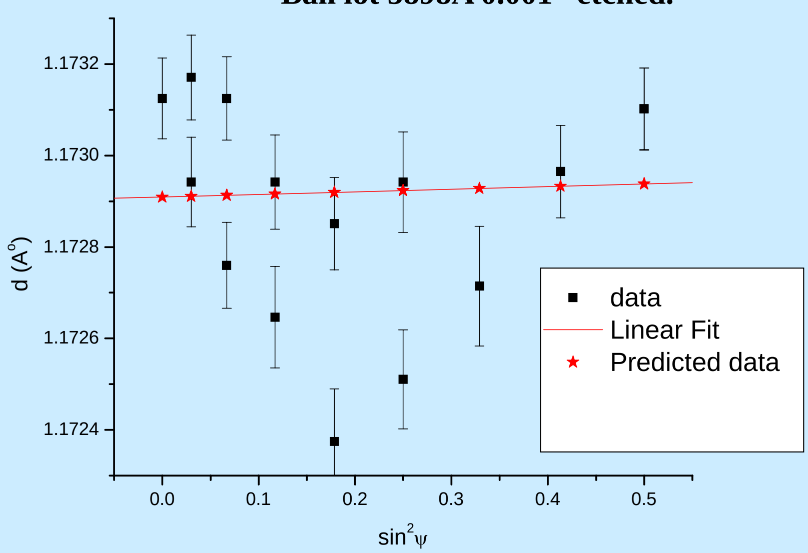
There are also oscillatory d vs. $\sin^2\psi$ plots.

One should not treat oscillatory data as a straight line.

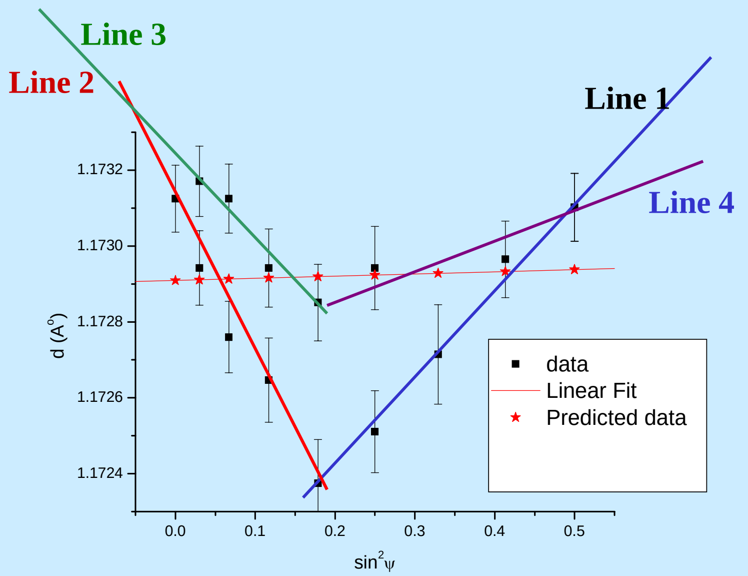


. Data that do not fit indicate that the model is inappropriate.

Ball lot 5898A 0.001" etched.



Stress from slope~1 ksi

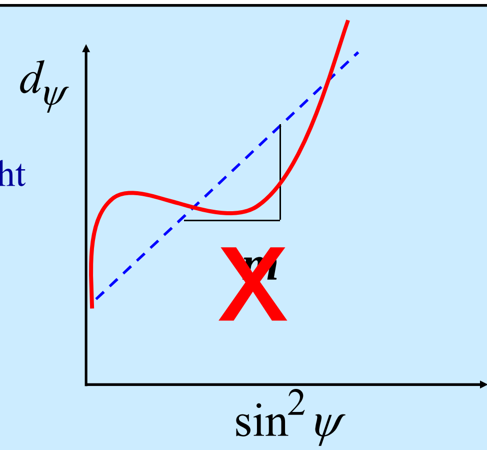


Ball lot 5898A 0.001” etched.

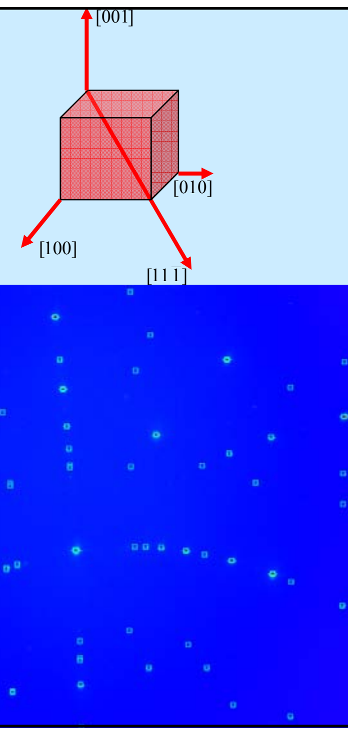
One should not treat oscillatory data as a straight line.

Neither should one cherry-pick the data and discard the points that do not fit.

Measuring only two tilts is OK if you know that the data in-between is linear.

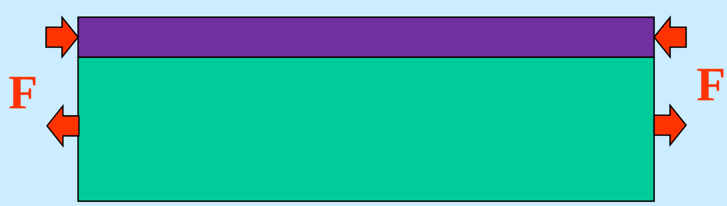


The treatment given so far does not need to assume the type of sample.
Applicable to single crystals as well.
For these materials, the ψ-angles are not arbitrary; they are given by the symmetry of the material.
One can simply measure the plane spacing from various reflections and then use the angles between these lattice vectors and the sample coordinates to do the tensor transformation.
The lattice parameter vs. angle plots, however, must obey the previous equations.



Curvature Methods

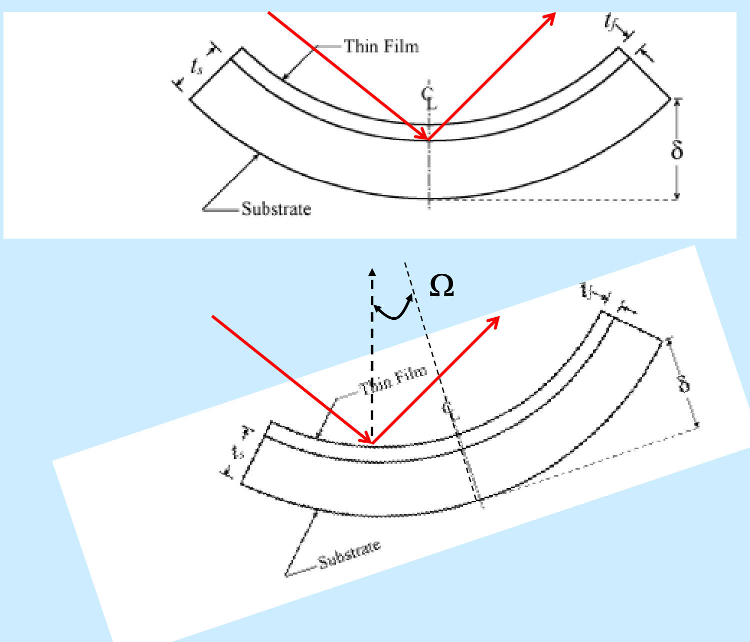
Macro residual stresses existing at one surface cause curvature.
One can note that the moments taken at the sample mid-plane are not balanced.



One can measure the curvature and calculate the stress using various equations. Stoney's equation is used frequently.

$$\sigma_f = \left(\frac{E_s}{3(1-\nu_s)}\right)\left(\frac{t_s^2}{t_f a^2}\right)\delta$$

E_s: Young's modulus of substrate,
ν_s: Poisson's ratio of substrate,
t_s: substrate thickness
t_f:film thickness
a: initial curvature of substrate
δ: deflection of substrate



For single crystal substrates, one can translate the sample and measure the sample rotation angle, Ω_x, at which the Bragg reflection is obtained.

If this is repeated at various positions, and Ω_x vs. x is plotted, the radius of curvature is obtained from the slope.

One can also obtain the slope from laser interferometry or optical comparator measurements.

X-rays yield very accurate measurements for rotations.

This is a differential measurement.

Topographic Stress/Strain Determination

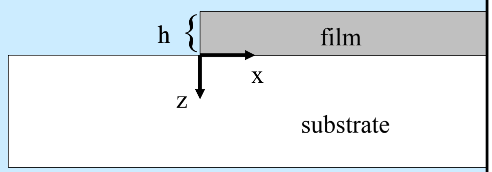
This technique is applicable to single crystal systems scattering in the dynamical regime.

In topographic measurement, we measure the integrated intensity from the substrate as a function of position.

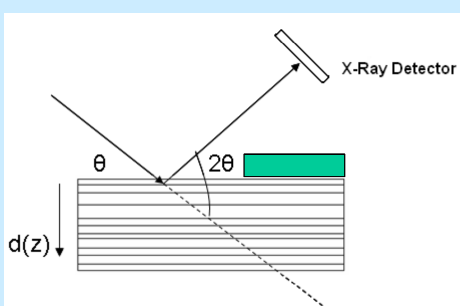
This intensity is then modeled using mechanics and diffraction equations.

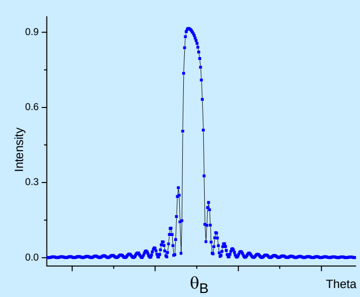
Residual stress profile of a step-edge

- Sample: Etched step-edge;pseudomorphic film of $Si_{0.8}Ge_{0.2}$ on 001 Si, 90 nm thick.
- 11.2keV incident energy.
- 5µm x-ray spot.
- We did both "d" spacing and incident intensity maps.

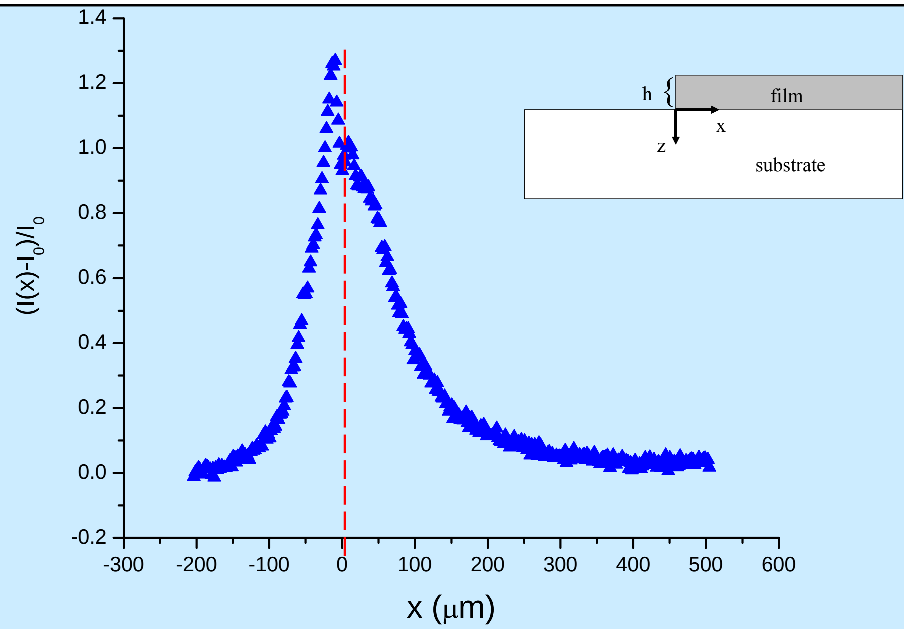


- The "d" spacing analysis yielded nonsensical results.

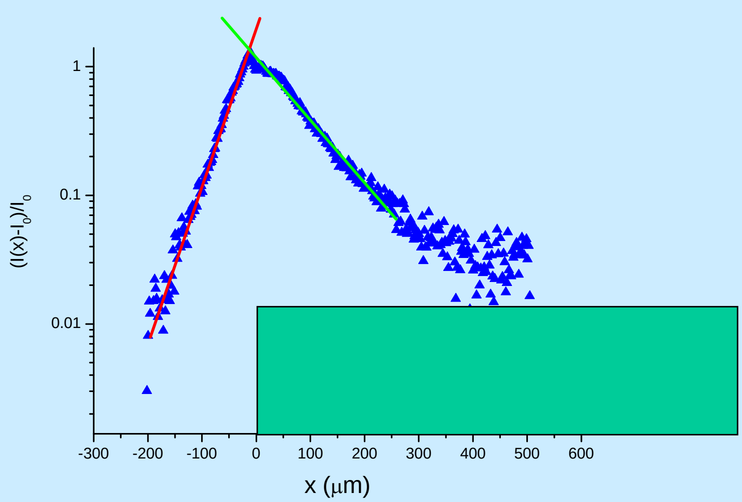




In an intensity mapping measurement we measure the integrated intensity from the substrate as a function of position.



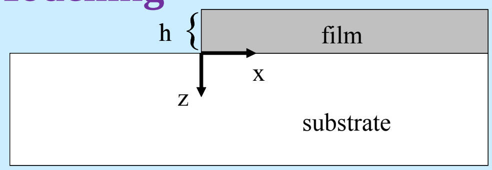
- The profile consists of two distinct intensity maxima bracketing the film edge.



The decay rate outside the feature is 2x the rate inside the feature,

Mechanics Modelling

The strain distribution can be obtained from the edge-force model:



$$\varepsilon_{zz}(x,z)=\frac{1}{E_s}\left[(1-\nu_s^2)\sigma_{zz}-\nu_s(1+\nu_s)\sigma_{xx}\right]=$$

$$-\Delta\varepsilon\frac{2E_f\ h}{E_s\pi(1-\nu_f)}\left[\frac{(1-\nu_s^2)xz^2-\nu_s(1+\nu_s)x^3}{(x^2+z^2)^2}\right]$$

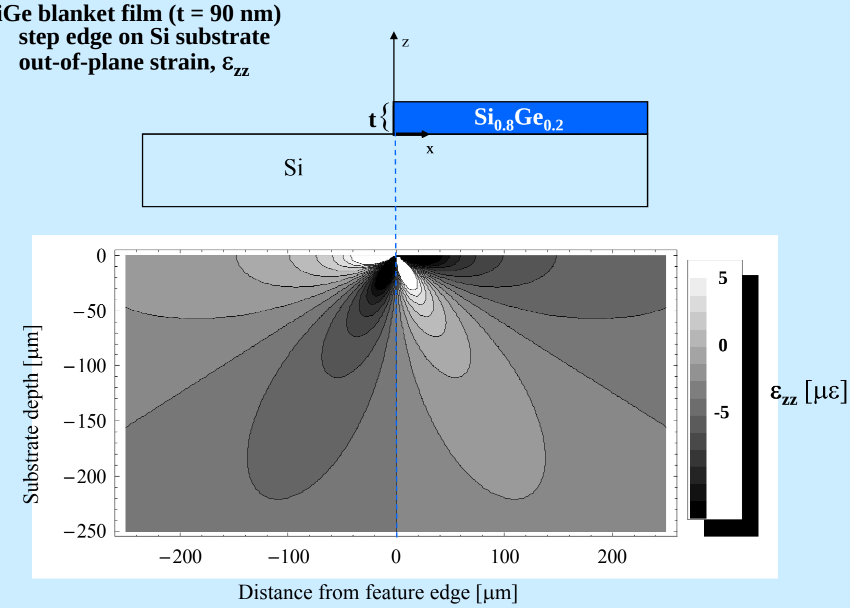
$\Delta\varepsilon$ is the mismatch strain between the film and substrate (0.0076 for Si0.8Ge 0.2).

ε_{zz} is an odd function of x about the feature edge; in the normal direction the substrate experiences compression outside the feature (x<0) or tension under it.

Substrate strain field: edge-force approximation

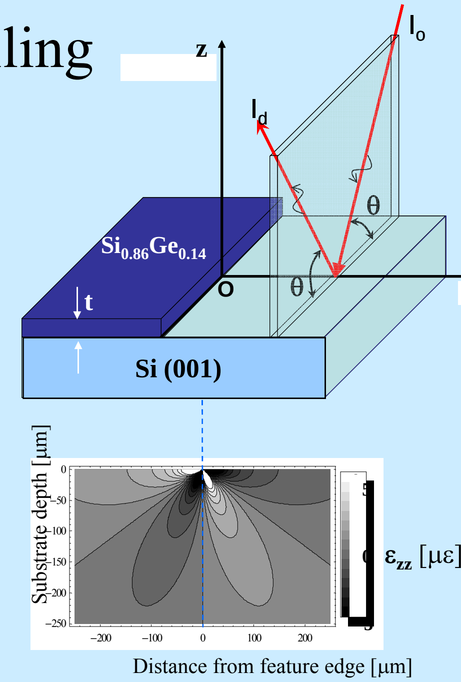
SiGe blanket film (t = 90 nm)

- **step edge on Si substrate**
- **out-of-plane strain, ε_{zz}**



Diffraction Modelling

- Approximate the distorted crystal as a stack of perfect crystal lamellae with different out-of-plane lattice constants, c(z).
- Obtain a recurrence relation that calculates the diffraction ratio (diffracted wave amplitude, D_h, over transmitted wave amplitude, D_0) on the top ($z = z_T$) and bottom ($z = z_B$) boundaries of a lamella by integrating the Takagi-Taupin equations.



$$X_T = \frac{wX_B - i(2C\chi_h + \xi X_B)\tan[\Phi(z_B - z_T)/2]}{w + i(2gC\chi_{\bar{h}} X_B + \xi)\tan[\Phi(z_B - z_T)/2]}$$

$$X = \frac{D_h}{D_0}$$

$$g = \cos\gamma_B / \cos\beta_B$$

$$\xi = (1+g)\chi_0 + 2\eta \sin 2\theta_B$$

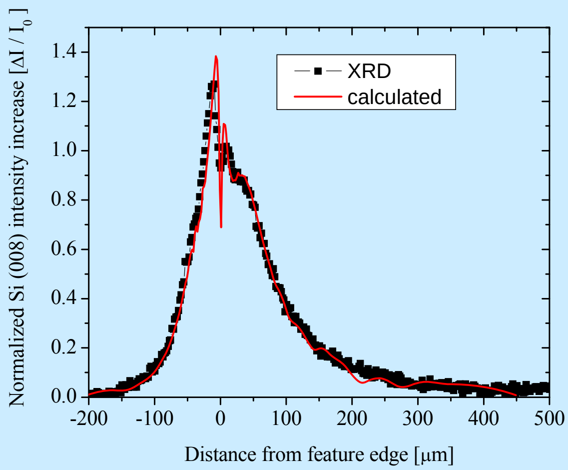
$$w = \sqrt{\xi^2 - 4C^2 g \chi_h \chi_{\bar{h}}}$$

$$\Phi = \pi K w / \cos\gamma_B$$

g is the geometry factor (β, γ represent the angles of the incident and diffracted x-ray beams).

χ are the Fourier coefficients of the susceptibility of the material,

θ_B is the Bragg angle of the unstrained crystal calculated from the Bragg's law, η is the deviation of the incidence angle from the *local* exact Bragg angle, *C* is the polarization factor, *K* is the wave number of the incident x-ray in vacuum.

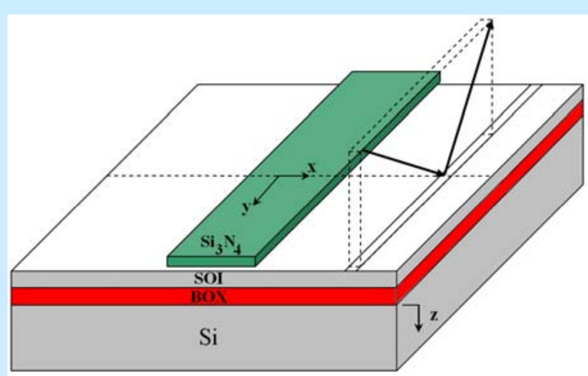


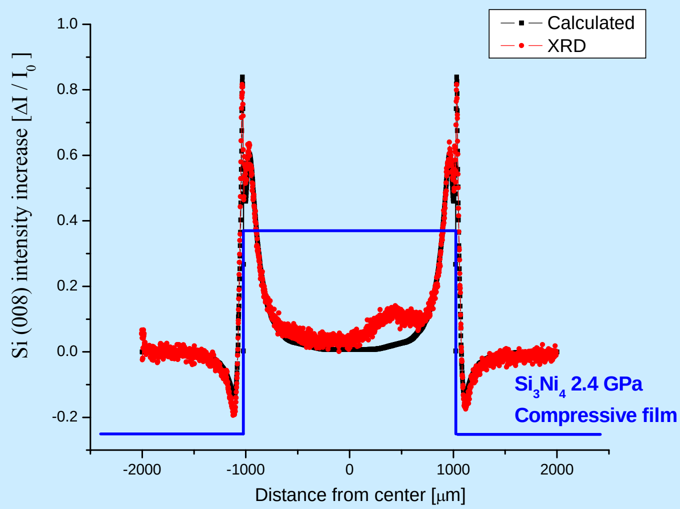
It fits rather well.

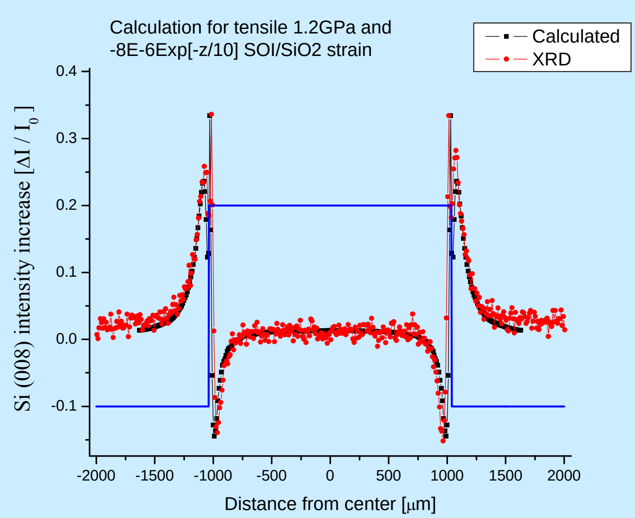
Testing the model

We then used a more complicated structure.

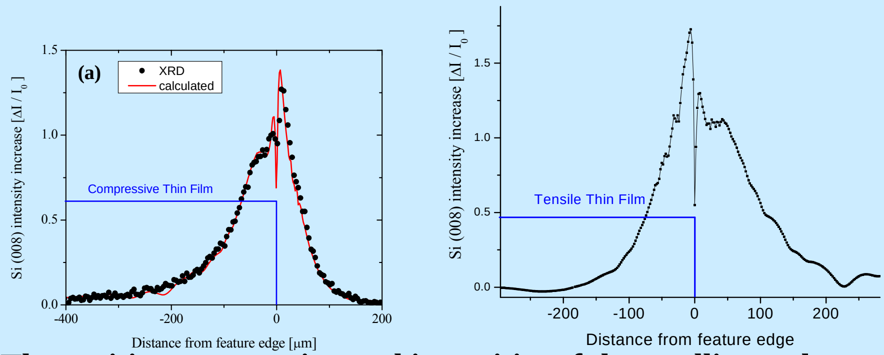
The nitride feature could be made to have tensile or compressive stresses.







Major and minor peaks switch if the film has "tensile" stresses.

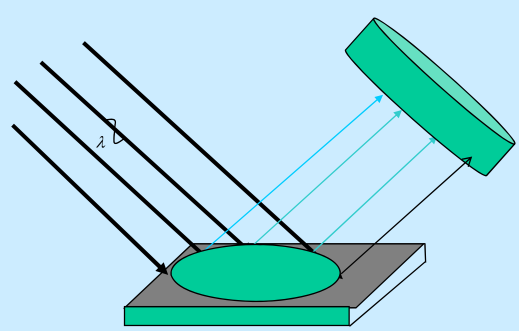


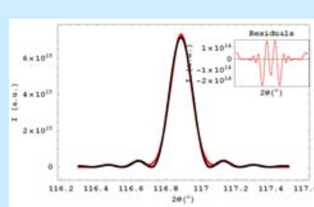
The positions, separation and intensities of the satellite peaks depend on the magnitude and sign of the strain field.
By comparing modeled and measured diffraction profiles, one can determine the full strain distribution.
Inverse problem: Agreement shows possible solution only.

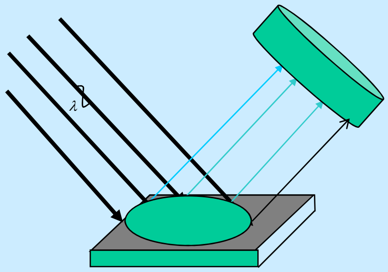
Kalenci, Murray, Noyan, JAP 104 063503 (2008)

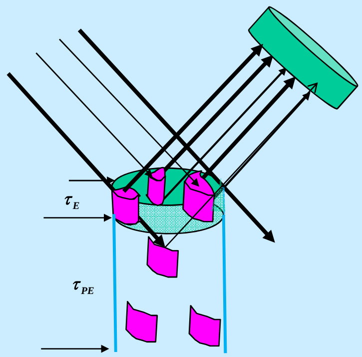
Information Volumes

- Diffraction peak/ plane spacing measurements
  - **Global averaging methods:** The diffracting volume is defined by the incident and diffracted beam optics , the penetration depth of the radiation and the sample scattering function.









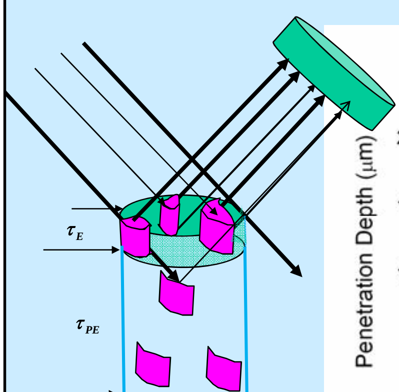
Scattering Volume Definition

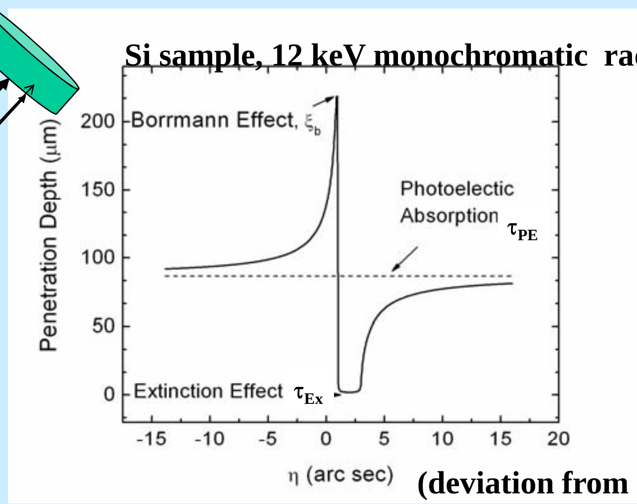
If the sample is a single crystal:

For $t < \tau_{extinction}$, the entire irradiated volume diffracts.

For $t > \tau_{extinction}$, the diffracting volume is a slanted cylinder with the thickness $\sim \tau_{extinction}$.

For a polycrystalline material, where the grain thickness is $\sim< \tau_{extinction}$, then only those grains in the diffraction condition within the photoelectric absorption depth scatter.



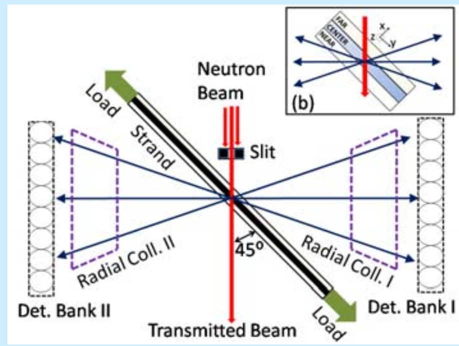


***Definition of the measurement volume is not simple** for samples with complex topologies, strain distributions, or when spherical incident beams are used.*

Diffraction peak / plane spacing measurements

- Real-space (ray tracing) methods: A subset within the diffracting volume is localized through the use of suitable apertures in the incident and diffracted beam optics

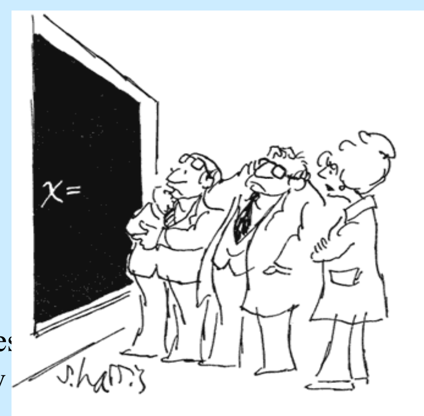
Radial or conical collimators in neutron diffraction.
Apertures or wire-scanning in x-rays



Definition of the measurement volume is simpler for kinematically *scattering samples.*
These methods do NOT work for dynamically scattering samples.

Observations

- Designing diffraction experiments to measure applied stress/strain distributions is straightforward.
- Measurement of residual strain distributions may require significant work.
- Cavalier application of these techniques can yield puzzling results; Bragg's law is more complicated that one would expect from $\lambda = 2d \sin\theta$
- When properly used, these techniques can yield invaluable data.



Acknowledgments:

IBM Watson Research:
- **Conal Murray**
- J. Chu
- G. Cohen
- J. Jordan-Sweet

Columbia University:
- **Ozgur Kalenci (GS)**
- **Hanfei Yan (NSLSII)**
- S. Polvino
- **Andrew J. Ying**

Argonne National Laboratory:
- J. Maser
- Z. Cai

D.O.E. Basic Energy Sciences, W-31-109-Eng-38
D.O.E. Materials Sci. & Chemicals Sci, DE-AC02-98CH1886

THANK YOU FOR YOUR ATTENTION.