Analytical Methods for Materials

Laboratory Module #6
Residual Stress Determination

Suggested Reading

B.D. Cullity and S.R. Stock, *Elements of X-ray Diffraction, 3rd Edition*, (Prentice-Hall, Upper Saddle River, NJ, 2001), Ch. 15, pages 435-470.

- **What about residual stress calculations?**
    - Best reference:

        I.C. Noyan and J.B. Cohen: <u>Residual Stress :</u>
        <u>Measurement by Diffraction and Interpretation</u>, New York :
        Springer-Verlag (1987).

- **Not a trivial undertaking**

General Stress/Strain Effects

- Elastic strain is related to the stretching of atomic bonds

    THUS,

- Elastic strain alters lattice spacing

- Rough estimate

    $$\beta = \Delta 2\theta = -2(\Delta d/d)\tan\theta$$

    or

 $$\Delta 2\theta \propto \Delta d/d \qquad \text{(Fractional variation in lattice spacing)}$$

- The actual relationship is:
    - $\Delta\sin\theta \propto \Delta d/d$, but $\Delta\theta$ is very small.

- For elastic strains, the change in lattice spacing is directly related to stress
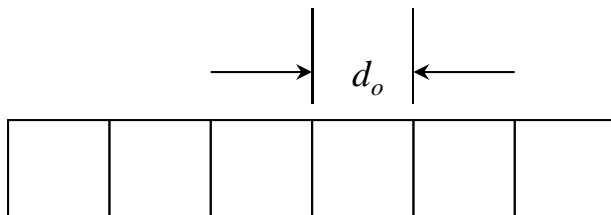   - $\sigma = \varepsilon E$ (Hooke's Law)

- To determine strain effects, we need an accurate determination of the lattice parameter in the unstressed material.

- We also need to consider the inhomogeneous nature of surface stresses

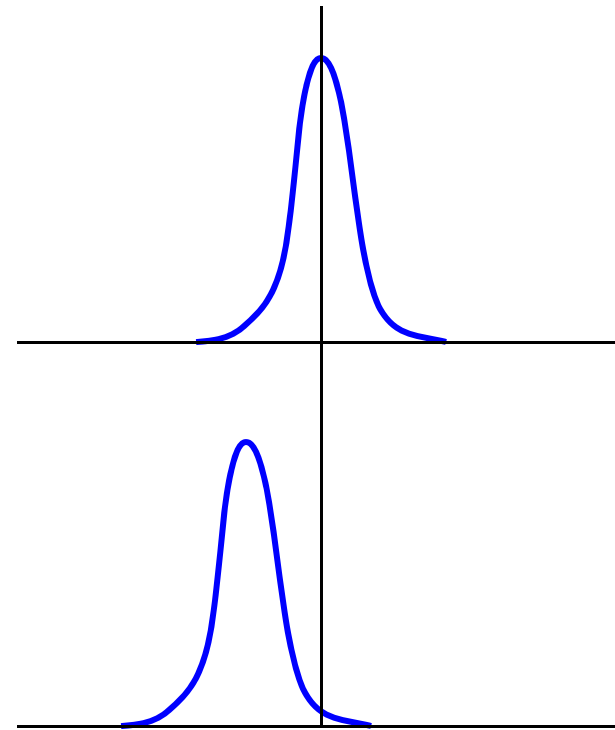- Difficulty in preparing suitable samples.

- ## There is no peak broadening with uniform strain, only peak shifting. Why?
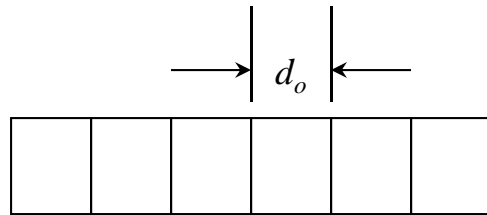
No lattice strain

Uniform lattice strain

- ## Strain is uniform for all planes

- There is peak broadening with non-uniform strain.

d_o

No lattice strain

tension

compression

Non-uniform lattice strain
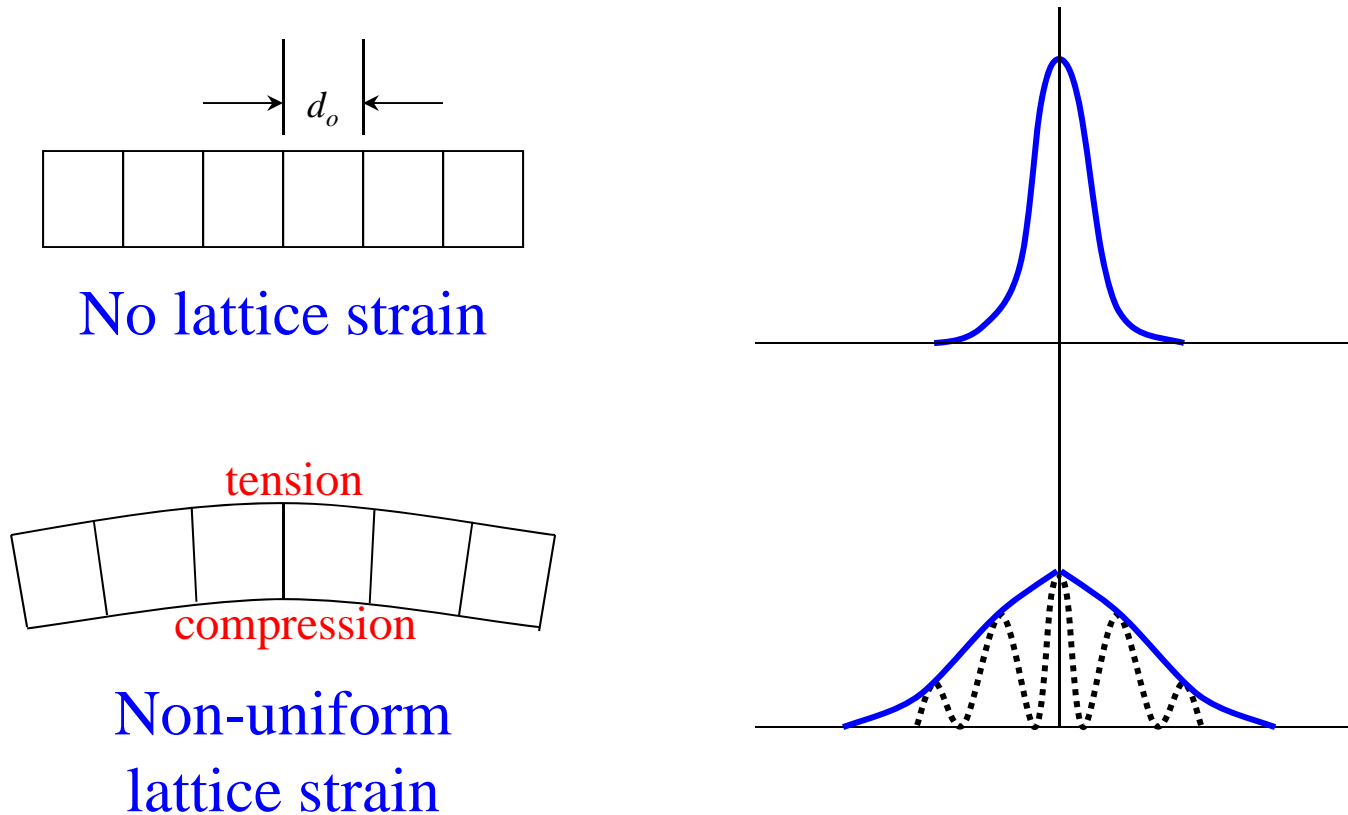
- Wide range of *d*-spacings $\Rightarrow$ peak broadening.

To do this accurately:

- We must take into account uniform and non-uniform strain.

- We must have:

  - accurate peak shapes

  - positions for multiple reflections.

RESIDUAL STRESS

- Lattice parameter changes are the result of elastic strains (*i.e.*, stretching of atomic bonds)

- Plastic strain causes inhomogeneous strains.
  - Nucleation and motion of dislocations (non-uniform strain fields).

- Stress is not intrinsic. Strain is intrinsic.
  We measure strain and use it to calculate stress.

Remember your basic mechanics

- Strain, $\varepsilon = \Delta l/l$, which is equivalent to $\Delta d/d$.

- We need to know d very accurately.

- Assume linear elastic behavior.
  - $\sigma = \varepsilon E$
  - $\tau = \gamma G$ (in terms of shear stress/strain)

X

Z

y

σ_x

σ_x
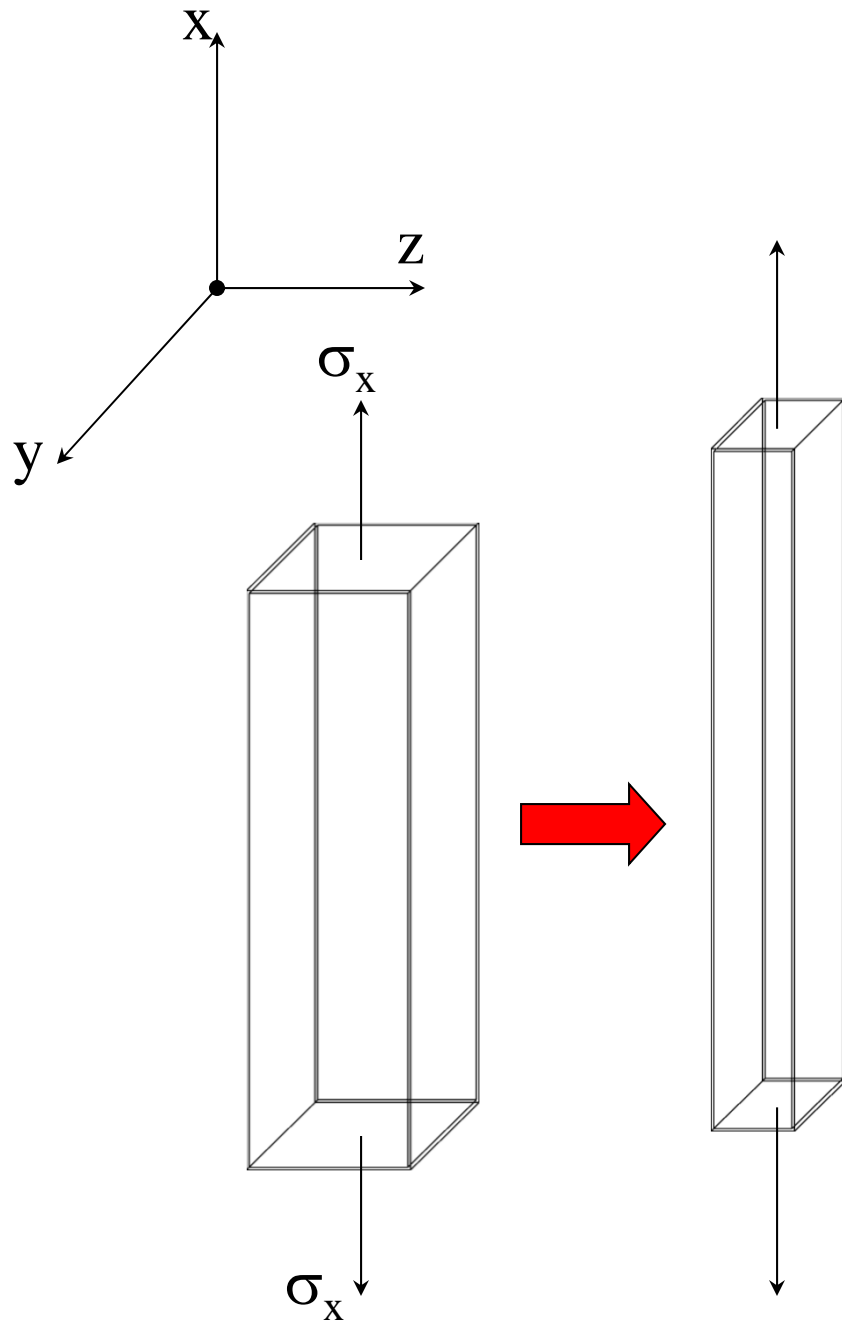
- During uniaxial tensile deformation a test bar must contract in the y and z directions as indicated.

- Poisson's Ratio

$$-\varepsilon_y = -\varepsilon_z = \nu\varepsilon_x$$

783

In terms of normal stresses

(select axes such that shear stress = 0)

- $\varepsilon_x = (1/E)[\sigma_x - \nu(\sigma_y + \sigma_z)]$
- $\varepsilon_y = (1/E)[\sigma_y - \nu(\sigma_x + \sigma_z)]$
- $\varepsilon_z = (1/E)[\sigma_z - \nu(\sigma_x + \sigma_y)]$

OR

- $\varepsilon_1 = (1/E)[\sigma_1 - \nu(\sigma_2 + \sigma_3)]$
- $\varepsilon_2 = (1/E)[\sigma_2 - \nu(\sigma_1 + \sigma_3)]$
- $\varepsilon_3 = (1/E)[\sigma_3 - \nu(\sigma_1 + \sigma_2)]$

Method

- YOU CANNOT MEASURE INTERNAL RESIDUAL STRESS! It is an extrinsic property

- Measure the strain in the crystal lattice

- calculate the residual stress producing the strain assuming linear elastic distortion of the crystal.

XRD residual stress

- Strain must be measured for at least two precisely known orientations relative to the surface of interest.

- XRD residual stress is suitable for fine-grained crystalline materials.

- NON-DESTRUCTIVE!

Macroscopic & microscopic stresses

- Macro-
  - extend over large distances relative to the grain size
  - tensor quantities that vary in magnitude with direction at a single point in a body.

- Micro-
  - scalar quantity resulting from imperfections within the crystal lattice.
  - Associated with "short range" strains
    (< dimensions of lattice).
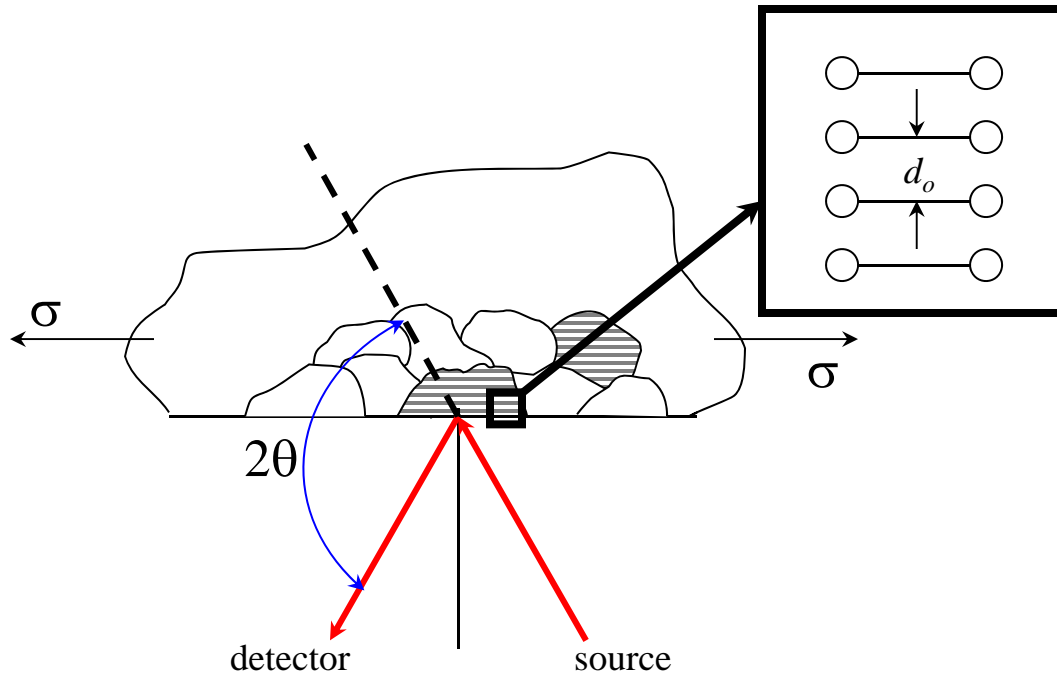
Sample at angle ψ=0
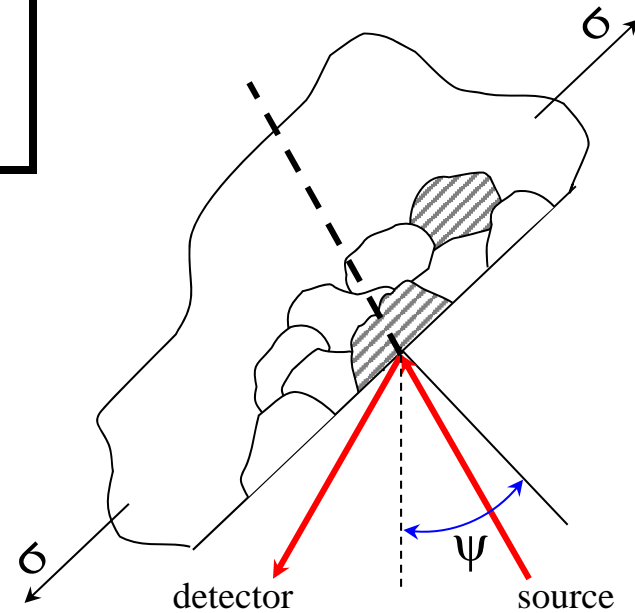
Sample rotated through a known angle ψ=0

- At $\psi=0$, tensile stress causes a decrease in the local d-spacing (THUS an increase in 2θ).

- Rotation through some angle, ψ, causes an increase in lattice spacing over the stress-free state (THUS a decrease in 2θ)

- MEASURE d at several ψ's. Calculate the stress at that specific volume by considering orientations of grains being sampled.

- **Residual stress determined using XRD is an average stress in a volume material which is defined by:**
  - the irradiated area
  - the penetration depth of the x-ray beam.
    - Governed by linear absorption coefficient of the materials for the radiation used.

- **Choice of diffraction peak for measurement is important!**
  - Higher angles lead to greater precision.

Plane-Stress Elastic Model

- Recall from MTE 455
  - Plane stress: $\sigma_3 = 0$, $\varepsilon \neq 0$

- Strain in direction defined by angles ϕ and ψ

$$\varepsilon_{\phi\psi} = \left[\frac{1+\nu}{E}(\sigma_1 \alpha_1^2 + \sigma_2 \alpha_2^2)\right] - \left[\frac{\upsilon}{E}(\sigma_1 + \sigma_2)\right]$$

$$\alpha_1 = \cos\phi\sin\psi$$

$$\alpha_2 = \sin\phi\sin\psi$$

Plane-Stress Elastic Model

- Substituting for the angle cosines (α_x) allows us to simplify the expression and to express it in terms of the orientation angles:

$$\varepsilon_{\phi\psi} = \left[\frac{1+\nu}{E}(\sigma_1 \cos^2\phi + \sigma_2 \sin^2\phi)\sin^2\psi\right] - \left[\frac{\upsilon}{E}(\sigma_1 + \sigma_2)\right]$$

- At ψ=90°, the strain vector lies in the plane of the surface.
- The surface stress component, σ_ϕ becomes:

$$\sigma_\phi = (\sigma_1 \cos^2\phi) + (\sigma_2 \sin^2\phi)$$

- Substitute this expression into the previous equation yields the strain in the sample surface at an angle ϕ from the principal stress σ_1

$$\varepsilon_{\phi\psi} = \left[\frac{1+\nu}{E}(\sigma_\phi \sin^2 \psi) \right] - \left[\frac{\upsilon}{E}(\sigma_1 + \sigma_2) \right]$$

- This expression relates the surface stress in any direction defined by ψ to the strain in the direction (ϕ,ψ) and the principal stresses in the surface.

- If $d_{\phi\psi}$ is the lattice spacing measured in the direction (ϕ,ψ), then the strain is:

$$\varepsilon_{\phi\psi} = \frac{\Delta d}{d} = \frac{d_{\phi\psi} - d_o}{d_o}$$

- If d_o is the stress-free lattice spacing

- Substituting,

$$\frac{d_{\phi\psi} - d_o}{d_o} = \left[\left(\frac{1+\nu}{E}\right)_{hkl}(\sigma_\phi \sin^2 \psi)\right] - \left[\left(\frac{\upsilon}{E}\right)_{hkl}(\sigma_1 + \sigma_2)\right]$$

- The elastic constants vary with crystallographic direction.

- The lattice spacing for any orientation is:

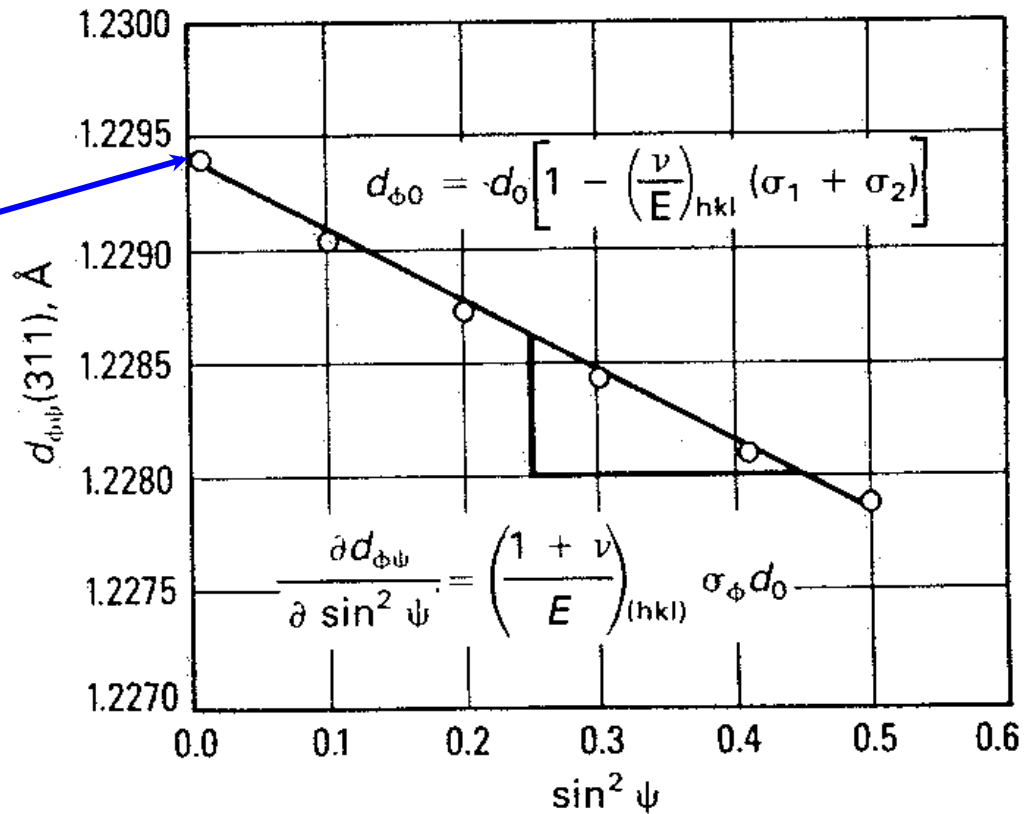$$d_{\phi\psi} = \left[\left(\frac{1+\nu}{E}\right)_{(hkl)}\sigma_\phi d_o \sin^2 \psi\right] - \left[\left(\frac{\upsilon}{E}\right)_{(hkl)}d_o(\sigma_1 + \sigma_2) + d_o\right]$$

- Equation relates lattice spacing and biaxial stresses at sample surface.

- The lattice spacing is a linear function of $\sin^2\psi$

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)

Intercept corresponds to unstressed lattice spacing

$$d_{\phi 0} = d_0\left[1 - \left(\frac{\nu}{E}\right)_{hkl}(\sigma_1 + \sigma_2)\right]$$

$$\frac{\partial d_{\phi\psi}}{\partial \sin^2\psi} = \left(\frac{1 + \nu}{E}\right)_{(hkl)}\sigma_\phi d_0$$

Vertical axis: $d_{\phi\psi}(311)$, Å

Values: 1.2300, 1.2295, 1.2290, 1.2285, 1.2280, 1.2275, 1.2270

Horizontal axis: $\sin^2\psi$

Values: 0.0, 0.1, 0.2, 0.3, 0.4, 0.5, 0.6

- At $\sin^2\psi = 0$

$$d_{\phi\psi} = d_o - \left[\left(\frac{\upsilon}{E} \right)_{(hkl)} d_o(\sigma_1 + \sigma_2) \right]$$

- This represents the unstressed lattice spacing minus the Poisson's ratio contraction.

- The slope of the plot is:

$$\frac{\partial d_{\phi\psi}}{\partial \sin^2\psi} = \left[\left(\frac{1+\upsilon}{E} \right)_{(hkl)} \sigma_\phi d_o \right]$$

which is the same as

$$\sigma_\phi = \left(\frac{E}{1+\nu} \right)_{(hkl)} \frac{1}{d_o} \left(\frac{\partial d_{\phi\psi}}{\partial \sin^2\psi} \right)$$

- The unstressed lattice spacing is generally unknown.

- However, $E >> (\sigma_1 + \sigma_2)$

- Thus, $d_{\phi\psi}$ usually differs from d_o by less than 1%

- Therefore:

$$\sigma_\phi = \left(\frac{E}{1+\nu}\right)_{(hkl)} \frac{1}{d_{\phi o}} \left(\frac{\partial d_{\phi\psi}}{\partial \sin^2 \psi}\right)$$

- You don't have to collect stress free measurements!

Techniques for Measurement

- Single-angle
  - Not as sensitive as other methods

- Two-angle
  - Well accepted in the USA
  - Very sensitive within limits of sample geometry and Bragg angle

Two-angle technique

- Measure $d_{\phi 0}$ ($\psi = 0°$) and $d_{\phi 45}$ ($\psi = 45°$)

- Assume linearity of the following equation

$$\sigma_\phi = \left(\frac{E}{1+\nu} \right)_{(hkl)} \frac{1}{d_{\phi o}} \left(\frac{\partial d_{\phi\psi}}{\partial \sin^2 \psi} \right)$$
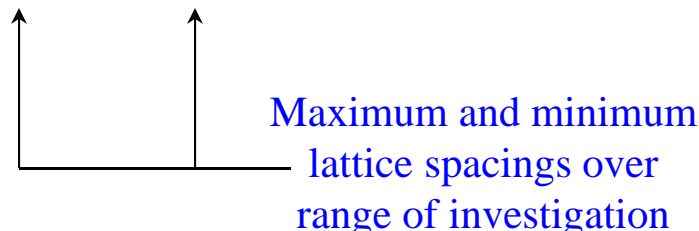
Methods

- $\sin^2 \psi$ method

  - Collect data for multiple ψ's and plot.

  - Do regression

  - Well accepted in Germany and Japan

  - No more sensitive than 2 angle method.

Another Method

- ## Marion-Cohen Method
    - for textured materials
    - characterizes the dependence of lattice spacing on stress.
    - Assumes a biaxial stress field and a dependence of interplanar spacings on texture distribution function, $f(\psi)$
    - $f(\psi)$ is a measure of the (hkl) pole density over the range of ψ tilts used for stress measurement

This model assumes the following lattice spacing dependence:

$$d_{\psi\phi} = \left(\frac{1+\nu}{E}\right)_{(hkl)} \sigma_\phi d_o \sin^2 \psi + (d_{max} - d_{min}) f(\psi) + d_{min}$$

Maximum and minimum lattice spacings over range of investigation

Requires SIMULTANEOUS measurement of texture along with lattice spacing

Solve by linear regression over the functions $f(\psi)$ and $d_{\psi\phi}$ as functions of $\sin^2 \psi$ to determine σ_ϕ, σ_{max}, and σ_{min}.

Practical application of this method is limited!

Assumes lattice spacing and preferred orientation present at the time of measurement result from the same origin

All of these methods yield nearly identical results for stresses produced by shot peening, grinding, or machining

You can find more details in
P.S. Prevey, *Advances in X-ray Analysis*, Vol. 19, 1976, p. 709

Sample Preparation

- Minimal if sample does not interfere with diffracted beam.

- Preparation of sample surface depends upon the stresses that you want to measure.

  - For stresses from surface treatments such as shot peening, grinding, or machining, the stress distribution is usually limited to <500 μm of the sample surface

- Must protect the surface from secondary abrasion, corrosion, etching, etc.

- Should oil samples to prevent oxidation or corrosion.

- Should pack samples carefully to protect the surface during handling.

- Should electropolish if post-treatment oxidation occurs to remove oxidized layer

- Should avoid cutting if possible. Sectioning can lead to relaxation effects.

- Should use strain gages to record strain relaxation during sectioning.

- Samples should be positioned precisely such that the true center of rotation of the ψ and 2θ axes and the angle ψ remains constant

Major sources of error

- ## Positioning of sample
  - needs to be ±0.025 mm

- ## Specimen
  - surface roughness and curvature lead to systematic error
  - coarse grains lead to peak asymmetry

Major sources of error

- ## XRD elastic constants
  - bulk values often differ by ±40% due to anisotropy

- ## Volume and Relaxation Effects

- **Use intermediate beam size.**
  - Small irradiated area: poor counting statistics
  - large irradiated area: larger area for averaging
  - intermediate size provides best of both

- **Peak locations must be determined precisely!**

- **More information can be found in the _ASM METALS HANDBOOK_, 10th Edition, Vol. 10, pp. 380-392**

RECAP!

- ## Setting up and running an x-ray scan

  - Two Approaches:
    - continuous scan $\Rightarrow$ good qualitative information
    - step scan $\Rightarrow$ good quantitative information

  - On newer instruments, the differences between continuous and step scans are less distinct.

Cont'd

- Scan rate, step size, and count time control your peak-to-background ratio/noise

- Can I resolve "small" peaks?

Cont'd

- Typical scan values
  - Rate: 1⁰ - 2⁰ 2θ/min
  - Step size: 0.02⁰ 2θ

- Value can be varied depending upon what you want to measure.

- Each experimenter must determine what is necessary.

- "THINK"