Measurement of Residual Stress by X-ray Diffraction

C-563

Colorado State University
Knowledge to Go Places

Overview

- Definitions
- Origin
- Methods of determination of residual stresses
- Method of X-ray diffraction (details)
- References
- End

Stress and Strain

- **Stress:** Force per unit area within materials that arises from externally applied forces, uneven heating, or permanent deformation and that permits an accurate description and prediction of elastic, plastic, and fluid behavior. **(Units N/m^2)**
- **Strain:** Relative deformation or change in shape and size of elastic, plastic, and fluid materials under applied forces **($\Delta L/L$).**

$$\frac{Stress}{Strain} = Young's\ Modulus$$

Stress

- **Tensile stress** is the stress that can be applied to an object by pulling on it, or attempting to stretch it. Positive values of stress indicate **tensile** stress

- **Compressive stress** is the stress applied to materials resulting to their compaction (decrease of volume). Negative values of stress indicate **compressive** stress

Residual Stresses

- **Definition**: Stresses that remain in material or body without application of an external load (applied force, displacement of thermal gradient).
- **Origin:** Usually originates during manufacturing and processing of materials due to heterogeneous plastic deformations, thermal contractions and phase transformations

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Types of Residual Stresses

- **Macro Residual Stresses (Type-I; σ^{I})**:
  - These stresses vary within the body of the component over a range much larger than the grain size
- **Micro Residual Stresses:**
  - Results from differences within the microstructure of a material.
  - Can change sign and/or magnitude over distances comparable to grain size of the material under analysis
  - *They are of two types, namely* **Type II or III**

Type II and III Residual Stresses

- **Type II (σ^{II}) :**
  - Vary on the scale on an individual grain
  - May be expected to exist in single phase materials because of anisotropy in the behavior of each grain
  - May also develop in multi-phase materials as a result of the different properties of the different phases
- **Type III (σ^{III})**
  - Exists within a grain, essentially as a result of the presence of dislocations and other crystalline defects

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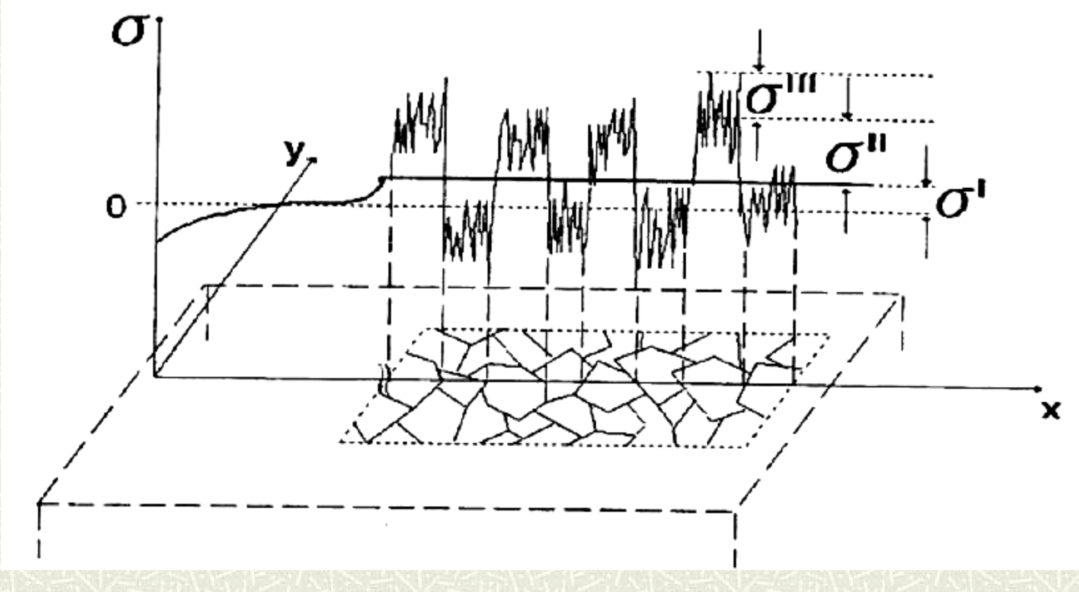
Stress definition according to length



Origin of Residual Stress

- ✓ Mechanical
- ✓ Thermal
- ✓ Chemical

Mechanically Generated

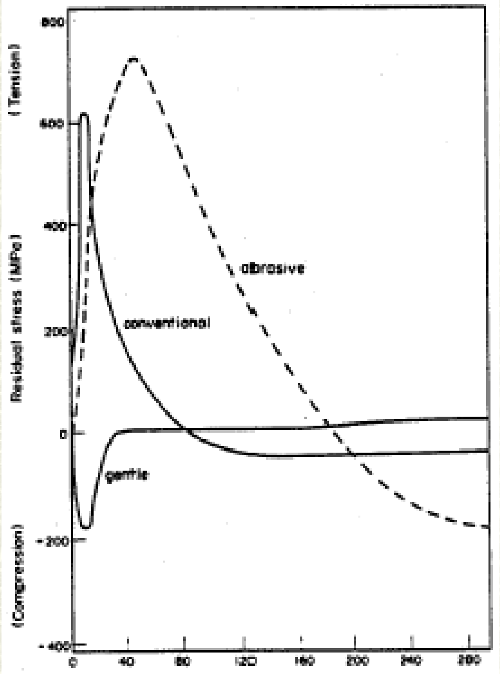
- Occurs due to manufacturing process that produce non-uniform plastic deformation
- May develop naturally during processing or treatment or may be introduced deliberately to develop a particular stress profile in a component
- Operations that produce “**undesirable**” **surface tensile stresses or residual stress gradients** are rod or wire drawing, welding, machining (turning, milling) and grinding
- Compressive residual stresses can be introduced by shot peening, autofrettage of pressure vessels, toughening of glass or cold expansion of holes

Compressive Vs Tensile

- In any free standing body stress equilibrium is maintained; *presence of a tensile residual stress in the component will be balanced by a compressive stress elsewhere.*
- **Tensile residual** stress in the surface of component are generally **undesirable** since they contribute to *fatigue failure, quench cracking and stress-corrosion cracking*
- **Compressive residual stresses** in the surface of component **increase** both the *fatigue strength and resistance to stress-corrosion cracking, and increase the bending strength of brittle ceramics and glass*

✓ ***In general, residual stresses are beneficial when they operate in the plane of applied load and are opposite in sense***

Mechanically Generated Stress



While, the methods of conventional and highly abrasive grinding produced ***undesirable*** *tensile stresses near the surface, gentle grinding produced* ***desirable*** *compressive stresses*

Ref:

E.B. Evans, “Residual Stress in Processing” Encyclopedia of Materials Science and Engineering Vol. 6, 4183-88 (1986)

Thermally Generated

- **Macroscopically:** Occurs as a result of non-uniform heating or cooling operations
- **Microscopic level:** Can also develop in a material during manufacture and processing as a consequence of Coefficient of thermal expansion mismatch between different phases or constituents.

Chemically Generated

- Develops due to volume changes associated with chemical reactions, precipitation or phase transformations
- Chemical surface treatments and coatings can lead to the generation of substantial residual stress gradients in the surface layers of components.
- For example, nitriding produces compressive stress in the diffusion region due to expansion of the lattice and precipitation of nitrides.

Common Methods of Residual Stress Determination

- Hole Drilling
- Curvature Method
- Diffraction
    - **X-ray Diffraction**
    - Neutron Diffraction
    - Electron Diffraction
- Other Methods
- Method Selection Guide

Hole Drilling

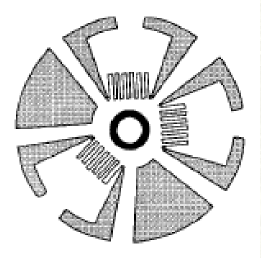
- *Principle*: The undisturbed regions of a sample containing residual stresses will relax into different shape when the locality is machined, thereby providing data for back calculation of residual stress.
- The process involves drilling a hole into a residually stressed body with a depth which is about equal to its diameter and small compared to the thickness of the test object.
- Strain is measured using either a rosette of strain gauges; moire interferometry, laser interferometry based on a rosette of indentations or holography

Hole Drilling: Equation

$$\sigma = (\sigma_{\max} + \sigma_{\min})\bar{A} + (\sigma_{\max} - \sigma_{\min})\bar{B}\cos 2\beta$$

Where $\bar{A}$ and $\bar{B}$ are hole drilling constants, and β is the angle from the axis to the direction of maximum principal stress $\sigma_{\max}$.

For the general case of a hole drilled in an infinite plate $\bar{A}$ and $\bar{B}$ must be calculated numerically



An arrangement of strain gauges

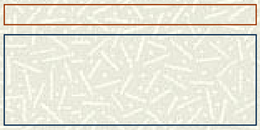
Curvature Method

- Frequently used to determine the stresses within coatings and layers.
- *Deposition of a layer can induce stresses which cause substrate to curve*
- Curvatures can be measured using **contact methods** (profilometry, strain gauges) or **without direct contact** methods (video, **laser scanning**, grids, double crystal diffraction topology)
- Curvature is related to the **residual stress** using **Stoney's equation**

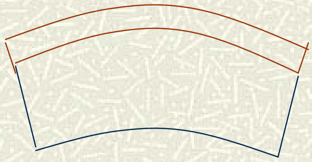
Curvature Method: Stoney's Equation

$$\sigma = \frac{E_s}{(1-\nu_s)}\frac{t_s^2}{t_l}\left[\frac{1}{R_2}-\frac{1}{R_1}\right]$$

Where, $E_s/(1 - \nu_s)$ is the substrate biaxial modulus, t_s and t_l are substrate and film thickness, R_1 and R_2 are radii of curvature of substrate before and after deposition respectively. It is assumed that $t_l \ll t_s$



Free and unstressed film and substrate



Stressed structure

X-ray Diffraction Method

- One of the most widely used non-destructive techniques for residual stress measurement
- Residual stress in the material causes the interplanar spacing of the material to change
- Changes in the interplanar spacing "d" can be used with the Bragg's equation to detect elastic strain "ε" through a change in the Bragg scattering angle Δθ

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

giving

$$\varepsilon = \frac{\Delta d}{d} = -\cot\theta\,\Delta\theta$$

"Accurate determination of stress free spacing "d_0" is required"

X-ray Diffraction Method, Contd.

- Stress is evaluated from strain values using Young’s modulus, Poisson ratio and taking into consideration Elastic Anisotropy of the material
- Typically single peak, available at highest value of 2θ, is used for analysis.
- Diffraction is selective and hence biased towards a particular sets of grains
- The peak shift sample both Type I and average Type II stresses, while Type III stresses give peak broadening.

Neutron Diffraction

- Measurements are carried out in much the same way as with X-Ray diffraction.
- Records the changes in the spacing of the lattice planes for a polycrystalline material from their stress free value.
- Neutrons have very **large penetration depths** as compared to X-ray, which makes them capable of measuring **at near surface depths** of around 0.2 mm **down to bulk measurements** of up to 250 mm in aluminum or 37mm in steel.

Neutron Diffraction, Contd.

- With **high spatial resolution**, neutron diffraction can provide complete three-dimensional strain maps of engineered components.
- However, compared to other diffraction techniques such as X-ray diffraction the relative **cost is much higher** and the **general availability** of facilities very much lower.

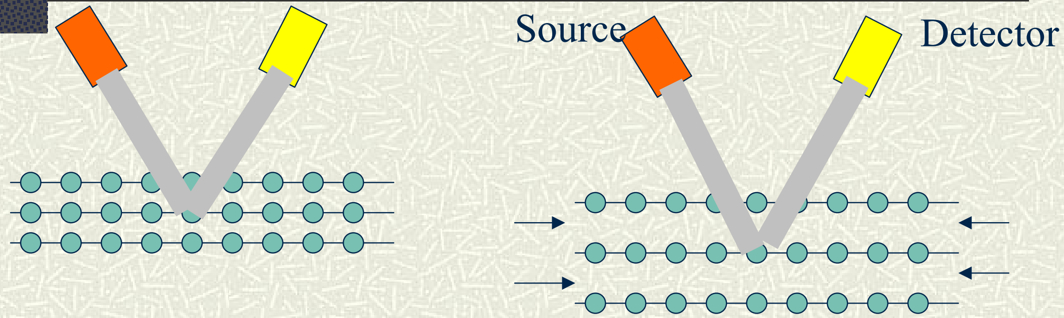
Electron Diffraction Techniques

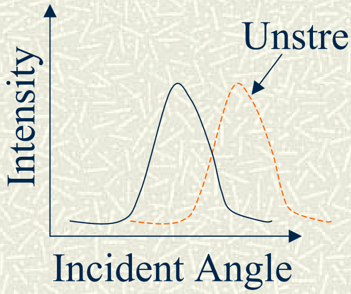
- An extremely powerful technique for very high lateral spatial resolution
- The convergent beam **electron diffraction is used for highest resolution**
- **Type II and Type III** stresses can be measured or macro stresses in very small electronic devices
- Only very thin films can be used; hence results are **vulnerable** to surface relaxation effects and strain values represent integral through thickness.

Other Techniques

- ✓ Compliance Methods
- ✓ Magnetic and Electrical Techniques
- ✓ Ultrasonic Method
- ✓ Piezospectroscopic Techniques *(Raman or Fluorescence luminescence)*
- ✓ Thermoelastic Methods
- ✓ Photoelastic Methods

Residual Stress by X-ray Diffraction Techniques



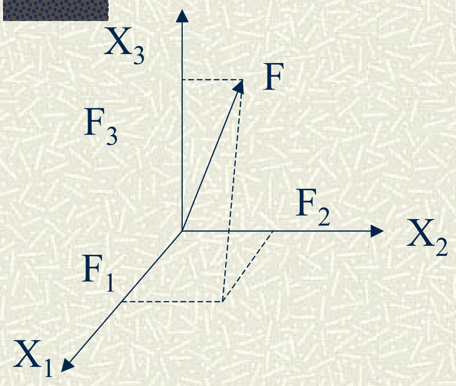


The compressive stress spreads apart the planes of atoms parallel to the surface (and perpendicular to the stress), which results in diffraction peaks at lower angles of incidence

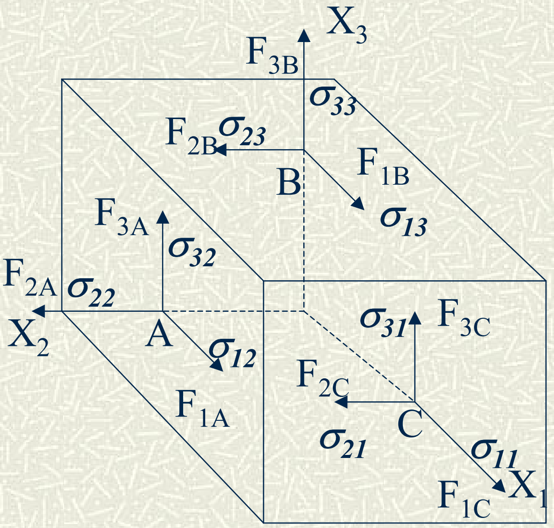
Tensors: Force and Stress

- **Tensors:** Physical quantities that are completely defined with the specifications of 3^n components. “n” denotes the rank of tensor
- **Forces** $\vec{F}$ are the vector quantities that are specified by magnitude and direction in an orthogonal coordinate system. Force component along an axis requires only one subscript to be defined. Hence force is a tensor of 1st rank.
- **Stresses** (σ) caused by application of forces require 9 components to be defined, hence are 2nd ranked tensors. Stresses are denoted by σ_{ij} where the 1st subscript denotes the direction of stress and 2nd denotes the normal of the plane in which it acts.

Forces and Stresses in an Orthogonal Coordinate System



Force F and its resolved components



Resolved forces and stresses caused on the faces of a homogenously loaded unit cube in static equilibrium

Forces and Stresses in an Orthogonal Coordinate System…

- Consider a unit cube with in a homogenously stressed body at static equilibrium. No body force (or torque) distribution exists within the body
- All the faces of the cube experience the force. However, since the body is at static equilibrium, parallel faces will experience the forces equal in magnitude but opposite in direction. Hence, only forces acting on three non-parallel faces of cube need to be considered.
- Force component acting on the face can be resolved into its normal (acting in normal direction) and shear component (in plane).
- Similar arguments are applicable for stress components.

Stress: Symmetric Tensor

- Stress is a symmetric 2nd rank sensor ($\sigma_{ij} = \sigma_{ji}$)

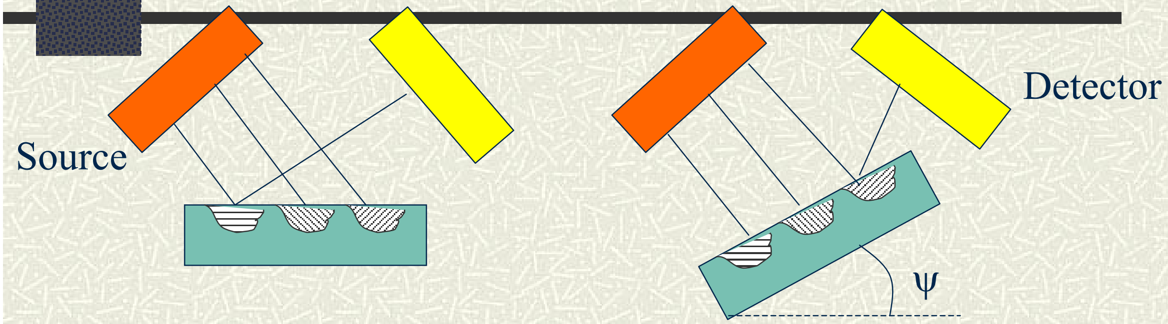
$$\sigma_{ij} = \begin{pmatrix} \sigma_{11} & \sigma_{12} & \sigma_{13} \\ \sigma_{21} & \sigma_{22} & \sigma_{23} \\ \sigma_{31} & \sigma_{32} & \sigma_{33} \end{pmatrix} = \begin{pmatrix} \sigma_{11} & \sigma_{12} & \sigma_{13} \\ \sigma_{12} & \sigma_{22} & \sigma_{23} \\ \sigma_{13} & \sigma_{23} & \sigma_{33} \end{pmatrix}$$

where the diagonal components (σ_{ii}) are called normal stresses, while off diagonal components (σ_{ij} $i \neq j$) are shear stresses)

Measuring Complete Stress tensor

- Strain values are recorded for different sample tilt angle (ψ) at constant azimuth angle φ. Strain Vs $\sin^2\psi$ is plotted to estimate the stress values.
- In order to completely evaluate at least 6 measurements are needed on Strain- $\sin^2\psi$ plot for three different values of φ. Typically 6 measurements are recorded for better statistics
- Three equidistant values of φ are chosen
- Negative and positive values of tilt are preferred (?)

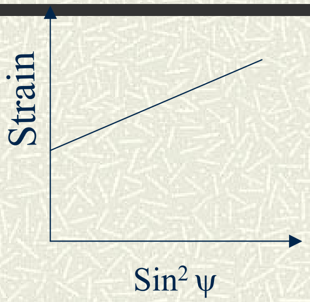
Resolving Multi-axial Stresses

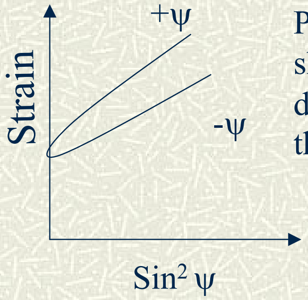


When the *sample is level, only one set of planes diffract*. But when the *sample is tilted, a different sets of crystallites diffract*. Thus each set of crystallites resolve a different directional component of the stress to which the sample is subjected. If the sample is compressed from the sides, the lattice spacing of crystallites oriented parallel to the surface will increase more than those oriented at an angle to the surface. *The peak position will shift during tilting*, since crystallites are subjected to different magnitude of stress.

Strain-Sin^2 ψ Curves

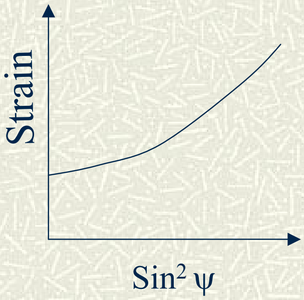
Normal stress only in the plane of surface

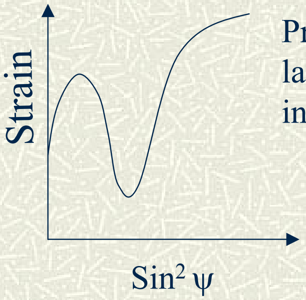




Presence of shear stress directed ⊥ to the surface

Stress gradient ⊥ to the surface





Presence of large variations in stress

Equation of Ellipse

- Strain-sin^2 ψ curves are fitted using the following equation of ellipse and complete stress tensor is evaluated

$$\varepsilon_{\varphi\psi} = \frac{1}{2}s_2(\sigma_{11}\cos^2\varphi + \sigma_{12}\sin 2\varphi + \sigma_{22}\sin^2\varphi)\sin^2\psi$$
$$+\frac{1}{2}s_2\sigma_{33}\cos^2\psi + s_1(\sigma_{11}+\sigma_{22}+\sigma_{33})$$
$$+\frac{1}{2}s_2(\sigma_{13}\cos\varphi + \sigma_{23}\sin\varphi)\sin 2\psi$$

X-ray elastic constants s_1 and ½ s_2 in above are defined as

$$s_1 = -\frac{\nu}{E} \qquad and \qquad \frac{1}{2}s_2 = \frac{\nu+1}{E}$$

where ν = Poisson's ratio and E= Young's modulus

Instrumentation

- Parallel X-ray Beam
- Goniometer capable of rotation and tilt
- ψ- tilting is performed either in **iso-inclination mode** or **side-inclination** mode
- **Side-inclination is preferred** because
  - The effect of misalignments of the sample height on the stress result is less pronounced
  - The range of tilt angles is not restricted by small Bragg Angles
  - The measurements on samples with concave surface are less hindered by shading effects
- Stress free sample of the material under investigation is needed

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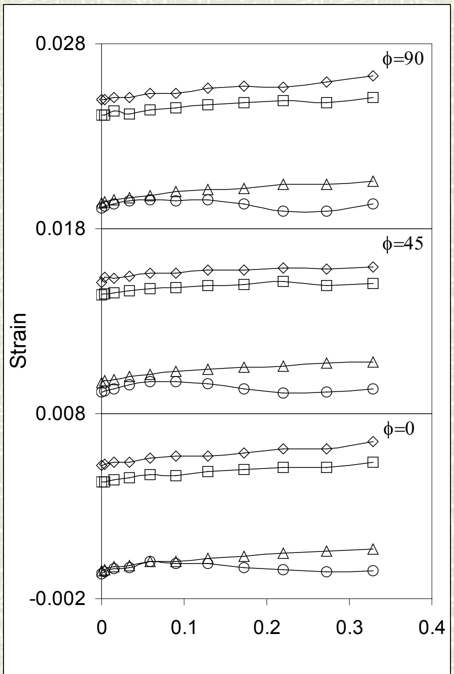
Example: Strain-Sin^2 ψ Curves



Strain–sin^2 ψ curves for 20 nm Ir films annealed in air for 1 h at (□) 673 K, (Δ) 873 K, (O) 1073 K, and (◊) as-deposited. Strain values for f=45° have an offset of 0.01 while f=90° have an offset of 0.02 strain units for the sake of presentation only.

Example: Stress Tensor

$$\sigma_{ij}(as-deposited) = \begin{pmatrix} 1300 & -1300 & 300 \\ -1300 & 1300 & 200 \\ 300 & 200 & 0 \end{pmatrix} \pm \begin{pmatrix} 300 & 300 & 100 \\ 300 & 300 & 100 \\ 100 & 100 & 0 \end{pmatrix}$$

$$\sigma_{ij}(673K) = \begin{pmatrix} 800 & -800 & 300 \\ -800 & 600 & 300 \\ 300 & 300 & 0 \end{pmatrix} \pm \begin{pmatrix} 200 & 200 & 100 \\ 200 & 200 & 100 \\ 100 & 100 & 0 \end{pmatrix}$$

$$\sigma_{ij}(873K) = \begin{pmatrix} 700 & -200 & 300 \\ -200 & 500 & 400 \\ 300 & 400 & 0 \end{pmatrix} \pm \begin{pmatrix} 200 & 100 & 100 \\ 100 & 200 & 100 \\ 100 & 100 & 0 \end{pmatrix}$$

Due to small penetration depth of X-rays, a plane-stress state in material can be assumed $\sigma_{33} \approx 0$

References

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