بسم الله الرحمن الرحیم

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K. N. Toosi University of Technology
Faculty of Materials Science and Engineering

Materials Characterization Methods

Fifth Session
(Application of X-ray in Materials Analysis)

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Quantitative Analyses of Materials Using X-Ray Diffraction

- Using the diagram of radiation intensity in terms of 2θ from the diffractometry method or the Debby Scherrer method, the percentage of different materials in the study composition can be calculated with XRD.
- For each test sample, it is necessary to prepare standard samples whose composition is the same as the composition of the test sample but with different percentages.

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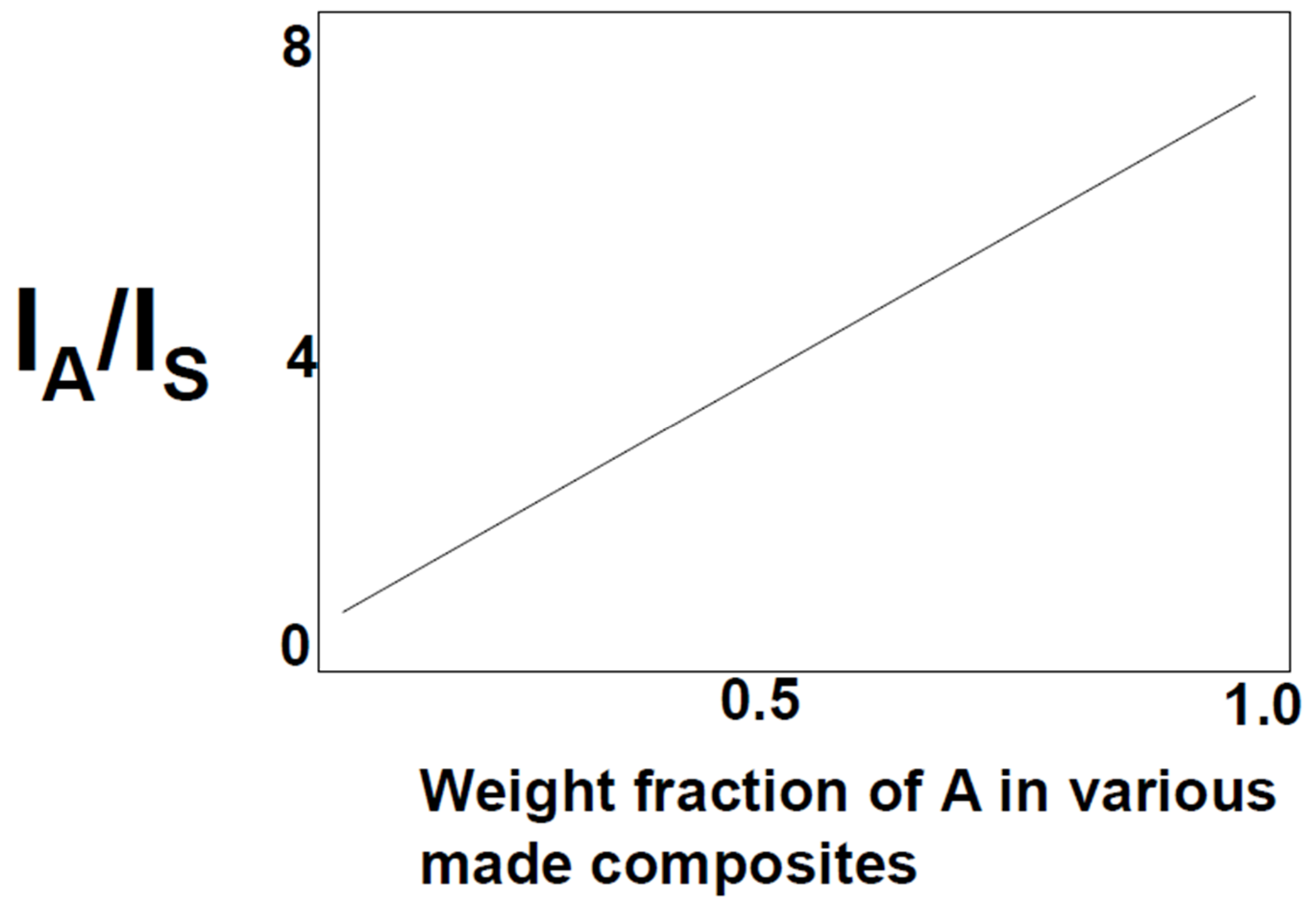
Quantitative Analyses of Materials Using X-Ray Diffraction

- For a combination of A, B and C, if we want to find the percentages of components, we need to prepare at least 4 or 5 combinations with specific percentages of A, B and C as standard compounds. Then the standard samples are irradiated with X-rays and the curves are compared with the curves of the original sample and the percentage of compounds in the sample is determined by means of related calculations.

Quantitative Analyses of Materials Using X-Ray Diffraction



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Peak Broadening in XRD

- Bragg's law assumes that the conditions of constructive interference are met, namely: The crystal is perfect and there are no defects in it. The beam is monochromatic and strikes the sample in parallel.

- **In practice, these conditions never exist completely.**

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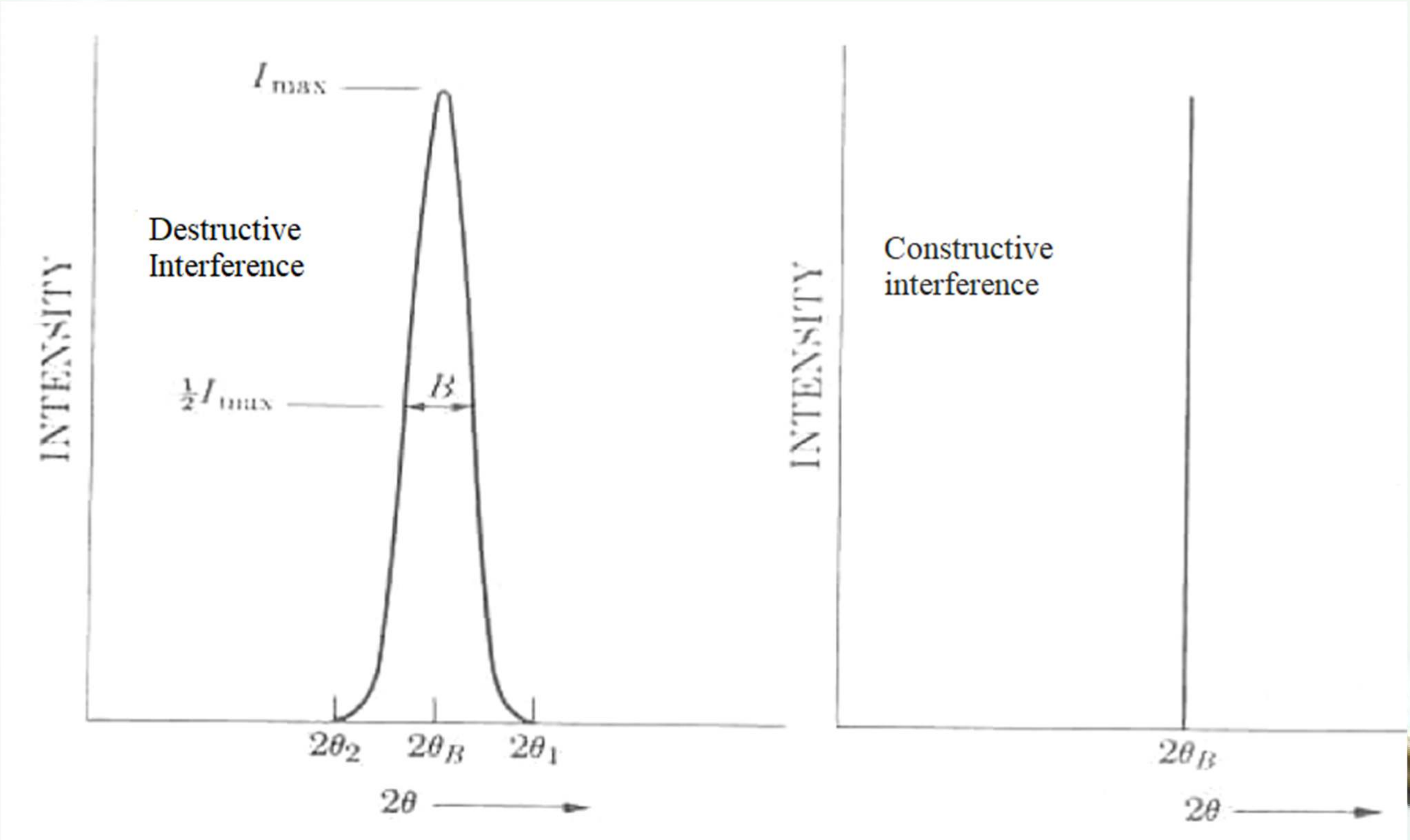
Peak Broadening in XRD

Destructive Interference

- X-ray diffraction under non-ideal conditions (angles close to Bragg's angle). The input rays are not parallel. If the phase difference between the two waves is large, then we will not have diffraction.

- In the case of destructive interference, if the phase difference is very small, we will have diffraction, but the peak will wide. This means that fewer waves are eliminated and more angles (close to 2θ) are diffracted with less intensity (due to non-compliance with Bragg's law).

Peak Broadening in XRD



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Peak Broadening in XRD

- The peak at certain angles represents the crystalline structure.
- Amorphous and liquids materials both have structures characterized by a lack of almost complete periodic order.
- There is no periodic structure in monoatomic gases. In such gases, the atoms are randomly placed together. Their scattering curve not only does not show a pick, but also shows a regular decrease in intensity with increasing scattering angle.

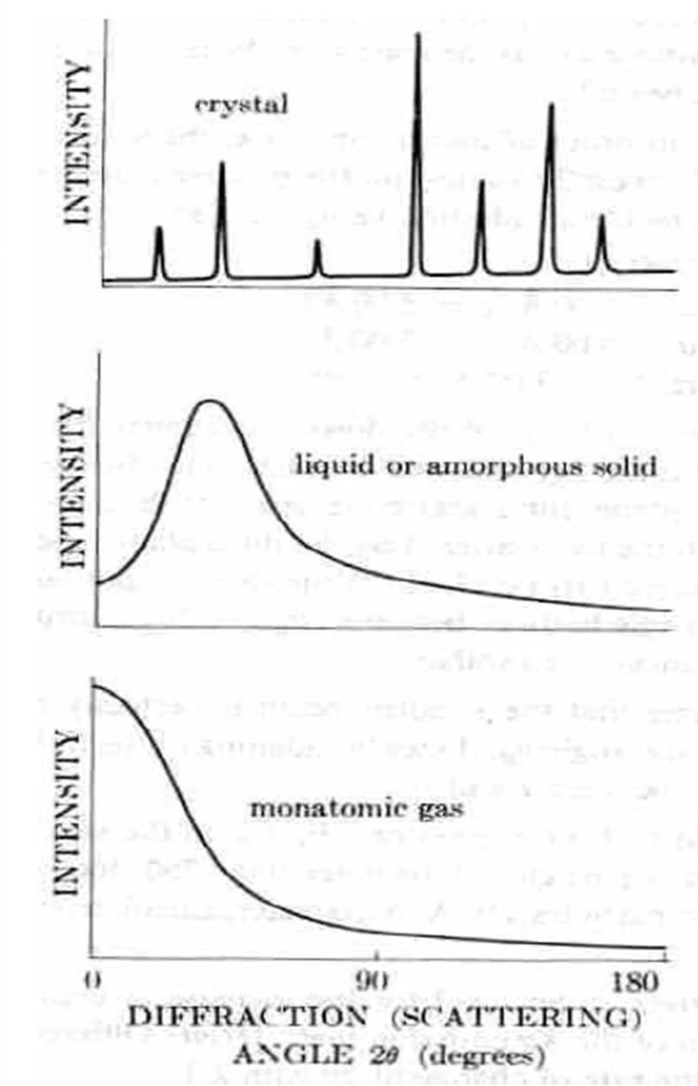
Peak Broadening in XRD

X-Ray Diffraction Curve

Crystal

Amorphous Solid or Liquid

Monoatomic Gas



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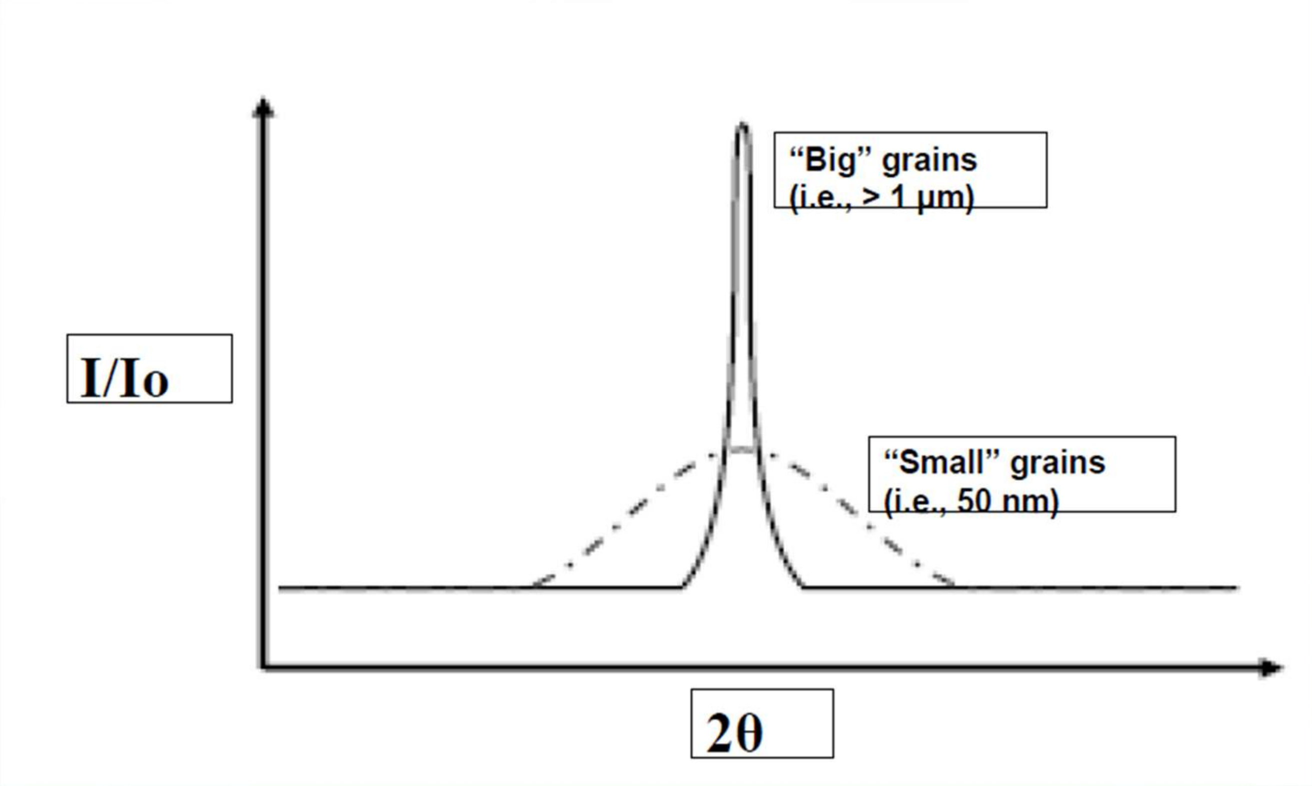
Peak Broadening in XRD

Factors Involved in the Peak Broadening:

- ✓ **Decrease in Crystallite Size**
- ✓ **Lattice Strain**



Peak Broadening in XRD

Peak Broadening =

Used Equipment + Decrease in Crystallite Size + Lattice Strain

$$\beta_{observed} = \beta_{instrumental} + \beta_{strain\ and\ particle\ size}$$

$$\beta_r = \beta_{crys.\ size} + \beta_{strain}$$

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Peak Broadening in XRD

Other Relationships:

$$\beta_r^2 = \beta_o^2 - \beta_i^2$$

$$\beta_r = \sqrt{(\beta_\circ - \beta_i\sqrt{(\beta_\circ^{\,2} - \beta_i^{\,2})}}$$

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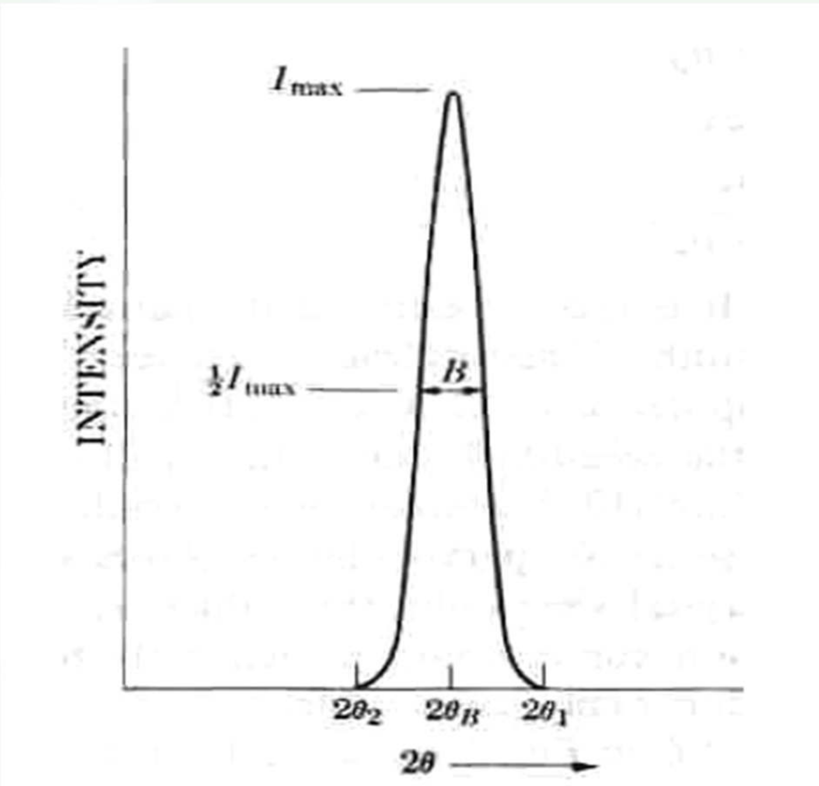
Peak Broadening in XRD

Scherrer Formula: **Peak broadening equation only due to crystallite size.**



$$D = \frac{k\lambda}{\beta_r \cos\theta}$$

D = grain diameter
β_r = **peak width in radians**

Based on grain shape: K can be 0.9 to 1

Best Case: Grain size range between 2 to 300 nm

Peak Broadening in XRD

If peak broadening is due to the strain:

$$\beta_{strain} = \eta \tan\theta$$

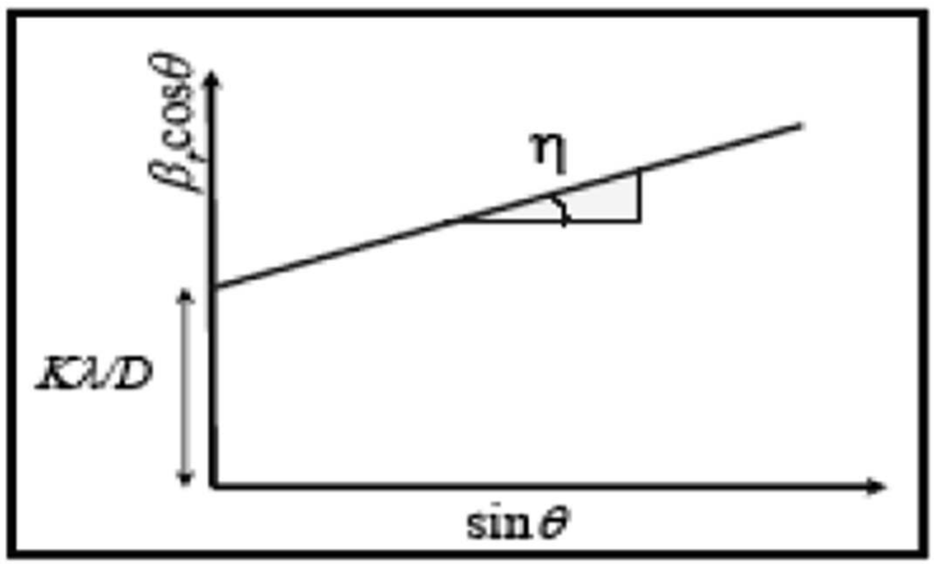
η= Strain in material

Deformation causes a local change in atomic spaces, and these lattice strains cause the peak broadening.

Peak Broadening in XRD

$$\beta_r = \beta_{\text{crys. size}} + \beta_{\text{strain}}$$

$$\beta_r = \frac{k\lambda}{D\cos\theta} + \eta\tan\theta \;\Rightarrow\; \beta_r\cos\theta = \frac{k\lambda}{D} + \eta\sin\theta$$

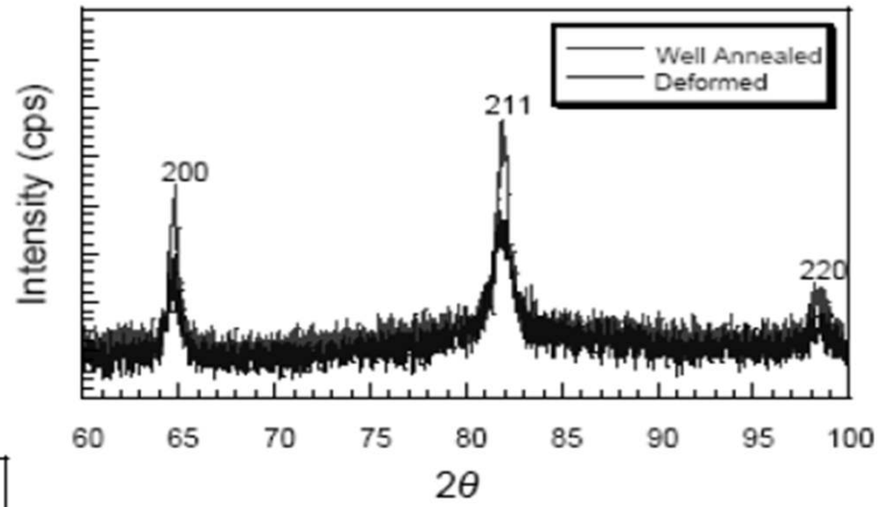


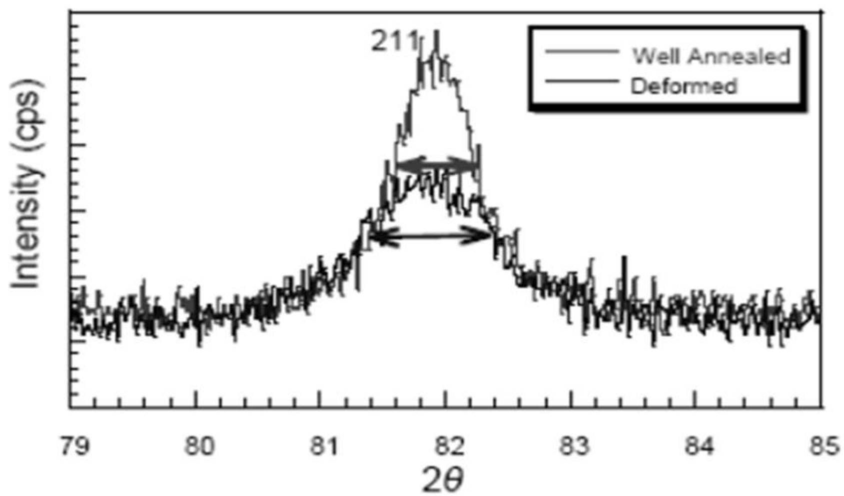
Peak Broadening in XRD

Example:

Annealing mode: determine the broadening caused by the equipment

Deformation mode: Observed broadening





Peak Broadening in XRD

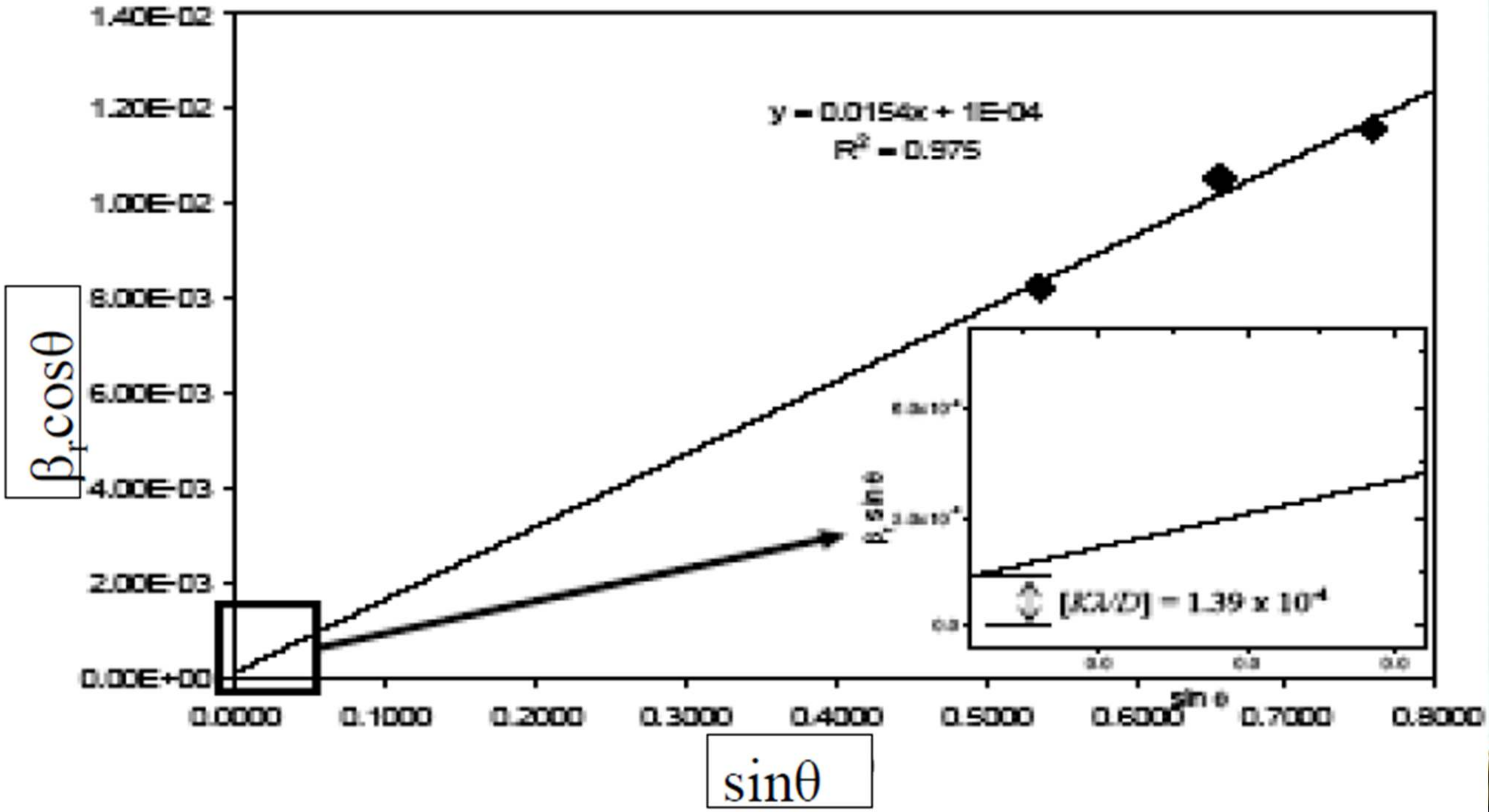
Annealed particles | CuKa radiation: 1.54056 Å

Peak#	2θ (°)	*hkl*	FWHM (°)	FWHM (radians) = β_i
1	64.9	200	0.5	8.73E-03
2	82.0	211	0.6	1.05E-02
3	98.5	220	1.1	1.92E-02

Deformed particles | CuKα 1.54056 Å

Peak #	2θ (°)	sin θ	*hkl*	FWHM (°) = β_o	FWHM (radians) = β_o	$\beta_r = \sqrt{\beta_o^2 - \beta_i^2}$	$\beta_r \cos\theta$
1	64.9	0.5366	200	0.75	1.31E-02	9.76E-03	8.23E-03
2	82.0	0.6561	211	1.0	1.75E-02	1.40E-02	1.05E-02
3	98.5	0.7576	220	1.5	2.62E-02	1.78E-02	1.16E-02

Peak Broadening in XRD



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Peak Broadening in XRD

Particle Size

$$\frac{k\lambda}{D} = 1.39\times10^{-4} = \frac{(\approx 1)(1.54056)}{D}$$

$$D = 11{,}083\,\mathring{A} \ \text{or}\ 1.1\,\mu m$$

Strain

Slope of line = 0.0154 or 1.5%

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