Orientation and quality of polycrystalline aggregates

Structure of the polycrystalline

- metals and alloys are produced and used is in the form of polycrystalline aggregates

→ composed of a great many individual crystals usually of microscopic size.

- The properties of the material depend on the weather these grains are large or small, strained or unstrained, oriented at random or in some particular way.

Grain size:

- X-ray diffraction photograph → semiquantitative information about grain size and craystal quality and orientation (see Fig. 1)
- The number of grains which take part in diffraction is governing effect

→**Fig. 1(a):** grain size is quite coarse

→**Fig. 1(b):** Finer grain size

→**Fig. 1(c):** The grain size further reduced→ the Laue spots merge into a general background and only Debye rings are visible(these rings are spotty since not enough crystal are present in the irradiated volume of the specimen to refelct all parts of ring).

→**Fig.1(d):** a finer grain size→ smooth and continuse Debye rings

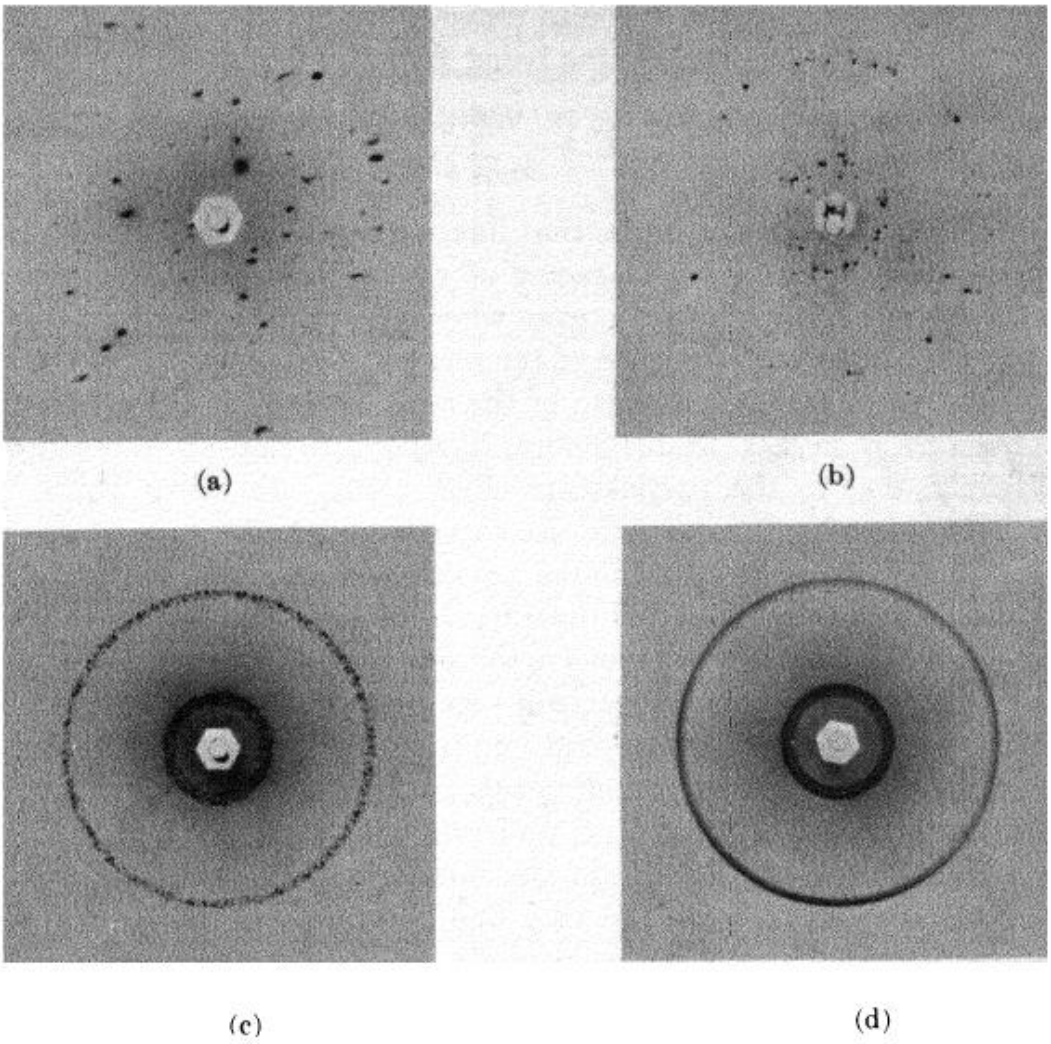

Fig. 1. Back-reflection pattern of recrystallized aluminum specimens, see text. (B. D. Cullity (1977))

Particle size

- When the size of the individual crystal is less than about 0.1 μm (1000 Å) the term **"** particle size" is used
- Crystals in this range → broadening of Debye rings

$$\beta = 0.9\lambda / t \cos\theta$$

(*t* = diameter of crystal particle, β = *FWHM*)

Crystal quality

- So characteristic imperfection of the *cold-worked* of metals and alloys is nonuniform strain

- **Schematic of tensile and compressive strain**

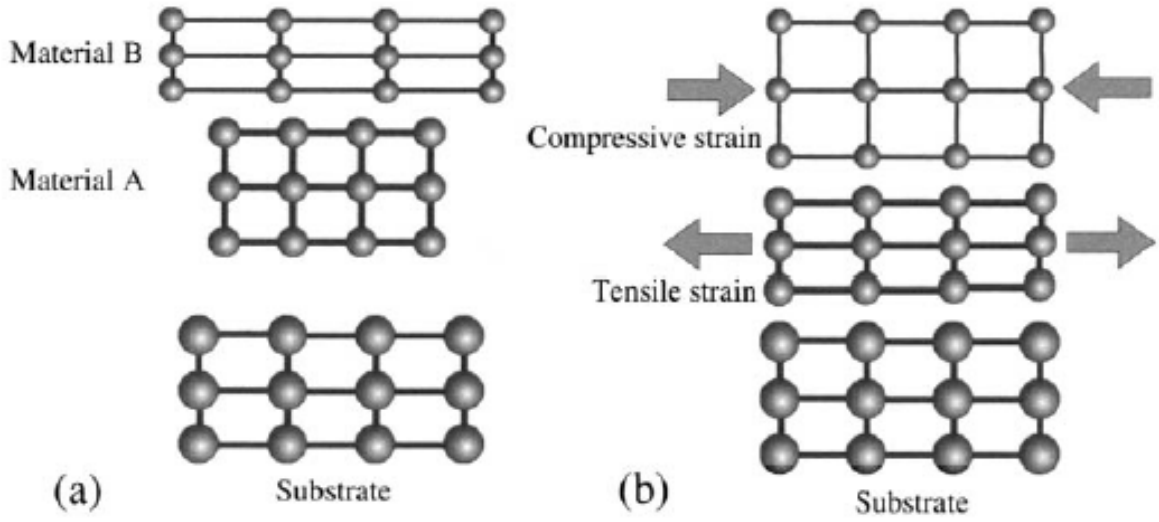

Fig. 2. Schematic illustration of a substrate with two mismatched layers. (a): free standing unstrained layers (b): the materials with different lattice constant are grown on the substrate (Paul Harrison, 2009)

The effect of strain on the direction of x-ray reflection:

- See Fig. 3:

→**Fig. 3(a):** unstrained grain→ equilibrium spacing d_0

→ **Fig. 3(b)**: if the grain is given a uniform tensile strain their spacing become larger than d_0 corresponding diffraction line shifts to lower angle.

→**Fig. 3(c)**: the grain is bent and the strain is nonuniform: on the top (tensile) side the plane spacing exceeds d_0, on the bottom (compression) side it is less than d_0 and somewhere in between is equal d_0.

- The relation between the broadening produced and the non-uniformity of the strain:

$$\Delta 2\theta = -2\frac{\Delta d}{d}\, tan\theta$$

→ $\frac{\Delta d}{d}$ includes both tensile and compressive strain and must be divided by two to obtain the maximum tensile strain alone, or maximum compressive strain alone, if these two are assumed equal.

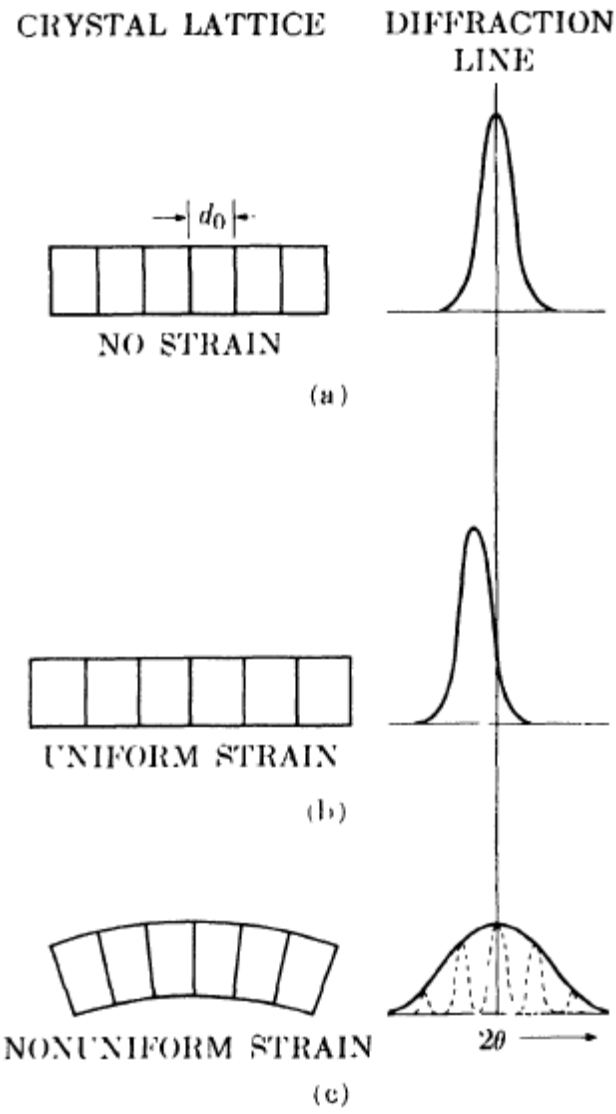

Fig. 3 Effect of lattice strain on Debye-line width and position (B. D. Cullity (1977))

Determination of Size and Strain

- How the mean size and strain within a powder can be calculated from the diffraction pattern when both are present simultaneously.

Williamson-Hall Plot

- It relies on the principle that the **approximate formulae** for size broadening, β_L, and strain broadening, β_e, vary quite differently with respect to Bragg angle, θ:

→ The simplification of Williamson and Hall is to assume the convolution is a simple sum

$$\beta_{tot} = \beta_e + \beta_L = C_\epsilon \, tan\theta + \mathrm{k}\lambda/\mathrm{t}\cos\theta$$

If we multiply this equation by cosθ we get:

$$\beta_{tot} cos\theta = C_\epsilon \, sin\theta + \mathrm{k}\lambda/\mathrm{t}$$

by plotting $\beta_{tot}\cos\theta$ versus $\sin\theta$ we obtain the strain component from the slope C_ϵ and the size component from the intercept $\mathrm{k}\lambda/\mathrm{t}$. Such a plot is known as a **Williamson-Hall plot**.

Warren-Averbach method

- If the observed line profile (corrected for instrumental broadening) are expressed as Fourier series, then an analysis of the Fourier coefficients disclose both particle size and strain.

Depth of x-ray penetration

- **What is the effective depth of x-ray penetration?**
- No precise answer because the intensity of the incident beam does not suddenly become zero at any one depth but rather decreases exponentially with distance below the surface
- The integrated intensity diffracted by an infinitesimally thin layer located at a depth *x* below the surface :

$$dI_D = \frac{I_0 ab}{sin\ \alpha} e^{-\mu x(\frac{1}{sin\alpha}+\frac{1}{sin\ \beta})} dx$$

- Where the incident beam has intensity I_0 is 1 cm square in cross section, and is incident on the powder plate at angle α and β is exit angle. *a* is volume fraction of the specimen containing particles having the correct orientation for reflection of the incident beam, and *b* the fraction of the incident energy which is diffracted by unit volume

- The total diffracted intensity is obtained by integrating over an infinity thick specimen:

$$I_D = \int_{x=0}^{x=\infty} dI_D = \frac{I_0 ab}{2\mu}$$

→ The unknown $I_0, a, and\ b$ constants will be cancel out if we express the intensity diffracted by the layer considered as a fraction of the total integrated intensity diffracted by a specimen of infinite thickness.

$$G_x = \frac{\int_{x=0}^{x=x} dI_D}{\int_{x=0}^{x=\infty} dI_D} = [1 - e^{-\mu x\left(\frac{1}{sin\alpha}+\frac{1}{sin\,\beta}\right)}]$$

→ The information recorded on the diffraction pattern (or, more precisely, 95 percent of the information) refers to the layer of depth x and not to the material below it.

→ In the case of diffractometer, $\alpha = \beta = \theta$ and Eq. reduces to

$$G_x = [1 - e^{\left(\frac{-2\mu x}{sin\theta}\right)}]$$

→ the more suitable for calculation for diffractometer

$$\frac{2\mu x}{sin\theta} = ln\left(\frac{1}{1 - G_x}\right) = K_x$$

$$x = \frac{K_x\, sin\theta}{2\mu}$$

Values of K_x corresponding to various assumed values of G_x are :

$\boldsymbol{G_x}$	0.50	0.75	0.90	0.95	0.99	0.999
$\boldsymbol{K_x}$	0.69	1.39	2.30	3.00	4.61	6.91