Particle Size and Strain Analysis by X-Ray Diffraction

Background

When crystallites are less than approximately 1,000 Å in size, appreciable broadening in the x-ray diffraction lines will occur. These regions may in fact correspond to the actual size of the particles. At other times, however, these regions form "domains" in the larger particle and may be a distinguishing and important feature. In either case, the observed line broadening can be used to estimate the average size. In the simplest case where the particles are stress-free, the size is estimated from a single diffraction peak. But in those cases where stress may be present, a more robust method involving several diffraction peaks is required.

Example 1

Figure 1 shows the X-ray diffraction pattern of nanocrystalline silicon which exhibits significant line broadening. Shown for comparison is the diffraction pattern from bulk Si with a particle size greater than 20 mm. Note that the $K\alpha_2$ line is well resolved in the bulk sample, but indistinguishable in the n-Si case. The extent of broadening is described by β, which is the full width at half maximum intensity of the peak.

Applications

Corrosion products	Amorphous/crystalline contents
Forensic analysis	Quality control
Intermetallics	Phase transformations
Contaminants	Catalysts
Pharmaceuticals	Fiber analysis

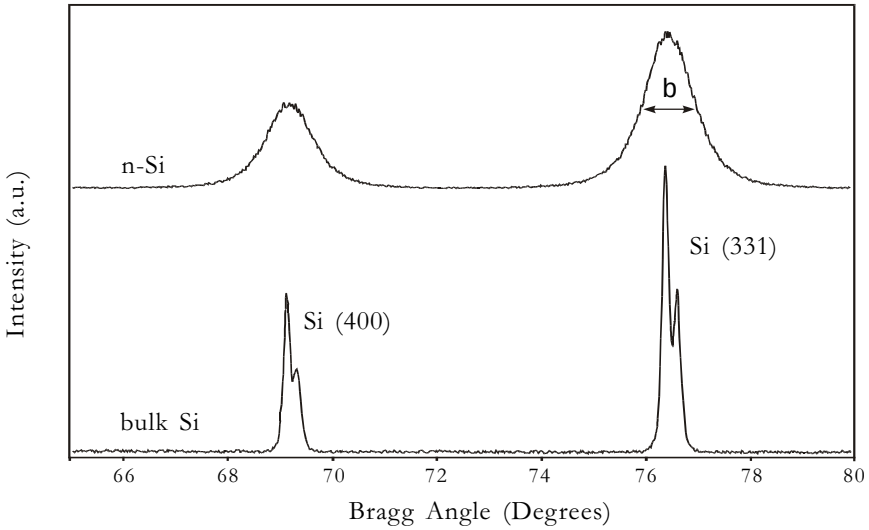

Figure 1 *Diffraction patterns of nanocrystalline silicon showing broadening because of particle size.*

After the value of β (in radians) is corrected for the instrumental contribution, it can be substituted into Scherrer's equation:

$$D \approx \frac{0.9\,\lambda}{\beta \cos\Theta}$$

where λ is the wavelength and Θ is the diffraction angle. For the diffraction pattern shown above, Θ = 38.226°, β = 0.0190 rad (after correction), and λ = 1.54178 Å, yielding a particle size D of 93Å, which is in close agreement with the value (90Å) observed by TEM.

Example 2

The simple method based upon Scherrer's formula described in Example 1 is only valid when the diffracting material is stress free. In those cases where both stress and particle size lead to broadening of the diffraction peaks, a more comprehensive method must be used to separate the contributions.

The most common method of strain/size analysis utilizes the fact that the broadening from the two different sources have different angular relationships. For instance, the size broadening as described earlier has a 1/cos(Q) relationship while the strain follows a tan(Q) function. Finally, the instrument also contributes to the broadening. Thus, the total broadening b_t can be described by

$$\beta_t^2 \approx \left\{\frac{0.9\,\lambda}{D\,cos\,\Theta}\right\}^2 + \left\{4\varepsilon\,tan\,\Theta\right\}^2 + \beta_0^2$$

where e is the strain and b_0 is the instrumental broadening. By a least squares method, the experimentally observed broadening of several peaks can be used to compute the average particle size D and the strain e simultaneously.

An example of this method is shown in Figure 2 for a nanoscale Al_2O_3/ZrO_2 composite. After partial sintering, the broadening of each diffraction peak was measured and plotted as a function of the Bragg angle. From this data, it was possible to determine the strain and particle size for *each* phase:

for Al_2O_3: D=415 Å
e=0.00083

for ZrO_2: D=327Å
e=0.00120

This method can also be used to study many other phenomena such as stacking fault density and non-uniform deformation.

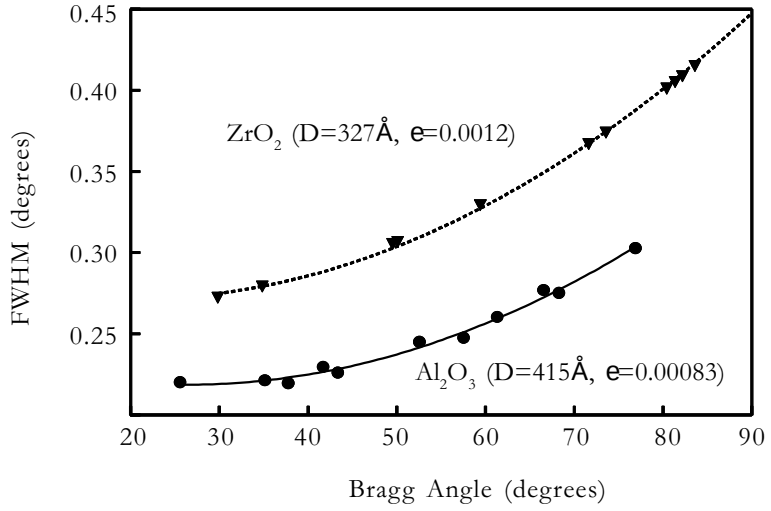

Figure 2 Plot of the peak widths (FWHM) for Al_2O_3 and ZrO_2 in a partially sintered composite at various diffraction angles. By use of a least squares fit, the particle size and strain can be computed.

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H & M Analytical Services, Inc.
35 Hutchinson Road
Allentown, NJ 08501-1415
Tel: (609) 758-5700
Fax: (609) 758-5708

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