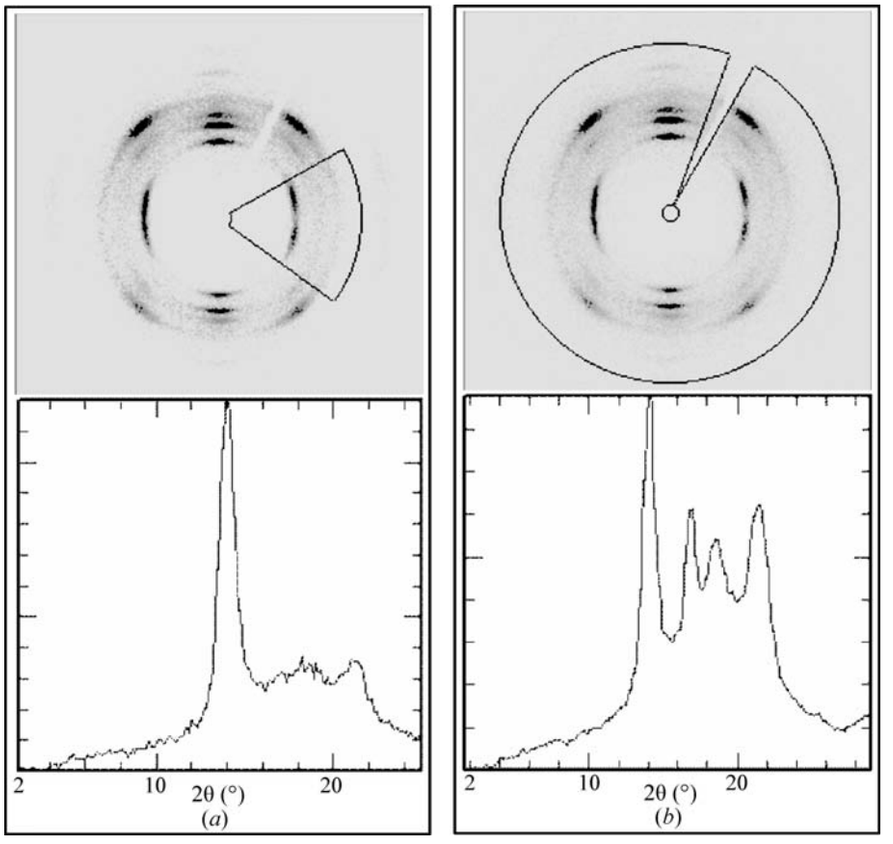

Figure 2.5.28
2D diffraction pattern from an oriented polycrystalline polymer sample. (*a*) Diffraction profile integrated from a horizontal region analogous to a profile collected with point detector. (*b*) Diffraction profile integrated from all parts of the 2D frame.

Fig. 2.5.29(*a*) shows a diffraction profile collected from gold nanoparticles and regular gold metal. The 2θ profile from the gold nanoparticles is significantly broader than the profile from regular gold metal. The crystallite size can be calculated by measuring the broadening and using the Scherrer equation:

$$B = \frac{C\lambda}{t\cos\theta}, \qquad (2.5.91)$$

where λ is the X-ray wavelength (in Å), B is the full width at half maximum (FWHM) of the peak (in radians) corrected for instrumental broadening and strain broadening, θ is the Bragg angle, C is a factor, typically from 0.9 to 1.0, depending on the crystallite shape (Klug & Alexander, 1974), and t is the crystallite size (also in Å). This equation shows an inverse relationship between crystallite size and peak-profile width. The wider the peak is, the smaller the crystallites. The 2θ diffraction profiles can be obtained either by using a conventional diffractometer with a point or line detector, or by γ integration from a diffraction pattern collected with 2D detector. When a 2D detector is used, a long sample-to-detector distance should be used to maximize the resolution. A small beam size and low convergence should also be used to reduce instrument broadening.

Fig. 2.5.29(*b*) shows a frame collected from an SRM660a (LaB_6) sample with a 2D-XRD system. The spotty diffraction rings are observed with average crystallite size of 3.5 μm. The number of spots in each diffraction ring is determined by the crystallite size and diffraction volume. Introducing a scaling factor covering all the numeric constants, the incident-beam divergence and the calibration factor for the instrument, we obtain an equation for the crystallite size as measured in reflection mode:

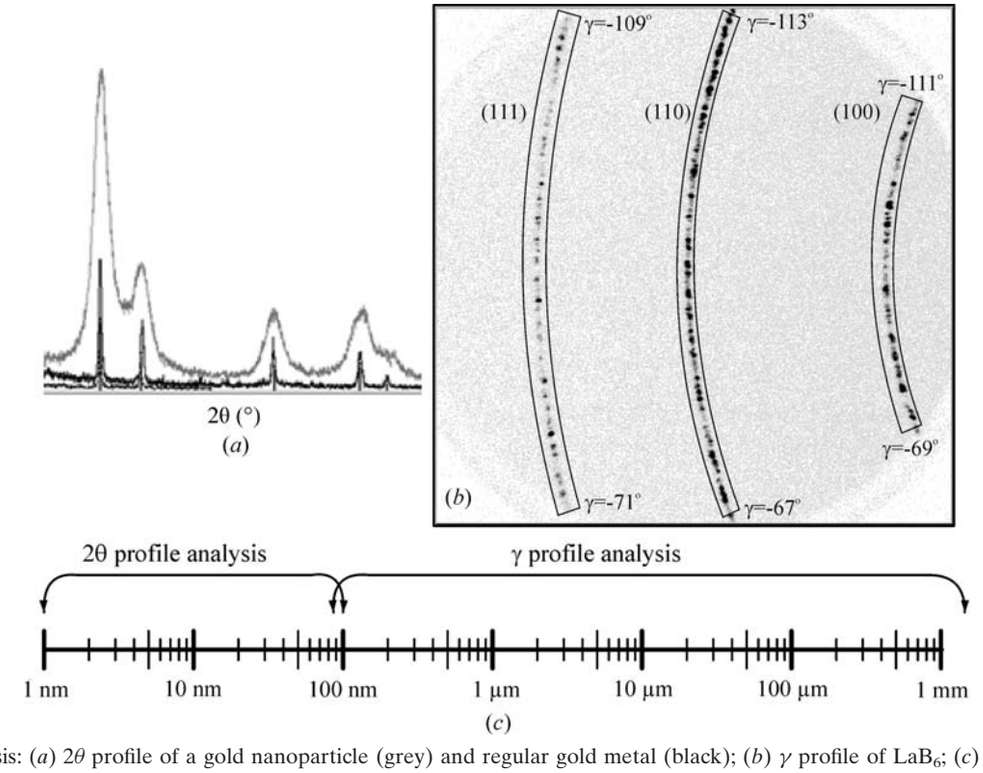

Figure 2.5.29
Crystallite-size analysis: (*a*) 2θ profile of a gold nanoparticle (grey) and regular gold metal (black); (*b*) γ profile of LaB_6; (*c*) measurement range.

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