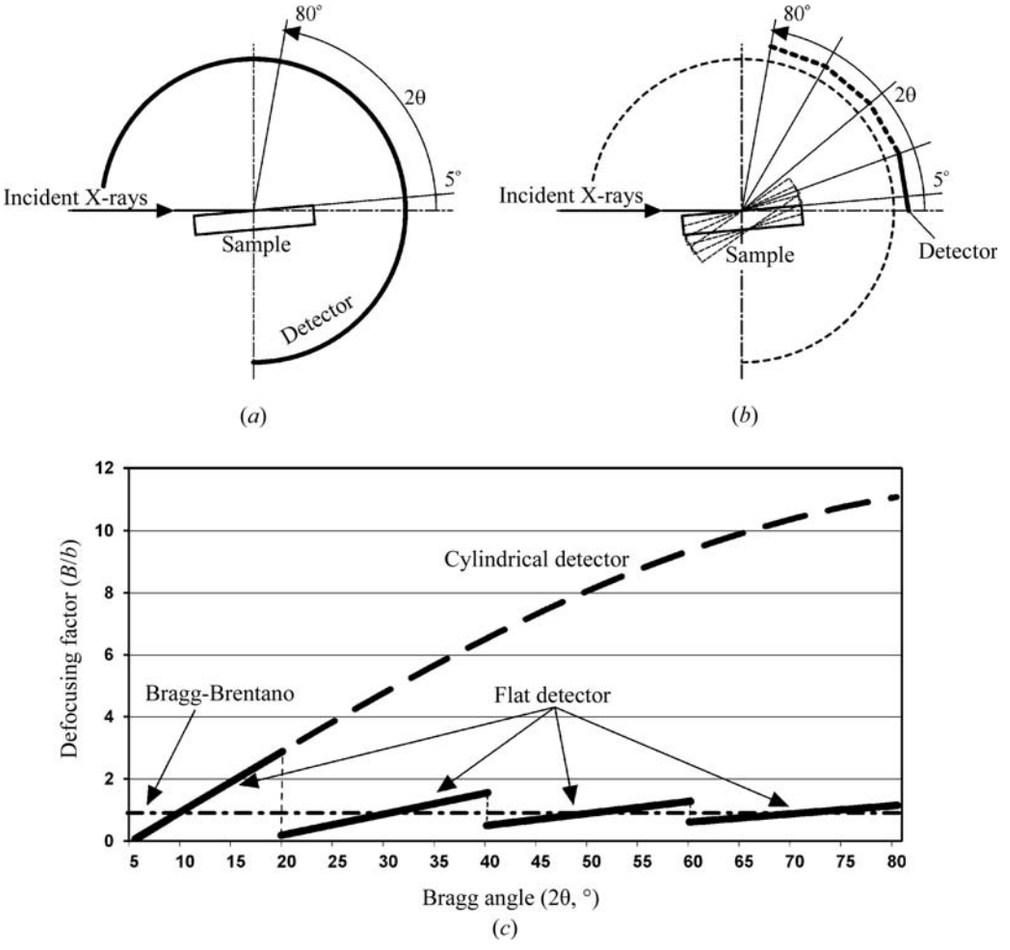

Figure 2.5.17
Defocusing effects: (*a*) cylindrical detector; (*b*) flat detector at various incident angles and detector swing angles; (*c*) comparison of defocusing factors.

statistics). Sampling statistics are determined by both the structure of the sample and the instrumentation. For a powder sample in which the crystallites are perfectly randomly oriented, the number of contributing crystallites for a diffraction peak can be given as

$$N_s = p_{hkl}\frac{Vf_i}{v_i}\frac{\Omega}{4\pi}, \tag{2.5.47}$$

where p_{hkl} is the multiplicity of the diffracting planes, V is the effective sampling volume, f_i is the volume fraction of the measuring crystallites (f_i = 1 for single-phase materials), v_i is the volume of individual crystallites and Ω is the angular window of the instrument (given as a solid angle). The multiplicity term, p_{hkl}, effectively increases the number of crystallites contributing to the integrated intensity from a particular set of (hkl) planes. The volume of individual crystallites, v_i, is an average of various crystallite sizes. The combination of the effective sampling volume and the angular window makes up the instrumental window, which determines the total volume of polycrystalline material making a contribution to a Bragg reflection. For 2D-XRD, the instrumental window is not only determined by the incident beam size and divergence, but also by the detective area and the sample-to-detector distance (γ angular coverage).

In B-B geometry, the effective irradiated volume is a constant,

$$V_{\rm BB} = A_o A_{\rm BB} = A_o/2\mu, \tag{2.5.48}$$

where A_o is the cross-section area of the incident beam measured on the sample surface, $A_{\rm BB} = 1/(2\mu)$ is the transmission coefficient for B-B geometry, and μ is the linear absorption coefficient. For 2D-XRD, the effective volume is given as

$$V = A_o A = A_o T/2\mu, \tag{2.5.49}$$

where A is the transmission coefficient and T is the transmission coefficient with B-B normalization for either transmission or reflection as given previously.

The angular window is given as a solid angle. The incident beam has a divergence angle of β_1 within the diffraction plane and β_2 in the perpendicular direction. The angular window corresponding to the incident-beam divergence is given by

$$\Omega = \beta_1\beta_2/\sin\theta \text{ or } \Omega = \beta^2/\sin\theta \text{ if } \beta = \beta_1 = \beta_2. \tag{2.5.50}$$

For 2D-XRD, the angular window is not only determined by the incident-beam divergence, but also significantly increased by γ integration. When γ integration is used to generate the diffraction profile, it actually integrates the data collected over a range of various diffraction vectors. Since the effect of γ integration on sampling statistics is equivalent to the angular oscillation on the ψ axis in a conventional diffractometer, the effect is referred to as virtual oscillation and $\Delta\psi$ is the virtual oscillation angle. In conventional oscillation, mechanical movement may result in some sample-position error. Since there is no actual physical movement of the sample stage during data collection, virtual oscillation can avoid this error. This is crucial for micro-diffraction. The angular window with the contributions of both the incident-beam divergence and the virtual oscillation is

$$\Omega = \beta\Delta\psi = 2\beta\arcsin[\cos\theta\sin(\Delta\gamma/2)], \tag{2.5.51}$$