The spatial calibration must be done at the same sample-to-detector distance as the diffraction-data collection.

2.5.3.3.3. *Frame integration*

2D frame integration is a data-reduction process which converts a two-dimensional frame into a one-dimensional intensity profile. Two forms of integration are generally of interest in the analysis of a 2D diffraction frame from polycrystalline materials: γ integration and 2θ integration. γ integration sums the counts in 2θ steps ($\Delta 2\theta$) along constant 2θ conic lines and between two constant γ values. γ integration produces a data set with intensity as a function of 2θ. 2θ integration sums the counts in γ steps ($\Delta\gamma$) along constant γ lines and between two constant 2θ conic lines. 2θ integration produces a data set with intensity as a function of γ. γ integration may also be carried out with the integration range in the vertical direction as a constant number of pixels. This type of γ integration may also be referred to as slice integration. A diffraction profile analogous to the conventional diffraction result can be obtained by either γ integration or slice integration over a selected 2θ range. Phase ID can then be done with conventional search/match methods. 2θ integration is of interest for evaluating the intensity variation along γ angles, such as for texture analysis, and is discussed in more depth in Chapter 5.3.

The γ integration can be expressed as

$$I(2\theta) = \int_{\gamma_1}^{\gamma_2} J(2\theta, \gamma)\,\mathrm{d}\gamma, \quad 2\theta_1 \le 2\theta \le 2\theta_2, \qquad (2.5.23)$$

where $J(2\theta, \gamma)$ represents the two-dimensional intensity distribution in the 2D frame and $I(2\theta)$ is the integration result as a function of intensity *versus* 2θ. γ_1 and γ_2 are the lower limit and upper limit of integration, respectively, which are constants for γ integration. Fig. 2.5.13 shows a 2D diffraction frame collected from corundum (α-Al_2O_3) powder. The 2θ range is from 20 to 60° and the 2θ integration step size is 0.05°. The γ-integration range is from 60 to 120°. In order to reduce or eliminate the dependence of the integrated intensity on the integration interval, the integrated value at each 2θ step is normalized by the number of pixels, the arc length or the solid angle. γ integration with normalization by the solid angle can be expressed as

$$I(2\theta) = \frac{\int_{\gamma_1}^{\gamma_2} J(2\theta, \gamma)(\Delta 2\theta)\,\mathrm{d}\gamma}{\int_{\gamma_1}^{\gamma_2} (\Delta 2\theta)\,\mathrm{d}\gamma}, \quad 2\theta_1 \le 2\theta \le 2\theta_2. \qquad (2.5.24)$$

Since the $\Delta 2\theta$ step is a constant, the above equation becomes

$$I(2\theta) = \frac{\int_{\gamma_1}^{\gamma_2} J(2\theta, \gamma)\,\mathrm{d}\gamma}{\gamma_2 - \gamma_1}, \quad 2\theta_1 \le 2\theta \le 2\theta_2. \qquad (2.5.25)$$

There are many integration software packages and algorithms available for reducing 2D frames into 1D diffraction patterns for polycrystalline materials (Cervellino *et al.*, 2006; Rodriguez-Navarro, 2006; Boesecke, 2007). With the availability of tremendous computer power today, a relatively new method is the bin method, which treats pixels as having a continuous distribution in the detector. It demands more computer power than older methods, but delivers much more accurate and smoother results even with $\Delta 2\theta$ integration steps significantly smaller than the pixel size. Depending on the relative size of $\Delta 2\theta$ to the pixel size, each contributing pixel is divided into several 2θ 'bins'. The intensity counts of all pixels within the $\Delta 2\theta$ step are summarized. All the normalization methods in the above integration, either by pixel, arc or solid angle, result in an intensity level of one pixel or unit solid angle. Since a pixel is much smaller than the active area of a typical point detector, the normalized integration tends to result in a diffraction pattern with fictitiously low intensity counts, even though the true counts in the corresponding $\Delta 2\theta$ range are significantly higher. In order to avoid this misleading outcome, it is reasonable to introduce a scaling factor. However, there is no accurate formula for making the integrated profile from a 2D frame comparable to that from a conventional point-detector scan. The best practice is to be aware of the differences and to try not to make direct comparisons purely based on misleading intensity levels. Generally speaking, for the same exposure time, the total counting statistics from a 2D detector are significantly better than from a 0D or 1D detector.

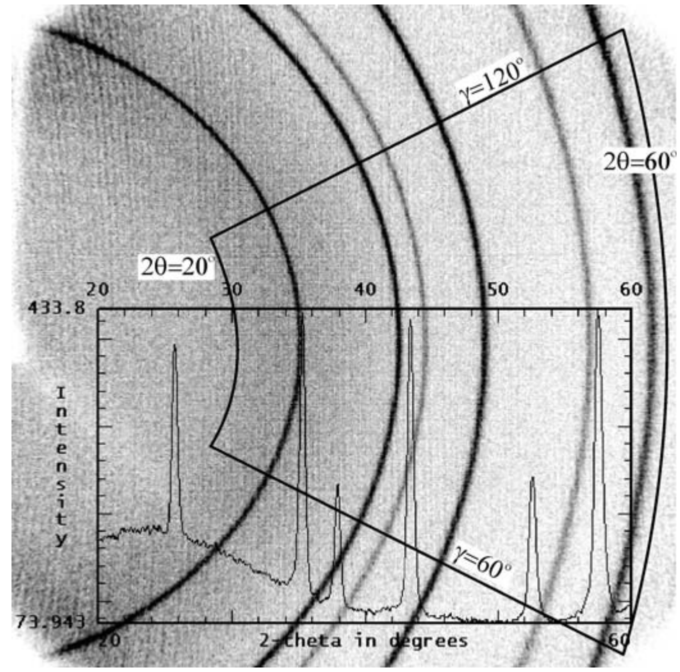

Figure 2.5.13
A 2D frame showing γ integration.

2.5.3.3.4. *Lorentz, polarization and absorption corrections*

Lorentz and polarization corrections may be applied to the diffraction frame to remove their effect on the relative intensities of Bragg peaks and background. The 2θ angular dependence of the relative intensity is commonly given as a Lorentz–polarization factor, which is a combination of Lorentz and polarization factors. In 2D diffraction, the polarization factor is a function of both 2θ and γ, therefore it should be treated in the 2D frames, while the Lorentz factor is a function of 2θ only. The Lorentz correction can be done either on the 2D frames or on the integrated profile. In order to obtain relative intensities equivalent to a conventional diffractometer with a point detector, reverse Lorentz and polarization corrections may be applied to the frame or integrated profile.

The Lorentz factor is the same as for a conventional diffractometer. For a sample with a completely random orientation distribution of crystallites, the Lorentz factor is given as

$$L = \frac{\cos\theta}{\sin^2 2\theta} = \frac{1}{4\sin^2\theta\cos\theta}. \qquad (2.5.26)$$

The Lorentz factor may be given by a different equation for a different diffraction geometry (Klug & Alexander, 1974). The forward and reverse Lorentz corrections are exactly reciprocal