equivalent, the keyword *mosaicity* describes the sum of both effects.

DENZO assumes the following model of angular shape of diffraction peaks for the oscillation angle φ:

$$M = \frac{\text{mos}}{\pi}\left[1 + \cos\frac{\pi(\varphi - \varphi_c)}{\text{mos}}\right], \qquad (11.4.6.9)$$

where mos is the observed angular width of a diffraction peak yet to be increased by the Lorentz factor, φ_c is the predicted oscillation angle at which the diffraction condition is fulfilled, φ is in the range$(\varphi_c - \text{mos}/2; \varphi_c + \text{mos}/2)$, otherwise $M = 0$. The actual width of the reflection is different due to Lorentz-factor variability over the image, so equation (11.4.6.9) describes only the common component of the angular width.

Using equation (11.4.6.9) we can calculate

$$P_{\varphi_1\varphi_2} = \int_{\varphi_1}^{\varphi_2} M(\varphi)\, \mathrm{d}\varphi. \qquad (11.4.6.10)$$

P is the predicted partiality of data collected by oscillating from φ_1 to φ_2. It is a number that represents what fraction of the reflection intensity is present in one image. If the partiality is 1, such reflections are called fully recorded; otherwise, they are called partials. For partials, predictions of partiality can be compared with the observed fraction P_0 of the reflection intensity present in one image. The partiality model contributes the following term to the refinement:

$$\chi^2_P = (P_{\varphi_1\varphi_2} - P_0)^2/\sigma^2_{P_0}. \qquad (11.4.6.11)$$

The combined positional [described by equations (11.4.6.7) and (11.4.6.8)] and partiality refinement [equation (11.4.6.11)] used in *DENZO* is both stable and very accurate. The power of this method lies in proper weighting by estimated errors of two very different terms – one describing positional differences and the other describing intensity differences. Both detector and crystal variables are uniformly treated in the refinement process.

11.4.6.3. Detector distortions

The design of detectors results in pixels not being positioned on an exact square or rectangular grid. A correct understanding of the detector distortions is essential to accurate positional refinement. The types of distortions are detector-specific. The primary sources of error include misalignment of the detector position sensors and optical or magnetic distortion in CCD-based detectors. If the detector distortion can be parameterized, then these parameters should be added to the refinement. For example, in the case of spiral scanners, there are two parameters describing the end position of the scanning head. In a perfectly adjusted scanner, these parameters would be zero. In practice, however, they may deviate from zero by as much as 1 mm. Such misalignment parameters can correlate very strongly with other detector and crystal parameters, particularly for low-symmetry lattices or for low-resolution data. If the distortions are stable, it is better to determine them in a separate experiment optimized for that task.

Fibre-optic tapers used in many CCD detectors have distortions that have to be individually determined for each instrument. The distortion is stable over time and its spatial characteristics are dominated by a smooth component and a small local shear. In high-quality tapers used in X-ray instruments, the small local shear can be ignored. The smooth component can be parameterized in a number of ways, for example by splines or polynomials (Messerschmidt & Pflugrath, 1987). *DENZO* uses two-dimensional Chebyschev polynomials (Press *et al.*, 1989) in $\{x, y\}$ or $\{p, q\}$ coordinates, normalized to the range $\{[-1, +1], [-1, +1]\}$. Typically, fifth- or seventh-order polynomials result in a positional error (r.m.s.) lower than 7 μm, which is about one tenth of a typical detector pixel. *DENZO* can use either a grid mask pattern or the X-ray diffraction pattern to refine the coefficients of the Chebyschev polynomials. If a grid mask is used, it has to be precisely made and positioned. The use of crystallographic data requires precise knowledge of detector and crystal parameters that are not known *a priori* with the required precision. The crystal and detector parameters can be determined in the same experiment as detector distortion. However, this experiment needs to be designed to minimize the impact of correlations between the parameters involved. The data analysis requires the description of the distortion function and its inverse. In *DENZO*, both are approximated in terms of Chebyschev polynomials. The magnitude of the approximation error is the same for the distortion function and its inverse.

11.4.7. Integration of diffraction maxima by profile fitting

To accurately integrate diffraction peaks, the spot position has to be predicted accurately. Each integration program has its own procedure for predicting and fitting diffraction profiles. In *DENZO*, profile-shape prediction is defined by a weighted average of other reflection profiles present within some radius from the spot of interest. Each spot has its own prediction, which is continuously adjusted to variations of spot shapes over the detector. The profiles are added by shifting them to the same position, generating a normalized profile P_i, where $\sum_i P_i = 1$. In the second step, the measured pixels' values are fitted to a function

$$B_i + IP_i, \qquad (11.4.7.1)$$

where B_i is the predicted value for the background in pixel i and I is the diffraction intensity of the spot. The profile-fitting procedure minimizes the function

$$\sum_i \frac{[M_i - (B_i + IP_i)]^2}{V_i}, \qquad (11.4.7.2)$$

where M_i are the measured pixel values and V_i are the variances of these measurements. The minimum of this function defines the value of the profile-fitted intensity (Otwinowski & Minor, 1997):

$$I = \frac{\sum_i P_i(M_i - B_i)/V_i}{\sum_i P_i^2/V_i}. \qquad (11.4.7.3)$$

11.4.8. Scaling – multiplicative corrections

The relation between the measured intensity $I(hkl)$ of reflection hkl and its squared structure-factor amplitude $|\mathbf{F}(hkl)|^2$ is described by

$$I(hkl) = I_b r_e^2 \frac{\lambda^2}{|\mathbf{S} \times \boldsymbol{\omega}'|} PT \frac{V}{v_u^2} |\mathbf{F}(hkl)|^2 D_A D_S, \qquad (11.4.8.1)$$

where I_b is the flux density of the primary beam; $r_e = e^2/m_e c^2$ is the classical electron radius (2.818×10^{-12} mm); λ is the wavelength of the beam; $\mathbf{S} \times \boldsymbol{\omega}'$ is a cross product between the diffraction vector $\mathbf{S}$ and $\boldsymbol{\omega}'$ (the projection of the crystal-rotation-speed vector $\boldsymbol{\omega}$ on the plane perpendicular to the primary beam;