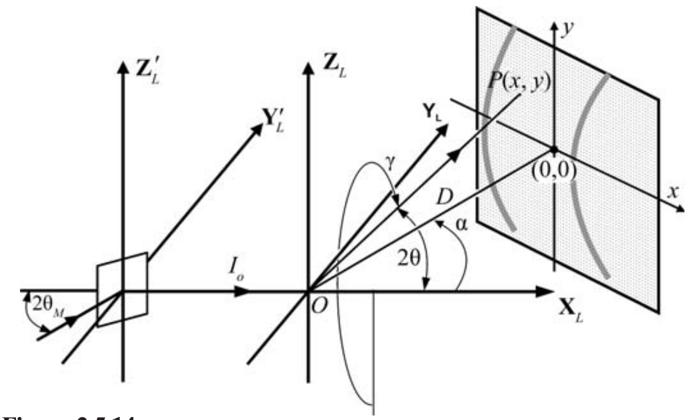

Figure 2.5.14
Geometric relationship between the monochromator and detector in the laboratory coordinates.

and effectively cancel each other. Therefore, it is not necessary to perform the Lorentz correction to the frame before integration if relative intensities equivalent to a conventional Bragg–Brentano diffractometer are expected. The Lorentz correction can be done on the integrated diffraction profiles in the same way as on the diffraction profiles collected with conventional diffractometers.

When a non-polarized X-ray beam is scattered by matter, the scattered X-rays are polarized. The intensity of the diffracted beam is affected by the polarization; this effect is expressed by the polarization factor. In two-dimensional X-ray diffraction the diffraction vectors of the monochromator diffraction and sample crystal diffraction are not necessarily in the same plane or perpendicular planes. Therefore, the overall polarization factor is a function of both 2θ and γ. Fig. 2.5.14 illustrates the geometric relationship between the monochromator and detector in the laboratory coordinates, X_L, Y_L, Z_L. The monochromator is located at the coordinates X_L, Y'_L, Z'_L, which is a translation of the laboratory coordinates along the X_L axis in the negative direction. The monochromator crystal is rotated about the Z'_L axis and $2\theta_M$ is the Bragg angle of the monochromator crystal. The diffracted beam from the monochromator propagates along the X_L direction. This is the incident beam to the sample located at the instrument centre O. The 2D detector location is given by the sample-to-detector distance D and swing angle α. The pixel $P(x, y)$ represents an arbitrary pixel on the detector. 2θ and γ are the corresponding diffraction-space parameters for the pixel. Since a monochromator or other beam-conditioning optics can only be used on the incident beam, the polarization factor for 2D-XRD can then be given as a function of both θ and γ:

$$P(\theta, \gamma) = \frac{(1 + \cos^2 2\theta_M \cos^2 2\theta)\sin^2\gamma + (\cos^2 2\theta_M + \cos^2 2\theta)\cos^2\gamma}{1 + \cos^2 2\theta_M}. \quad (2.5.27)$$

If the crystal monochromator rotates about the Y'_L axis, *i.e.* the incident plane is perpendicular to the diffractometer plane, the polarization factor for two-dimensional X-ray diffraction can be given as

$$P(\theta, \gamma) = \frac{(1 + \cos^2 2\theta_M \cos^2 2\theta)\cos^2\gamma + (\cos^2 2\theta_M + \cos^2 2\theta)\sin^2\gamma}{1 + \cos^2 2\theta_M}. \quad (2.5.28)$$

In the above equations, the term $\cos^2 2\theta_M$ can be replaced by $|\cos^n 2\theta_M|$ for different monochromator crystals. For a mosaic crystal, such as a graphite crystal, $n = 2$. For most real monochromator crystals, the exponent n takes a value between 1 and 2. For near perfect monochromator crystals, n approaches 1 (Kerr & Ashmore, 1974). All the above equations for polarization factors may apply to multilayer optics. However, since multilayer optics have very low Bragg angles, $|\cos^n 2\theta_M|$ approximates to unity. The γ dependence of the polarization factor diminishes in this case. The polarization factor approaches

$$P(\theta, \gamma) \simeq \frac{1 + \cos^2 2\theta}{2}. \quad (2.5.29)$$

2.5.3.3.5. *Air scatter*

X-rays are scattered by air molecules in the beam path between the X-ray source and detector. Air scatter results in two effects: one is the attenuation of the X-ray intensity, the other is added background in the diffraction pattern. Air scatter within the enclosed primary beam path – for instance, in the mirror, monochromator housing or collimator – results in attenuation of only the incident beam. The enclosed beam path can be purged by helium gas or kept in vacuum to reduce the attenuation so that no correction is necessary for this part of the air scatter. The open beam between the tip of the collimator and the sample generates an air-scatter background pattern, which is the major part of the air scatter. In the secondary beam path, the air scatter from the diffracted beam may generate background too, but the main effect of the air scatter is inhomogeneous attenuation of the diffraction pattern due to the different beam path lengths between the centre and the edge of the detector.

The background generated by air scattering from the open incident-beam path has a strong 2θ dependence. The specific scattering curve depends on the length of the open primary beam path, the beam size and the wavelength of the incident beam. There are two approaches to correct air scatter. One is to collect an air-scatter background frame under the same conditions as the diffraction frame except without a sample. The background frame is then subtracted from the diffraction frame. Another approach is to remove the background from the integrated profile, since the background is 2θ dependent.

The attenuation of the diffracted beam by air absorption depends on the distance between the sample and pixel. For a flat detector, air absorption can be corrected by

$$p_c(x, y) = p_o(x, y)\exp\left[\mu_{\rm air}(D^2 + x^2 + y^2)^{1/2}\right], \quad (2.5.30)$$

where $p_o(x, y)$ is the original pixel intensity of the pixel $P(x, y)$ and $p_c(x, y)$ is the corrected intensity. The detector centre is given by (0, 0). $\mu_{\rm air}$ is the linear absorption coefficient of air. The value of $\mu_{\rm air}$ is determined by the radiation wavelength. By approximation, for air with 80% N_2 and 20% O_2 at sea level and at 293 K, $\mu_{\rm air} = 0.01$ cm^{-1} for Cu $K\alpha$ radiation. Air scatter and absorption increases with increasing wavelength. For example, $\mu_{\rm air} = 0.015$ cm^{-1} for Co $K\alpha$ radiation and 0.032 cm^{-1} for Cr $K\alpha$ radiation. The absorption coefficient for Mo $K\alpha$ radiation, $\mu_{\rm air} = 0.001$ cm^{-1}, is only one-tenth of that for Cu $K\alpha$ radiation, so an air-absorption correction is not necessary. Alternatively, the absorption correction may be normalized to the absorption level in the beam centre as

$$p_c(x, y) = p_o(x, y)\exp\left\{\mu_{\rm air}\left[(D^2 + x^2 + y^2)^{1/2} - D\right]\right\}. \quad (2.5.31)$$