walls and containers in X-ray work. Most of the X-ray investigations have therefore been made on quenched samples. Because TDS is dominating at high temperatures, also in the presence of a static disorder problem, the quantitative separation can hardly be carried out in the case of high experimental background. Calculation and subtraction of the TDS is possible in principle, but difficult in practice.

A quantitative analysis of diffuse-scattering data is essential for a definite decision about a disorder model. By comparison of calculated and corrected experimental data the magnitudes of the parameters of the structural disorder model may be derived. A careful analysis of the data requires, therefore, corrections for polarization (X-ray case), absorption and resolution. These may be performed in the usual way for polarization and absorption. Very detailed considerations, however, are necessary for the question of instrumental resolution which depends, in addition to other factors, on the scattering angle and implies intensity corrections analogous to the Lorentz factor used in structure analysis from sharp Bragg reflections.

Resolution is conveniently described by a function, $R(\mathbf{H}-\mathbf{H}_0)$, which is defined as the probability of detecting a photon or neutron with momentum transfer $h\mathbf{H}=h(\mathbf{k}-\mathbf{k}_0)$ when the instrument is set to measure $\mathbf{H}_0$. This function R depends on the instrumental parameters (collimations, mosaic spread of monochromator, scattering angle) and the spectral width of the source. Fig. 4.2.5.1 shows a schematic sketch of a diffractometer setting. Detailed considerations of resolution volume in X-ray diffractometry are given by Sparks & Borie (1966). If a triple-axis (neutron) instrument is used, for example in a purely elastic configuration, the set of instrumental parameters is extended by the mosaic of the analyser and the collimations between analyser and detector (see Chapter 4.1).

If photographic (X-ray) techniques are used, the detector aperture is controlled by the slit width of the microdensitometer. A general formulation of R in neutron diffractometry is given by Cooper & Nathans (1968):

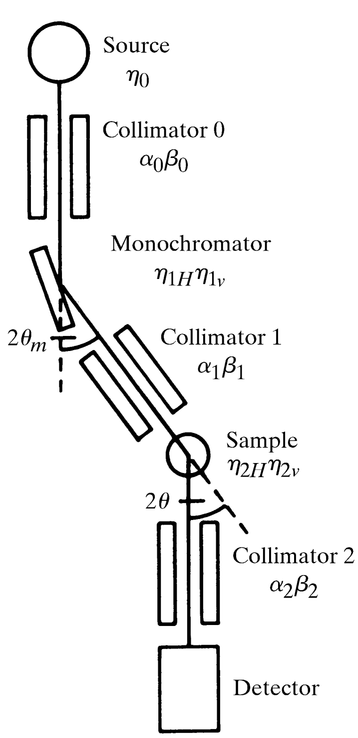

Fig. 4.2.5.1. Schematic sketch of a diffractometer setting.

$$R'(\mathbf{H}-\mathbf{H}_0)=R'_0\exp\left\{-\tfrac{1}{2}\sum_k\sum_l M'_{kl}\Delta H_k\Delta H_l\right\}. \qquad (4.2.5.1)$$

Gaussians are assumed for the mosaic distributions and for the transmission functions the parameters are involved in the coefficients R'_0 and M'_{kl}.

The general assumption of Gaussians is not too serious in the X-ray case (Iizumi, 1973). Restrictions are due to absorption which makes the profiles asymmetric. Box-like functions are considered to be better for the spectral distribution or for large apertures (Boysen & Adlhart, 1987). These questions are treated in some detail by Klug & Alexander (1954). The main features, however, may also be derived by the Gaussian approximation. In practice the function R may be obtained either by calculation from the known instrumental parameters or by measuring Bragg peaks of a perfect unstrained crystal. In the latter case [*cf.* equation (4.2.5.2)] the intensity profile is given solely by the resolution function. A normalization with the Bragg intensities is also useful in order to place the diffuse-scattering intensity on an absolute scale.

In single-crystal diffractometry the measured intensity is given by the convolution product of $\mathrm{d}\sigma/\mathrm{d}\Omega$ with R,

$$I(\mathbf{H}_0)=\int\frac{\mathrm{d}\sigma}{\mathrm{d}\Omega}(\mathbf{H})\cdot R(\mathbf{H}-\mathbf{H}_0)\,\mathrm{d}\mathbf{H}, \qquad (4.2.5.2)$$

where $\mathrm{d}\sigma/\mathrm{d}\Omega$ describes the scattering cross section for the disorder problem. In more accurate form the mosaic of the sample has to be included:

$$\begin{aligned}I(\mathbf{H}_0)&=\int\frac{\mathrm{d}\sigma}{\mathrm{d}\Omega}(\mathbf{H}-\Delta\mathbf{k})\cdot\eta(\Delta\mathbf{k})R(\mathbf{H}-\mathbf{H}_0)\,\mathrm{d}\mathbf{H}\,\mathrm{d}(\Delta\mathbf{k})\\&=\int\frac{\mathrm{d}\sigma}{\mathrm{d}\Omega}(\mathbf{H}')\cdot R'(\mathbf{H}'-\mathbf{H}_0)\,\mathrm{d}\mathbf{H}'. \qquad (4.2.5.2a)\end{aligned}$$

$R'(\mathbf{H}'-\mathbf{H}_0)=\int\eta(\Delta\mathbf{k})R(\mathbf{H}'+\Delta\mathbf{k}-\mathbf{H}_0)\,\mathrm{d}(\Delta\mathbf{k})$. $\eta(\Delta\mathbf{k})$ describes the mosaic block distribution around a most probable vector $\mathbf{k}_0$: $\Delta\mathbf{k}=\mathbf{k}-\mathbf{k}_0$; $\mathbf{H}'=\mathbf{H}-\Delta\mathbf{k}$.

In formulae (4.2.5.1) and (4.2.5.2) all factors independent of 2θ are neglected. All intensity expressions have to be calculated from equations (4.2.5.2) or (4.2.5.2*a*). In the case of a dynamical disorder problem, *i.e.* when the differential cross section also depends on energy transfer $\hbar\omega$, the integration must be extended over energy.

The intensity variation of diffuse peaks with 2θ was studied in detail by Yessik *et al.* (1973). In principle all special cases are included there. In practice, however, some important simplifications can be made if $\mathrm{d}\sigma/\mathrm{d}\Omega$ is either very broad or very sharp compared with R, *i.e.* for Bragg peaks, sharp streaks, 'thin' diffuse layers or extended 3D diffuse peaks (Boysen & Adlhart, 1987).

In the latter case the cross section $\mathrm{d}\sigma/\mathrm{d}\Omega$ may be treated as nearly constant over the resolution volume so that the corresponding 'Lorentz' factor is independent of 2θ:

$$L_{3D}=1. \qquad (4.2.5.3)$$

For a diffuse plane within the scattering plane with very small thickness and slowly varying cross section within the plane, one derives for a point measurement in the plane:

$$L_{2D,\parallel}=(\beta_1'^2+\beta_2^2+\eta_{2v}^2-\sin^2\theta)^{-1/2}, \qquad (4.2.5.4)$$

exhibiting an explicit dependence on θ ($\beta'_1,\beta_2,\eta_{2v}$ determining an effective vertical divergence before the sample, the divergence before the detector and the vertical mosaic spread of the sample, respectively).

In the case of *relaxed* vertical collimations $\beta'_1,\beta_2\gg\eta_{2v}$

$$L_{2D,\parallel}=(\beta_1'^2+\beta_2^2)^{-1/2}, \qquad (4.2.5.4a)$$

i.e. again independent of θ.

references