2.5.1.3. *Resolution*

The momentum resolution in energy-dispersive diffraction is limited by the angular divergence of the incident and diffracted X-ray beams and by the energy resolution of the detector system. The observed profile is a convolution of the profile due to the angular divergence and the profile due to the detector response. For resolution calculations, it is usually assumed that the profiles are Gaussian, although the real profiles might exhibit geometrical and physical aberrations (Subsection 2.5.1.5). The relative full width at half-maximum (FWHM) of a diffraction peak in terms of energy is then given by

$$\delta E/E = [(e_n/E)^2 + 5.546F\varepsilon/E + (\cot\theta_0\Delta\theta_0)^2]^{1/2}, \quad (2.5.1.2)$$

where e_n is the electronic noise contribution, F the Fano factor, ε the energy required for creating an electron–hole pair (*cf.* Subsection 7.1.5.1), and $\Delta\theta_0$ the overall angular divergence of the X-ray beam, resulting from a convolution of the incident- and the diffracted-beam profiles. For synchrotron radiation, $\Delta\theta_0$ can usually be replaced by the divergence of the diffracted beam because of the small divergence of the incident beam.

Fig. 2.5.1.3 shows $\delta E/E$ as a function of Bragg angle θ_0. The curves have been calculated from equations (2.5.1.1) and (2.5.1.2) for two values of the lattice-plane spacing and two values of $\Delta\theta_0$, typical for *Bremsstrahlung* and synchrotron radiation, respectively. It is seen that in all cases $\delta E/E$ decreases with decreasing angle (*i.e.* increasing energy) to a certain minimum and then increases rapidly. It is also seen that the minimum point of the $\delta E/E$ curve is lower for the small d value and shifts towards smaller θ_0 values for decreasing $\Delta\theta_0$. Calculations of this kind are valuable for optimizing the Bragg angle for a given sample and other experimental conditions (*cf.* Fukamachi, Hosoya & Terasaki, 1973; Buras, Niimura & Olsen, 1978).

The relative peak width at half-height is typically less than 1% for energies above 30 keV. When the observed peaks can be fitted with Gaussian functions, one can determine the centroids of the profiles by a factor of 10–100 better than the $\delta E/E$ value of equation (2.5.1.2) would indicate. Thus, it should be possible to achieve a relative resolution of about 10^{-4} for high energies. A resolution of this order is required for example in residual-stress measurements.

The detector broadening can be eliminated using a technique where the diffraction data are obtained by means of a scanning crystal monochromator and an energy-sensitive detector (Bourdillon, Glazer, Hidaka & Bordas, 1978; Parrish & Hart, 1987). A low-resolution detector is sufficient because its function (besides recording) is just to discriminate the monochromator harmonics. The Bragg reflections are not measured simultaneously as in standard XED. The monochromator-scan method can be useful when both a fixed scattering angle (*e.g.* for samples in special environments) and a high resolution are required.

2.5.1.4. *Integrated intensity for powder sample*

The kinematical theory of diffraction and a non-absorbing crystal with a 'frozen' lattice are assumed. Corrections for thermal vibrations, absorption, extinction, *etc.* are discussed in Subsection 2.5.1.5. The total diffracted power, $P_{\mathbf{h}}$, for a Bragg reflection of a powder sample can then be written (Buras & Gerward, 1975; Kalman, 1979)

$$P_{\mathbf{h}} = hcr_e^2 VN_c^2 [i_0(E) jd^2 |F|^2]_{\mathbf{h}} C_p(E, \theta_0) \cos\theta_0 \Delta\theta_0, \quad (2.5.1.3)$$

where $\mathbf{h}$ is the diffraction vector, r_e the classical electron radius, $i_0(E)$ the intensity per unit energy range of the incident beam evaluated at the energy of the diffraction peak, V the irradiated sample volume, N_c the number of unit cells per unit volume, j the multiplicity factor, F the structure factor, and $C_p(E, \theta_0)$ the polarization factor. The latter is given by

$$C_p(E, \theta_0) = \tfrac{1}{2}[1 + \cos^2 2\theta_0 - P(E)\sin^2 2\theta_0], \quad (2.5.1.4)$$

where $P(E)$ is the degree of polarization of the incident beam. The definition of $P(E)$ is

$$P(E) = \frac{i_{0,p}(E) - i_{0,n}(E)}{i_0(E)}, \quad (2.5.1.5)$$

where $i_{0,p}(E)$ and $i_{0,n}(E)$ are the parallel and normal components of $i_0(E)$ with respect to the plane defined by the incident- and diffracted-beam directions.

Generally, $C_p(E, \theta_0)$ has to be calculated from equations (2.5.1.4) and (2.5.1.5). However, the following special cases are sometimes of interest:

$$P = 0: \quad C_p(\theta_0) = \tfrac{1}{2}(1 + \cos^2 2\theta_0) \quad (2.5.1.6a)$$

$$P = 1: \quad C_p(\theta_0) = \cos^2 2\theta_0 \quad (2.5.1.6b)$$

$$P = -1: \quad C_p = 1. \quad (2.5.1.6c)$$

Equation (2.5.1.6*a*) can often be used in connection with *Bremsstrahlung* from an X-ray tube. The primary X-ray beam can be treated as unpolarized for all photon energies when there is an angle of 45° between the plane defined by the primary and the diffraced beams and the plane defined by the primary beam and the electron beam of the X-ray tube. In standard configurations, the corresponding angle is 0° or 90° and equation (2.5.1.6*a*) is generally not correct. However, for $2\theta_0 < 20°$ it is correct to within 2.5% for all photon energies (Olsen, Buras, Jensen, Alstrup, Gerward & Selsmark, 1978).

Equations (2.5.1.6*b*) and (2.5.1.6*c*) are generally acceptable approximations for synchrotron radiation. Equation (2.5.1.6*b*) is used when the scattering plane is horizontal and (2.5.1.6*c*) when the scattering plane is vertical.

The diffraction directions appear as generatrices of a circular cone of semi-apex angle $2\theta_0$ about the direction of incidence. Equation (2.5.1.3) represents the total power associated with this

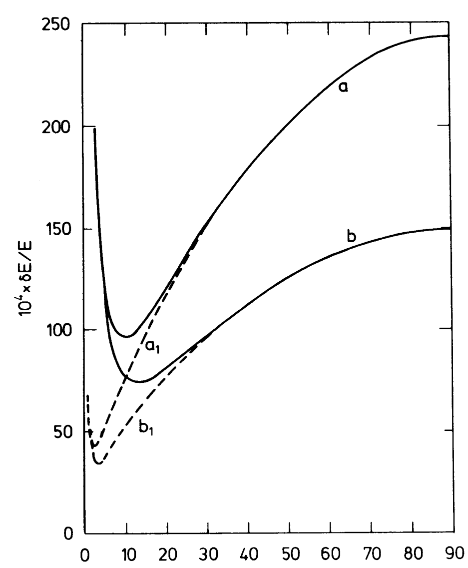

Fig. 2.5.1.3. Relative resolution, $\delta E/E$, as function of Bragg angle, θ_0, for two values of the lattice plane spacing: (*a*) 1 Å and (*b*) 0.5 Å. The full curves have been calculated for $\Delta\theta_0 = 10^{-3}$, the broken curves for $\Delta\theta_0 = 10^{-4}$.

references