dispersive diffraction (EDD) described in Section 2.5.1, the specimen and detector remain fixed at selected angles during the recording. This makes it possible to design special experiments that would not be possible with specimen-scanning methods. It also simplifies the design of specimen-environment chambers for high and low temperatures. The advantages of the method over conventional EDD are the order-of-magnitude higher resolution that can be controlled by the X-ray optics, the ability to handle high peak count rates with a high-speed scintillation counter and conventional circuits, and much lower count times for good statistical accuracy.

The accessible range of *d*'s that can be recorded using a selected wavelength range is determined by the 2θ setting of the detector. Changing 2θ causes the separation of the peaks to expand or compress in a manner similar to a variation of λ in conventional diffractometry. This is illustrated in Figs. 2.3.2.9(*a*)–(*d*) for a quartz powder specimen using an Si(111) channel monochromator and $\theta_M = 19$ to 5° (2.04 to 0.55 Å, 6.1 to 22.7 keV) and four detector 2θ settings. At small 2θ settings, only the large *d*'s are recorded and the peak separation is large. Increasing the 2θ setting decreases the *d* range and the separation of the peaks as shown in Fig. 2.3.2.9(*e*). These patterns were recorded with the pulse-height analyser set to discriminate only against scintillation counter noise.

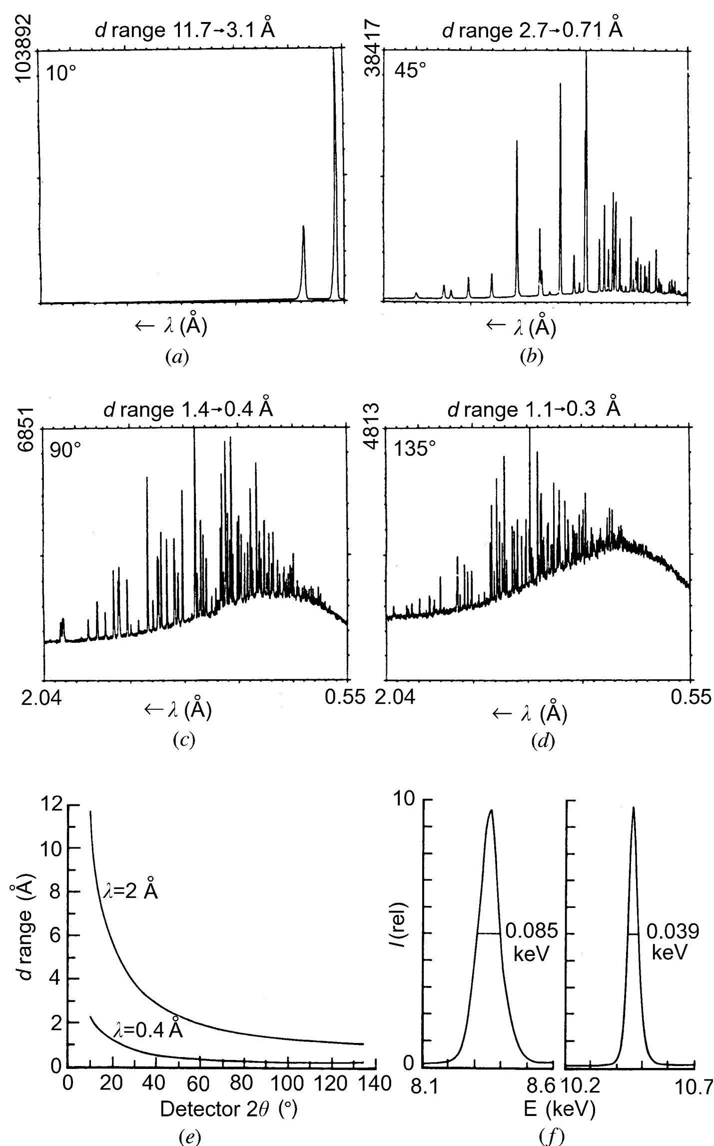

Fig. 2.3.2.9. (*a*)–(*d*) High-resolution energy-dispersive diffraction patterns of quartz powder sample obtained with 2θ settings shown in upper left corners. (*e*) *d* range as a function of detector 2θ setting for $\lambda = 0.4$ to 2 Å. (*f*) Effect of 2θ setting and *E* on profile widths of quartz. Right: 121 reflection, $20°2\theta$, E_p 10.45 keV; left: 100 reflection, $45°2\theta$, e_p 8.35; both reflections broadened by X-ray optics and peak intensity of 100 twice that of 121.

For given X-ray optics, the profiles symmetrically broaden with decreasing X-ray photon energy and with θ. This type of broadening remains symmetrical if *E* is increased and 2θ decreased, or *vice versa*, Fig. 2.3.2.9(*f*). The two profiles shown have been broadened by the X-ray optics but the intrinsic resolution is far better. The number of points recorded per profile thus decreases with decreasing profile width since $\Delta\theta_M$ is constant. At the higher energies, it may be desirable to use smaller $\Delta\theta_M$ steps to increase the number of points to define better the profile. Alternatively, increments in $\sin\theta$ steps rather than θ steps would eliminate this variation.

Many electronic solid-state devices use thin films that are purposely prepared to have single-crystal structure (*e.g.* epitaxic growth), or with a selected lattice plane oriented parallel or normal to the film surface to enhance certain properties. The properties vary with the degree of orientation and textural characterization is essential to make the correct film preparation. Preferred orientation can be detected by comparing the relative intensities of the thin-film pattern with those of a random powder. The pattern can be recorded with conventional θ–2θ scanning (λ fixed) or by EDD. However, this only gives information on the planes oriented parallel to the surface. To study inclined planes requires uncoupling the specimen surface and detector angles. This can be done with the EDD method described above without distorting the profiles (Hart *et al.*, 1987).

The principle of the method is illustrated in Fig. 2.3.2.10. The set of lattice planes (*hkl*) oriented parallel to the surface has its highest intensity in the symmetric θ–2θ position. Rotating the specimen by an angle θ_r while keeping 2θ fixed reduces the intensity of (*hkl*) and brings another set of planes (*pqr*), which are inclined to the surface, to its symmetrical reflecting position. The required rotation is determined by the interplanar angle between (*hkl*) and (*pqr*). The angular distribution of any plane can be measured with respect to the film surface by step scanning at small θ_r steps. The specimen is rotated clockwise with the limitation $\theta_s + \theta_r < 2\theta$. A computer automation program is desirable for large numbers of measurements.

Fig. 2.3.2.3(*c*) shows the appearance of a pattern of a specimen containing elements with absorption edges in the recording range and using electronic discrimination only against electronic noise. Starting at the incident high-energy side, the Zn and Ni *K* fluorescence increases as the energy approaches the edges (λ^3 law), decreases abruptly when the energy crosses each edge, and disappears beyond the Ni *K* edge. Long-wavelength fluorescence is absorbed in the windows and air path.

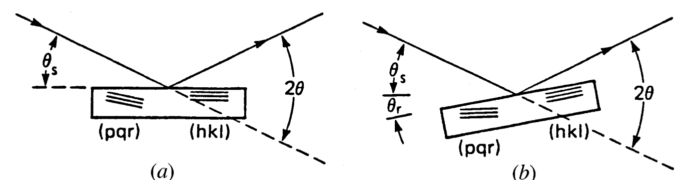

Fig. 2.3.2.10. Specimen orientation for symmetric reflection (*a*) from (*hkl*) planes and (*b*) specimen rotated θ_r for symmetric reflection from (*pqr*) planes.

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