The easy wavelength selection makes it possible to avoid specimen fluorescence, to record data on both sides of an absorption edge for anomalous-scattering studies, to select optimum angles and wavelengths for lattice-parameter measurements, and to vary the dispersion. Short-wavelength radiation can be used for uncomplicated patterns without the background occurring in X-ray tube spectra. Fig. 2.3.2.2 shows a silicon pattern obtained with 1.0 Å X-rays in which there are twice as many reflections as can be recorded with Cu $K\alpha$, and the background remains very low out to the highest $2\theta$ angles. The short wavelengths ($\sim 0.7$ Å) are especially useful for samples mounted in cryostats, furnaces, and pressure cells.

Using an incident-beam tunable monochromator, no continuous radiation reaches the specimen and a wavelength can be selected that gives a high peak-to-background ratio and no specimen fluorescence. If the specimen contains different chemical phases, patterns can be recorded using wavelengths on both sides of the absorption edge to enhance one of the patterns as an aid in identification. This is illustrated in Fig. 2.3.2.3 for a mixture of Ni and ZnO powders. A pattern (*a*) with maximum peak-to-background ratio is obtained with a wavelength slightly longer than the Ni *K*-absorption edge but using a wavelength shorter than the edge (*b*) causes high Ni *K* fluorescence background. The relative intensities of the peaks in each compound are the same with both wavelengths. However, the large change in the Ni absorption across the edge caused a large difference in the ratio of Ni/ZnO intensities. The Ni(111) decreased by 85% and the intensity ratio Ni(111)/ZnO(102) dropped from 4.2 to 1.3.

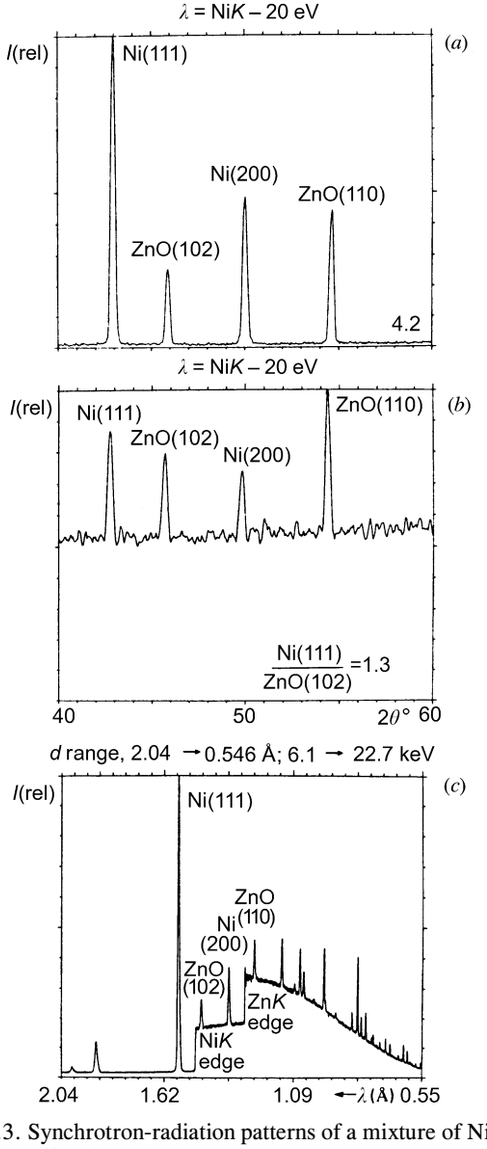


Fig. 2.3.2.3. Synchrotron-radiation patterns of a mixture of Ni and ZnO powders. Diffraction pattern using a wavelength (*a*) slightly longer than the Ni *K*-absorption edge and (*b*) slightly shorter. (*c*) High-resolution energy-dispersive diffraction (EDD) pattern.

Modified conventional vertical-scanning diffractometers are used to avoid intensity losses from the strong polarization in the horizontal plane. The six basic powder diffraction methods that have been used are:

(*a*) Monochromatic X-rays with $\theta$–$2\theta$ scanning and flat specimen as in conventional X-ray tube methods but using parallel-beam X-ray optics. This is the most widely applicable method for polycrystalline materials.

(*b*) Monochromatic X-rays with fixed specimen and $2\theta$ detector scan, used for analysing texture, preferred orientation, and grazing-incidence diffraction.

(*c*) Monochromatic X-rays with a capillary specimen and scanning receiving slit or position-sensitive detector.

(*d*) Energy-dispersive diffraction using a step-scanned channel monochromator, selectable fixed $\theta$–$2\theta$ positions, and conventional scintillation counter and electronics. The instrumentation is the same as (*a*) and may be used in methods that require a stationary specimen.

(*e*) Energy-dispersive diffraction using the white beam, solid-state detector and multichannel analyser, and selected fixed $\theta$–$2\theta$. This is the method frequently used with synchrotron and X-ray tube sources but it has low pattern resolution (Giessen & Gordon, 1968).

(*f*) Angle-dispersive or energy-dispersive experiments with an imaging-plate detector, whereby complete Debye–Scherrer rings are recorded simultaneously, as in some film methods (Subsection 2.3.4.1) (*e.g.* Piltz *et al.*, 1992). This is a particularly useful technique for studies under non-ambient conditions, such as experiments at ultra-high pressure (*e.g.* McMahon & Nelmes, 1993).

### 2.3.2.1. *Monochromatic radiation, θ–2θ scan*

The X-ray optics of a plane-wave parallel-beam diffractometer is shown schematically in Fig. 2.3.2.4(*a*). The primary white beam is limited by slits at *C*1. A channel monochromator CM is used because it has the important property of maintaining the same direction and position for a wide range of wavelengths. It may be used in the dispersive setting with respect to the specimen or in the parallel setting [Fig. 2.3.2.4(*b*)]. The monochromatic beam is larger than the entrance slit ES and it is unnecessary to realign the powder diffractometer when changing wavelengths. The monochromator can be mounted on a stripped diffractometer for easy alignment and step scanning.

There are no characteristic spectral lines and the wavelength calibration of the monochromator is made by step scanning the monochromator across absorption edges of elements in a specimen or pure element foils placed in the beam. The wavelength accuracy is limited by the uncertainty as to what feature of the edge should be measured and which one was used for the wavelength tables. A standard powder sample such as NIST silicon 640b whose lattice parameter is known with moderately high precision can also be used. An alternative method is to measure the reflection angle of a single-crystal plate of float-zones oxygen-free silicon whose lattice parameter is known to 1 part in $10^{-7}$ and to determine the wavelength from





references