Table 8.2.2.1. *Advantages and disadvantages of the Laue technique*

This table is adapted from Moffat (1997). See also Ren *et al.* (1999).

Advantages
Shortest possible exposure time, well suited to rapid time-resolved studies that require high time resolution.
Insensitive to all temporal fluctuations in the beam incident on the crystal, whether arising from the source itself, the optical components of the beamline or the shutter train. (Sensitive only to unusual fluctuations of the shape of the incident spectrum with time.)
All spots in a local region of the detector have an identical profile; none are (geometrically) partial.
Requires a stationary crystal and relatively simple optical components, therefore images are easy to acquire.
A large volume of reciprocal space is surveyed per image, hence fewer images are necessary to survey the entire unique volume.
High redundancy of measurements readily obtained, particularly at high resolution.
Disadvantages
Energy overlaps must be deconvoluted into their components if complete data are to be obtained, particularly at low resolution.
Spatial overlaps are numerous, particularly for mosaic crystals, and must be resolved.
Completeness at low resolution may be low, which would lead to significant series-termination errors in Fourier maps.
The rate of heating owing to X-ray absorption can be very high.
The wider the wavelength range, the higher the background under each spot; a trade-off is unavoidable between coverage of reciprocal space and accuracy of intensity measurements.
Spot shape is quite sensitive to crystal disorder.
More complicated wavelength-dependent corrections must be derived and applied to spot intensities to yield structure amplitudes.

increasing the average spot-to-spot distance and potentially increasing the signal-to-noise ratio. There are, however, trade-offs. More ordered crystals may not be readily available, a narrower wavelength range means that more images are required for a complete data set and the detector must continue to intercept all of the high-angle diffraction data (which consist largely of single spots stimulated by longer wavelengths).

As a third example, consider radiation damage. This can be purely thermal, arising from heating due to X-ray absorption. The rate of temperature rise may easily reach several hundred kelvin per second from a focused pink bending-magnet beam at second-generation sources such as the National Synchrotron Light Source (NSLS) (Chen, 1994; Moffat, 1997) or several thousand kelvin per second from a focused wiggler source at third-generation sources such as the ESRF. Fast shutters are required to provide an individual exposure of one millisecond or less in the latter case, and hence to limit the temperature rise to a readily survivable value of several kelvin (Bourgeois *et al.*, 1996; Moffat, 1997). Primary radiation damage (arising directly from X-ray absorption and hence from energy deposition) cannot be eliminated, but it may be modified by selection of the wavelength range and by lowering λ_{max}. Secondary radiation damage, arising from the chemical and structural damage generated by highly reactive, rapidly diffusing free radicals, hydrated electrons and other chemical species, can be greatly minimized by the use of very short exposures which allow little time for damaging reactions to occur, and by working at cryogenic temperatures where diffusion is greatly reduced (see *e.g.* Garman & Schneider, 1997). However, the last strategy may not be an option in a time-resolved Laue experiment, where the desired structural transitions may be literally frozen out at cryogenic temperatures.

Extraction of structure amplitudes from a Laue image or data set proceeds through five stages, reviewed in detail by Clifton *et al.* (1997) and Ren *et al.* (1999), and outlined in Fig. 8.2.3.1. First comes the purely geometrical process of indexing, in which each spot is associated with the appropriate *hkl* value and the unit-cell parameters, crystal orientation matrix, λ_{min}, geometric parameters of the detector and X-ray camera, λ_{max} and d^*_{max} are refined, also yielding λ, the wavelength stimulating that spot. In the second stage, each spot is integrated using appropriate profile-fitting algorithms. Thirdly, the wavelength normalization curve is derived, usually by comparison of the recorded intensities of the same (single) spots or symmetry-related spots at several crystal orientations, applied to each image, and the images in each data set scaled together. In the fourth stage, the intensities of spots identified in the first stage as multiple are resolved (or deconvoluted) into the intensity of each individual component or harmonic. The total intensity of a multiple Laue spot is the weighted sum of the intensities of each component, and the weights are known from the wavelength assigned to each component (Stage 1) and the wavelength normalization curve (Stage 3). In the fifth stage,

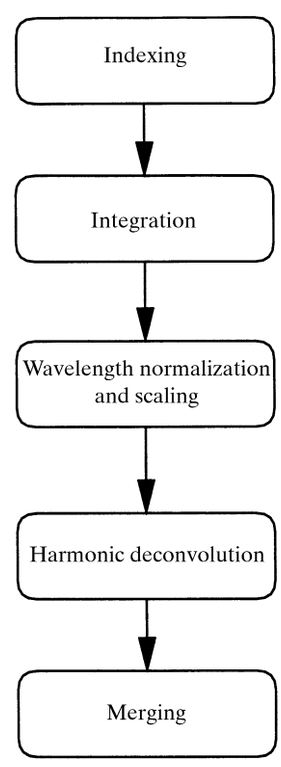

Fig. 8.2.3.1. Flow chart of typical Laue data processing.

references