5.1. Introduction

BY A. J. C. WILSON

Precise and accurate lattice parameters can be obtained by X-ray methods, both polycrystalline and single crystal. The literature on X-ray lattice-parameter determination is voluminous, involving hundreds of publications. For powder-camera methods most of it is fairly old; summaries with sufficient detail for most purposes will be found in Klug & Alexander (1974) and Peiser, Rooksby & Wilson (1960). The proceedings of the symposium on *Accuracy in Powder Diffraction* held in Gaithersburg in 1979 (Block & Hubbard, 1980) are particularly valuable as a source of information on developments of all aspects of powder diffraction up to 1979; the publication includes a review of accuracy in lattice-parameter measurements (Wilson, 1980), which contains about twice as many references as Chapter 5.2. A more recent symposium was held in Fremantle in 1987 (CSIRO, 1988); it contains several papers relevant to accuracy in lattice-parameter determination, as well as papers on other applications of powder diffractometry. References to papers published in these symposia are included in Chapter 5.2 where appropriate. A second Gaithersburg conference with the same title (Prince & Stalick, 1992) covered many aspects of powder diffraction, but not lattice-parameter determination. Unfortunately there seem to be no comparable reviews of single-crystal methods, so that Chapter 5.3 is somewhat more detailed than Chapter 5.2.

Electron- and neutron-diffraction methods are less used and are (in general) less precise than X-ray methods. They are, however, applicable to some problems for which X-ray methods are inappropriate, and are described in Chapters 5.4 and 5.5.

Whatever the radiation, perhaps the most important factor in obtaining accurate and precise lattice parameters is careful experimental technique; lack of care can produce larger errors than lack of the latest equipment. Measurements should be repeated to check the precision (reproducibility) of the determination; periodical check measurements of a standard specimen (for example, high-purity silicon) should be undertaken to check the accuracy (absence of, or satisfactory correction for, systematic errors).

The lattice parameter of silicon is discussed in Section 4.2.1. There is said to be a difference between the lattice parameters of high-quality single-crystal silicon plates and silicon powder samples. The lattice parameter of the powder is reported as 1.5×10^{-5} nm lower at room temperature (Okada & Tokumaru, 1984; see also Hubbard, 1983).

5.5. Neutron methods

By B. T. M. Willis

In general, one would not expect to measure lattice parameters as precisely with neutrons as with X-rays. The main reason for this is the need to relax the resolution of the diffraction peaks observed in neutron diffraction, in order to obtain reasonable count rates. However, the high-resolution powder diffractometer D2B (on the reactor source at the Institut Laue–Langevin) and the high-resolution powder instrument HRPD (on the pulsed source at the Rutherford Appleton Laboratory) have resolutions approaching that of X-ray diffractometers. Using Rietveld refinement, lattice parameters can be determined to a precision of a few parts in 10^4 (Fischer *et al.*, 1986).

Neutron methods are better suited to the indexing of the powder pattern. This requires the accurate measurement of the d spacings of the lowest-index lines in the pattern. Whereas d spacings measured with X-rays at low values of $(\sin\theta)/\lambda$ tend to have systematic errors, this is not such a serious problem with neutrons. It is relatively straightforward, using the time-of-flight pulsed-neutron method, to measure the d spacings of the first 20–30 lines of a powder pattern to better than 0.1%.

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5.1

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