1964; Slade *et al.*, 1964; Newman & Weissmann, 1968; Berg & Hall, 1975). A special case of strains is an extensional deformation of the lattice in the direction of crystal growth (Isherwood, 1968).

A typical metallurgical problem is the effect of heat treatment on the microstructure of alloys. An example of the application of the Kossel method to the task is given by Shinoda, Isokawa & Umeno (1969), who reported a study of precipitation of α from β in copper–zinc alloys. The lattice parameters and thermal expansion of α-iron and its alloys were examined by Lutts & Gielen (1971). Structure defects resulting from over-pressure experiments and annealing were investigated by Potts & Pearson (1966). Irradiation effects caused by neutrons were the subject of papers of Hanneman, Ogilvie & Modrzejewski (1962), Yakowitz (1972), and Spooner & Wilson (1973); those caused by electron bombardment were reported by Ullrich (1967).

Divergent-beam techniques are considered to be a suitable tool for studying strains in epitaxic layers (Hart, 1981), since corresponding lines of the layer and substrate, observed on one photograph, can be readily identified. Relevant examples are given by Brühl (1978), Chang, Patel, Nannichi & de Prince (1979), and Chang (1979), who examined lattice mismatch in LPE heterojunction systems, and by Brown, Halliwell & Isherwood (1980), and Isherwood, Brown & Halliwell (1981, 1982), who reported characterization of distortions in hetero-epitaxic structures together with a theoretical basis (multiple diffraction) for the method.

Another task of real-structure examination is the determination of angles between crystal blocks. A method has been worked out by Aristov, Shmytko & Shulakov (1974*a*,*b*).

Divergent-beam techniques can also be used in X-ray topographic studies, realized either by means of Kossel-line scanning (Rozhansky, Lider & Lyutzau, 1966) or by line-profile analysis (Glass & Weissmann, 1969).

Schetelich & Geist (1993) used the Kossel method for lattice-parameter determination and a qualitative estimation of the crystal perfection of *quasicrystals* and showed that the fine structure of Kossel lines of quasicrystals is the same as observed for conventional crystals.

Mendelssohn & Milledge (1999) used a Dingley–Kossel camera for quick and simple computer-aided measurements of cell parameters of isotopically distinct samples of LiF over a wide temperature range of 15–375 K.

5.3.3. Methods with counter recording

5.3.3.1. *Introduction*

Although, theoretically, the limit of accuracy in all methods based on the Bragg law [equation (5.3.1.1)] is given by the accuracy of the wavelength measurement ($\delta\lambda/\lambda \sim 10^{-6}$), with photographic recording this limit is not attained. Surprisingly high accuracy may be offered by accurately applied Kossel or divergent-beam techniques. In practice, however, even in this case the accuracy achieved is poorer by an order of magnitude.

The use of Geiger–Müller, proportional, or scintillation counters together with a step-scanning motor makes it possible to record the diffraction profile in a quantitative numerical form convenient for data processing, to locate it with better accuracy and precision and, as a consequence, to obtain better accuracy and precision for the Bragg angle and thus for the lattice parameter. To make the most of this possibility, theoretical papers concerning methods of peak location, estimation of systematic and statistical errors, and optimization of the measurement were developed in parallel with constructional and experimental methods.

Methods of lattice-parameter determination with counter recording form a large and heterogeneous group. As well as measurements on two- or four-circle standard diffractometers, a separate method developed by Bond (1960) and a variety of non-dispersive (X-ray and optical interferometry) and pseudo-non-dispersive methods (two- and three-circle spectrometers, multiple-beam techniques, and combined methods) are included in this group.

5.3.3.2. *Standard diffractometers*

The determination of lattice parameters by the use of a standard diffractometer is based, as in the case of photographic methods, on (5.3.1.1) and (5.3.1.2), and the main task is to measure a sufficient number of reflections (the θ values for various *hkl* indices) for determining and solving the equations and for calculating the unknown parameters. The reflections can be chosen arbitrarily or in a special way (high θ angle, axial or non-axial reflections).

The characteristic feature of measurements performed on a diffractometer is, however, that to satisfy the Ewald condition for a given reflection the crystal and the detector are rotated or, depending on the geometry (equatorial or inclination), shifted round their axes as well. Basic and more detailed information about the geometry of diffractometers is given elsewhere (Arndt & Willis, 1966, Chap. 3; Stout & Jensen, 1968, Section 6.3; Kheiker, 1973, Chap. 4; Luger, 1980, Chap. 4; Section 2.2.6 of this volume). For calculating the *setting angles* for given *hkl* reflections, the lattice parameters (at least preliminary values) have to be known, and conversely, if the setting angles are known, it is possible to calculate or to refine lattice parameters. Therefore, not only the θ values (given by the angle 2θ of rotation of the detector about the goniometer axis) but also the values of the remaining setting angles (*i.e.* ω, φ, and χ of the crystal rotation in equatorial geometry, or μ and φ for the crystal and ν for the detector in inclination geometry) can be used for lattice-parameter determination. This problem can be treated by a matrix analysis.

5.3.3.2.1. *Four-circle diffractometer*

In the case of an automated four-circle (equatorial geometry) diffractometer, the setting angles are calculated by means of the orientation matrix $\boldsymbol{U}$, *i.e.* a matrix such that

$$\boldsymbol{A}^* = \boldsymbol{U}\boldsymbol{A}_G, \tag{5.3.3.1}$$

where

$$\boldsymbol{A}^* = \begin{bmatrix} a^* \\ b^* \\ c^* \end{bmatrix} \tag{5.3.3.1a}$$

is the reciprocal-axis system with metric

$$\boldsymbol{G}^{-1} = \begin{bmatrix} a^{*2} & a^*b^*\cos\gamma^* & a^*c^*\cos\beta^* \\ a^*b^*\cos\gamma^* & b^{*2} & b^*c^*\cos\alpha^* \\ a^*c^*\cos\beta^* & b^*c^*\cos\alpha^* & c^{*2} \end{bmatrix} \tag{5.3.3.1b}$$

and

$$\boldsymbol{A}_G = \begin{bmatrix} a_G \\ b_G \\ c_G \end{bmatrix} \tag{5.3.3.1c}$$

is the crystal-fixed orthonormal system. As can be proved (Busing & Levy, 1967; Hamilton, 1974; Luger, 1980, Section

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