International Tables for Crystallography (2018). Vol. H, Section 2.5.2.1.1, pp. 119–120.

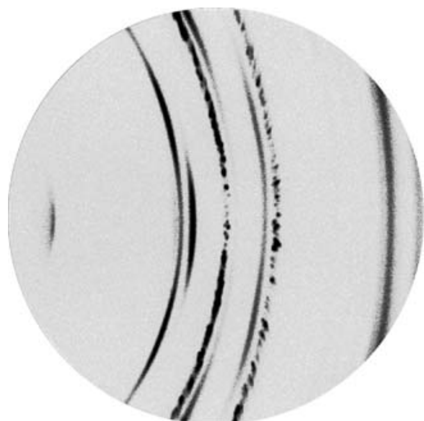

Figure 2.5.2
Diffraction pattern from a battery component containing multiple layers.

1996; Rodriguez-Navarro, 2006). The integrated data give better intensity and statistics for phase ID and quantitative analysis, especially for those samples with texture or large grain sizes, or where the sample is small. Then the integrated diffraction profiles can be analysed with existing algorithms and methods: profile fitting with conventional peak shapes and fundamental parameters, quantification of phases, and lattice-parameter indexing and refinement. The results can be used to search and match to entries in a powder-diffraction database, typically the Powder Diffraction File.

Texture measurement with 2D-XRD is extremely fast compared to measurement using a point or linear detector. The area detector collects texture data and background values simultaneously for multiple poles and multiple directions. Owing to the high measurement speed, pole figures can be measured at very fine steps, allowing detection of very sharp textures (Smith & Ortega, 1993; Bunge & Klein, 1996; He, 2009).

Stress measurement with 2D-XRD is based on a direct relationship between the stress tensor and distortion of the diffraction cones. Since the whole or a part of the diffraction ring is used for stress calculation, 2D-XRD can measure stress with high sensitivity, high speed and high accuracy (He & Smith, 1997; He, 2000). It is highly suitable for samples containing large crystals and textures. Simultaneous measurement of stress and texture is also possible, since 2D data contain both stress and texture information.

Concentrations of crystalline phases can be measured faster and more accurately with data analysis over 2D frames, especially for samples with an anisotropic distribution of crystallite orientations and/or amorphous content. The amorphous region can be defined by the user to consist of regions with no Bragg peaks, or the amorphous region can be defined with the crystalline region included when the crystalline region and the amorphous region overlap.

Microdiffraction data are collected with speed and accuracy. Collection of X-ray diffraction data from small sample amounts or small sample areas has always been a slow process because of limited beam intensity. The 2D detector captures whole or a large portion of the diffraction rings, so spotty, textured or weak diffraction data can be integrated over the selected diffraction rings (Winter & Squires, 1995; Bergese *et al.*, 2001; Tissot, 2003; Bhuvanesh & Reibenspies, 2003; He, 2004). The point beam used for microdiffraction allows diffraction mapping with fine space resolution, even on a curved surface (Allahkarami & Hanan, 2011).

Data can be collected from thin-film samples containing a mixture of single-crystal and polycrystalline layers with random orientation distributions, and highly textured layers, with all the features appearing simultaneously in diffraction frames (Dickerson *et al.*, 2002; He, 2006). The pole figures from different layers and the substrate can be overlapped to reveal the orientation relationships. The use of a 2D detector can dramatically speed up the data collection for reciprocal-space mapping on an in-plane reciprocal-lattice point (Schmidbauer *et al.*, 2008).

Because of the penetrating power of the X-ray beam, fast nondestructive data collection and the abundant information about atomic structure, two-dimensional X-ray diffraction can be used to screen a library of materials with high speed and high accuracy. Two-dimensional X-ray diffraction systems dedicated for combinatorial screening are widely used in the pharmaceutical industry for drug discovery and process analysis (Klein *et al.*, 1998; He *et al.*, 2001).

Forensic science and archaeology have benefited from using two-dimensional X-ray diffraction for identifying materials and structures from small specimens (Kugler, 2003; Bontempi *et al.*, 2008). It is nondestructive and does not require special sample treatment, so the original evidence or sample can be preserved. Two-dimensional diffraction patterns contain abundant information and are easy to observe and explain in the courtroom.

2.5.2. Fundamentals

A conventional powder-diffraction pattern is displayed as the scattering intensity *versus* scattering angle 2θ or d-spacing. A 2D-XRD pattern contains the scattering-intensity distribution as a function of two orthogonal dimensions. One dimension can be expressed in 2θ, which can be interpreted by Bragg's law. The distribution in the dimension orthogonal to 2θ contains additional information, such as the orientation distribution, strain states, and crystallite-size and -shape distribution. In order to understand and analyse 2D diffraction data, new geometry conventions and algorithms are introduced. The geometry conventions and algorithms used for 2D-XRD should also be consistent with conventional XRD, so that many existing concepts and algorithms are still valid when 2D diffraction data are used.

The geometry of a 2D-XRD system can be explained using three distinguishable and interrelated geometry spaces, each defined by a set of parameters (He, 2003). The three geometry spaces are the diffraction space, detector space and sample space. The laboratory coordinate system $\mathbf{X}_L$, $\mathbf{Y}_L$, $\mathbf{Z}_L$ is the basis of all three spaces. Although the three spaces are interrelated, the definitions and corresponding parameters should not be confused. Except for a few parameters introduced specifically for 2D-XRD, many of these parameters are used in conventional X-ray diffraction systems. Therefore, the same definitions are maintained for consistency. The three-circle goniometer in Eulerian geometry is the most commonly used, and all the algorithms for data interpretation and analysis in this chapter are based on Eulerian geometry. The algorithms can be developed for the geometries of other types (such as kappa) by following the same strategies.

2.5.2.1. *Diffraction space and laboratory coordinates*

2.5.2.1.1. *Diffraction cones in laboratory coordinates*

Fig. 2.5.3(*a*) describes the geometric definition of diffraction cones in the laboratory coordinate system $\mathbf{X}_L$, $\mathbf{Y}_L$, $\mathbf{Z}_L$. The laboratory coordinate system is a Cartesian coordinate system.

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The plane given by X_L and Y_L is the diffractometer plane. The axis Z_L is perpendicular to the diffractometer plane. The axes X_L, Y_L and Z_L form a right-handed rectangular coordinate system with the origin at the instrument centre. The incident X-ray beam propagates along the X_L axis, which is also the rotation axis of all diffraction cones. The apex angles of the cones are determined by the 2θ values given by the Bragg equation. The apex angles are twice the 2θ values for forward reflection ($2\theta \leq 90^\circ$) and twice the value of $180^\circ - 2\theta$ for backward reflection ($2\theta > 90^\circ$). For clarity, only one diffraction cone of forward reflection is displayed. The γ angle is the azimuthal angle from the origin at the six o'clock direction with a right-handed rotation axis along the opposite direction of incident beam ($-X_L$ direction). A given γ value defines a half plane with the X_L axis as the edge; this will be referred to as the γ plane hereafter. The diffractometer plane consists of two γ planes at γ = 90° and γ = 270°. Therefore many equations developed for 2D-XRD should also apply to conventional XRD if the γ angle is given as a constant of 90° or 270°. A pair of γ and 2θ values represents the direction of a diffracted beam. The γ angle takes a value of 0 to 360° for a complete diffraction ring with a constant 2θ value. The γ and 2θ angles form a spherical coordinate system which covers all the directions from the origin of sample (instrument centre). The γ–2θ system is fixed in the laboratory system $\mathbf{X}_L$, $\mathbf{Y}_L$, $\mathbf{Z}_L$, which is independent of the sample orientation and detector position in the goniometer. 2θ and γ are referred to as the diffraction-space parameters. In the laboratory coordinate system $\mathbf{X}_L$, $\mathbf{Y}_L$, $\mathbf{Z}_L$, the surface of a diffraction cone can be mathematically expressed as

$$y_L^2 + z_L^2 = x_L^2 \tan^2 2\theta, \tag{2.5.1}$$

with $x_L \geq 0$ or $2\theta \leq 90^\circ$ for forward-diffraction cones and $x_L < 0$ or $2\theta > 90^\circ$ for backward-diffraction cones.

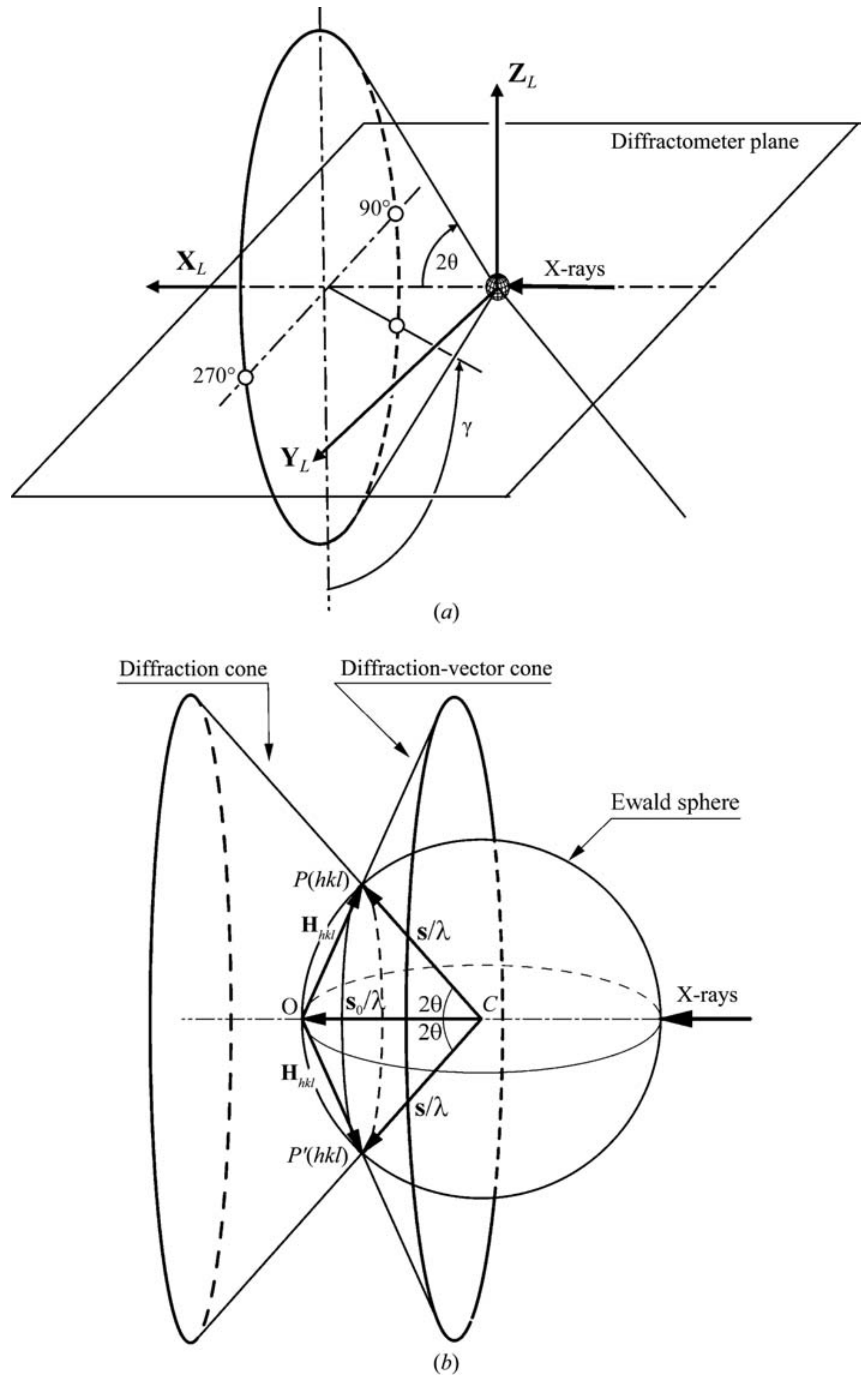

Figure 2.5.3
The diffraction cone and the corresponding diffraction-vector cone.

2.5.2.1.2. *Diffraction-vector cones in laboratory coordinates*

Fig. 2.5.3(*b*) shows the diffraction-vector cone corresponding to the diffraction cone in the laboratory coordinate system. *C* is the centre of the Ewald sphere. The diffraction condition can be given by the Laue equation as

$$\frac{\mathbf{s} - \mathbf{s}_0}{\lambda} = \mathbf{H}_{hkl}, \tag{2.5.2}$$

where $\mathbf{s}_0$ is the unit vector representing the incident beam, $\mathbf{s}$ is the unit vector representing the diffracted beam and $\mathbf{H}_{hkl}$ is the reciprocal-lattice vector. Its magnitude is given as

$$\left|\frac{\mathbf{s} - \mathbf{s}_0}{\lambda}\right| = \frac{2\sin\theta}{\lambda} = \left|\mathbf{H}_{hkl}\right| = \frac{1}{d_{hkl}}, \tag{2.5.3}$$

in which d_{hkl} is the *d*-spacing of the crystal planes (*hkl*). It can be easily seen that it is the Bragg law in a different form. Therefore, equation (2.5.2) is the Bragg law in vector form. In the Bragg condition, the vectors $\mathbf{s}_0/\lambda$ and $\mathbf{s}/\lambda$ make angles θ with the diffracting planes (*hkl*) and $\mathbf{H}_{hkl}$ is normal to the (*hkl*) crystal plane. In order to analyse all the X-rays measured by a 2D detector, we extend the concept to all scattered X-rays from a sample regardless of the Bragg condition. Therefore, the index (*hkl*) can be removed from the above expression. $\mathbf{H}$ is then a vector which takes the direction bisecting the incident beam and the scattered beam, and has dimensions of inverse length given by $2\sin\theta/\lambda$. Here 2θ is the scattering angle from the incident beam. The vector $\mathbf{H}$ is referred to as the scattering vector or, alternatively, the diffraction vector. When the Bragg condition is