2.5. Two-dimensional powder diffraction

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2.5.1. Introduction

2.5.1.1. *The diffraction pattern measured by an area detector*

The diffracted X-rays from a polycrystalline or powder sample form a series of cones in three-dimensional space, since large numbers of crystals oriented randomly in the space are covered by the incident X-ray beam. Each diffraction cone corresponds to the diffraction from the same family of crystal planes in all the participating grains. The apex angles of cones are given by Bragg's law for the corresponding crystal interplanar *d*-spacing. A conventional X-ray powder-diffraction pattern is collected by scanning a point or linear detector along the 2θ angle. The diffraction pattern is displayed as scattering intensity *versus* 2θ angle (Klug & Alexander, 1974; Cullity, 1978; Warren, 1990; Jenkins & Snyder, 1996; Pecharsky & Zavalij, 2003). In recent years, use of two-dimensional (2D) detectors for powder diffraction has dramatically increased in academic and industrial research (Sulyanov *et al.*, 1994; Rudolf & Landes, 1994; He, 2003, 2009). When a 2D detector is used for X-ray powder diffraction, the diffraction cones are intercepted by the area detector and the X-ray intensity distribution on the sensing area is converted to an image-like diffraction pattern, also referred to as a frame. Since the diffraction pattern collected with a 2D detector is typically given as an intensity distribution over a two-dimensional region, so X-ray diffraction with a 2D detector is also referred to as two-dimensional X-ray diffraction (2D-XRD) or 2D powder diffraction. A 2D diffraction pattern contains far more information than a conventional diffraction pattern, and therefore demands a special data-collection strategy and data-evaluation algorithms. This chapter covers the basic concepts and recent progress in 2D-XRD theory and technologies, including geometry conventions, X-ray source and optics, 2D detectors, diffraction-data interpretation, and various applications, such as phase identification and texture, stress, crystallinity and crystallite-size analysis. The concepts and algorithms of this chapter apply to both laboratory and synchrotron diffractometers equipped with 2D detectors.

2.5.1.2. *Comparison between 2D-XRD and conventional XRD*

Fig. 2.5.1 is a schematic of X-ray diffraction from a powder (polycrystalline) sample. For simplicity, it shows only two diffraction cones; one represents forward diffraction ($2\theta \leq 90^\circ$) and one represents backward diffraction ($2\theta > 90^\circ$). The diffraction measurement in a conventional diffractometer is confined within a plane, here referred to as the diffractometer plane. A point (0D) detector makes a 2θ scan along a detection circle. If a line (1D) detector is used in the diffractometer, it will be mounted on the detection circle. Since the variations in the diffraction pattern in the direction (**Z**) perpendicular to the diffractometer plane are not considered in a conventional diffractometer, the X-ray beam is normally extended in the *Z* direction (line focus). Since the diffraction data out of the diffractometer plane are not detected, the structures in the material that are represented by the missing diffraction data will either be ignored, or extra sample rotation and time are needed to complete the measurement.

With a 2D detector, the diffraction measurement is no longer limited to the diffractometer plane. Depending on the detector size, the distance to the sample and the detector position, the whole or a large portion of the diffraction rings can be measured simultaneously. Diffraction patterns out of the diffractometer plane have for a long time been recorded using Debye–Scherrer cameras, so the diffraction rings are referred to as Debye rings. However, when a Debye–Scherrer camera is used, only the position of the arches in the 2θ direction and their relative intensities are measured for powder-diffraction analysis. The diffraction rings collected with a large 2D detector extend further in the 'vertical' direction and the intensity variation in the vertical direction is also used for data evaluation. Therefore, the terms 'diffraction cone' and 'diffraction ring' will be often be used in this chapter as alternatives to 'Debye cone' and 'Debye ring'.

2.5.1.3. *Advantages of two-dimensional X-ray diffraction*

A 2D diffraction frame contains far more information than a diffraction pattern measured using a conventional diffraction system with a point detector or a linear position-sensitive detector. In addition to the significantly higher data-collection speed, the intensity and 2θ variation along the diffraction rings can reveal abundant structural information typically not available from a conventional diffraction pattern. Fig. 2.5.2 shows a 2D pattern collected from a battery component containing multiple layers of different phases. Some diffraction rings have strong intensity variation due to preferred orientation, and the spotty diffraction rings are from a phase that contains large crystal grains. It is apparent that different diffraction-ring patterns are from different phases. 2D-XRD analyses commonly performed on polycrystalline materials include phase identification, quantitative phase analysis, preferred-orientation quantification and characterization of residual stresses.

Phase identification (phase ID) can be done by integration in a selected 2θ range along the diffraction rings (Hammersley *et al.*,

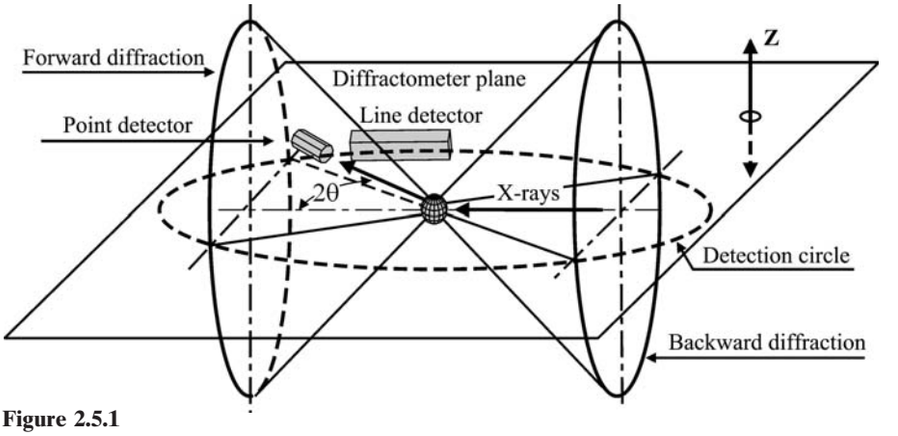

Figure 2.5.1
Diffraction patterns in 3D space from a powder sample and the diffractometer plane.

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