

## 2. INSTRUMENTATION AND SAMPLE PREPARATION

If the texture has a rotational symmetry with respect to an axis of the sample, the texture is referred to as a fibre texture and the axis is referred to as the fibre axis. The sample orientation containing the symmetry axis is referred to as the fibre axis. The fibre texture is mostly observed in two types of materials: metal wires or rods formed by drawing or extrusion, and thin films formed by physical or chemical deposition. The fibre axis is the wire axis for a wire and normal to the sample surface for thin films. Fibre texture can also be artificially formed by rotating a sample about its normal. If the fibre axis is aligned to the  $S_3$  direction, the pole-density distribution function becomes independent of the azimuthal angle  $\beta$ . For samples with fibre texture, or artificially formed fibre texture by rotating, the pole-density function is conveniently expressed as a function of a single variable,  $g_{hkl}(\chi)$ . Here,  $\chi$  is the angle between the sample normal and pole direction.

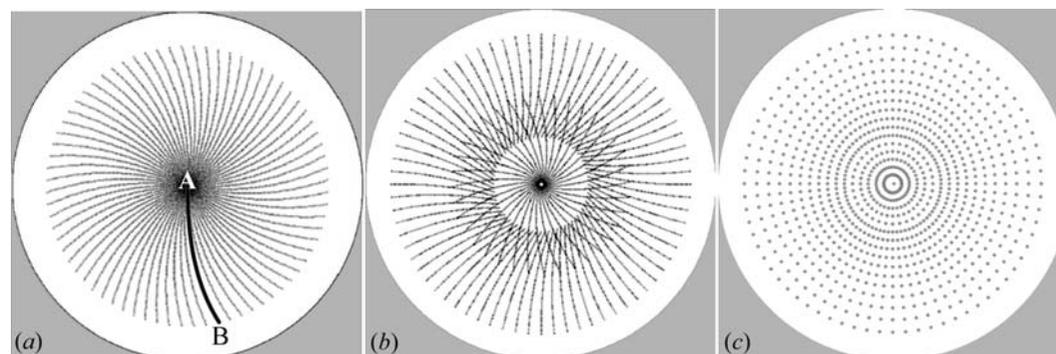
$$\chi = 90^\circ - \alpha \text{ or } \chi = \cos^{-1}|h_3|. \quad (2.5.59)$$

The pole-density function for fibre texture can be expressed as a fibre plot. The fibre plot  $g_{hkl}(\chi)$  can be calculated from the relative intensity of several peaks (He, 1992; He *et al.*, 1994) and artificial fibre texture can be achieved by sample spinning during data collection.

## 2.5.4.2.3. Data-collection strategy

Since a one-dimensional pole-density mapping is created from each 2D frame, it is important to lay out a data-collection strategy so as to have the optimum pole-figure coverage and minimum redundancy in data collection. The pole-figure coverage can be simulated from the diffraction  $2\theta$  angle, detector swing angle, detector distance, goniometer angles and scanning steps. When a large 2D detector is placed close to the sample, it is possible to collect a pole figure with a single  $\varphi$  scan. Fig. 2.5.20(a) shows an example of a scheme generated by a single  $\varphi$  scan of  $5^\circ$  steps with a detector 10.5 cm in diameter and  $D = 7$  cm. The data collected with a single exposure at  $\varphi = 0^\circ$  would generate a one-dimensional pole figure as shown in the curve marked by A and B. The pole figure can be generated by a full-circle rotation of  $360^\circ$ . The pole density at the centre represents the diffraction vector perpendicular to the sample surface. It is important to have the pole-density information in the centre region of the pole figure, especially for fibre texture. The pole-figure angle at the centre is  $\alpha = 90^\circ$ , and the best strategy is to put point A at the centre of pole figure. That is

$$h_3^A = \sin \theta \cos \psi \sin \omega - \cos \theta \sin \gamma_A \cos \psi \cos \omega - \cos \theta \cos \gamma_A \sin \psi = 1. \quad (2.5.60)$$



**Figure 2.5.20**  
Data-collection strategy: (a) 2D detector with  $D = 7$  cm; (b) 2D detector with  $D = 10$  cm; (c) point detector.

In some cases, a single  $\varphi$  scan is not enough to cover sufficient pole-figure angles because of a large detector distance or limited detector area, so it is necessary to collect a set of data with  $\varphi$  scans at several different sample tilt angles. Fig. 2.5.20(b) illustrates the data-collection scheme with a detector that is 10.5 cm in diameter and  $D = 10$  cm for the (111) plane of a Cu thin film. In this case, each pole figure requires two  $\varphi$  scans at different sample orientations. The data-collection strategy should also be optimized for several crystallographic planes if all can be covered in a frame. The step size of the data-collection scan depends highly on the strength of the texture and the purpose of the texture measurements. For a weak texture, or quality control for metal parts,  $\varphi$  (or  $\omega$ , or  $\psi$ ) scan steps of  $5^\circ$  may be sufficient. For strong textures, such as thin films with epitaxial structure, scan steps of  $1^\circ$  or smaller may be necessary.

The effectiveness of two-dimensional data collection for a texture can be compared with that using a point detector with the data-collection strategy of the Cu thin film as an example. Fig. 2.5.20(c) shows the pole-figure data-collection strategy with a point detector. For the same pole-figure resolution, significantly more exposures are required with a point detector. Considering that several diffraction rings are measured simultaneously with a 2D detector, the pole-figure measurement is typically 10 to 100 times faster than with a point detector. Therefore, quantitative high-resolution pole-figure measurements are only practical with a 2D-XRD system (Bunge & Klein, 1996).

## 2.5.4.2.4. Texture-data processing

For a specific diffraction ring,  $2\theta$  is a constant or at least assumed to be constant for texture analysis, and the sample-orientation angles ( $\omega$ ,  $\psi$ ,  $\varphi$ ) for a frame are also constants. Therefore, the pole-density information is given by the diffraction-intensity distribution as a function of  $\gamma$  only, or  $I = I(\gamma)$ . Integration of the diffraction intensities in the  $2\theta$  direction converts 2D information into the function  $I(\gamma)$ .

Fig. 2.5.21(a) shows a 2D diffraction ring for texture analysis. The low and high background and diffraction-ring  $2\theta$ - $\gamma$  range are defined by three boxes, noted as  $B_L$ ,  $B_H$  and  $I(\gamma)$ , respectively. All three boxes have the same  $\gamma$  range from  $\gamma_1$  to  $\gamma_2$ . The  $2\theta$  ranges for the diffraction ring, low background and high background should be determined based on the width of the  $2\theta$  peak and available background between adjacent peaks. Assuming a normal distribution, a  $2\theta$  range of 2 times the FWHM covers 98% of the intensity peak, and 3 times the FWHM covers more than 99.9%. The  $2\theta$  range should also be broad enough to cover the possible  $2\theta$  shifts caused by residual stresses in the sample. Fig. 2.5.21(b) is the  $2\theta$  profile integrated over the section  $\Delta\gamma$  in

Fig. 2.5.21(a). The background ranges on the low and high  $2\theta$  sides are given by  $2\theta_{L1}$ - $2\theta_{L2}$  and  $2\theta_{H1}$ - $2\theta_{H2}$ , respectively. The  $2\theta$ -integrated diffraction intensities as a function of  $\gamma$  are plotted in Fig. 2.5.21(c). The background can be calculated and removed from the intensity values of the low and high backgrounds or ignored if the contribution of the background is very small.

$2\theta$  integration without a background correction can be