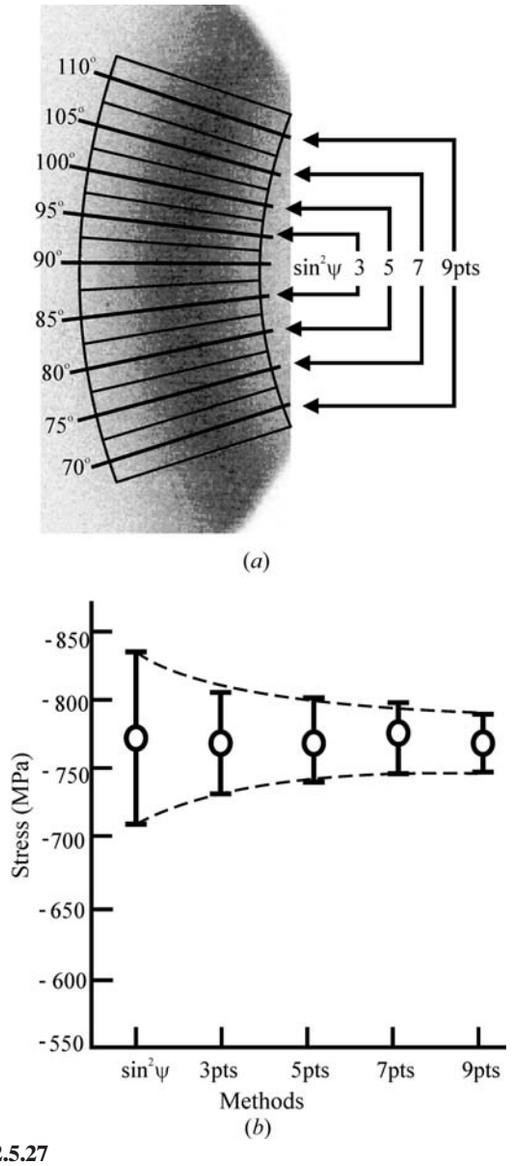

Figure 2.5.27
Stress calculation with the 2D method and the $\sin^2\psi$ method: (*a*) nine data points (abbreviated as pts) on the diffraction ring; (*b*) measured stress and standard deviation by different methods.

dimensional diffraction system, more crystallites can contribute to the diffraction because of the larger γ range.

An example of a stress calculation is provided by the measurement of the residual stress on the end surface of a carbon steel roller. One of the seven frames taken with an ω scan is shown in Fig. 2.5.27(*a*). The (211) ring covering the γ range 67.5 to 112.5° was used for stress analysis. First, the frame data were integrated along γ with an interval of $\Delta\gamma = 5°$. A total of nine diffraction profiles were obtained from γ integration. The peak position 2θ for each γ angle was then obtained by fitting the profile with a Pearson-VII function. A total of 63 data points can be obtained from the seven frames. The data points at $\gamma = 90°$ from seven frames, a typical data set for an ω diffractometer, were used to calculate the stress with the conventional $\sin^2\psi$ method. In order to compare the gain from having increased data points with the 2D method, the stress was calculated from 3, 5, 7 and 9 data points on each frame. The results from the conventional $\sin^2\psi$ method and the 2D method are compared in Fig. 2.5.27(*b*). The measured residual stress is compressive and the stress values from different methods agree very well. With the data taken from the same measurement (seven frames), the 2D method gives a lower standard error and the error decreases with increasing number of data points from the diffraction ring.

2.5.4.4. *Quantitative analysis*

2.5.4.4.1. *Crystallinity*

The crystallinity of a material influences many of its characteristics, including mechanical strength, opacity and thermal properties. Crystallinity measurement provides valuable information for both materials research and quality control in materials processing. The diffraction pattern from a material containing both amorphous and crystalline solids has a broad feature from the amorphous phase and sharp peaks from the crystalline phase. The weight percentage of the crystalline phases in a material containing both crystalline and amorphous phases can be determined by X-ray diffraction (Chung & Scott, 1973; Alexander, 1985; Murthy & Barton, 2000; Kasai & Kakudo, 2005). Assuming that the X-ray scattering intensity from each phase in such a material is proportional to its weight percentage, and that the scattering intensities from all phases can be measured within a given 2θ range, the per cent crystallinity is given by

$$x_{\rm pc} = 100\% \frac{I_{\rm crystal}}{I_{\rm crystal} + I_{\rm amorphous}}, \qquad (2.5.90)$$

where $x_{\rm pc}$ is the per cent crystallinity, $I_{\rm crystal}$ is the integrated intensity of all crystalline peaks and $I_{\rm amorphous}$ is the integrated intensity of the amorphous scattering. The accuracy of the measured per cent crystallinity depends on the integrated diffraction profile. Since most crystalline samples have a preferred orientation, it is very difficult to obtain a consistent measurement of crystallinity with a conventional diffractometer. Fig. 2.5.28 shows a 2D diffraction frame collected from an oriented polycrystalline sample. The diffraction is in transmission mode with the X-ray beam perpendicular to the plate sample surface. Fig. 2.5.28(*a*) shows a diffraction profile integrated from a horizontal region analogous to a profile collected with a conventional diffractometer. Only one peak from the crystalline phase can be observed in the profile. It is also possible that a different peak or no peak is measured if the sample is loaded in other orientations. Fig. 2.5.28(*b*) is the diffraction profile integrated from the region covering all peaks from the crystalline phase over almost all azimuthal angles. A total of four peaks from the crystalline phase are observed. This shows that a 2D-XRD system can measure per cent crystallinity more accurately and with more consistent results (Pople *et al.*, 1997; Bruker, 2000) than a conventional system.

2.5.4.4.2. *Crystallite size*

The size of the crystallites in a polycrystalline material has a significant effect on many of its properties, such as its thermal, mechanical, electrical, magnetic and chemical properties. X-ray diffraction has been used for crystallite-size measurement for many years. Most methods are based on diffraction-line broadening and line-profile analysis (Wilson, 1971; Klug & Alexander, 1974; Ungár, 2000). Another approach to crystallite-size measurement is based on the spotty diffraction rings collected with two-dimensional detectors when a small X-ray beam is used (Cullity, 1978; He, 2009). Line-profile analysis is based on the diffraction profile in the 2θ direction, while crystallite-size analysis with a spotty 2D diffraction pattern is based on the diffraction profile in the γ direction. The latter may be referred to as γ-profile analysis.