determine the stress-free d-spacing d_o, the instrument should be first calibrated with a stress-free standard of a similar material.

2.5.4.3.4. *Data-collection strategy*

The practice of stress analysis with 2D-XRD involves the selection of the diffraction-system configuration and the data-collection strategy, frame correction and integration, and stress calculation from the processed data points. Most concepts and strategies developed for a conventional diffractometer are still valid for 2D-XRD. We will focus on the new concepts and practices due to the nature of the 2D detectors.

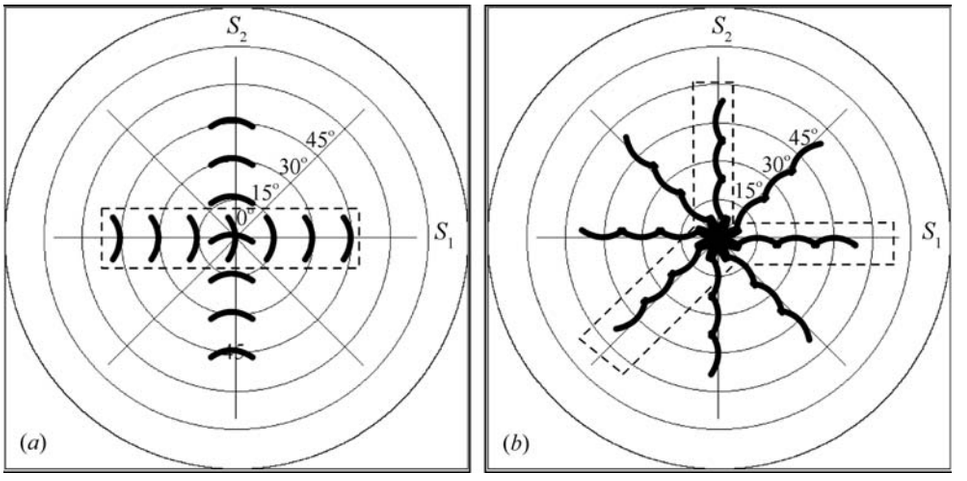

Figure 2.5.25
Data-collection strategy schemes: (*a*) $\omega + \varphi$ scan; (*b*) $\psi + \varphi$ scan.

The diffraction vector is in the normal direction to the measured crystalline planes. It is not always possible to have the diffraction vector in the desired measurement direction. In reflection mode, it is easy to have the diffraction vector normal to the sample surface, or tilted away from the normal, but impossible to have the vector on the surface plane. The stress on the surface plane, or biaxial stress, is calculated by elasticity theory from the measured strain in other directions. The final stress-measurement results can be considered as an extrapolation from the measured values. In the conventional $\sin^2\psi$ method, several ψ-tilt angles are required, typically at 15° steps from −45° to +45°. The same is true with a 2D-XRD system. The diffraction vectors corresponding to the data scan can be projected onto a 2D plot in the same way as the pole-density distribution in a pole figure. The 2D plot is called a data-collection strategy scheme.

By evaluating the scheme, one can generate a data-collection strategy suitable for the measurement of the intended stress components. Fig. 2.5.25 illustrates two schemes for data collection. In the bisecting condition ($\omega = \theta$ or $\theta_1 = \theta$ and $\psi = 0°$), the trace of the diffraction vector falls in the vicinity of the scheme centre. Either an ω tilt or a ψ tilt can move the vectors away from the centre. The circles on the scheme are labelled with the tilt angle of 15°, 30° and 45°. Scheme (*a*) is for an ω tilt of 0°, ±15°, ±30° and ±45° with the φ angle at 0° and 90°. It is obvious that this set of data would be suitable for calculating the biaxial-stress tensor. The data set with $\varphi = 0°$, as shown within the box enclosed by the dashed lines, would be sufficient on its own to calculate σ_{11}. Since the diffraction-ring distortion at $\varphi = 0°$ or $\varphi = 90°$ is not sensitive to the stress component σ_{12}, strategy (*a*) is suitable for the equibiaxial stress state, but is not able to determine σ_{12} accurately. In scheme (*b*), the ψ scan covers 0° to 45° with 15° steps at eight φ angles with 45° intervals. This scheme produces comprehensive coverage on the scheme chart in a symmetric distribution. The data set collected with this strategy can be used to calculate the complete biaxial-stress tensor components and shear stress (σ_{11}, σ_{12}, σ_{22}, σ_{13}, σ_{23}). The scheme indicated by the boxes enclosed by the dashed lines is a time-saving alternative to scheme (*b*). The rings on two φ angles are aligned to S_1 and S_2 and the rings on the third φ angle make 135° angles to the other two arrays of rings. This is analogous to the configuration of a stress-gauge rosette. The three φ angles can also be separated equally by 120° steps. Suitable schemes for a particular experiment should be determined by considering the stress components of interest, the goniometer, the sample size, the detector size and resolution, the desired measurement accuracy and the data-collection time.

2.5.4.3.5. *Data integration and peak evaluation*

The purpose of data integration and peak evaluation is to generate a set of data points along distorted diffraction rings. Data integration for stress analysis is γ integration over several defined segments so as to generate diffraction profiles representing the corresponding segments. The peak position can be determined by fitting the diffraction profile to a given analytic function. Fig. 2.5.26 illustrates data integration over a diffraction frame. The total integration region is defined by $2\theta_1$, $2\theta_2$, γ_1 and γ_2. The integration region is divided into segments given by $\Delta\gamma$. One data point on the distorted diffraction ring is generated from each segment. The γ value in the centre (denoted by the dot-dashed line) of each segment is taken as the γ value of the data point. γ integration of the segment produces a diffraction profile and the 2θ value is determined from the profile. The number of segments and the segment size ($\Delta\gamma$) are selected based on the quality of the data frame. The larger the segment size $\Delta\gamma$ is, the better the integrated diffraction profile as more counts are being integrated. γ integration also produces a smearing effect on the diffraction-ring distortion because the counts collected within the segment size $\Delta\gamma$ are considered as a single γ value at the segment

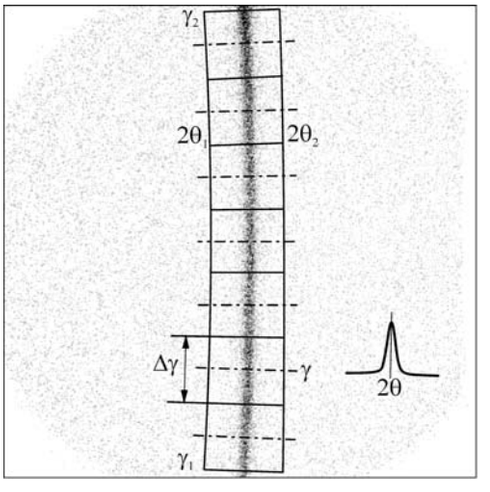

Figure 2.5.26
Data integration for stress measurement.

centre. The 2θ shift in the segment is averaged. The segment size $\Delta\gamma$ should be sufficient to produce a smooth diffraction profile, but not so large as to introduce too much smearing. For data frames containing high pixel counts, the integration segment can be small, *e.g.* $\Delta\gamma \leq 2^\circ$, and still have a smooth profile for each segment. For data frames having low pixel counts, for example the frames collected from a micron-sized area, from a sample with large grains or with a short data-collection time, it is critical to choose a sufficiently large segment size. The segment size can be determined by observing the smoothness of the integrated profile.

Peak evaluation in each segment can be done using the same algorithm used in the conventional method. The corrections to the integrated profiles are performed before or during the peak evaluation. Absorption correction eliminates the influence of the irradiated area and the diffraction geometry on the measured intensity distribution. The absorption for a given material and radiation level depends on the incident angle to the sample and the reflected angle from the sample. For 2D-XRD, the reflected angle is a function of γ for each frame. The polarization effect is also a function of γ. Therefore, the correction for polarization and absorption should be applied to the frame before integration. (Details of these corrections were discussed in Section 2.5.4.3.4.) The polarization and absorption correction is not always necessary if the error caused by absorption can be tolerated for the application, or if the data-collection strategy involves only ψ and φ scans.

In most cases, $K\alpha$ radiation is used for stress measurement, in which case the weighted average wavelength of $K\alpha_1$ and $K\alpha_2$ radiation is used in the calculations. For samples with a broad peak width, diffraction of $K\alpha_1$ and $K\alpha_2$ radiation is merged together as a single peak profile, and the profile can be evaluated as if there is a single $K\alpha$ line without introducing much error to the measured d-spacing. For samples with a relatively narrow peak width, the diffraction profile shows strong asymmetry or may even reveal two peaks corresponding to the $K\alpha_1$ and $K\alpha_2$ lines, especially at high 2θ angles. In this case the profile fitting should include contributions from both the $K\alpha_1$ and $K\alpha_2$ lines. It is common practice to use the peak position from the $K\alpha_1$ line and the $K\alpha_1$ wavelength to calculate the d-spacing after $K\alpha_2$ stripping.

Background correction is necessary if there is a strong background or the peak-evaluation algorithms are sensitive to the background, such as in $K\alpha_2$ stripping, peak fitting, and peak-intensity and integrated-intensity evaluations. Background correction is performed by subtracting a linear intensity distribution based on the background intensities at the lower 2θ side and the higher 2θ side of the diffraction peak. The background region should be sufficiently far from the 2θ peak so that the correction will not truncate the diffraction profile. The 2θ ranges of the low background and high background should be determined based on the width of the 2θ peak and available background in the profile. Based on a normal distribution, a 2θ range of 2 times the FWHM covers 98% of the peak intensity, and 3 times the FWHM covers more than 99.9%, so the background intensity should be determined at more than 1 to 1.5 times the FWHM away from the peak position. The background correction can be neglected for a profile with a low background or if the error caused by the background is tolerable for the application. The peak position can be evaluated by various methods, such as gravity, sliding gravity, and profile fitting by parabolic, pseudo-Voigt or Pearson-VII functions (Lu, 1996; Sprauel & Michaud, 2002).

2.5.4.3.6. *Stress calculation*

The final data set after integration and peak evaluation should contain many data points describing the diffraction-ring shape for all collected frames. Each measured data point contains three goniometer angles (ω, ψ, φ) and the diffraction-ring position (γ, 2θ). The peak intensity or integrated intensity of the diffraction profile is another value to be determined and may be used in the stress calculation. In most cases the number of data points is more than the number of unknown stress components, so a linear least-squares method can be used to calculate the stresses. In a general least-squares regression, the residual for the ith data point is defined as

$$r_i = y_i - \hat{y}_i, \tag{2.5.86}$$

where y_i is the observed response value, $\hat{y}_i$ is the fitted response value and r_i is the residual, which is defined as the difference between the observed value and the fitted value. The summed square of residuals is given by

$$S = \sum_{i=1}^{n} r_i^2 = \sum_{i=1}^{n} (y_i - \hat{y}_i)^2, \tag{2.5.87}$$

where n is the number of data points and S is the sum-of-squares error to be minimized in the least-squares regression. For stress calculation, the observed response value is the measured strain at each data point,

$$y_i = \ln\left(\frac{\sin\theta_0}{\sin\theta_i}\right), \tag{2.5.88}$$

and the fitted response value is given by the fundamental equation as

$$\hat{y}_i = p_{11}\sigma_{11} + p_{12}\sigma_{12} + p_{22}\sigma_{22} + p_{13}\sigma_{13} + p_{23}\sigma_{23} + p_{33}\sigma_{33} + p_{\rm ph}\sigma_{\rm ph}, \tag{2.5.89}$$

where all possible stress components and stress coefficients are listed as a generalized linear equation. Since the response-value function is a linear equation of unknown stress components, the least-squares problem can be solved by a linear least-squares regression. In order to reduce the impact of texture, large grains or weak diffraction on the results of the stress determination, the standard error of profile fitting and the integrated intensity of each profile may be introduced as a weight factor for the least-squares regression (He, 2009).

2.5.4.3.7. *Comparison between the 2D method and the conventional method*

Stress measurement on a polycrystalline material by X-ray diffraction is based on the strain measurements in a single or in several sample orientations. Each measured strain is calculated from the average d-spacing of specific lattice planes $\{hkl\}$ over many crystallites (grains). A larger number of contributing crystallites gives better accuracy and sampling statistics (also referred to as particle statistics). The sampling statistics are determined by both the crystal structure and the instrumentation. The instrument window is mainly determined by the divergence of the incident X-ray beam. Lattice-plane families with high multiplicity will also effectively improve the sampling statistics. The number of contributing crystallites measured by a conventional diffractometer is limited by the sizes and divergences of the incident and diffracted beams to the point detector. In a two-