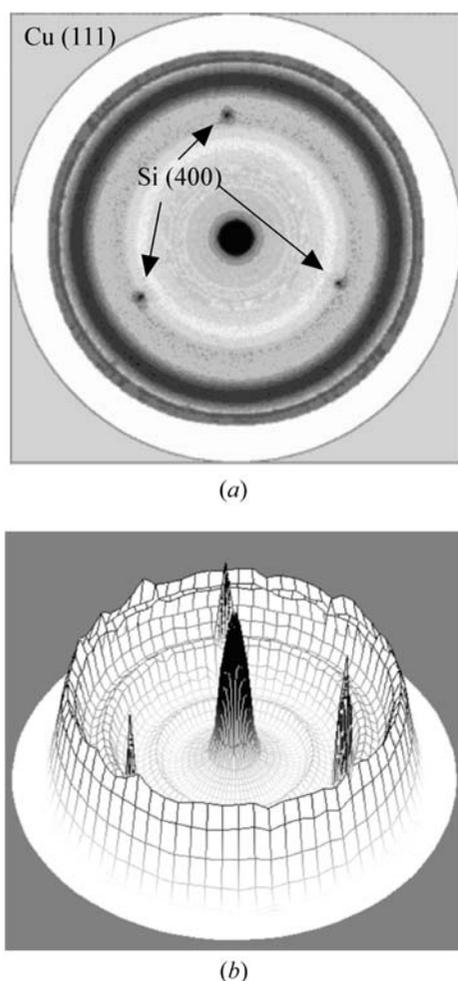


## 2. INSTRUMENTATION AND SAMPLE PREPARATION



**Figure 2.5.23**  
Combined pole figure of a Cu (111) film on an Si (400) substrate: (a) regular 2D projection; (b) 3D surface plot.

mized in the centre of the pole figure shows a strong (111) fibre texture. The orientation relationship between the film fibre axis and the substrate is clearly described by the combined pole figures. For samples containing multiple thin-film layers, the orientation relationships between the different layers of the films and substrate can be revealed by superimposing their pole figures.

### 2.5.4.3. Stress measurement

When a solid material is elastically deformed by a force, each crystallite in it changes shape or size. Assuming that the stresses in each crystallite represent the stresses in the solid, the stresses can be measured by measuring the lattice  $d$ -spacing changes in the crystallites. These  $d$ -spacing changes can be measured by the changes in diffraction-peak positions based on Bragg's law. In this case, the  $d$ -spacing serves as a gauge of the deformation. Stress measurement by X-ray diffraction is typically done using a point detector or line detector (Walter, 1971; James & Cohen, 1980; Noyan & Cohen, 1987; Lu, 1996); this will be referred to as the conventional method. The stress or stress tensor is calculated from many strain measurements from diffraction-peak  $2\theta$  shifts of a specific lattice-plane family. With a point or line detector, only a small cross section of the diffraction cone is measured at one sample orientation ( $\psi, \varphi$ ). Compared to using a conventional detector, 2D detectors have many advantages in stress measurement (Borgonovi, 1984; Korhonen *et al.*, 1989; Yoshioka & Ohya, 1992; Fujii & Kozaki, 1993; He & Smith, 1997; Kämpfe *et*

*al.*, 1999; Hanan *et al.*, 2004). Since a 2D diffraction pattern covers the whole or a large portion of the diffraction rings, it can be used to measure stress with higher accuracy and can be collected in a shorter time than a conventional diffraction pattern, especially when dealing with highly textured materials, large grain sizes, small sample areas, weak diffraction, stress mapping and stress-tensor measurement. The 2D method for stress measurement is based on the fundamental relationship between the stress tensor and the diffraction-cone distortion (He & Smith, 1997; He, 2000; European Standard, 2008).

There are two kinds of stresses, which depend on the source of the loading forces that produce them. One kind is applied stress, caused by external forces acting on the solid object. Applied stress changes when the loading forces change and it disappears once the forces are removed. The stresses measured by X-ray diffraction method are mostly residual stresses. Residual stress is caused by internal forces between different parts of a solid body. Residual stress exists without external forces or remains after the external forces have been removed. The net force and moment on a solid body in equilibrium must be zero, so the residual stresses in the body must be balanced within the body. This means that a compressive stress in one part of the body must come with a tensile stress in another part of the body. For example, the residual stress in a thin film is balanced by the stresses in the substrate. When residual stress in a solid body is mentioned it typically refers to a specific location.

Residual stresses are generally categorized as macroscopic or microscopic depending on the range over which the stresses are balanced. The macroscopic residual stress is the stress measured over a large number of grains. This kind of stress can be measured by X-ray diffraction through the shift of the Bragg peaks. The microscopic stress is the stress measured over one or a few grains, or as small a range as micro- or nanometres. This kind of stress alone will not cause a detectable shift of diffraction peaks, but is reflected in the peak profiles. In this chapter, we will focus on the X-ray diffraction method for stress measurement at the macroscopic level.

#### 2.5.4.3.1. Stress and strain relation

Stress is a measure of the deforming force applied to a solid per unit area. The stress on an elemental volume in the sample coordinates  $S_1, S_2, S_3$  contains nine components, given by

$$\sigma_{ij} = \begin{bmatrix} \sigma_{11} & \sigma_{12} & \sigma_{13} \\ \sigma_{21} & \sigma_{22} & \sigma_{23} \\ \sigma_{31} & \sigma_{32} & \sigma_{33} \end{bmatrix}. \quad (2.5.63)$$

A component is normal stress when the two indices are identical, or shear stress when the two indices differ. The group of the nine stress components is called the stress tensor. The stress tensor is a tensor of the second order. Under equilibrium conditions, the shear components must maintain the following relations:

$$\sigma_{12} = \sigma_{21}, \quad \sigma_{23} = \sigma_{32} \quad \text{and} \quad \sigma_{31} = \sigma_{13}. \quad (2.5.64)$$

Therefore, only six independent components define the stress state in a solid. The following stress states are typically measured:

Uniaxial: all stress components are zero except one normal stress component.

Biaxial: all nonzero components are within the  $S_1S_2$  plane.

Biaxial with shear:  $\sigma_{33} = 0$ , all other components are not necessarily zero.

Equibiaxial: a special case of biaxial stress where  $\sigma_{11} = \sigma_{22} = \sigma$ .

## 2.5. TWO-DIMENSIONAL POWDER DIFFRACTION

Triaxial: all components are not necessarily zero.

Equitriaxial: a special case of triaxial stress where  $\sigma_{11} = \sigma_{22} = \sigma_{33} = \sigma$ .

Strain is a measure of the resulting deformation of a solid body caused by stress. Strain is calculated from the change in the size and shape of the deformed solid due to stress. Analogous to normal stresses and shear stresses are normal strains and shear strains. The normal strain is calculated from the change in length of the solid body along the corresponding normal stress direction. Like the stress tensor, the strain tensor contains nine components:

$$\varepsilon_{ij} = \begin{bmatrix} \varepsilon_{11} & \varepsilon_{12} & \varepsilon_{13} \\ \varepsilon_{21} & \varepsilon_{22} & \varepsilon_{23} \\ \varepsilon_{31} & \varepsilon_{32} & \varepsilon_{33} \end{bmatrix}. \quad (2.5.65)$$

The directions of all strain components are defined in the same way as for the stress tensor. Similarly, there are six independent components in the strain tensor. Strictly speaking, X-ray diffraction does not measure stresses directly, but strains. The stresses are calculated from the measured strains based on the elasticity of the materials. The stress–strain relations are given by the generalized form of Hooke's law:

$$\sigma_{ij} = C_{ijkl}\varepsilon_{kl}, \quad (2.5.66)$$

where  $C_{ijkl}$  are elastic stiffness coefficients. The stress–strain relations can also be expressed as

$$\varepsilon_{ij} = S_{ijkl}\sigma_{kl}, \quad (2.5.67)$$

where  $S_{ijkl}$  are the elastic compliances. For most polycrystalline materials without texture or with weak texture, it is practical and reasonable to consider the elastic behaviour to be isotropic and the structure to be homogeneous on a macroscopic scale. In these cases, the stress–strain relationship takes a much simpler form. Therefore, the Young's modulus  $E$  and Poisson's ratio  $\nu$  are sufficient to describe the stress and strain relations for homogeneous isotropic materials:

$$\begin{aligned} \varepsilon_{11} &= \frac{1}{E}[\sigma_{11} - \nu(\sigma_{22} + \sigma_{33})], \\ \varepsilon_{22} &= \frac{1}{E}[\sigma_{22} - \nu(\sigma_{33} + \sigma_{11})], \\ \varepsilon_{33} &= \frac{1}{E}[\sigma_{33} - \nu(\sigma_{11} + \sigma_{22})], \\ \varepsilon_{12} &= \frac{1+\nu}{E}\sigma_{12}, \quad \varepsilon_{23} = \frac{1+\nu}{E}\sigma_{23}, \quad \varepsilon_{31} = \frac{1+\nu}{E}\sigma_{31}. \end{aligned} \quad (2.5.68)$$

It is customary in the field of stress measurement by X-ray diffraction to use another set of macroscopic elastic constants,  $S_1$  and  $\frac{1}{2}S_2$ , which are given by

$$\frac{1}{2}S_2 = (1 + \nu)/E \text{ and } S_1 = -\nu/E. \quad (2.5.69)$$

Although polycrystalline materials on a macroscopic level can be considered isotropic, residual stress measurement by X-ray diffraction is done by measuring the strain in a specific crystal orientation of the crystallites that satisfies the Bragg condition. The stresses measured from diffracting crystallographic planes may have different values because of their elastic anisotropy. In such cases, the macroscopic elasticity constants should be replaced by a set of crystallographic plane-specific elasticity constants,  $S_1^{\{hkl\}}$  and  $\frac{1}{2}S_2^{\{hkl\}}$ , called X-ray elastic constants (XECs). XECs for many materials can be found in the literature, measured or calculated from microscopic elasticity constants (Lu, 1996). In the case of materials with cubic crystal symmetry, the

equations for calculating the XECs from the macroscopic elasticity constants  $\frac{1}{2}S_2$  and  $S_1$  are

$$\begin{aligned} \frac{1}{2}S_2^{\{hkl\}} &= \frac{1}{2}S_2[1 + 3(0.2 - \Gamma(hkl))\Delta] \\ S_1^{\{hkl\}} &= S_1 - \frac{1}{2}S_2[0.2 - \Gamma(hkl)]\Delta, \end{aligned} \quad (2.5.70)$$

where

$$\Gamma(hkl) = \frac{h^2k^2 + k^2l^2 + l^2h^2}{(h^2 + k^2 + l^2)^2} \text{ and } \Delta = \frac{5(A_{RX} - 1)}{3 + 2A_{RX}}.$$

In the equations for stress measurement hereafter, either the macroscopic elasticity constants  $\frac{1}{2}S_2$  and  $S_1$  or the XECs  $S_1^{\{hkl\}}$  and  $\frac{1}{2}S_2^{\{hkl\}}$  are used in the expression, but either set of elastic constants can be used depending on the requirements of the application. The factor of anisotropy ( $A_{RX}$ ) is a measure of the elastic anisotropy of a material (He, 2009).

### 2.5.4.3.2. Fundamental equations

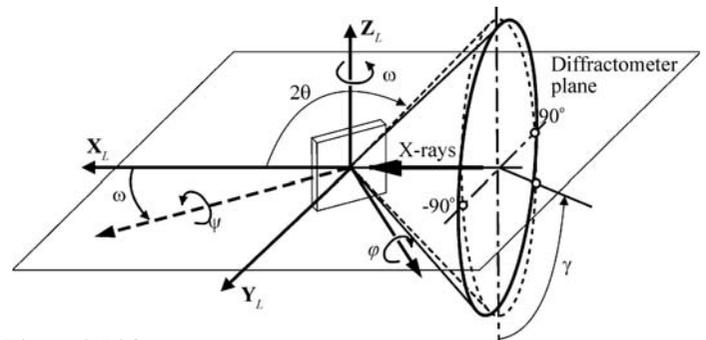
Fig. 2.5.24 illustrates two diffraction cones for backward diffraction. The regular diffraction cone (dashed lines) is from the powder sample with no stress, so the  $2\theta$  angles are constant at all  $\gamma$  angles. The diffraction ring shown as a solid line is the cross section of a diffraction cone that is distorted as a result of stresses. For a stressed sample,  $2\theta$  becomes a function of  $\gamma$  and the sample orientation ( $\omega$ ,  $\psi$ ,  $\varphi$ ), i.e.  $2\theta = 2\theta(\gamma, \omega, \psi, \varphi)$ . This function is uniquely determined by the stress tensor. The strain measured by the  $2\theta$  shift at a point on the diffraction ring is  $\varepsilon_{(\gamma, \omega, \psi, \varphi)}^{\{hkl\}}$ , based on the true strain definition

$$\varepsilon_{(\gamma, \omega, \psi, \varphi)}^{\{hkl\}} = \ln \frac{d}{d_o} = \ln \frac{\sin \theta_o}{\sin \theta} = \ln \frac{\lambda}{2d_o \sin \theta}, \quad (2.5.71)$$

where  $d_o$  and  $\theta_o$  are the stress-free values and  $d$  and  $\theta$  are measured values from a point on the diffraction ring corresponding to  $(\gamma, \omega, \psi, \varphi)$ . The direction of  $\varepsilon_{(\gamma, \omega, \psi, \varphi)}^{\{hkl\}}$  in the sample coordinates  $S_1, S_2, S_3$  can be given by the unit-vector components  $h_1, h_2$  and  $h_3$ . As a second-order tensor, the relationship between the measured strain and the strain-tensor components is then given by

$$\varepsilon_{(\gamma, \omega, \psi, \varphi)}^{\{hkl\}} = \varepsilon_{ij} \cdot h_i \cdot h_j. \quad (2.5.72)$$

The scalar product of the strain tensor with the unit vector in the above equation is the sum of all components in the tensor multiplied by the components in the unit vector corresponding to the first and the second indices. The expansion of this equation for  $i$  and  $j$  values of 1, 2 and 3 results in



**Figure 2.5.24** Diffraction-cone distortion due to stresses.