

## 2. INSTRUMENTATION AND SAMPLE PREPARATION

where  $\beta$  is the divergence of the incident beam. While increasing the divergence angle  $\beta$  may introduce instrumental broadening which deteriorates the  $2\theta$  resolution, virtual oscillation improves sampling statistics without introducing instrumental broadening.

In the cases of materials with a large grain size or preferred orientation, or of microdiffraction with a small X-ray beam size, it can be difficult to determine the  $2\theta$  position because of poor counting statistics. In these cases, some kind of sample oscillation, either by translation or rotation, can bring more crystallites into the diffraction condition. Angular oscillation is an enhancement to the angular window of the instrument. The effect is that the angular window scans over the oscillation angle. Any of the three rotation angles ( $\omega$ ,  $\psi$ ,  $\varphi$ ) or their combinations can be used as oscillation angles. Angular oscillation can effectively improve the sampling statistics for both large grain size and preferred orientation. As an extreme example, a powder-diffraction pattern can be generated from single-crystal sample if a sufficient angular window can be achieved by sample rotation in such a way as to simulate a Gandolfi camera (Guggenheim, 2005). Sample oscillation is not always necessary if virtual oscillation can achieve sufficient sampling statistics.

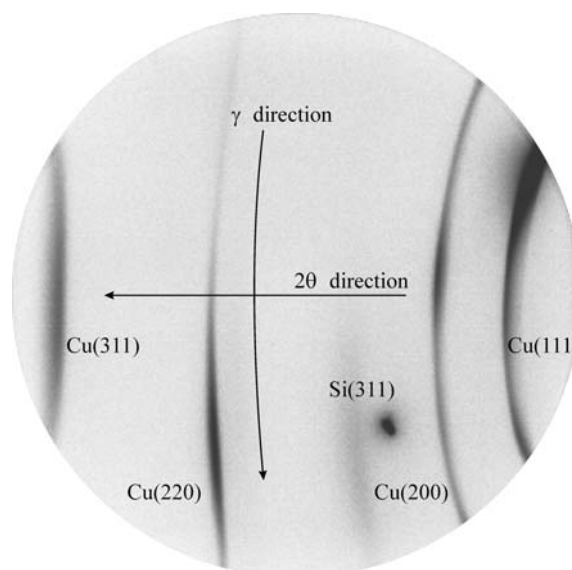
## 2.5.4.2. Texture analysis

Most natural or artificial solid materials are polycrystalline, consisting of many crystallites (also called grains) of various sizes, shapes and orientations. When the orientations of the crystallites in a material have a random distribution, it presents isotropic properties. The anisotropic orientation distribution of crystallites is referred to as preferred orientation or texture. Depending on the degree of the preferred orientation, a sample is referred to as having a weak, moderate or strong texture. Many electrical, optical or mechanical properties of materials are affected or determined by their texture. The determination and interpretation of textures are therefore of fundamental importance in materials science and technology (Bunge, 1983).

When a conventional X-ray diffractometer with a point detector is used for texture measurement, the crystallite orientation distribution in one direction is measured at a time, and full texture information is measured by rotating the sample to all the desired orientations. When a two-dimensional X-ray diffraction system is used for texture measurement, the orientation distributions of several crystallographic planes over a range of angles can be measured simultaneously so as to get better measurement results in a shorter data-collection time (Smith & Ortega, 1993; Blanton, 1994; Bunge & Klein, 1996; Helming *et al.*, 2003; Wenk & Griggull, 2003; He, 2009). The orientation relationships between different phases or between different layers of thin films and substrates can also be easily revealed. The texture effect may be observed and evaluated directly from the 2D diffraction frames without data processing.

## 2.5.4.2.1. Pole density and pole figures

XRD results from an 'ideal' powder in which the crystallites are randomly oriented normally serve as a basis for determining the relative intensity of each diffraction peak. The deviation of the grain orientation distribution of a polycrystalline material from that of an ideal powder is measured as texture. The pole figure for a particular crystallographic plane is normally used to represent the texture of a sample. Assuming that all grains have the same volume, each 'pole' represents a grain that satisfies the Bragg condition. The number of grains satisfying the Bragg condition at a particular sample orientation can be larger or



**Figure 2.5.18**

Diffraction frame collected from a Cu film on an Si substrate showing intensity variation along  $\gamma$  due to texture.

smaller than the number of grains for an ideal sample, and likewise for the integrated intensity of that peak. The measured 2D diffraction pattern contains two very important parameters at each  $\gamma$  angle: the partially integrated intensity  $I$  and the Bragg angle  $2\theta$ . Fig. 2.5.18 shows a 2D frame for a Cu thin film on an Si wafer collected with a microgap 2D detector. It contains four Cu lines and one Si spot. The diffraction intensity varies along  $\gamma$  because of the anisotropic pole-density distribution. For each diffraction ring, the intensity is a function of  $\gamma$  and the sample orientation ( $\omega$ ,  $\psi$ ,  $\varphi$ ), *i.e.*  $I = I(\gamma, \omega, \psi, \varphi)$ .

Plotting the intensity of each ( $hkl$ ) line with respect to the sample coordinates in a stereographic projection gives a qualitative view of the orientation of the crystallites with respect to a sample direction. These stereographic projection plots are called pole figures. As is shown in Fig. 2.5.19(a), the sample orientation is defined by the sample coordinates  $S_1$ ,  $S_2$  and  $S_3$ . For metals with rolling texture, the axes  $S_1$ ,  $S_2$  and  $S_3$  correspond to the transverse direction (TD), rolling direction (RD) and normal direction (ND), respectively. Let us consider a sphere with unit radius and the origin at  $O$ . A unit vector representing an arbitrary pole direction starts from the origin  $O$  and ends at the point  $P$  on the sphere. The pole direction is defined by the radial angle  $\alpha$  and azimuthal angle  $\beta$ . The pole density at the point  $P$  projects to the point  $P'$  on the equatorial plane through a straight line from  $P$  to the point  $S$ . The pole densities at all directions are mapped onto the equatorial plane by stereographic projection as shown in Fig. 2.5.19(b). This two-dimensional mapping of the pole density onto the equatorial plane is called a pole figure. The azimuthal angle  $\beta$  projects to the pole figure as a rotation angle about the centre of the pole figure from the sample direction  $S_1$ . When plotting the pole density into a pole figure of radius  $R$ , the location of the point  $P'$  in the pole figure should be given by  $\beta$  and

$$r = R \tan\left(\frac{\pi}{4} - \frac{\alpha}{2}\right) = R \tan \frac{\chi}{2}. \quad (2.5.52)$$

For easy computer plotting and easy angular readout from the pole figure, the radial angle  $\alpha$  may be plotted on an equally spaced angular scale, similar to a two-dimensional polar coordinate system. Other pole-figure mapping styles may be used, but must be properly noted to avoid confusion (Birkholz, 2006).