

## 2.5. ELECTRON DIFFRACTION AND ELECTRON MICROSCOPY IN STRUCTURE DETERMINATION

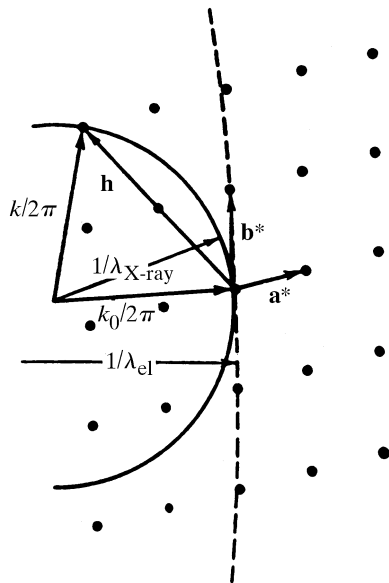


Fig. 2.5.4.1. Ewald spheres in reciprocal space. Dotted line: electrons, solid line: X-rays.

approximation holds true or if the contributions of the dynamic and secondary scattering are not too large.

The indexing of reflections and the unit-cell determination are carried out according to the formulae relating the RL to the DL (direct lattice) (Vainshtein, 1964; Pinsker, 1953; Zvyagin, 1967).

Under electron-diffraction conditions crystals usually show a tendency to lie down on the substrate plane on the most developed face. Let us take this as (001). The vectors  $\mathbf{a}$  and  $\mathbf{b}$  are then parallel, while vector  $\mathbf{c}^*$  is normal to this plane, and the RL points are considered as being disposed along direct lines parallel to the axis  $c^*$  with constant  $hk$  and variable  $l$ .

The interpretation of the point patterns as respective RL planes is quite simple in the case of orthogonal lattices. If the lattice is triclinic or monoclinic the pattern of the crystal in the position with the face (001) normal to the incident beam does not have to contain  $hk0$  reflections with nonzero  $h$  and  $k$  because, in general, the planes  $ab$  and  $a^*b^*$  do not coincide. However, the intersection traces of direct lines  $hk$  with the plane normal to them (plane  $ab$ ) always form a net with periods

$$(a \sin \gamma)^{-1}, (b \sin \gamma)^{-1} \text{ and angle } \gamma' = \pi - \gamma \quad (2.5.4.2a)$$

(Fig. 2.5.4.2). The points  $hkl$  along these directions  $hk$  are at distances

$$\eta = ha^* \cos \beta^* + kb^* \cos \alpha^* + lc^* \quad (2.5.4.3)$$

from the  $ab$  plane.

By changing the crystal orientation it is possible to obtain an image of the  $a^*b^*$  plane containing  $hk0$  reflections, or of other RL planes, with the exception of planes making a small angle with the axis  $c^*$ .

In the general case of an arbitrary crystal orientation, the pattern is considered as a plane section of the system of directions  $hk$  which makes an angle  $\varphi$  with the plane  $ab$ , intersecting it along a direction  $[uv]$ . It is described by two periods along directions  $0h, 0k$ ;

$$(a \sin \gamma \cos \psi_h)^{-1}, (b \sin \gamma \cos \psi_k)^{-1}, \quad (2.5.4.2b)$$

with an angle  $\gamma''$  between them satisfying the relation

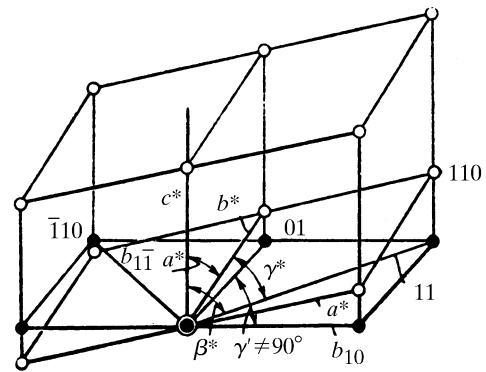


Fig. 2.5.4.2. Triclinic reciprocal lattice. Points: open circles, projection net: black circles.

$$\cos \gamma'' = \sin \psi_h \sin \psi_k - \cos \psi_h \cos \psi_k \cos \gamma, \quad (2.5.4.2c)$$

and by a system of parallel directions

$$p_h h + p_k k = l; \quad l = 0, \pm 1, \pm 2, \dots \quad (2.5.4.4)$$

The angles  $\psi_h, \psi_k$  are formed by directions  $0h, 0k$  in the plane of the pattern with the plane  $ab$ . The coefficients  $p_h, p_k$  depend on the unit-cell parameters, angle  $\varphi$  and direction  $[uv]$ . These relations are used for the indexing of reflections revealed near the integer positions  $hkl$  in the pattern and for unit-cell calculations (Vainshtein, 1964; Zvyagin, 1967; Zvyagin *et al.*, 1979).

In RED patterns obtained with an incident beam nearly parallel to the plane  $ab$  one can reveal all the RL planes passing through  $c^*$  which become normal to the beam at different azimuthal orientations of the crystal.

With the increase of the thickness of crystals (see below, Chapter 5.1) the scattering becomes dynamical and Kikuchi lines and bands appear. Kikuchi ED patterns are used for the estimation of the degree of perfection of the structure of the surface layers of single crystals for specimen orientation in HREM (IT C, 2004, Section 4.3.8). Patterns obtained with a convergent beam contain Kossel lines and are used for determining the symmetry of objects under investigation (see Section 2.5.3).

*Texture ED patterns* are a widely used kind of ED pattern (Pinsker, 1953; Vainshtein, 1964; Zvyagin, 1967). Textured specimens are prepared by substance precipitation on the substrate, from solutions and suspensions, or from gas phase in vacuum. The microcrystals are found to be oriented with a common (developed) face parallel to the substrate, but they have random azimuthal orientations. Correspondingly, the RL also takes random azimuthal orientations, having  $c^*$  as the common axis, *i.e.* it is a rotational body of the point RL of a single crystal. Thus, the ED patterns from textures bear a resemblance, from the viewpoint of their geometry, to X-ray rotation patterns, but they are less complicated, since they represent a plane cross section of reciprocal space.

If the crystallites are oriented by the plane  $(hkl)$ , then the axis  $[hk]l^*$  is the texture axis. For the sake of simplicity, let us assume that the basic plane is the plane (001) containing the axes  $a$  and  $b$ , so that the texture axis is  $[001]^*$ , *i.e.* the axis  $c^*$ . The matrices of appropriate transformations will define a transition to the general case (see IT A, 2005). The RL directions  $hk = \text{constant}$ , parallel to the texture axis, transform to cylindrical surfaces, the points with  $\eta_{hkl} = \text{constant}$  are in planes perpendicular to the texture axis, while any 'tilted' lines transform to cones or hyperboloids of rotation. Each point  $hkl$  transforms to a ring lying on these surfaces. In practice, owing to a certain spread of  $c^*$  axes of single crystals, the rings are blurred into small band sections of a