within a wedge of tens of milliradians. Thus, powder electron data generally tend to be more kinematical than single-crystal data.

2.4.3. Electron powder diffraction techniques

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The basic setup for electron powder diffraction uses a transmission electron microscope equipped with an area electron detector (photographic film, CCD camera *etc.*). Thin films, such as amorphous carbon or holey carbon films supported on metal grids, are typically used to support powder samples, which are then mounted and inserted into the transmission electron microscope inside a TEM sample holder. Solid free-standing thin films can be placed directly on top of a metal grid.

The electron beam used for a powder electron diffraction experiment is shaped using electromagnetic lenses. A modern transmission electron microscope uses at least three sets of magnetic lenses for the illumination system: condensers I and II, and the objective prefield. The prefield is part of the objective lens system before the sample acting as a lens. Some transmission electron microscopes come with an additional condenser lens (condenser III, or condenser mini-lens), which can be used for nanodiffraction. These lenses are used in various combinations to set up electron illumination for selected-area electron diffraction (SAED) or nano-area electron diffraction (NAED) (Zuo, 2004). The major difference between these two is the area of illumination, which is controlled by the strength (or focal length) of the condensers II and III.

An issue to be considered during setup of the electron beam for powder diffraction is the electron lateral coherence length. In a transmission electron microscope, the electron coherence is defined by the coherence length seen at the condenser aperture. According to the Zernike–Van Cittert theorem, the degree of coherence between electron wavefunctions at two different points far away from a monochromatic electron source is given by the Fourier transform of the source intensity distribution (Cowley, 1999). If we assume that the source has a uniform intensity within a circular disc, the coherence function is then given by $\lambda J_1(\pi\beta r/\lambda)/\beta r$ with J_1 being the first-order Bessel function, r the radial distance at the aperture and β the angle sustained by the electron source. The lateral coherence length L, which is often referred to in the literature, is defined by r at the first zero of J_1, which has the value of $L = 1.2\lambda/\beta$. The source seen by the condenser aperture inside a transmission electron microscope is the source image formed after the condenser-I lens. For a Schottky emission source, the emission diameter is between 20 and 30 nm according to Botton (2007). For a condenser aperture placed 10 cm away from the electron source image, a factor of 10 source demagnification provides a coherence length from 100 to 150 µm. When a smaller condenser aperture is used, such as in NAED, the electron beam can be considered as approximately coherent and the lateral coherence length on the same is limited by the beam convergence angle α with $L_{\rm sample} = 1.2\lambda/\alpha$.

2.4.3.1. *Selected-area electron diffraction (SAED)*

SAED is formed using the transmission electron microscope illumination, which is spread out over a large area of the specimen to minimize the beam convergence angle. The diffraction pattern is first formed at the back focal plane of the objective lens and then magnified by the intermediate and projector lenses (only one is shown) onto the screen or electron detector (Fig. 2.4.3). The recorded diffraction pattern is from an area of interest selected by placing an aperture in the conjugate (imaging) plane of the objective lens. Only electron beams passing through this aperture contribute to the diffraction pattern. For a perfect lens without aberrations, electron beams recorded in the diffraction pattern come from an area that is defined by the image of the selected-area aperture at the specimen plane. The aperture image is demagnified by the objective lens. In a conventional electron microscope, rays at an angle to the optic axis are displaced away from the centre because of the spherical aberration of the objective lens (C_s) as shown in Fig. 2.4.3. The displacement is proportional to $C_s\alpha^3$, where α is twice the Bragg angle. The smallest area that can be selected in SAED is thus limited by the objective lens aberrations. This limitation is removed by using an electron microscope equipped with a transmission electron microscope aberration corrector placed after the objective lens (Haider *et al.*, 1998).

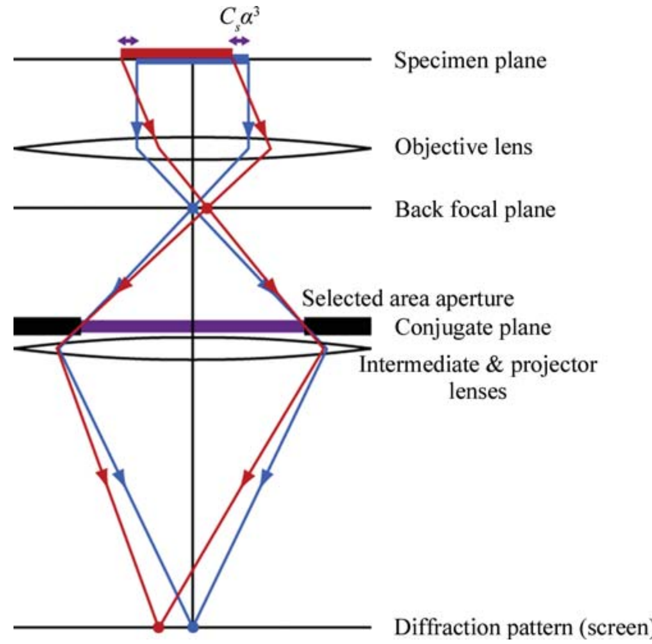

Figure 2.4.3
Schematic illustration of selected-area electron diffraction in conventional TEM. (Provided by Jun Yamasaki of Nagoya University, Japan.)

The major feature of SAED is that it provides a large illumination area, which is beneficial for recording diffraction patterns from polycrystalline samples as it leads to averaging over a large volume (for example, a large number of nanoparticles). SAED can also be used for low-dose electron diffraction, which is required for studying radiation-sensitive materials such as organic thin films.

2.4.3.2. *Nano-area electron diffraction (NAED)*

NAED uses a small (nanometre-sized) parallel illumination with the condenser/objective setup shown in Fig. 2.4.4 (Zuo *et al.*, 2004). The small beam is achieved by reducing the convergence angle of the condenser-II crossover and placing it at the focal plane of the objective prefield, which then forms a parallel-beam illumination on the sample for an ideal lens. A third condenser lens, or a mini-lens, is required for the formation of a nanometre-sized parallel beam. For a condenser aperture of 10 µm in diameter, the probe diameter is ~50 nm with an overall magnification factor of 1/200 in the JEOL 2010 electron microscopes (JEOL, USA). The smallest beam convergence angle in NAED is