*International Tables for Crystallography* (2018). Vol. H, Figure 2.4.5, p. 106.



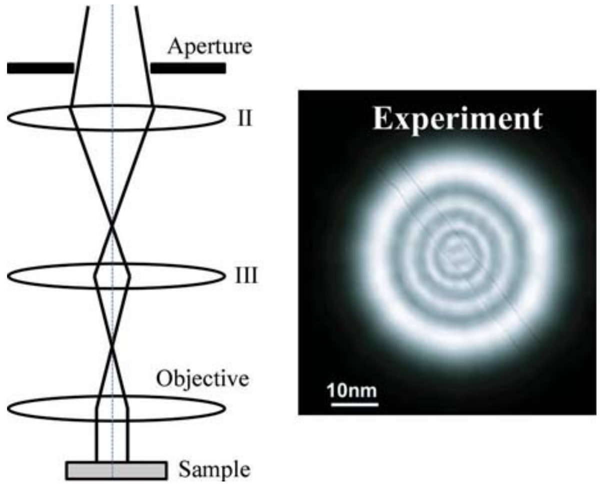


**Figure 2.4.4**
Schematic illustration of electron nanoprobe formation using a combination of condenser lenses (II and III) and the objective lens. The beam divergence angle is kept at a minimum by forming a crossover at the front focal plane of the objective lens. An image of an experimental electron nanoprobe is shown on the right with a carbon nanotube contained inside the probe.

limited by the aberrations of the illumination lenses. A beam convergence angle as small as ~0.05 mrad has been reported (Zuo *et al.*, 2004). A diffraction pattern recorded using NAED is similar to one recorded by SAED. The major difference is that the diffraction volume is defined directly by the electron probe in NAED. Since all electrons illuminating the sample are recorded in the diffraction pattern, NAED in an FEG microscope also provides higher beam intensity than SAED (the probe current intensity using a 10 µm condenser-II aperture in a JEOL 2010F is $\sim 10^5$ e $s^{-1}$ $nm^{-2}$) (Zuo *et al.*, 2004).

The small probe size is most useful for studying a small section of thin films or for selection of nanoparticles for powder diffraction. The small beam size reduces the background in the electron diffraction pattern from the surrounding materials.

### 2.4.3.3. *Sample preparation*

The success of an electron powder diffraction experiment to a large extent depends on sample preparation. The powder sample has to be suitable for electron-beam observation, and the sample also needs to be compatible with the vacuum environment of the microscope. *In situ* experiments can be carried out using special holders for cooling, heating and cryogenic or environmental transfer. Special microscopes are also available to provide a gaseous or ultra high vacuum environment for the investigation of structures under a gas or at ultra low pressure, or *in situ* sample preparation.

The observed area of the sample must be electron transparent, *i.e.* have a thickness of less than or comparable to the inelastic mean free path of electrons. The inelastic mean free path increases with the electron voltage (Egerton, 2011). The typical sample thickness ranges from a few tens to hundreds of nanometres for 200 kV high-energy electrons (see Table F.1 in Zuo & Spence, 2017).

The sample-preparation techniques can be divided into three categories: (i) bulk-based for bulky materials and supported thin films, (ii) powder-based techniques and (iii) free-standing thin films over a supporting grid prepared by vacuum evaporation or sputtering.

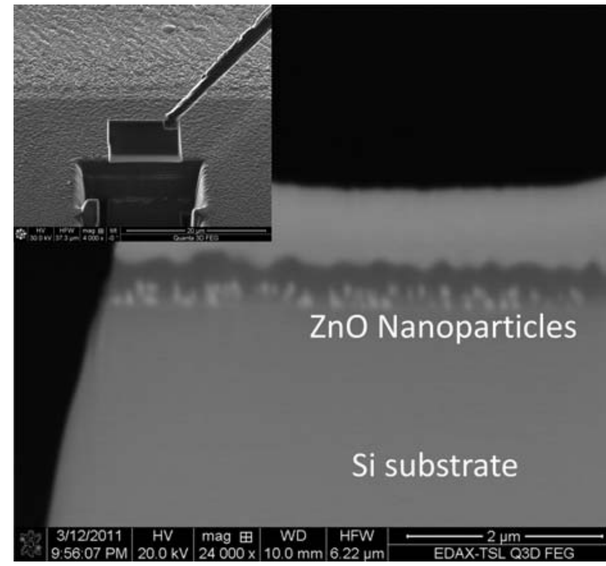


**Figure 2.4.5**
Sample preparation and lift-out using a focused ion beam (FIB). A thin section of the sample is cut out using the FIB and attached to a mechanical probe for lift-out (inset). The image shows the lift-out section containing ZnO nanoparticles in bright dot-like contrast supported on an Si substrate.

The bulk-based techniques involve mechanical cutting, thinning/polishing and perforation. An ion beam is typically used in the last step of perforation to create a thin area around the edge of a hole for electron-beam observation. Chemical and electrolytic methods are also often used for preparing electron-transparent samples. While these methods have been applied to a broad range of materials, they are mostly used for metals or semiconductors to create smooth sample surfaces free from defects or sample heating caused by ion-beam irradiation. Mechanical thinning and polishing are sometimes done with a wedge angle with the help of a tripod. The thin region next to the edge only requires a brief ion-beam bombardment to make it electron transparent. A detailed description of traditional sample-preparation techniques for TEM can be found in Barna & Pécz (1997). The above techniques are applicable to both thin films and bulk nanocrystalline materials. The powder-based techniques use dispersion of powders on thin supporting films placed on metal grids specially made for TEM observations. This technique is most suitable for nanoparticles. For micron or larger-sized powders, additional grinding is used to produce smaller particles. The most commonly used supporting films are continuous amorphous carbon films, holey carbon films, networked carbon fibres (lacey carbon), amorphous silicon nitride and $SiO_x$. For amorphous carbon films, an ultra thin version is available which is especially useful for nanoparticle samples.

A recent development in TEM sample preparation is the use of a focused ion beam of $Ga^+$ ions for cross-sectioning a sample. The focused ion beam can drill a precise hole in the sample. The same ion beam can also be scanned over a sample surface to form an image by collecting the secondary electrons or ions generated by the beam. The ion column can be integrated into an electron column in a scanning electron microscope in the so-called dual-beam configuration. An image can be formed using either electrons or ions. Most often the electron beam is used for sample inspection, while the ion beam is used for patterning and milling. This allows precise control over the position and thickness of the cross section, which is very practical for characterization of

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