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semiconductor devices or failure analysis in general (Fig. 2.4.5). Further details about ion-beam techniques can be found in Lábár & Egerton (1999) and Orloff *et al.* (2002). For a comprehensive review of sample-preparation techniques for TEM, see Őzdöl *et al.* (2012).

2.4.3.4. *Diffraction data collection, processing and calibration*

Experimental electron powder diffraction data are collected using two-dimensional area electron detectors. Experimental issues involved in the diffraction-pattern recording procedure are electron optical alignment, diffraction-pattern collection and calibration, with particular care taken in adjusting the specimen height position (eucentric position), selection of a suitable illumination-beam convergence angle and diffraction-camera length, and finally projector-lens focusing. The diffraction-camera length is determined by the setting of intermediate and projector lenses in combination with the objective lens. To calibrate the diffraction-camera length, a standard sample is placed in the eucentric position of the objective lens at the standard focus. At this setting, the specimen plane is conjugate to the selected-area aperture (Fig. 2.4.3) and the sample image appears in focus. To obtain a sharp diffraction pattern, the detector plane must be conjugate to the back focal plane of the objective lens. This can be achieved by setting up a parallel-beam illumination and adjusting the intermediate-lens focus length to bring the direct beam into a sharp focus.

Currently available area electron detectors are CCD and CMOS cameras, imaging plates (IPs) and photographic film. While photographic film has a long history of use in electron microscopy, its limited dynamic range makes it less useful for electron diffraction data collection. Both CCD cameras and IPs are digital recorders capable of collecting electron intensity over a large dynamic range. The crucial characteristics of digital recording systems are the gain (g), linearity, resolution, detector quantum efficiency (DQE) and the dynamic range. The gain of a CCD or CMOS camera can be normalized using a flat-field illumination; the gain in IPs is assumed to be constant. The detector resolution is characterized by the point-spread function (PSF), which is roughly the detector's response to a point-like illumination. These characteristics for CCDs and IPs have been compared by Zuo (2000). The intensity of an electron diffraction pattern recorded with a digital detector is given by

$$I^{\rm recorded}(i,j) = g(i,j)H(i,j)\otimes I^{\rm original}(i,j) + n(i,j), \qquad (2.4.14)$$

where $g(i, j)$ is the detector gain image, H is the PSF of the detector, n is the detector noise and $I^{\rm original}$ is the intensity of scattered electron beams originally received by the detector. The i and j are the pixel coordinates of the detector. The PSF is experimentally characterized and measured by the amplitude of its Fourier transform, or the so-called modulated transfer function (MTF). The effects of the PSF can be removed by deconvolution. The Richardson–Lucy method is specifically targeted for Poisson processes, which can be applied to CCD images (Zuo, 2000). The alternative to the removal of the PSF is to treat it as part of the peak broadening that can be used to fit the powder pattern.

The noise in the experimental data is characterized by the DQE:

$$\mathrm{var}(I) = \frac{m\bar{g}I}{\mathrm{DQE}(I)}. \qquad (2.4.15)$$

Here I is the experimentally measured intensity, var stands for the variance, m is the area under the MTF and $\bar{g}$ is the average gain of the detector. Once the DQE is known, this expression allows an estimation of the variance in measured intensity, which is essential for quantitative intensity analysis where the variance is often used as the weight for comparing experimental and fitted data.

The performances of CCDs and IPs for electron diffraction pattern recording are different at different electron dose rates. At low dose rates, the DQE of the CCD camera is limited by the readout noise and the dark current of the CCD. IPs have better performance in the low dose range due to the low dark current and low readout noise of the photomultipliers used in IP readers. At medium and high dose rates, the IP signal is affected mostly by the linear noise due to the granular variation in the phosphor and instability in the readout system, while for CCDs the noise is mostly linear noise in the gain image.

Electromagnetic lenses are not perfect and have aberrations affecting the collected data. In most transmission electron microscopes, electron diffraction patterns are produced using the post-specimen magnetic lenses. For electron diffraction, the most important aberration is the distortion of the projector lens, causing a shift of an image point. There is no blurring in diffraction patterns associated with the lens distortion. However, the distortion affects the overall shape of diffraction patterns. The distortion is most obvious at low camera lengths, where the pattern may seem stretched or twisted at high scattering angles. There are three types of distortion of the same order as the spherical aberration of the lens. They are called pin-cushion, barrel and spiral distortions (Reimer, 1984). A distortion can also arise from the use of an electron energy filter, where a lower order of distortion can be introduced with the use of non-spherical lenses (Rose & Krahl, 1995).

For quantitative analysis an electron powder diffraction pattern recorded on an area detector needs to be integrated into one-dimensional powder diffraction data (Fig. 2.4.6). The integration involves four separate steps: (i) identifying areas of the diffraction pattern for integration, (ii) centring the diffraction pattern, (iii) applying a diffraction pattern distortion correction, if there is any, and (iv) integrating intensities for a constant diffraction angle. Electron powder diffraction patterns can be recorded on a crystalline support film, which gives sharp diffraction spots distinct from the powder diffraction rings. The sharp diffraction patterns from the support film can be excluded from the powder diffraction intensity integration in step (i) by using a mask. The same approach can be used to eliminate any alien features from a diffraction pattern caused, for instance, by the aperture or the energy filter. The diffraction pattern centring is based on the analysis of the transmitted beam in the centre of the pattern. As the transmitted beam is usually very strong and is often overexposed, finding its centre may be a non-trivial task. In order to prevent detector damage in the area of the transmitted beam a beam stop is often used. In this case, the central area in the pattern may have an irregular shape not suitable for the centring procedure. Non-distorted diffraction patterns can be centred by finding the centre of the concentric diffraction rings either by locating the position of the maximum diffraction peak intensity along the ring and using these positions to determine the centre of the ring, or by searching for the centre that gives the maximum correlation between $I(g)$ and $I(-g)$. For distorted diffraction patterns, the centring and the distortion correction must be carried out simultaneously.

The distortion correction requires a powder sample with known d-spacings. The amount of distortion can be obtained by

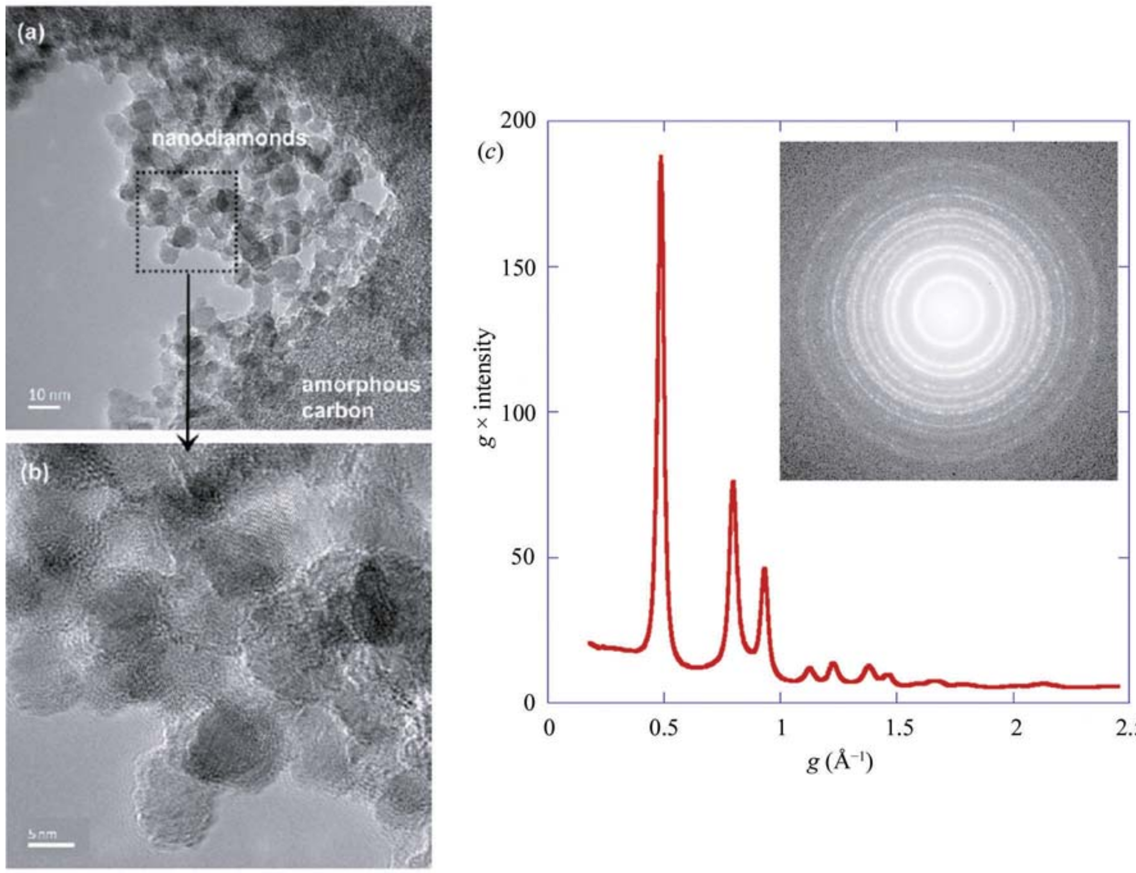

Figure 2.4.6
An example of electron powder diffraction recording for nanodiamonds. (*a*) A TEM image showing nanodiamond particles supported on amorphous carbon, (*b*) the magnified image from the boxed region of (*a*), and (*c*) the recorded electron powder diffraction pattern from nanodiamond particles and the obtained radial intensity profile.

fitting the diffraction ring position $R_d(\varphi)$ using a cosine expansion with

$$R_d(\varphi) = R + \sum_{n=1}^{N} \Delta R_n \cos n(\varphi - \varphi_n), \qquad (2.4.16)$$

where R is the average radius (zero order) of the diffraction ring, ΔR represents the amplitude of distortion of order n and φ is the azimuthal angle. Once the distortion is calibrated and excluded from the data, the diffraction intensity integration can be simply carried out by summing the recorded diffraction intensity according to the radius using

$$I_n = \frac{1}{N} \sum I[i, j], \qquad (2.4.17)$$

where the sum is taken over $R(i, j, i_0, j_0, \Delta R) \in \{n\delta, (n+1)\delta\}$. Here the powder diffraction intensity is integrated in fine discrete steps along the radius of a diffraction pattern (corresponding to increasing scattering angle) with an interval of δ, the summation is done over all diffraction pixels that fall between the radius of $n\delta$ and $(n + 1)\delta$ and N is the number of these pixels.

Filtering the inelastic background is an option for electron microscopes equipped with an electron energy filter. A major contribution to the inelastic background in electron diffraction patterns comes from bulk plasmon excitation (Egerton, 2011). This can be filtered out by dispersing the electrons according to their energies using magnetic or electrostatic fields inside an electron energy filter and using a slit of a few eV in width around the elastic (zero-loss) electron beam. For use with an area electron detector for electron diffraction, the filter must also have a double focusing capability to function as an imaging lens. There are two types of electron imaging energy filters that are currently employed: one is the in-column Ω energy filter and the other is the post-column Gatan imaging filter (GIF). The in-column Ω filter is placed between the transmission electron microscope's intermediate and projector lenses and can be used in combination with IPs, as well as with a CCD or CMOS camera. The GIF is placed after the projector lens and the use of a GIF for electron diffraction typically requires the transmission electron microscope to be switched to a special low-camera-length setting. For electron diffraction, geometric distortions, isochromaticity and the angular acceptance are important characteristics of the imaging filter (Rose & Krahl, 1995). Geometrical distortions arise from the use of non-cylindrical lenses inside the energy filter. The distortion can be caused by optical misalignment, which is an issue with the GIF with its low camera-length setting. The amount of distortion can be measured using a standard calibration sample and corrected using numerical methods. Isochromaticity defines the range of electron energies for each detector position. Ideally, this should be the same across the whole detector area. The angular acceptance defines the maximum range of diffraction angles that can be recorded on the detector without a significant loss of isochromaticity (Rose & Krahl, 1995).

2.4.4. Phase identification and phase analysis

By J. L. Lábár

For known structures, powder diffraction patterns can be used for identification of the crystalline phases and quantification of their

references