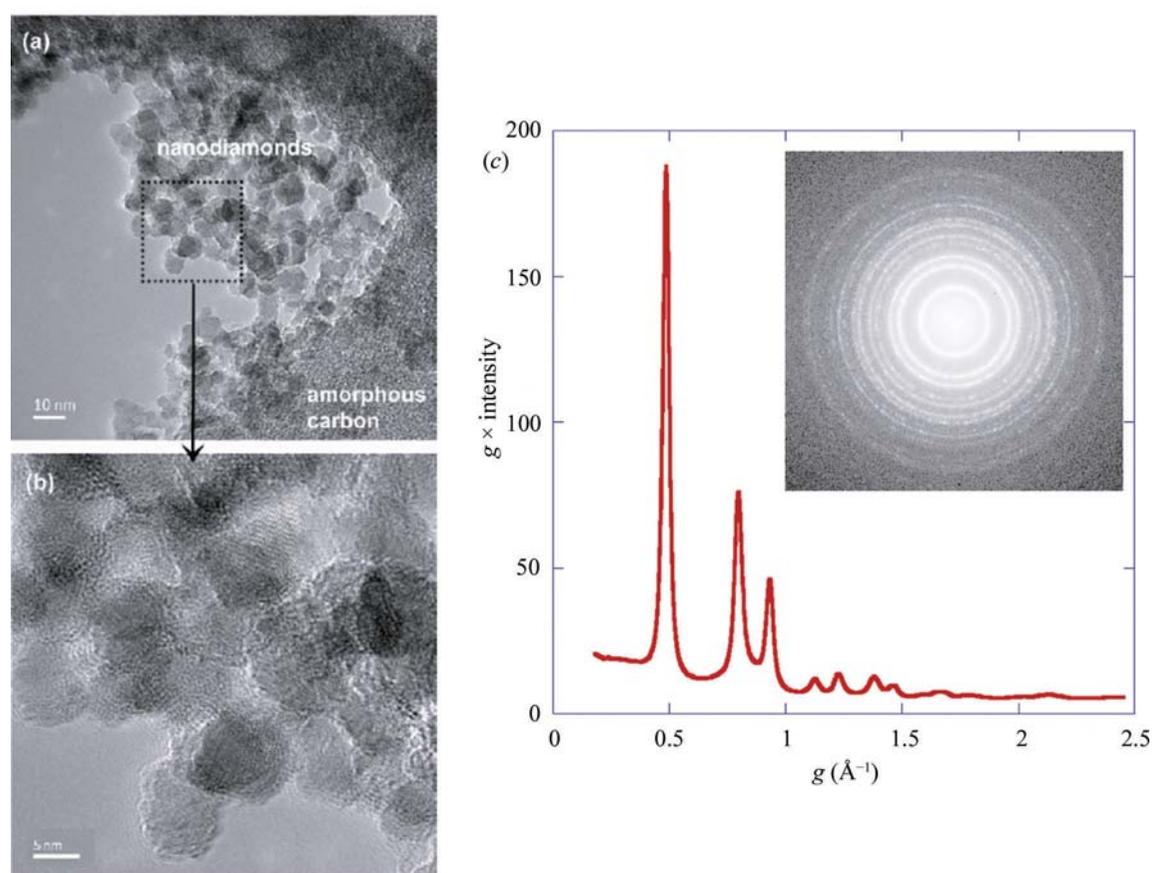


2. INSTRUMENTATION AND SAMPLE PREPARATION

**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

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

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

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

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

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