SHORT RANGE ORDER IN AMORPHOUS AND NANOCRYSTALLINE MATERIALS

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Atomic pair-distribution functions are determined from electron diffraction in TEM to obtain the nearest neighbour distances in amorphous and nanocrystalline materials. This total scattering approach is generally called ePDF analysis. Experimental pitfalls are examined, which are generally not dealt with in most of the publications. Possibilities and limitations are illustrated on nanocrystalline Ni and on different forms of amorphous carbon thin films. Density and thickness of the thin a-C films are determined by EELS from plasmon energy and energy distribution of scattered electrons [1].

Diffraction patterns in both selected area mode (SAED) and in nanoprobe mode (when illumination is restricted to the examined volume) are recorded in a Philips CM-20 TEM on imaging plates (IP). The same LN-holder was used both at room temperature (RT) and cooled to liquid nitrogen temperature (LN). The measured 2D diffraction pattern was converted into a 1D distribution for further processing [2].

Calibration of the camera length was carried out prior to each set of measurements using nanocrystalline nickel. Cross-checking of that calibration was also provided by the ePDF analysis of the same nanocrystalline sample (Ni), which in itself provided Bragg-peaks for calibration. When completely amorphous samples were examined without any Bragg-peaks, reliability of the used camera length was estimated from comparing calibration values prior to and after the measurement set and was found to be 0.5% relative.

The effect of the read-out noise of the IP was found to be order of 10 for read-out of empty IPs. The effect of damage to the IP by previous local overexposure was found to be more serious, warning that the IP must also be protected against excessive dose, similarly to cameras, even though at a higher dose level. This limit seems to be 1 million read out counts per pixel in binning 1 mode.

Effect of the supporting film (typically a-C) is compared for a uniform 15 nm thick carbon film and lacey carbon support structure with 3 nm a-C film over the holes. *In the latter case* a strange effect of the selected area aperture was observed, which was not detected with large area uniform supporting films. The presence of the observed stray radiation limits the useful Q-range for ePDF analysis. In such cases (e.g. ePDF analysis of amorphous particles) usage of localized illumination seems to be preferable to SAED.

Blocking of the direct beam is limited to as small angular range as possible (in accordance with the extension of the direct beam) and the missing part is approximated by a Gaussian continuation of the measured pattern down to zero length scattering vector (zero scattering angle).

Slowly varying inconsistencies in the measured shape of the diffracted intensity distribution are attributed to multiple scattering and an empirical correction was used to remove them.

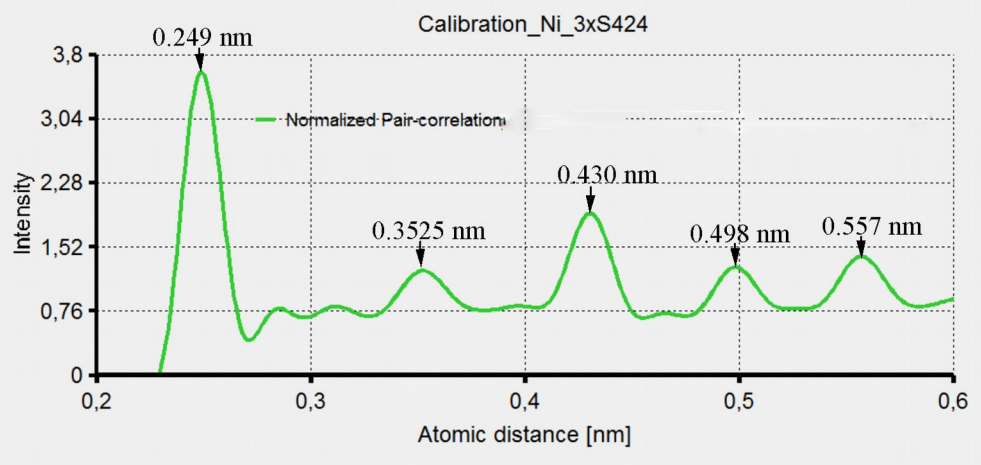

Figure 1: Normalized pair-correlation function measured from nanocrystalline Ni at RT. Measured distances are in perfect agreement with those calculated for bulk fcc Ni.

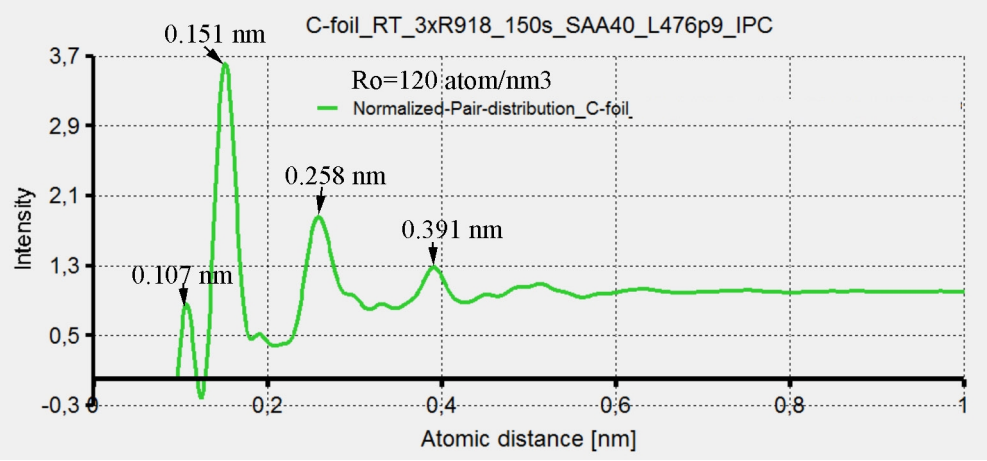

Figure 2: Normalized pair-correlation function measured from an amorphous carbon support film at RT. Measured distances are between those at graphite and diamond and the peak at 0.107 nm probably indicates the presence of hydrogen in the film.

References

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