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Quantitative Structural Analysis of Nanoparticles Using Electron Pair Distribution Function (ePDF)

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Quantitatively determining the atomic arrangement of nanomaterials is essential in nanoscience. While the traditional method of X-ray crystallography fails because of limited size of coherent structural domains within nanomaterials, a specialized approach, known as the atomic pair distribution function (PDF) method, has emerged as a powerful tool to obtain quantitative 3D structural information of nanomaterials [1]. However, currently, most PDF experiments are carried out at X-ray synchrotron or neutron sources from a large user facility, where access is limited. Here, we present that electron diffraction data that are collected in a standard transmission electron microscope (TEM) can be used to quantitatively determine nanostructures by using the ePDF technique [2].

The main procedure used for quantitative structural analysis by ePDF is described as follows: the experimentally recorded 2D electron diffraction patterns are first integrated into 1D powder diffraction patterns. Then, a home-written program, PDFgetE (unpublished) is used to calculate reduced structure function, $F(Q)$, and PDF, $G(r)$. Finally, G(r) is modeled by using PDFgui [3] to extract structural information.

For reliable ePDF measurements, different calibration samples were tested. The NIST-standard Au nanoparticle sample is recommended to calibrate camera length (CL) and determine instrument resolution parameter, Q_{damp}, in TEM. A typical electron diffraction of the Au nanoparticle sample is shown in Fig. 1(a). Fig. 1(b) and (c) show the TEM image at low and high magnification, respectively. We can see uniform size distribution of Au nanoparticles. Once the electron diffraction of the calibration Au sample was recorded, our real sample, SnO_2 nanoparticles, was then loaded. A typical electron diffraction of the SnO_2 sample is shown in Fig. 2(a). With CL calibrated by taking the Au diffraction as a reference, the reduced structure function was calculated, which is shown in Fig. 2(b). $F(Q)$ shows good statistics up to 17 $Å^{-1}$. The resulting G(r) is shown as blue symbol in Fig. 2(c), and the best-fit PDF from a structural model with space group $P4_2/mnm$ is plotted in red with a difference curve offset below. The lattice constant a and b were refined to be 4.75Å and 3.23Å, respectively. With the Q_{damp} calibrated, the particle size was refined to be ~ 2nm. Other refined parameters will also be discussed.

Furthermore, different protocols for ePDF data recording, including common/possible TEM operation mistakes, were examined by comparing the refined parameters of SnO_2 with the X-ray PDF (xPDF) results, which will be discussed in the presentation.

References:

[1] T. Egami and S. J. L. Billinge, "Underneath the Bragg peaks: structural analysis of complex materials", (Pergamon Press, Elsevier).

[2] M. Abeykoon *et al*, Z. Kristallogr. **227** (2012), p. 248.
[3] C. L. Farrow *et al*, J. Phys: condens. Mat. **19** (2007), p. 335219.
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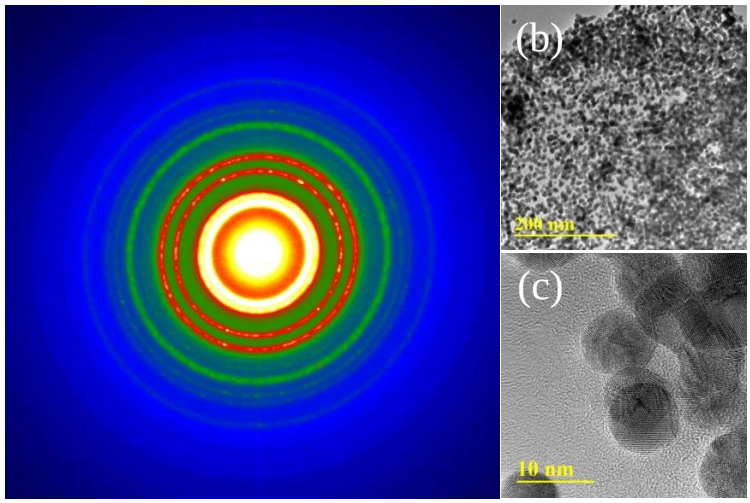

Figure 1. (a) A typical electron diffraction pattern of the 10 nm diameter Au nanoparticle sample. The TEM images at low and high magnification are shown in (b) and (c), respectively.

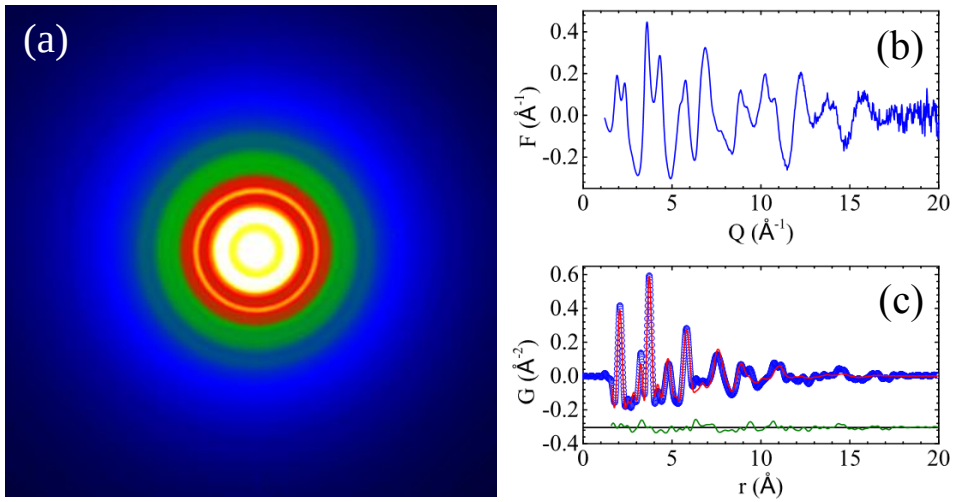

Figure 2. (a) A typical electron diffraction pattern of the SnO_2 nanoparticle sample. (b) Reduced electron structure function, *F(Q),* calculated from the integrated 2D electron diffraction pattern in (a). The resulting PDF, G(r), is shown as blue symbols in (c). The best-fit PDF from a structural model is plotted in red with a difference curve offset below.

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