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4D-STEM/PNBD: FAST AND EASY POWDER ELECTRON DIFFRACTION IN SEM

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Miroslav Slouf was born in Slany, Czech Republic, in 1973. He studied chemistry at Charles University in Prague, where he obtained his BSc degree in 1994 in the field of general chemistry, his MSc degree in 1996 in the field of inorganic chemistry, and finally PhD degree in 2001 in the field of single-crystal X-ray diffraction analysis. Since 1998 he has been working in the Institute of Macromolecular Chemistry, Czech Academy of Sciences, Prague. He started his work at IMC as a student (from 1998), continued as a head of Polymer Morphology Group (from 2002), and finally as a head of Polymer Morphology Department (from 2010 up to now). He has published >300 scientific papers with > 6,000 citations, 8 book chapters, and 5 patents (h-index = 37). So far, he has presented 6 invited lectures, mostly about micromechanical properties of polymers. He is currently teaching 2 courses at Charles University in Prague (the first about electron microscopy and the second on Python data processing and problem solving); once per circa 4 years he also teaches at the University of Trento, Italy, at the position of visiting professor (electron microscopy and micromechanical properties of polymers). He has supervised or co-supervised 3 PhD and 2 master theses. In 2013 he became an associate professor at the Faculty of Chemistry of Brno University of Technology. His research is focussed on bulk biocompatible polymers for medical applications (such as UHMWPE for total joint replacements or starch-based blends for local release of antibiotics) and development of microscopic methods (such as electron diffraction in SEM and micromechanical properties).

1. ABSTRACT

This contribution describes a novel scanning electron microscopy (SEM) method, which yields powder electron diffraction patterns [1] that are fully comparable to standard TEM/SAED powder diffractograms [2]. This opens quite new possibilities in the field of SEM microscopy. The only hardware requirement is that a SEM microscope must be equipped with a 2D-array detector of transmitted electrons (i.e., 2D-array STEM detector or pixelated STEM).

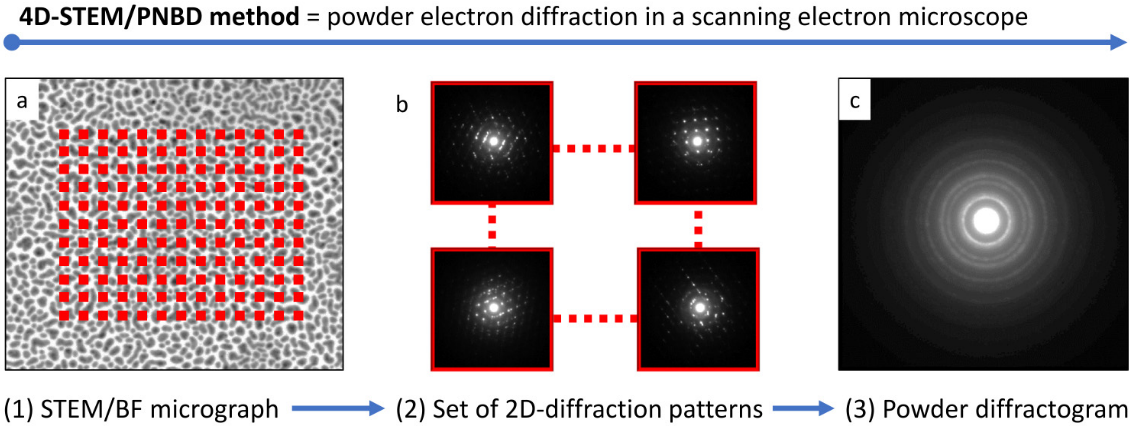

Figure 1. Principle of 4D-STEM/PNBD method: a) Standard STEM/BF image of a sample with a scanning sub-matrix (represented by red points). b) Diffraction patterns captured from each selected location (= each red point in Fig. 1a) by means of 2D-array STEM detector. c) Powder diffraction pattern obtained by summation of the individual diffraction patterns, which are shown schematically in Fig. 1b. The summation uses various computational tricks, such as entropy filtration to minimise noise and 2D-deconvolution to reduce the effect of primary beam spread.

The pixelated detectors (2D-array detectors) for STEM-in-SEM have been commercialised recently. They can be used routinely to collect a high number of electron diffraction patterns from individual nano-crystals and/or locations [3]. This is called 4D-STEM, as we obtain 2D diffractogram for each pixel of the 2D scanning array (Figs. 1a and 1b). The 4D-STEM datasets are easy to collect, but the individual 4D-STEM diffractograms (Fig. 1b) are difficult to analyse due to the random orientation of nano-crystalline material. In our method, all individual spotty diffractograms are combined into one composite powder diffraction pattern (Fig. 1c). Consequently, the method was called 4D-STEM/PNBD (powder nanobeam diffraction). The final 4D-STEM/PNBD diffractogram can be analysed easily by means of standard programmes for TEM/SAED, such as ProcessDiffraction [4] or EDIFF [5].

To make the 4D-STEM/PNBD analysis as simple as possible, we developed a freeware Python programme package STEMDIFF [6]. The package converts 4D-STEM datasets to powder diffractograms with a minimal user input. The recent STEMDIFF version includes a fast entropy-based filtering module (selecting of strongly diffracting locations and ignoring the amorphous regions) and deconvolution module (reducing the effect of primary beam spread), which improve the 4D-STEM/PNBD resolution to a TEM/SAED level (Fig. 2).

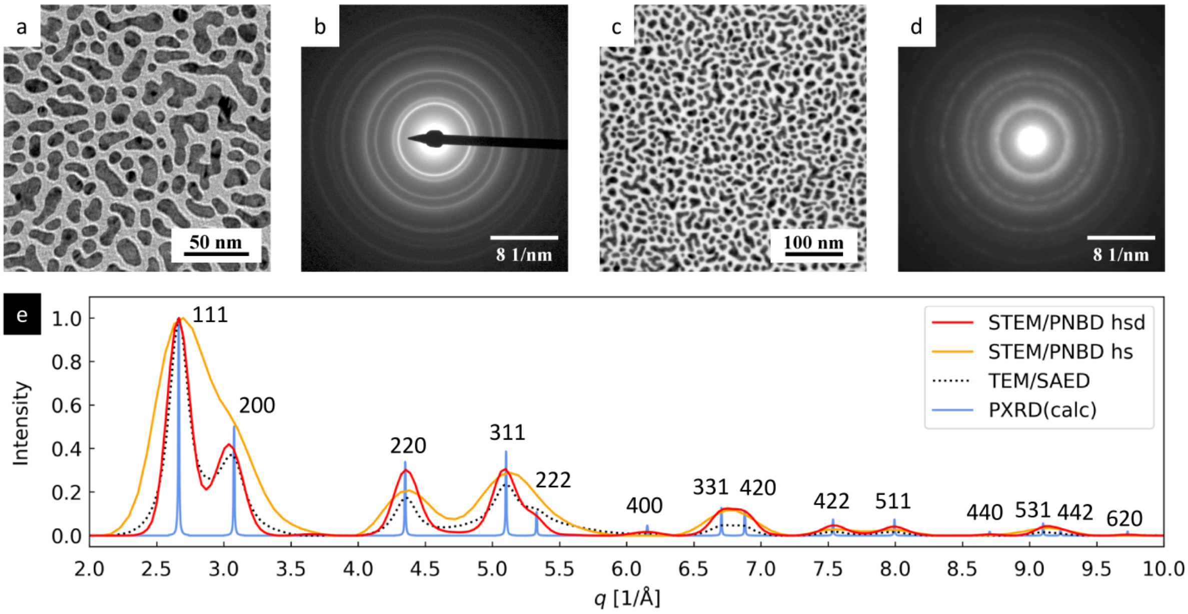

Figure 2. Comparison of 4D-STEM/PNBD, TEM and PXRD results for Au nano-islands: a) TEM/BF, b) TEM/SAED, c) STEM/BF, d) 4D-STEM/PNBD, and e) the comparison of radially averaged results from 4D-STEM/PNBD method with deconvolution (red line), 4D-STEM/PNBD without deconvolution (orange), TEM/SAED (black), and theoretically calculated PXRD (blue).

An interesting application of 4D-STEM/PNBD method is the identification of even very small amounts of nano-crystalline materials in amorphous matrices. Typical samples include crystalline nano-precipitates in various microorganisms [7] or external nano-particles deposited in biological tissues [8]. The trick consists in that we can process only the interesting (i.e., strongly diffracting) areas in 4D-STEM (which is impossible in classical TEM/SAED experiments). The 4D-STEM/PNBD method has already been successfully employed in confirmation of TiO_2 nano-particles in lung tissue [8] and ongoing experiments on similar samples show very promising results.

2. *ACKNOWLEDGEMENTS*

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