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4D-STEM/PNBD: POWDER ELECTRON DIFFRACTION IN SEM MICROSCOPES

Miroslav Slouf[1], P. Sikorova[2], M. Slouf[3], R. Skoupy[2], E. Pavlova[1] and V. Krzyzanek[2]

1 Czech Academy of Sciences, Institute of Macromolecular Chemistry
Heyrovskeho nam. 2, 162 06 Prague 6, Czech Republic
2 Czech Academy of Sciences, Institute of Scientific Instruments
Královopolská 147, 61200 Brno, Czech Republic
3 LiveSystems s.r.o.
Senovážné náměstí 978/23, 11000 Prague 1, Czech Republic
e-mail: slouf@imc.cas.cz

Miroslav Slouf was born in Slany, Czech Republic, in 1973. He studied chemistry at Charles University in Prague, where he obtained his BSc in 1994 in the field of general chemistry, his MSc in 1996 in the field of inorganic chemistry, and finally PhD 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 = 40). So far, he has presented 9 invited lectures in international conferences, mostly about electron microscopy and/or micromechanical properties of polymers. He is currently teaching two courses at Charles University in Prague (the first about electron microscopy and the second on Python data processing and problem solving); once per ca. 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 5 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. *INTRODUCTION*

We have developed a new method, named 4D-STEM/PNBD (i.e., four-dimensional scanning transmission electron microscopy/powder nanobeam diffraction), which can convert a modern SEM microscope to a simple, fast, and user-friendly powder electron diffractometer [1, 2]. The only hardware requirement is that the SEM microscope must be equipped with a 2D-array detector of transmitted electrons (also referred to as 2D-array STEM detector or pixelated STEM). In 4D-STEM/PNBD, we reduce a huge and complex 4D-STEM-in-SEM dataset, which is easy-to-collect, to a single 2D-powder diffraction pattern, which is easy-to-process, even without deep crystallographic knowledge. The principle of our method is shown in Fig. 1. The final powder diffraction patterns are equivalent to those from TEM/SAED (selected area electron diffraction), as documented in Fig. 2.

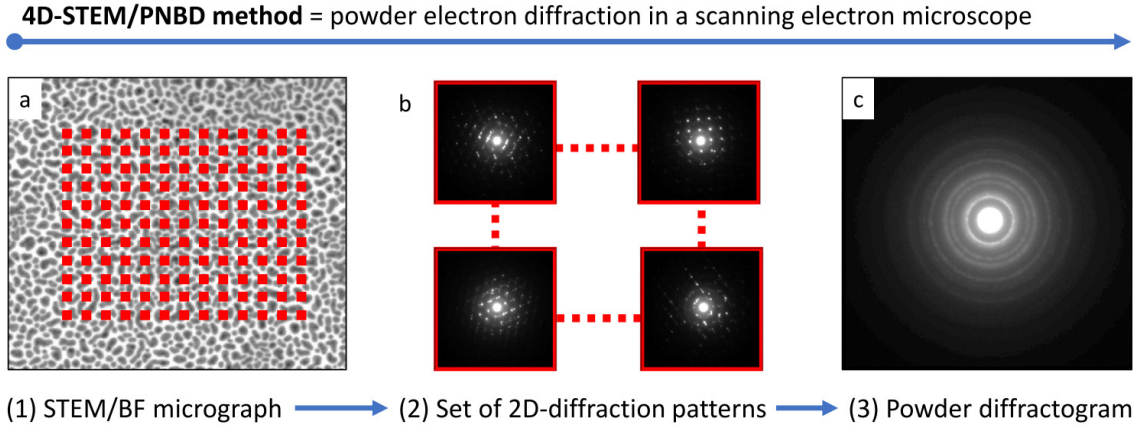

Figure 1. Principle of 4D-STEM/PNBD method: a) Standard STEM/BF image of a sample with a scanning sub-matrix (represented by red points). b) Diffraction patterns captured from each red point in Fig. 1a by means of 2D-array STEM detector. c) Powder diffraction pattern obtained by summation of the individual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

2. *RESULTS*

This contribution describes recent developments of the 4D-STEM/PNBD method, which are focussed at two areas: (*i*) To make the method as simple as possible, so that it could be used by virtually any user of a SEM microscope, and (*ii*) To enable processing of more difficult samples with worse signal-to-noise ratio, such as thicker inorganic crystals with high absorption or small nanocrystals in amorphous biological tissues [3, 4].

The first set of improvements was aimed at simplicity and user-friendliness of 4D-STEM/PNBD method. As mentioned above, the collection of 4D-STEM datasets is quite easy any modern SEM microscope equipped with a pixelated detector. The critical step that should be simplified for a common user is the processing of 4D-STEM datasets. In our case, the processing includes two sub-steps:

1. The reduction of the 4D-STEM dataset to the 2D-powder diffraction pattern, as illustrated in Fig. 1.
2. The conversion of the 2D-powder diffractogram to 1D-diffraction profile that can be compared with a theoretically calculated PXRD (powder X-ray diffraction pattern), as shown in Fig. 2.

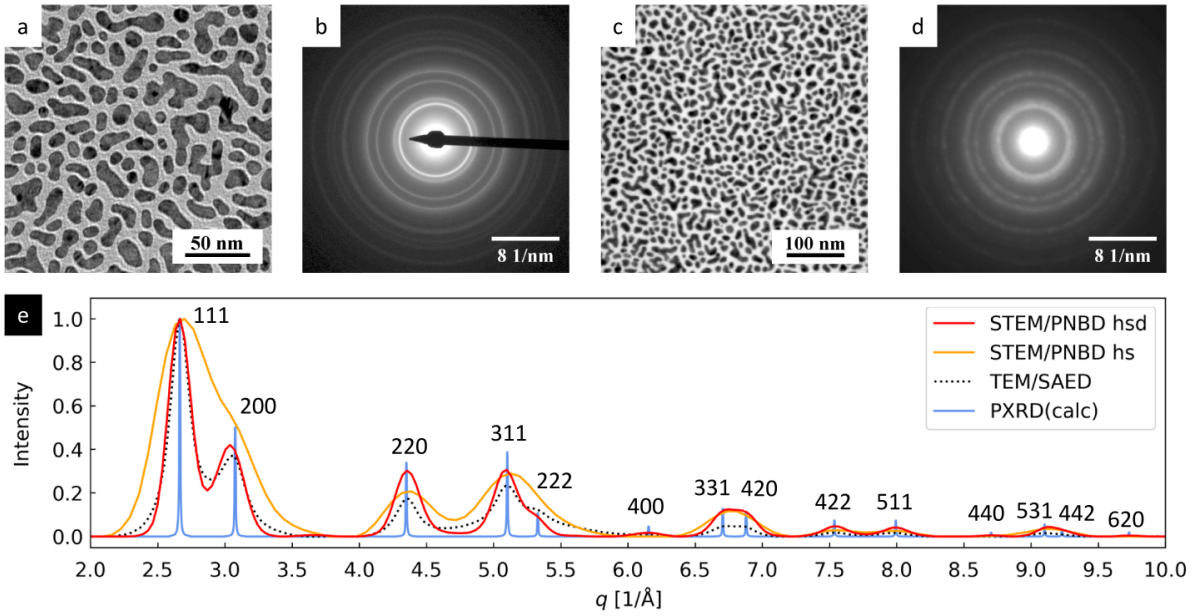

Figure 2. Comparison of 4D-STEM/PNBD, TEM and PXRD results for Au nano-islands: a) TEM/BF, b) TEM/SAED, c) STEM/BF, d) 4D-STEM/PNBD, and e) the comparison of radially averaged results from 4D-STEM/PNBD method with deconvolution (red line), 4D-STEM/PNBD without deconvolution (orange), TEM/SAED (black),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

To facilitate both processing steps, we developed two open-source Python packages, which can be incorporated to any Python installation. The first processing step (the 4D STEM → 2D-diffractogram reduction) can be done with our Python package STEMDIFF [5]. The second processing step (the 2D-diffractogram → 1D-profile conversion and comparison with PXRD) can be done with our Python package EDIFF [6].

STEMDIFF [5] and EDIFF [6] packages have been updated significantly towards the user-friendliness (a graphical user interface employing well-established Jupyter notebooks), the ease of use (templates downloadable for the websites of both packages, which can be applied – after some minimalistic modifications – to an arbitrary 4D-STEM dataset), the independence on the third party software (the whole processing, including PXRD calculations, can be performed just with a freeware Python programing language distribution combined with the two packages), and the documentation (both packages are fully documented at www using common Python standards [5, 6]).

The second set of improvements was connected with the processing of more difficult samples, which exhibit unfavourable signal-to-noise ratio, i.e., low intensity diffractions combined with high background due to absorption and/or inelastic scattering. In this area, we introduced better and more flexible user interface (current STEMDIFF and EDIFF versions are optimised for running within Jupyter notebooks), improved filtering of 4D-STEM/PNBD dataset (i.e., the selection of images with strong diffractions and omitting images containing mostly noise and/or just the primary beam), and increased speed (by introducing multicore processing, which decreases the time-consuming deconvolution step; the previous version of STEMDIFF running on a single core was ca 5× slower, which was rather inconvenient for larger datasets). Ongoing work is focussed on advanced background reduction and more efficient deconvolution methods.

3. CONCLUSION

The 4D-STEM/PNBD method brings a simple, fast, and easy-to-use electron diffraction technique to SEM users. The classical SEM microscopes offer imaging modes (such as SE or BSE) and spectroscopy modes (such as EDS). The modern SEM microscopes equipped with pixelated STEM detectors add also the third mode – the electron diffraction. The pixelated detectors can be installed in a common SEM port like any other detector. The 4D-STEM datasets are *easy-to-collect*, and the 4D-STEM/PNBD method reduces them to 2D-diffractograms, which are also *easy-to-process* [7]. Our experience shows that the analysis of complete 4D-STEM datasets requires a user with a strong crystallographic background, while the basic processing of 2D-diffractograms can be taught an average student within a half-day training.

4. ACKNOWLEDGEMENTS

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