MS41-O4 Investigation of layered and porous nanomaterials by diffraction tomography, simulations and HRTEM

Yaşar Krysiak[1], Bastian Barton[1], Haishuang Zhao[1], Reinhard Neder[2], Ute Kolb[1]

1. Institute of Inorganic Chemistry and Analytical Chemistry, Johannes Gutenberg University, Jakob-Welder-Weg 11, D-55128 Mainz, Germany
2. Chair for Crystallography and Structural Physics, Friedrich-Alexander-Universität Erlangen-Nürnberg, Staudtstr. 3, D-91058 Erlangen, Germany

email: krysiak@uni-mainz.de

Layered and porous nanomaterials are frequently characterized by disorder, pseudo symmetry and stress/strain effects, which often give rise to unique properties but make structural analysis difficult. For such materials, automated electron diffraction tomography[1] (ADT) can deliver 3D structure information at atomic resolution. For instance, a super structure in $CaCO_3$ polymorph vaterite or twinned domains for boron oxynitride could be solved with ADT.[2,3]

Currently, we aim for a more complete description of nanomaterial structures, including non-periodic effects through a combination of TEM imaging, ADT and structure simulation and modeling.

ADT data can be directly used for an *ab-initio* structure solution of the average structure. Disorder and distortion or intergrown phases are simulated using the DISCUS software package[4] through accurate analysis of the structure's variance. The results are compared to TEM holograms[5] reconstructed from focal series.

Zeolite Beta[6] was chosen as a test sample. The intergrown polymorphs A and B could be solved *ab-initio* from one single ADT dataset by integrating the relative reflection intensities with two distinct lattices. The disorder type was identified by exit wave reconstruction and confirmed by DISCUS simulations. Based on these results it was possible to solve the non-periodic structure of an unknown zeolite *ab-initio*.

The 3D atomic structure of different compounds forming highly distorted and disordered nanoparticles could be successfully described by combining ADT, layer shift simulations and TEM imaging. In future, we aim to fit the simulations automatically to the experimental diffraction data and to apply this method to different compounds such as alloys, organic pigments, and photoactive organics.

[1] E. Mugnaioli, T. Gorelik, U. Kolb, *Ultramicroscopy* **2009**, *109*, 758–765.

[2] E. Mugnaioli, I. Andrusenko, T. Schüler, N. Loges, R. E. Dinnebier, M. Panthöfer, W. Tremel, U. Kolb, *Angew. Chem.* **2012**, *124*, 7148–7152.

[3] S. Bhat, L. Wiehl, L. Molina-Luna, E. Mugnaioli, S. Lauterbach, S. Sicolo, P. Kroll, M. Duerrschnabel, N. Nishiyama, U. Kolb, et al., *Chem. Mater.* **2015**, *27*, 5907–5914.

[4] T. Proffen, R. B. Neder, *J. Appl. Crystallogr.* **1997**, *30*, 171–175.

[5] B. Barton, B. Jiang, C. Song, P. Specht, H. Calderon, C. Kisielowski, *Microsc. Microanal.* **2012**, *18*, 982–994.

[6] J. M. Newsam, M. M. J. Treacy, W. T. Koetsier, C. B. D. Gruyter, *Proc. R. Soc. Lond. Math. Phys. Eng. Sci.* **1988**, *420*, 375–405.

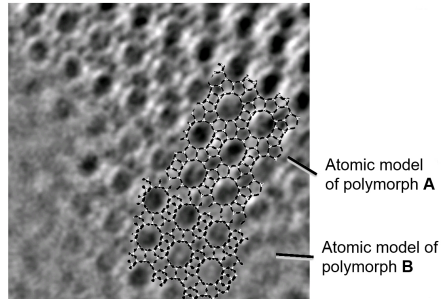

Figure 1. Intergrown polymorphic mixture of Zeolite Beta A and B: HRTEM hologram combined with structure solution models.

Keywords: automated electron diffraction tomography, disorder simulations, TEM holography, zeolite beta