# MS24- Defects and disorder quantification at the nanoscale

Chairs: Dr. Partha Pratim Das, Dr. Tatiana Gorelik

## MS24-P01

### Diffraction effects of powder nano-scale materials

Dmitriy Yatsenko[1], Sergey. Tsybulya[1]

1. Novosibirsk State University, Boreskov Institute of Catalysis SB RAS, Novosibirsk, Russia

email: yatsenko@catalysis.ru

Properties of nanomaterials are defined by features of the atomic structure and morphology. Investigation of crystal structure and nanostructure of such small objects is an actual problem. X-ray diffraction method can be used for this purpose. Anisotropic broadening of the diffraction peaks, redistribution of the intensities or appearance of diffuse scattering can appear for nanomaterials. Standard X-ray diffraction techniques used for bulk materials with periodic structures are often not applicable in this case.

These tasks can be solved by the Debye Function Analysis (DFA) method [1], based on Debye scattering equation (DSE) [2]. It is full-profile method which is applicable for any an arbitrary atoms collection, and therefore can be used for crystalline objects, non-crystalline materials or nanostructures.

Possibilities of modelling diffraction patterns by the DFA by our software [3] will be shown for specific examples of various nanocrystalline materials: hydroxides of magnesium and tungsten, layered structures, metastable forms of aluminum oxide, ultradispersed iron oxides et al. [4]. It is public-domain software available on the website: www.sourceforge.net/projects/dianna.

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## MS24-P02

### Analysis of chemical short range order using single crystal diffuse scattering

Ella Mara Schmidt[1], Reinhard B Neder[1]

1. Institute for Crystallography and Structural Physics, FAU Erlangen-Nürnberg, Erlangen, Germany

email: ella.schmidt@fau.de

In the average structure refinement of molecular materials, split positions are often encountered. As a consequence a molecule on one site can be present in more than one orientation. The typical structure refinement stops there and does not consider diffuse scattering, which allows statements about short range order interactions. Building and refining a short range order model to analyze the diffuse scattering takes the structure refinement to the next level.

A complex molecular crystal will usually consist of several components on several sites within the unit cell. Furthermore, a single site may be occupied by molecules in different orientations e.g. a first molecule in orientations A and B on site 1 and a second molecule in orientations C and D on site 2, see Fig. 1. In such systems short range order is common: On a local scale, the molecules tend to show preferred pair-wise arrangements. Characterizing this local order enables profound statements about molecular interactions [1].

With the help of molecular form factors [1] and the theory of diffuse scattering (e.g. Warren [2]), we developed a method that characterizes correlated chemical short range occupational disorder directly in reciprocal space. The diffuse scattering IDiff can be expressed as a function of the indices $h,k,l$ in reciprocal space:

$$I_{Diff}(h,k,l)=I_{Laue}(h,k,l)+I_{SRO}(h,k,l)$$

Where $I_{Laue}$ is the Laue scattering

$$\left( I_{Laue}(h,k,l)\propto\sum_{i=1}^{n_s}\sum_{A=1}^{k_i}\sum_{B>A}^{k_i}|F_{A_i}-F_{B_i}|^2 \right)$$

and $I_{SRO}$ is the short range order scattering:

$$I_{SRO}(h,k,l)\propto\sum_{(u,v,w)}\sum_{i=1}^{n_s}\sum_{i=1}^{n_s}\sum_{A_i=1}^{k_i}\sum_{B_i=1}^{k_i} m_{A_i} m_{B_i} F_{A_i} F_{B_i}^{*} \alpha_{uvw}^{A_iB_i} \exp\left(2\pi i(h\cdot u+k\cdot v+l\cdot w)\right)$$

Here $(u,v,w)$ are vectors in direct space, $n_s$ is the number of different sites and $k_i$ is the number of possible components on site $i$. $m_{Ai}$ is the concentration of component $A$ on site $i$, $F_{Ai}$ is the molecular form factor of the molecule type $A$ on site $i$. $\alpha_{uvw}^{A_iB_i}$ are the Warren-Cowley short range order parameters, that encode the probability to find an AB pair separated by vector $(u,v,w)$. All parameters, except the $\alpha_{uvw}^{A_iB_i}$ can directly be determined from the average structure refinement.

We apply this formula for the analysis of the diffuse scattering of 9-Bromo-10-Methylanthracene [3]. The model for the short range order can be developed directly in reciprocal space and the formula can be used to perform a least squares regression analysis to fit the short range order parameters quantitatively.

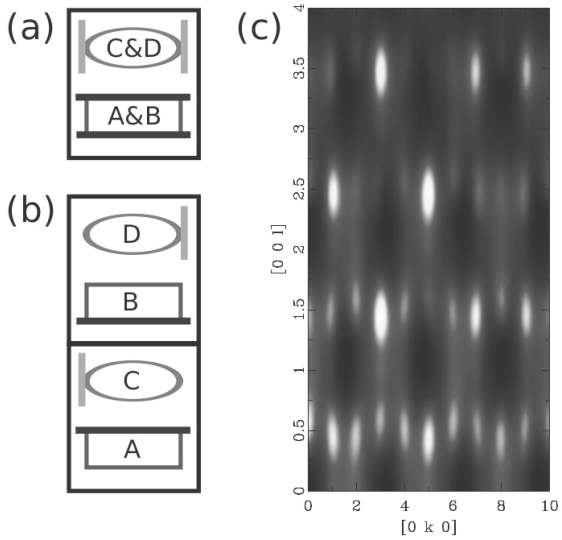
Our method to characterize complex molecular disorder using single crystal diffuse scattering is a powerful tool to understand and model molecular interactions in disordered crystals. As the method treats data directly in reciprocal space and enables least squares fitting of disorder models, calculations can be performed on desktop computers without the excessive use of computation time.

Figure 1:

(a) Refined average unit cell with overlayed components A and B on site 1 and 2.

(b) Possible configuration of disordered unit cells.

(c) Diffuse scattering of 9-Bromo-10-Methylanthracene in the 0kl-layer.

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## MS24-P03

# NanoMAX Beamline, a nanoprobe beamline for scattering and imaging at MAX IV

Angel Rodriguez Fernandez[1], Ulf Johansson[1], Gerardina Carbone[1], Alexander Björling[1], Sebastian Kalbfleisch[1], Tomas Stankevic[1], Björn Bring[1], Anders Mikkelsen[2], Ulrich Vogt[3]

1. MAX IV LABORATORY, Lund University, Lund, Sweden
2. Synchrotron Radiation Research, Department of Physics, Lund University, Lund, Sweden
3. Biomedical & X-Ray Physics, Dept. of Appl. Physics, KTH Royal Inst. of Tech, Stockholm, Sweden

**email: angel.rodriguez_fernandez@maxiv.lu.se**

NanoMAX is a hard X-ray nanoprobe beamline at the 3 GeV multi-bend achromat storage ring MAX IV, Lund, Sweden [1]. The beamline is designed to utilize the uniquely high brilliance of the facility to achieve nanometer-sized coherent foci with high photon intensity. The small focus is used for scanning imaging with the main methods nano-diffraction, phase and absorption contrast, coherent diffractive imaging and ptychography - in forward and Bragg condition. The beamline optics has been briefly presented earlier [2].

The beamline will have two experimental stations when the buildup phase ends 2019/2020. One experimental station using Kirkpatrick-Baez mirror optics (KB) for focusing. The KB system gives a diffraction limited probe of 40 nm (24 keV) - 200 nm (5 keV) with 100 mm working distance from optics to sample position. The generous space will allow for versatile sample environments. Three main detectors are planned for or installed at the station; a megapixel photon counting area detector in forward direction, a compact photon counting area detector in off-axis position on a commercial industry robot and a 3-element Germanium X-ray fluorescence detector. A compact two axis high precision goniometer will allow advanced studies of ordered samples with diffractive methods. Continuous sample scanning is implemented in a basic version and will be further developed for efficient data acquisition. The design of the second experimental station is ongoing and first tests are anticipated early 2019.

We have provided beamtime to a handful user experiments during the first year of operation. Experiments in X-ray fluorescence, wide-angle scattering, nano-diffraction and ptychography have been executed. To achieve optimal performance from the super-polished KB-mirrors we have developed a simple procedure to measure focus astigmatism by scanning a Siemens star like test structure in ptychographic mode. The test sample image and the probe are reconstructed at the sample position using diffraction data from an inline pixel detector [3]. The probe is then propagated along the beam direction to show the beam profile, in vertical and horizontal plane, as seen in figure 1 bottom parts.