# Oral Contributions

## [MS46] In situ methods and transient effects in chemical crystallograpy

***Chairs: Simone Techert (DE), Marijana Dakovic (HR)***

### [MS46-01] Microstructure Defects in Graphitic BN and their Impact on the Transition to the Dense Phases.

Christian Schimpf[a], Mikhaylo Motylenko[a], Marcus Schwarz[b] and Edwin Kroke[b], David Rafaja[a]

[a] *Institute of Materials Science, TU Bergakademie Freiberg, Germany*
[b] *Institute of Inorganic Chemistry, TU Bergakademie Freiberg, Germany*
E-mail: schimpf@iww.tu-freiberg.de

Microstructure defects in hexagonal graphitic boron nitride (h-BN) are analysed in detail using the characteristic dependence of the X-ray diffraction line broadening on the crystallographic direction. The most often observed structural defects are puckering of basal layers, turbostratic disorder, dislocations and stacking faults on basal planes. A detailed model of the anisotropic X-ray line broadening caused by puckering (= waviness of basal planes in h-BN) will be discussed. Furthermore, it will be shown how the above microstructure defects influence the anisotropy of the XRD line broadening and how this can be used for the quantification of the defects. *Ex-situ* and *in-situ* XRD experiments performed on high pressure/high temperature synthesised samples showed the impact of the microstructure defects on the transition of h-BN to the high pressure modifications crystallising in wurtzitic (w-BN) or sphaleritic (c-BN) structure [1,2]. Principally, h-BN containing a low density of microstructure defects (mainly dislocations and stacking faults) converts to the metastable wurtzitic modification [3]. The conversion is accompanied by a fragmentation of original h-BN crystallites. The formed w-BN has an increased dislocation density (~ 2 orders of magnitude) and stacking fault probability (~ 1 order of magnitude) compared to the precursor.

Starting materials containing a high density of either puckering type defects or turbostratic disorder convert from h-BN directly to c-BN during high pressure/high temperature processes via diffusion processes being assisted by the high density of microstructure defects [4]. The resulting c-BN phase includes some stacking faults during the nucleation stage; their density decreasing with increasing c-BN volume fraction. The activation energy of the conversion at a pressure of ~ 10 GPa was obtained from the time-dependent change of the phase composition of the nanocomposites measured during *in-situ* synchrotron radiation diffraction experiments at DESY/HASYLAB. Complementary investigations of the microstructure of the h-BN precursors and synthesised BN (nano)composites were conducted using transmission electron microscopy with high resolution (HRTEM). The synchrotron and HRTEM investigations revealed detailed information on the microstructure of h-BN and its impact on a nanometre scale on the phase transition to the high pressure BN phases.

[1] Rafaja,D., Wüstefeld, C., Motylenko, M., Schimpf, C., Barsukova, T., Schwarz, M.R. & Kroke, E. (2012). *Chem. Soc. Rev.*

[2] Rafaja, D., Klemm, V., Motylenko, M., Schwarz, M.R., Barsukova, T., Kroke, E., Frost, D., Dubrovinsky, L. & Dubrovinskaia, N. (2008). *J. Mater. Res.* **23**, 981-993.

[3] Britun, V.F. & Kurdyumov, A.V. (2000). *High Pressure Res.* **17**, 101-111.

[4] Eremets, M., Takemura, K., Yusa, H., Golberg, D., Bando, Y., Blank, V., Sato, Y. & Watanabe, K. (1998). *Phys. Rev. B* **57**, 5655-5660.

**Keywords:** X-ray powder diffraction, boron nitride, microstructure defects