



	Experiment title: Toward New UV LEDs through the High Pressure High Temperature Synthesis of Boron Nitride Nanoconfined in Zeolite Matrix	Experiment number: CH-5798
Beamline:	Date of experiment: from: 21.01.2021 to: 25.01.2021	Date of report: 25.03.2021
Shifts:	Local contact(s): Davide Comboni, Tomasz Poręba	<i>Received at ESRF:</i>
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Report:

Due to the high content of hydrogen (19.6 wt.%), ammonia borane (AB) is considered as one of the greatest hydrogen storage materials. Upon temperature AB undergoes two-step polymerization reaction resulting in the formation of polyaminoborane, polyiminoborane, hydrogen and, at still elevated temperature, boron nitride (material of choice in the Far UV due to its high band gap of 6 eV). [1-3]

Within the project we intended to synthesize new functional composite materials through high pressure confinement and further high temperature treatment of ammonia borane inserted to a zeolite framework. Our aim was to investigate two different binary systems using ammonia borane AB as molecular precursors of BN and silica zeolites: MFI (silicalite-1F), and TON as host materials.

At ID15B High Pressure Beamline we have performed a series of variable high pressure high temperature X-ray diffraction experiments on MFI/AB and TON/AB composites loaded under inert-gas atmosphere in a resistively heated membrane-driven Diamond Anvil Cells at 1 and 3 GPa and elevated temperature up to 300°C.

Powder XRD experiments confirmed the insertion and polymerization of ammonia borane trapped inside the pores of host materials.

We have observed pressure-induced phase transition of ammonia borane (the bulk ammonia borane transforms to its orthorhombic high pressure phase upon compression) and its further polymerization inside the porous material manifested by the disappearance of ammonia borane diffraction peaks upon high temperature.

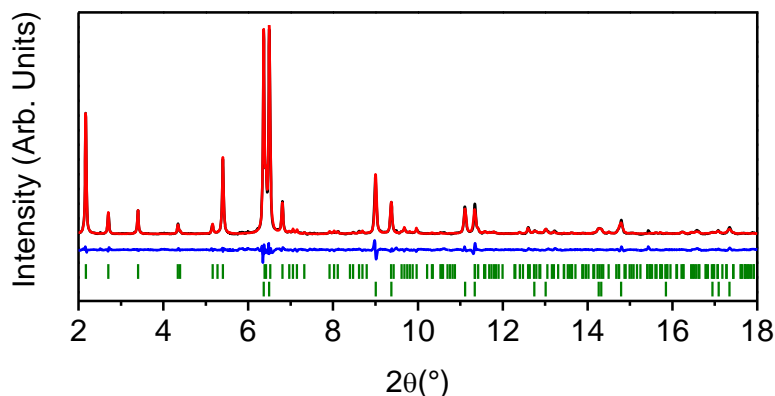


Figure 1. Experimental (black), calculated (red) and difference (blue) profiles ($\lambda=0.4104$ Å) for the $Cmc2_1$ structure of TON- NH_3BH_3 ($\lambda=0.4104$ Å) at 0.17 GPa and 23°C.

The anomalous p, T -evolution of a unit cell dimensions and significant increase of unit-cell volume confirmed dehydrocoupling of AB, its polymerization and hydrogen release inside the pores of the zeolites.

Structural refinement have been performed for TON/AB composite material, together with detailed Raman spectroscopic studies, the results have been described and submitted for publication. [4]

Detailed analysis of MFI/AB composite material and its further spectroscopic characterization is still in progress.

References:

- [1] Hamilton, C. W.; Baker, R. T.; Staubitz, A.; Manners, I. B-N compounds for chemical hydrogen storage. *Chem Soc Rev* **2009**, *38* (1), 279.
- [2] Staubitz, A.; Robertson, A. P. M.; Manners, I. Ammonia-Borane and Related Compounds as Dihydrogen Sources. *Chem Rev* **2010**, *110* (7), 4079.
- [3] Kubota, Y., Watanabe, K., Tsuda, O., Taniguchi, T., Deep Ultraviolet Light-Emitting Hexagonal Boron Nitride Synthesized at Atmospheric Pressure, *Science* **2007**, *317*, 932.

[4] Paliwoda, D., Comboni, D., Poręba, T., Hanfland, M., Alabarse, F., Maurin, D., Michel, T., Demirci, U. B., Rouquette, J., di Renzo, F., van der Lee, A., Bernard, S., Haines, J, Anomalous Volume Changes in the Siliceous Zeolite TON due to Hydrogen Insertion under High-Pressure, High-Temperature Conditions, *submitted for publication.*