

MS18-P06

1-Dimensional sp^3 -carbon nanostructures synthesized through nanocasting at high pressure

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One-dimensional diamond mesostructures (1D-diamond) are of interest as heat-conducting wires, and as mechanically reinforcing material in diamond/polymer composites because of the superlative mechanical strength and thermal conductivity of diamond. Thus far, 1D-diamond has been produced by CVD methods.¹ However, CVD methods do not allow for the economic production of bulk quantities of 1D-diamond, and it is difficult to control the diameters and length of the produced 1D-diamond structures. Currently there is no high-pressure synthesis available for the bulk synthesis of one-dimensional diamond mesostructures. The basic problem is that at high-pressure conditions it is difficult to direct the growth of diamond crystals in a specific direction. In addition, any one-dimensional structure formed can easily aggregate with another which prevents any further processing after the high-pressure synthesis.²

Herein, we present the synthesis of diamond-like 1D sp^3 -carbon mesostructures from benzene inside the 1-dimensional channels of periodic mesoporous silica SBA-15 at pressures above 20 GPa and room temperature (Figure 1). Inside the silica template, the 1D- sp^3 -carbon nanostructures are spatially separated from each other preventing their aggregation at the high-pressure conditions. Small Angle and X-Ray Scattering data collected for SBA-15/benzene composite clearly shows that silica mesostructure retains its periodic order upon compression, while X-ray diffraction experiments allow to track pressure-induced structural transformations of benzene.

Acknowledgements: This work was supported by the EFree, an Energy Frontier Research Center of the US Department of Energy Office of Basic Energy Science (SC-0001057)



Figure 1. A schematic pathway for the nanocasting of 1D- sp^3 -carbons within the 1-dimensional channels of periodic mesoporous silica SBA-15.

References:

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Keywords: one-dimensional nanomaterials, high pressure, diamond anvil cell

MS18-P07

Integrated solutions for most efficient in house high-pressure single crystal experiments

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Within the last decade high pressure studies have received significant increase of interest. Present typical applications range from the investigation of solid-state organics as part of the pharmaceutical drug development to the study of rocks and minerals with applied pressures of up to 50 GPa. While in the past it has been almost impossible to accommodate and extend standard in-house solutions, i.e. hardware and software, to the needs of high-pressure crystallography, today this picture has changed completely. Modern high-performance software, such as APEX3, has been developed to address the intrinsic challenges of high pressure crystallography, particularly with respect to sample alignment, indexing, data integration and scaling. This complements the significantly higher flexibility available in the hardware setup. Current instrumentation can easily be used for routine low temperature data collections, fully optimized with respect to minimizing air scattering and achieving full completeness and desired data multiplicity. Within just a few minutes the same instrument can be converted into an optimized solution for high pressure data collection, e.g. for the investigation of organic solid state material in a diamond anvil cell (DAC). Electronic component recognition ensures automated reconfiguration and validation of the instrument. This approach avoids invalid and useless configurations on the one hand, while on the other hand taking the special requirements of a high-pressure experiment into account. For example, even with the limitations implied by the DAC best possible, completeness and data redundancy is automatically ensured.

Using a D8 VENTURE with a fixed-chi stage we will demonstrate the advanced hardware capabilities and processing methods based on the monoclinic polymorph of the sulfonium ylid. The high flexibility of the D8 VENTURE design will be further demonstrated in the second part of the presentation: For the investigation of miniscule mineral crystals enclosed in diamond anvil cells, functionality typically only available at synchrotron facilities has been added to the in-house system. These features include highly accurate, motorized sample positioning and the ability to monitor intensity of the X-ray beam passing through diamond anvil cell. The latter is used both for DAC alignment, as well as for correcting measured intensity data. The availability of online pressure measurements based on ruby fluorescence completes the extension of the D8 VENTURE, making the system "a little synchrotron at home".

Lab-based experiments up to 50 GPa with synchrotron-sized high-pressure single-crystal samples of even triclinic symmetry will be presented.