

to their extended porosity and chemical and structural versatility.

Recently, we have been using high-pressure to probe these attributes on zeolitic imidazolate framework (ZIF) materials. The first of these to be studied was ZIF-8 ($\text{Zn}(\text{MeIM})_2$, MeIM = 2-methylimidazolate) which has a sodalite (zeolitic-type) topology.¹ In this experiment, we surrounded ZIF-8 with a liquid medium (methanol) in the pressure chamber in order to apply pressure evenly, so as not to crush the sample. On application of pressure, we discovered that we could force the medium to enter the pores, causing the sample to *expand*. On increasing pressure further, the framework underwent a phase transition between 0.96 and 1.47 GPa. This transition was driven by the rotation of the methylimidazole rings which dramatically increased both the available pore space and content.

Here, we present a combined experimental and computational approach to understand the behavior of ZIF-8, while presenting new results on the effect of pressure on ZIF-65 ($\text{Zn}(\text{NO}_2\text{IM})_2$, NO_2IM = 2-nitroimidazolate), to 4.77 GPa which, although topologically similar to ZIF-8, exhibits quite different behavior.

[1] S.A. Moggach, T.D. Bennett, A.K. Cheetham, *Angewandte Chemie, International Edition* **2009**, *48*, 7087-7089.

Keywords: porous, MOFs, high-pressure

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TEM study of pressure-induced C_{60} transformation into ill-ordered graphite phase

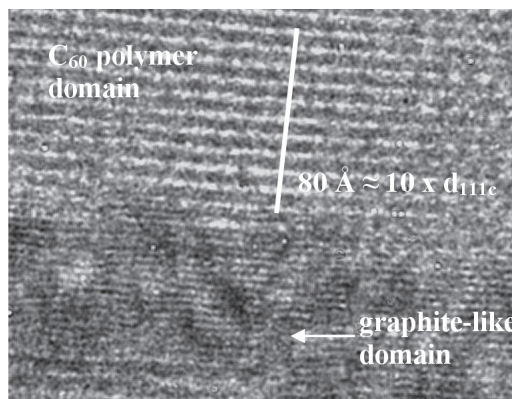
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Fullerenes C_{60} have been intensively studied during these last years because of its capacity of creating numerous phases depending on the pressure and temperature conditions during synthesis[1]. The different C_{60} polymers can adopt 1D, 2D or 3D structures in tetragonal, orthorhombic and rhomboedral systems, these structures having been summarized in a reaction phase diagram as reviewed by Moret in 2004.[2]. An interesting point is the transformation of these phases into ill-ordered “graphite-like” phase above certain values of temperature and pressure[3].

In order to understand the crystallographic relations between polymer and “graphitic” phases, as well as the conformation of the graphite planes on compounds obtained at different pressure conditions, several powder samples have been studied by transmission electron microscopy using electron diffraction (ED) and High Resolution Transmission Electron Microscopy (HRTEM). They were synthesized at more than 1100K in order to achieve the graphite phase transformation under 2 GPa, 5GPa, 8.5 GPa and 10 GPa respectively.

The ED studies for all these samples evidence mostly an ill-ordered graphite-like phase, textured or not, with traces of C_{60} polymer. In order to observe the evolution of interlayer distance between graphite-like planes with pressure, these distances were measured on ED patterns over many particles for each sample. It turns out that it gets shorter with the pressure increase, varying from 3.8 Å at 2 GPa to 3.4Å at 10 GPa. HRTEM imaging displays textured graphite-like undulated planes tangled to each other explaining the texture broadening of the interlayer reflections on ED patterns. On the 5 GPa sample, coexistence of ill-ordered graphite and crystallized C_{60} polymer is observed, both phases getting linked by a pseudo epitaxial relation along the $[111]_c$ direction (c related to cubic, the basic form of C_{60}) of the polymer. This phenomenon was confirmed by HRTEM

imaging showing graphite-like domains in a C_{60} polymer matrix (as seen in the figure below).



[1] J.L. Hodeau, J.M. Tonnerre, B. Bouchet-Fabre, M. Nunez Regueiro, J.J. Capponi, M. Perroux, *Physical Review B*, **1994**, *50*, 14, 10311-10314. [2] R. Moret, *Acta Cryst.*, **2005**, *A61*, 62-76. [3] E.V. Tat'yanin, A.G. Lyapin, V.V. Mukhamadiarov, V.V. Brazhkin, A.L. Vasiliev *J. Phys.: Condens. Matter* **2005**, *17*, 249-256.

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High pressure X-ray powder diffraction study of BaWO₄

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Barium tungstate (BaWO_4) has been shown to be an excellent laser-host material [1] and has been used in the construction of scintillating detectors [2]. In this work we present a study of the structural behavior of BaWO_4 under pressure. X-ray diffraction experiments have been carried out up to 20 GPa. Experiments were performed at the HPCAT (16 IDB beamline) using a diamond-anvil cell and under quasi-hydrostatic conditions - neon used as pressure-transmitting medium. Experiments have shown that the tetragonal ($I4_1/a$) structure remains stable up to 6 GPa. Upon further compression, phase transitions to lower symmetry structures are detected. The obtained results will be compared with previous results obtained using less hydrostatic pressure media [3, 4] and with *ab initio* calculations [3]. Comparison will be done not only for structural sequences but also for bulk and axial compressibilities. In addition, the effects of pressure in the tetragonal distortion of the low-pressure phase and bond compressibility will be discussed. Rietveld refinements of different structures at several pressures will be also reported.

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