

shares the same two stage densification mechanism as quartz. The first one from berlinite to a  $\text{CrVO}_4$  type structure where Al is six-fold coordinated and P still four-fold occurs around 14 GPa. In the final compression step, phosphorous becomes sixfold coordinated by oxygen. This previously unknown phosphorous coordination is of great importance from a fundamental point of view and opens new possibilities on the high pressure chemistry of phosphorus

#### MS22 P10

**The high pressure behaviour of gypsum: a single crystal X-ray study** Comodi P., Nazzareni S., Zanazzi P.F., Dept. of Earth Sciences, Università di Perugia, Italy  
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Gypsum is the most common sulphate mineral with molecular water which may be easily removed from the structure by heating. This can affect the pore pressure inside the rocks, greatly enhancing the probability of large-scale over thrusts. Most studies on gypsum were devoted to understand the dehydration mechanism, while its baric behaviour was less studied as well as the nature of phase transitions observed at different pressures by X-ray powder diffraction [1] and infrared and Raman spectroscopy [2].

A gypsum crystal was loaded at different pressures up to 5 GPa in a Merrill-Bassett DAC with ethanol: methanol mixture as hydrostatic fluid. Both quartz and  $\text{Sm}^{+2}$ : Ba FCI chips were used to monitor the pressure. Single-crystal X-ray diffraction experiments were performed by using a Xcalibur Oxford diffractometer (with a CCD detector for intensity data collection and a point detector for accurate cell parameters measurements).

The bulk modulus determined by fitting the volume-pressure data with a third order Birch-Murnaghan equation-of-state is 44 (1) GPa with  $K' = 3.3$  GPa, and 43(1) GPa when  $K'$  is fixed to 4. The principal linear compression coefficients calculated by STRAIN program [3] up to 4 GPa were  $\varepsilon_1 < \varepsilon_2 < \varepsilon_3$ , with  $\varepsilon_2$  parallel to the  $b$  axis and  $\varepsilon_3$  forming an angle of  $55^\circ$  with the  $a$  axis.

The structural refinements indicate that the  $\text{SO}_4$  volume and the average S-O bond distance remain almost unchanged from room pressure to 3.9 GPa: (in the range  $1.64\text{\AA}^3$  -  $1.66\text{\AA}^3$ ;  $1.473\text{\AA}$  -  $1.481\text{\AA}$ ) while they undergo a sensible distortion: the smaller distance decreases (from  $1.473\text{\AA}$  to  $1.45\text{\AA}$ ) and the larger increases (from  $1.474\text{\AA}$  to  $1.51\text{\AA}$ ). On the other hand the calcium polyhedra show a normal high-pressure behaviour, becoming more regular and reducing the volume (from  $25.84\text{\AA}^3$  at room  $P$  to  $24.67\text{\AA}^3$  at 3.9GPa). The largest variations are observed in the interlayer region where the water molecules are located. The two hydroxyls of the water molecule, under room conditions, form weak hydrogen bonds with the

oxygens of Ca and S polyhedra. Increasing pressure, the weakest hydrogen bond becomes the strongest one.

These structural results indicate that the water remain in the structure also at HP condition and explain raman and infrared spectra. In fact the distortion of sulphate tetrahedra explains the splitting of raman mode and the different compressibilities of the two hydrogen bonds explain the coalescence of the Raman stretching mode observed at HP (Knittle et al. 2001)

The diffraction pattern above 4 GPa changes. Indexing with the  $C2/c$  space group, some  $h+k$  odd reflections appear, the  $a$  and  $b$  lattice parameters, as well as the cell volume, show a discontinuity, while the  $c$  lattice parameter and the  $\beta$  angle, vary linearly.

[1]Huang et al. 2000 *High pressure research*, 17, 57-75,

[2]Knittle et al. 2001 *Phys. Chem. Min.*, 28, 630-640,

[3] Ohashi (1972) Strain tensor calculation program.

#### MS22 P11

**In situ diffraction study of high-pressure transformation of  $\text{C}_{60}$  to disordered  $\text{sp}^2$ -carbon** L. Marques<sup>a</sup>, M.Mezouar,<sup>b</sup> J-L.Hodeau,<sup>c</sup> <sup>a</sup>*Departamento de Física and Cicco, Universidade de Aveiro, Portugal.* <sup>b</sup>*E.S.R.F., Grenoble, France.* <sup>c</sup>*C.N.R.S., Grenoble, France.*  
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$\text{C}_{60}$ -2DR polymer amorphisation under high pressure has been studied by in situ diffraction techniques employing the Paris-Edinburgh press. The initial interest of this study was to find structural signatures of the so-called magnetic carbon phase, which would be formed by the  $\text{C}_{60}$ -2DR polymer phase close to the molecular collapse. Evolution of the transformation process was followed by 2D angular-dispersive diffraction. The recorded diffraction patterns show that the  $\text{C}_{60}$  polymer phase gradually amorphises into a disordered  $\text{sp}^2$ -carbon phase. Changes in the diffraction pattern of the 2DR  $\text{C}_{60}$  polymer during the amorphisation transition, which would be indicative of the magnetic phase, were not perceived. This must be confirmed by detailed data analysis, under way.

Samples quenched at different levels of transformation were recovered in order to perform x-ray diffraction and complementary magnetization measurements. Diffraction patterns of the partial amorphised samples show that both the amorphous  $\text{sp}^2$  carbon and the 2DR  $\text{C}_{60}$  polymer are highly oriented. This indicates an orientational relationship between the parent polymeric structure and the amorphous transformed structure: the graphitic planes and  $\text{C}_{60}$  polymerized planes have a defined orientational relationship typical of martensitic transformations. Therefore  $\text{C}_{60}$  molecules amorphise in a way that does not involve the complete collapse of the cage structure, in contrast to what one would expect. Detailed data analysis is under way in order to obtain the full orientational relationship between parent and transformed structures.