

incommensurate with each other along one direction. The group-V elements Bi, Sb and As are shown to have similar host-guest structures in their high-pressure phases [3], where both components exhibit displacive modulations [4].

Using synchrotron x-ray diffraction and diamond anvil cells, we study the host-guest structures of Sb and As under pressure [5-6], and find an incommensurate-to-incommensurate phase transition with change in symmetry from monoclinic to tetragonal in both host and guest components. In our Raman spectroscopy studies on lattice dynamics of these metallic phases we observed five modes with the frequencies in the range of 90-200  $\text{cm}^{-1}$  for Sb, shifting to higher values with pressure increase. We analyze the Raman modes with the help of first-principles calculations for commensurate approximants [6].

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**Keywords:** high pressure phases, Raman spectroscopy, incommensurate structures

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#### High-pressure Behavior of Feldspathoids: the Case of Analcite

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Feldspathoids are low silica minerals and, similar to zeolites, have large openings in the crystal structure. Elastic and structural behaviour of a natural cubic feldspatoid analcite ( $\text{NaAlSi}_2\text{O}_6 \cdot \text{H}_2\text{O}$ ) was investigated up to 8.5 GPa by *in situ* single-crystal X-ray diffraction. A first-order phase transition was observed at  $P = 0.98 \pm 0.07$  GPa. Lattice parameters and reflection conditions show that the HP-polymorph has a  $P\bar{1}$ , Sp. Gr. Volume data of the low- $P$  (cubic) and high- $P$  (triclinic) polymorphs were fitted with a second- and third-order Birch-Murnaghan Equation of State [1], respectively. The refined elastic parameters are:  $V_0 = 2571.2(4)\text{\AA}^3$ ,  $K_{T0} = 56(3)$  GPa and  $K' = 4$  (fixed), for the cubic polymorph,  $V_0 = 2613(10)\text{\AA}^3$ ,  $K_{T0} = 18(1)$  GPa and  $K' = 7.2(7)$ , for the triclinic polymorph. The elastic behaviour of the HP-polymorph, calculated on the basis of the linearised bulk moduli, appears to be strongly anisotropic ( $K(a):K(b):K(c) = 2.07:1.36:1.00$ ). Tetrahedral tilting produces the main deformation mechanism in response of the cubic  $\rightarrow$  triclinic phase transition. The distortion of the secondary building units gives rise to a change of the 8- and 6-ring channels ellipticity. As a consequence, the extra-framework topological configuration changes: it appears in fact that the coordination number of part of the Na atoms becomes 7 ( $2\text{H}_2\text{O} + 5$  framework oxygens) instead of 6 ( $2\text{H}_2\text{O} + 4$  framework oxygens).

[1] Birch F., *Phys. Rev.*, 1947, **71**, 809.

**Keywords:** analcite, high-pressure, compressibility

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#### New High-pressure Forms of Simple Salts-sulfates, Formates, and Acetates

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Over the last few years, we have been studying the effects of high pressure on the structures of a variety of molecular compounds that include: simple organic compounds [1], pharmaceuticals [2], amino acids [3], and simple inorganic compounds such as the oxoacids and their hydrates [4]. All of these systems have been studied principally with single-crystal x-ray diffraction techniques in combination with diamond-anvil cells (DAC). Methods for studying single crystals in

DACs include growth of single crystals *ex situ* followed by loading into the DAC or growth of single-crystals *in situ* from the melt. Both of these methods suffer from disadvantages and so we have recently developed methods for the high-pressure *in situ* growth of single crystals from solution [Ref]. Using these methods, we have studied the high-pressure recrystallisation of the sodium salts of the simple carboxylic acids, formic acid and acetic acid, and of the sodium salt of sulfuric acid. All of these compounds form previously unobserved hydrate phases at high pressure. For the new sodium sulfate hydrate phase, the growth of the single-crystal occurred *via* a high-pressure/high-temperature chemical reaction and its structure is certainly the most complex of all five known phases of  $\text{Na}_2\text{SO}_4$ , or its two previously observed hydrates,  $\text{Na}_2\text{SO}_4 \cdot 7\text{H}_2\text{O}$  and  $\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$ . These sulfates are all geologically relevant and so the identification of this new high-pressure phase is likely to be highly significant.

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**Keywords:** high-pressure, crystal structure, small molecule

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#### Hydrogen Storage in Molecular Compounds

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At low temperature ( $T$ ) and high pressure ( $P$ ), gas molecules can be held in ice cages to form crystalline molecular compounds that may have application for energy storage. We synthesized a hydrogen clathrate hydrate,  $\text{H}_2(\text{H}_2\text{O})_2$ , that holds 50 g/liter hydrogen by volume or 5.3 wt %. The clathrate, synthesized at 200–300 MPa and 240–249 K, can be preserved to ambient  $P$  at 77 K. The stored hydrogen is released when the clathrate is warmed to 140 K at ambient  $P$ . Low  $T$  also stabilizes other molecular compounds containing large amounts of molecular hydrogen, although not to ambient  $P$ , e.g., the stability field for  $\text{H}_2(\text{H}_2\text{O})$  filled ice (11.2 wt % molecular hydrogen) is extended from 2,300 MPa at 300 K to 600 MPa at 190 K, and that for  $(\text{H}_2)_4\text{CH}_4$  (33.4 wt% molecular hydrogen) is extended from 5,000 MPa at 300 K to 200 MPa at 77 K. These unique characteristics show the potential of developing low- $T$  molecular crystalline compounds as a new means for hydrogen storage.

**Keywords:** hydrogen storage, molecular compounds, high pressure synthesis

#### MS48 MICROBEAM X-RAY SCATTERING

**Chairpersons:** Christian Riekkel, Atsuo Iida

#### MS48.27.1

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#### Microbeam Diffraction of Hierarchical Nanocomposites

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Advanced composite materials with optimized mechanical properties are often hierarchically structured from the atomic/molecular level up to macroscopic length scales. Typical examples are biological tissues such as bone or wood, but also many complex technical composites, which often benefit from the imitation of natural materials by biomimetic principles or by biotemplating. Structural investigations of such materials require new experimental techniques with a position resolution covering several length scales. Beside electron microscopy, small- and wide-angle X-ray scattering (SAXS/WAXS) are well suited to study structural features in the nanometer regime. The high brilliance of third generation synchrotron radiation sources together with novel X-ray optics can be used to extend the position resolution to the micrometer regime by using microbeam scanning techniques in combination with SAXS/WAXS.

The present contribution reviews some recent experimental studies