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-pressures 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 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 ($NaAlSi_2O_6 \cdot H_2O$) 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±0.07GPa. 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)Å^3, K_{T0}= 56(3) GPa and K'= 4 (fixed), for the cubic polymorph, V_0= 2613(10)Å^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→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 ($2H_2O$ + 5 framework oxygens) instead of 6 ($2H_2O$ + 4 framework oxygens).

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Keywords: analcite, high-pressure, compresibility

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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 Na_2SO_4, or its two previously observed hydrates, $Na_2SO_4.7H_2O$ and $Na_2SO_4.10H_2O$. 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, $H_2(H_2O)_2$, 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 $H_2(H_2O)$ 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 $(H_2)_4CH_4$ (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 Riekel, 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

from different complex materials with scanning microbeam SAXS/WAXS. Results from the imaging of nanostructural parameters such as shape, size and orientation of nanoscale inhomogeneities in bone and other hierarchical biocomposites are presented. Moreover, a unique combination of *in-situ* bending deformation with X-ray nanobeam scanning is demonstrated for single carbon fibres.

Keywords: bone, carbon materials, small-angle scattering

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Engineered Bone from Bone Marrow Stromal Cells: a Structural Study by an Advanced X-ray Microdiffraction Technique

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One of the most recent therapeutic strategies for the reconstruction of damaged large bony segments includes the tissue engineering approach. It takes advantage of the patient's own cells, which are isolated, expanded in vitro, loaded onto a bioceramic scaffold and reimplanted into the lesion site. Bone marrow stromal cells (BMSC) are the most commonly used cell type.

A structural characterization of the engineered bone is largely desirable. An important point is to evaluate if the BMSC extracellular matrix deposition on a bioceramic scaffold recapitulates the ontogeny of the natural bone development. Moreover the investigation of the interaction between the newly deposited bone and the scaffold results particularly interesting. Indeed the chemistry and the geometry of the scaffold used to deliver BMSC in the lesion site determine spatial organization of the new bone and the bone-biomaterial integration.

We investigated for the first time the local interaction between the newly formed mineral crystals in the engineered bone and the biomaterial by means of microdiffraction, using a set-up based on an X-ray waveguide. We demonstrated that the newly formed bone is well organized inside the scaffold pore, following the growth model of natural bone, and that there is a good adhesion with the scaffold. Combining Wide Angle (WAXS) and Small Angle (SAXS) X-ray Scattering with high spatial resolution, we were able to determine the orientation of the crystallographic c-axis inside the bone grains, and the orientation of the mineral crystals and collagen micro-fibrils with respect to the scaffold. Moreover from a quantitative analysis of both the SAXS and WAXS patterns the grain size appears to be compatible with the model for early stage mineralization.

Keywords: SAXS, WAXS, bone mineralization

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Polychromatic Microdiffracion Measurements of Mesoscale Structure and Dynamics

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Polychromatic x-ray microdiffraction combined with differential aperture microscopy is a powerful new method for studying the local crystallographic structure of materials. This approach extends single-crystal methods to virtually all materials including materials characterized by heterogeneity at the atomic *and* mesoscopic length scales. Defects such as grain boundaries, surfaces, precipitates, second phases, strain, dislocations, vacancies, interstitials, site substitutions and other disruptions of perfect periodicity all have signatures best studied using single crystal methods. Here we describe emerging x-ray microbeam techniques that exploit "single-crystal like" x-ray diffraction measurements on subgrains in typical polycrystalline materials. We show how polychromatic and tunable monochromatic measurements on small sample volumes can bring single-crystal techniques to real materials and reveal their atomic and mesoscopic defect structures. This emerging revolution in materials science is certain to address long-standing issues of materials behavior.

Keywords: phase, texture, strain

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SR X-ray Microdiffraction Systems at SPring-8: Present Status and Applications

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In Hyogo beamline of SPring-8, we are developing research programs using an x-ray microbeam for applications to the wide range of science. Two types of the x-ray microbeam system have been developed; one is for a high spatial resolution and the other is for a high angular resolution. The former uses a Fresnel zone plate (FZP) for the x-ray focusing element, and the sub-micrometer beam is available [1]. By adopting a narrow slit in front of the FZP, a microbeam with a relatively small angular divergence can be also available [2]. The system is used, for example, for measurements of strain distribution in laser diodes with a è-2è diffractometer and structural analysis of polymers with a diffractometer using imaging plate detectors. The smallest beam size ever achieved is 70 nm in FWHM at 10 keV. The latter system uses a total reflection focusing mirror with a bent-cylindrical shape and the microbeam possesses very a small angular divergence of several arcsec with the beam size of a few micrometers [3]. The system is used for the estimation of local crystallinity of various semiconductor crystals for electronic devices. The both systems are opened for many kinds of users.

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Keywords: synchrotron radiation instrumentation, synchrotron X-ray diffraction, X-ray microanalysis

MS48.27.5

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Hard X-ray Nanoprobe with Refractive X-ray Lenses

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A hard x-ray nanoprobe based on refractive x-ray lenses allows one to apply hard x-ray analytical techniques, such as diffraction or fluorescence analysis, with high spatial resolution. This is particularly useful to investigate heterogeneous samples in materials, environmental and life science.

Using nanofocusing lenses (NFLs) [1-2], a nanobeam with a lateral extension down to 50 x 50 nm^2 is currently feasible at third generation synchrotron radiation sources in the hard x-ray range. The beam divergence of about 1 mrad is sufficient for many diffraction experiments. The beam size can be expected to be reduced to below 10 nm in the future [3].

We report on nanodiffraction experiments in materials science performed at beamline ID-13 of the ESRF. The coherence properties of the nanobeam are discussed in view of diffraction from small objects with coherent radiation.

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