

pressure, and it will be demonstrated that the guest-atom chains in the composite Rb-IV structure represent a realisation of the classic monatomic linear chain model. In modulated Te-III, a pronounced phonon anomaly is observed that will be discussed in the context of Fermi-surface nesting, Kohn anomalies and charge-density waves.

This work was performed in collaboration with M. I. McMahon, L. F. Lundegaard, S. R. Evans (University of Edinburgh) and A. Bossak, M. Krisch (ESRF, Grenoble).

Keywords: incommensurate crystals, lattice dynamics, inelastic X-ray scattering

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Phase transformations in silane — Hydrogen-dominant material

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The metallization of hydrogen directly would require pressure of 400 GPa, out of the reach of present experimental techniques. The dense group IVa hydrides attract considerable attention because hydrogen in these compounds is chemically precompressed and a metallic state is expected to be achievable at experimentally accessible pressures(1). We report the transformation of insulating molecular silane to a metal at 50 GPa, becoming superconducting at a transition temperature of $T_c = 17$ K at 96 and 120 GPa(2). The metallic phase has a hexagonal close-packed structure with a high density of atomic hydrogen, creating a three-dimensional conducting network. These experimental findings support the idea of modeling metallic hydrogen with hydrogen-rich alloy. The metallic $P6_3$ phase is apparently stable in the 50 to 110 GPa range. However it partly transforms into a transparent insulating phase at pressures ≥ 120 GPa. Concurrent with the onset of transparency, a pronounced Raman signal appears. Two distinct phases coexist to the highest experimental pressure of 192 GPa. We determined the transparent phase structure to be a $I4_1/a$ structure. The structure and its lattice parameters are in exact agreement with the $I4_1/a$ phase predicted to be thermodynamically stable in the pressure range 50 to 250 GPa(3). The observed transformations in stoichiometric silane with pressure are unusual. Molecular SiH_4 at a pressure of ~ 50 GPa does not transform to the predicted thermodynamically stable $I4_1/a$ phase(3) but instead collapses to a significantly denser $P6_3$ phase.

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Structure of drug-target proteins determined by both X-ray and neutron diffraction

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Crystallography enables us to obtain accurate atomic positions within proteins. High resolution X-ray crystallography provides

information for most of the atoms comprising a protein, with the exception of hydrogens. Neutron diffraction data can provide information of the location of hydrogen atoms to the structural information determined by X-ray crystallography. Here, we show the recent result of the structural determination of drug-target proteins, porcine pancreatic elastase (PPE) and human immunodeficiency virus type-1 protease (HIV-PR) by both X-ray and neutron diffraction. The structure of porcine pancreatic elastase with its potent inhibitor (FR13080) was determined to 0.94 Å resolution by X-ray diffraction and 1.75 Å resolution by neutron diffraction. It was found that there are two characteristic hydrogen bonding interactions in which hydrogen atoms were confirmed. One is located between a catalytic aspartate and histidine, another is involved in the inhibitor recognition site. The structure of HIV-PR with its potent inhibitor (KNI-272) was also determined to 0.93 Å resolution by X-ray diffraction and 2.3 Å resolution by neutron diffraction. The ionization state of the catalytic residues were clarified to show that Asp125 is protonated and Asp25 is deprotonated. The ionization state and the location of hydrogen atoms of the catalytic residue in HIV-PR were firstly determined by neutron diffraction. Furthermore, collaborative use of both X-ray and neutron to identify the location of ambiguous hydrogen atoms will be shown.

Keywords: neutron diffraction, protein, structure

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Neutron crystallographic analysis of deuterated and selectively CH_3 -protonated deuterated rubredoxin

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Neutron crystallography is used to locate hydrogen atoms in biological materials and can distinguish between negatively scattering hydrogen and positively scattering deuterium substituted positions in isomorphous neutron structures. Recently, Hauptman and Lang (2003) have shown that neutron diffraction data can be used to solve macromolecular structures by direct methods and that solution is aided by the presence of negatively scattering hydrogen atoms in the structure. Selective labeling protocols allow the design and production of group or residue specific H/D-labeled macromolecular structures in which the ratio of hydrogen to deuterium atoms can be precisely controlled. We have applied labeling protocols to selectively introduce protonated methyl groups into deuterated rubredoxin from *Pyrococcus furiosus* (PfRd). Perdeuterated and selectively CH_3 -protonated, deuterated rubredoxin were crystallized. High quality neutron data sets extending to 1.75 Å resolution were collected on the new LADI-III instrument at the Institut Laue-Langevin. Of special importance, the 1.75 Å data from the perdeuterated crystal required just 14 hours of beam time, a record which heralds a new era in neutron protein crystallography. We will present the production, crystallization, and neutron analysis of the perdeuterated and selectively CH_3 -protonated PfRd.

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