The wavefront of vibrational energy emanating from a point disturbance within the crystal is far from spherical, as classically observed in fluids. Although the mathematics of elastic-wave propagation in an anisotropic medium has been known for decades, experimental observation of a vibrational wavefront in a small (micro- or nano-sized) crystal has only been here achieved.

Based on the weird behaviour of acoustic phonons in anisotropic media like crystals, we here present the first development of phonon imaging at high pressure by means of picoseconds acoustics in diamond anvil cell, an advanced and unconventional technique to probe (with a pulsed laser) the structure and elastic behaviour of thin solids under extreme conditions.

Our method gives snapshots that portray the acoustic ray which provides an immediate indication on the complete elastic properties of thin compounds and its evolution under extreme mechanical condition.

The example of single-crystalline silicon up to 10 GPa is presented as a case study [1].

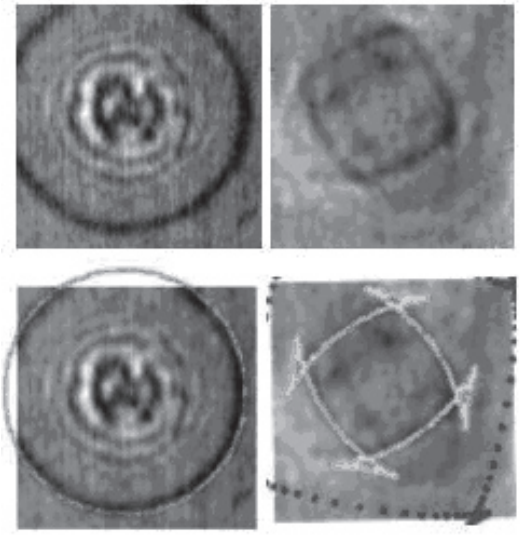

Figure : Top : experimental phonon imaging patterns in the (100) plane of silicon at 7.75 GPa at two different pump-probe delays. Bottom : same as top with superimposed calculation curves for longitudinal, fast and slow transversal group velocities (red, blue and green dashed lines respectively) using C11= 196.9 GPa, C_{12}= 104 GPa and C_{44}= 80 GPa.

[1] F. Decremps, L. Belliard, M. Gauthier, B. Perrin, *Phys. Rev. B* **2010,** *82,* 104119.

Keywords: phonon, pressure, elasticity

MS.47.2

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High-pressure neutron diffraction at the SNS

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To date, the role of neutron diffraction in high pressure research has been relatively peripheral, with the vast majority of structural studies using synchrotron-based x-ray diffraction. This is despite well known (and not so well known) advantages of neutrons for crystallography, including: sensitivity to positions of light atoms (in particular H^2, Be, B^{11}, C, N and O), exceptional real-space resolution and the ability to measure long-range magnetic order.

The principle reason for the lack of neutron science under extreme pressure conditions is the intrinsic weakness of available sources and the corresponding need for large sample sizes. To put this in context, current typical sample volumes for neutron powder diffraction are of order 25mm^3 compared with 0.0025mm^3 for diamond-anvil cell synchrotron diffraction. The realisation of such small volumes for neutron diffraction would greatly expand the capabilities of this unique probe.

Recently, the neutron landscape changed with the opening of the 'next generation' 1.4 MW Spallation Neutron Source (SNS) at Oak Ridge National Laboratory. Since then, a collaboration between the Geophysical Laboratory (GL) and the SNS has sought to exploit the SNS's unrivalled, pulsed flux of neutrons for high-pressure crystallography. A key component has been the incorporation of many aspects of GL's experience in high-pressure synchrotron beamlines onto the high-pressure neutron diffractometer, SNAP. This has been coupled with a new generation of diamond anvil cell (DAC) technology tailored to optimize sample volume, and minimise background scattering.

We will present the most recent achievements of this collaboration: diffraction measurements from powder samples, at pressure, inside DAC's with volumes below 0.1mm^3. We will also show the latest attempts to exploit these small volumes to achieve unprecedented pressures for neutron crystallography.

Keywords: neutron, pressure, diffraction

MS.47.3

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Towards the use of Laue microdiffraction intensities for structural studies at extreme conditions

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Single crystal X-ray diffraction is a most powerful technique to decipher the atomic structure of crystalline material. In its most mature and most precise flavor, monochromatic single crystal X-ray diffraction, the reciprocal lattice of a mounted crystal is rotated through a single Ewald sphere, representing one narrow band pass energy. The drawbacks of monochromatic single crystal X-ray diffraction are the need to rotate the sample through the Ewald sphere and - linked to this - the relatively time consuming data acquisition. The need for a mobile sample makes this technique ill suited for in situ studies such as e.g. laser heated diamond anvil experiments. One possible alternative for such situations is Laue diffraction. By using a broad band-pass X-ray source such as provided for example by a Synchrotron bending magnet, a large number of reciprocal lattice points can be simultaneously imaged in a single exposure.

The potential of Laue diffraction for time resolved in situ experiments has been recognized and exploited by the protein community. In their approach, the Laue specific problems were addressed by the large data redundancy stemming from the large unit cells of typical protein crystals. This approach is not applicable to inorganic substances with relatively small unit cells. We devise ways to extract the integrated intensities from a Laue pattern in cases where high reflection redundancy cannot be achieved. In order to properly interpret intensities, various specific issues such as energy dependent correction factors (absorption, Lorentz

coefficient) and a precise evaluation of the effective incident spectrum have to be addressed.

We determined a transferable effective flux curve by comparing the measured Laue intensities of a well characterized standard crystal (calcite) with the corresponding calculated structure factors [1]. The thus derived normalized flux curve proved to be transferable between different mineral structures. First applications involving the unambiguous indexation of primitive trigonal unit cells as well as work to enable routine structure solution and refinement based on intensities derived from Laue diffraction experiments will be discussed.

[1] C. Dejoie, M. Kunz, N. Tamura, C. Bousige, K. Chen, S. Teat, C. Beavers, C. Baerlocher, *J. Appl. Crist.* **2011**, *44*, 177-183.

Keywords: laue microdiffraction, structure solution, inorganic material

MS.47.4

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High pressure freezing of protein crystals

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The standard method to reduce radiation damage on biological samples is cryo cooling to cryogenic temperatures by immersing them in liquid nitrogen or a cold nitrogen gas stream [1]. Protein crystals typically contain up to 90% of solvent. In order to suppress the formation of crystalline ice upon cooling, which destroys the crystal lattice, cryoprotectants such as ethylene glycol or glycerol have to be applied. Finding a suitable cryoprotectant for a specific crystal is a very time and crystal consuming trial and error process. Moreover, the crystal quality is often degraded upon flash-cooling even if adequate cryoprotectants have been found. Such degradation manifests itself in an increase of the crystal mosaicity and a decrease in observable diffraction resolution which finally limits the ability to phase the structure.

High pressure freezing (HPF) allows cryogenic cooling of macromolecular samples by application of high pressures and low temperatures without formation of hexagonal ice and avoiding penetrative cryoprotectants [2-6].

A HPF protocol for several test proteins, *e.g.* thaumatin, hen egg-white lysozyme and porcine insulin, has been developed and established. For that purpose protein crystals are grown in cellulose carbonate microtubes *via* dialysis or in glas capillaries using counter diffusion techniques [7] to facilitate sample handling during the freezing procedure. Subsequently, the samples are frozen at 210 MPa while being cooled to liquid-nitrogen temperatures using a Bal-Tec HPM 010 instrument.

First X-ray diffraction experiments revealed a superior quality of the high pressure frozen samples. Due to the formation of high-density amorphous (HDA) ice, HPF crystals were diffracting to higher resolution and showed better R values compared to normally flash-cooled samples.

For the first time data sets of freeze substituted protein crystals have been successfully collected. Freeze substitution was performed on high pressure frozen lysozyme crystals at 183 K using ethanol as solvent. These crystals showed a different crystal symmetry and an increased cell volume compared to unsubstituted samples.

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Keywords: protein crystallography, high pressure, cryo cooling

MS.47.5

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Synchrotron X-ray diffraction tomography technique using diamond anvil cell

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The joint effort from multiple user groups and beamlines in Advanced Photon Source, Argonne National Laboratory will be introduced for the development of synchrotron x-ray diffraction tomographic method combined with high pressure diamond anvil cell (DAC) technique, which offers great opportunity for the deeper understanding on phase transition behaviors of materials under high pressure extreme conditions. These studies will broaden our horizons and perspectives of the states of materials upon compression. The procedure of the pressure-induced amorphous state to amorphous state transitions, crystallization from amorphous state under pressure, as well as the phase transition and equation of state studies for simple metal powder samples, such as iron, silver and titanium, was probed using the novel techniques. The structural evolution of amorphous materials, as well as powder samples under high pressure conditions up to couple ten GPa pressure range were studied by synchrotron x-ray diffraction and imaging tomography methods in 3-D space domain in the DAC. These novel researches will provide new insight on the nature of phase transition, highlight the relationship of deviatoric stress and anisotropic elastic strain in 3-D space between the old and new phases during these phase transition processes, provide new invitation for the electronic theoretical studies for the phase stability, and improve our understanding three dimensionally of the kinetic process of the crystallization, phase transition path and mechanism in various type materials at high pressure extreme conditions.

Keywords: high pressure, diffraction tomography, phase transition

MS.48.1

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Probing the electronic structure of correlated electron systems with synchrotron light

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When other methods for mapping the Fermi surface are excluded (for example, owing to sample quality, concerns about the surface, substitutional disorder, or simply the temperature at which the phase of interest exists), then the Fermi surface can be accessed through