

# Phonon Dispersion of Samples in Diamond Anvil Cells at SPring-8: meV-resolution IXS to 3000K / 300 GPa.

Alfred Q.R. Baron

[baron@spring8.or.jp](mailto:baron@spring8.or.jp)

Materials Dynamics Laboratory, RIKEN SPring-8 Center & Precision Spectroscopy Division, CSRR, SPring-8/JASRI

RIKEN's Materials Dynamics Laboratory started a program in 2006 to measure phonons (sound velocities) of samples in diamond anvil cells via meV-resolution inelastic x-ray scattering (IXS) [1,2,3]. Single crystal work was spearheaded by H. Fukui and co-workers, who applied Christoffel's equation to efficiently extract the elastic constants from IXS data [4]. This approach has been applied to multiple samples at room temperature and at pressures up to 54 GPa [5-10]. The method is mostly limited by the difficulty of preserving crystal quality at high pressures. In parallel, work was started with E. Ohtani and co-workers to measure powder (polycrystalline) samples of geologically relevant materials – iron compounds/alloys - at pressures and temperatures that are comparable to the Earth's core. This has included measurements of pure iron at both high T and high P with T. Sakamaki *et al.*, [11,12] (up to P~163 GPa at 3000K) and we recently were able to exceed 300 GPa at room temperature [13]. Various iron compounds have also been investigated [14-19]. More recently, K. Hirose began a program to measure liquids at high pressure. First work by Y. Nakajima *et al.* on Fe<sub>84</sub>C<sub>16</sub>[20] demonstrated that liquid measurements were possible up to 70 GPa. In fact, liquids are especially challenging since liquids tend to migrate in the DAC, so a sapphire cell is constructed *inside the DAC* to stabilize their position. That approach has been extended to investigate pure iron and other liquids [21-24]. Recently, D. Ikuta, *et al.*, measured phonons in rhenium to >200 GPa [25]. The observation of a transverse acoustic (TA) mode peak allows construction of a primary pressure scale. That new scale suggests previous work significantly over-estimated pressures (e.g., by 20% at 230 GPa) implying the light element content of the Earth's core may be underestimated by a factor of 2.

The talk will discuss some of these results in the context of the instrumentation that has made them possible. IXS [1] work with DACs at SPring-8 began at BL35XU [2] where, at present, a 4.5 m, short period insertion device provides high flux into a <20 μm diameter spot created using compound focusing [26]. The BL35 spectrometer has 12 independent analyzer crystals and a flat panel area detector for powder diffraction and crystal alignment. More recently, most DAC work migrated to BL43LXU [3] which has leading flux from 3x5m IDs [27], a 5x5 μm<sup>2</sup> beam size, and, now, up to 28 active analyzers. The small beam size (the smallest now available for IXS world-wide) is made using a carefully designed KB multilayer mirror pair [28] that specifically targeted experiments above 150 GPa. Another critical component for measurements at higher pressures is what we call a "Soller screen" [28]. This acts similarly to a Soller slit, but is straightforward and relatively inexpensive to make even with multiple channels on a ~60 μm pitch. While it takes some experience to align, the Soller screen strongly improves the signal to noise ratio, reducing the background from the diamond anvils, though at the cost of reducing the number of active analyzers from 28 to 16. There are two different setups for laser heating [29,20], as are run by specific user groups. There is also a membrane pressure setup, and a variety of off-line tools available for DAC experiments, including, e.g., Raman pressure measurement, a laser drill for gasket (including Be gasket) preparation, *etc.*

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