# Multi-Anvil High Pressure Beamlines at the NSLS II (XPD) and the APS (6BMB): COMPRES Annual Report

November 2016 – November 2017

Prepared by Donald J. Weidner, Matthew L. Whitaker, Haiyan Chen

Mineral Physics Institute, Stony Brook University, Stony Brook, NY 11794-2100

# Table of Contents

Table of Contents 1

Overview 2

Scientific Highlights 3

Beamline Personnel 4

Beamline Operations 4

APS 6-BM-B: 4

6-BM-B Beamtime Statistics: 4

NSLS-II XPD-D: 5

XPD-D Beamtime Statistics: 5

Beamline Performance Metrics 5

Beamline Community Activities 6

Beamline Development 6

DT25 6

XPD-D Development Timeline: 7

XPD-D Development Highlights: 10

Planned Development Activities 11

6-BM-B 11

XPD-D Development Timeline 11

Budget 12

Budget Justification 12

Appendices 13

Appendix 5 16

## Overview

Here we present the annual report for the combination of beamlines 6BMB at the APS and XPD-D at the NSLS II. We do this realizing that we have recently submitted a proposal for the continuation of 6BMB, but with the spirit of providing a complete annual report of the year’s activity. Thus, there will be some overlap between the two documents.

6BMB has completed a stable year of beam operation, with full implementation of the ultrasonic system, providing new data to a range of users. XPD-D remains on track to its first *in situ* high pressure experiment in early December. This will lead to commissioning experiments early next year and will be open to general users by mid-2018.

At the time that the NSLS closed down, this program at X17 was, by most measures, the world – leading synchrotron beamline for measuring mechanical properties of Earth materials. It used about 67% of available time, employing 2 beamline scientists, with significant support from Stony Brook University. The hutch was equipped with a DDIA or RDA on a white beam and an experimental DT10 on a monochromatic side station. By mid-year at the NSLS II and APS we will provide a white beam for the DDIA and RDA (at 6BMB) and a monochromatic beam for a new DT25 at XPD at the NSLS II. The total beamtime will be similar if not more than we had at the NSLS, and the total beamline scientist support from COMPRES will be the same as at the NSLS. Our program will be divided between two beamlines in much the same manner as the multi-anvil program at GSECARS, where one beamline provides a stable platform for many experiments (6BMB) and the other uses a world class beam (XPD) with a unique high pressure apparatus (DT25) allowing a new range of experiments. While the responsibilities of the beamline scientists are mostly divided with Haiyan Chen at APS and Matthew Whitaker at NSLS II, in fact, Whitaker spends about 25% of his time at APS assisting users and working to maintain the 6BMB system and Chen participates at NSLS II when we need ‘all hands’ available for focused projects.

Mechanical properties, as we use it here, represent properties that reflect the relationship between stress, strain, and time. The elastic properties that define elastic wave speeds stand at one end of the spectrum, where no permanent deformation is inflicted on the sample, while plastic flow laws represent the other end with the sample shape changing through time. The elastic properties translate to seismic velocities and are the necessary basis that turns seismic diagrams, coded in red and blue, into temperature or compositional gradients, or that allow us to map out phase transitions as a function of depth. Plastic properties are the agent of flow that enables plate tectonics on the one hand, and leads to earthquakes on the other.

The current facility (including 6BMB and XPD) represents the leading edge for mechanical studies in Earth sciences at this time. While we were the developers of the first high pressure, synchrotron based, deformation machine (the DDIA), many other synchrotrons have adapted this system. However, we are the only beamline program using the RDA (rotational Drickamer apparatus), which can reach lower mantle conditions and produce very high sample strains, and we will also be the only beamline using the DT25 which should also reach lower mantle conditions. We are the only deformation program using a conical slit for the cleanest diffraction patterns for stress measurements. We have the first ultrasonic system capable of measurements in lab times on the order of a second. We are the first lab program measuring elastic moduli and Q at frequencies between 1 Hz and 1 mHz (Spring8 has recently adapted our technique) into the multiple GPa range. In all of these cases, our designs have been published and other synchrotron facilities can and often do copy them. Our position in the field results from continued enhancement of experimental capabilities and is driven by the user community.

The 6-BM-B Beamline at the Advanced Photon Source (APS), Argonne National Laboratory, is a bending magnet "white beam" beamline (~20-100 keV) utilizing energy dispersive diffraction (EDD) and X-radiographic imaging techniques in mineral physics and Earth materials studies. This beamline is a dedicated high pressure multi-anvil facility equipped with a 250-ton hydraulic press with DIA, Deformation-DIA, and T-cup pressure modules, a Canberra 10-element Ge solid state detector array with conical slit system, and a Prosilica CCD camera for X-radiographic imaging. This beamline provides experimental capabilities for studying hydrostatic and dynamic phase transformations, equations-of-state, melting processes, steady-state and dynamic rheological properties, as well as transport, thermal, elastic, and acoustic properties to COMPRES affiliated users in United States and abroad.

We are building an endstation in hutch D of the XPD beamline at the NSLS II. XPD-D is located on an insertion device beamline at NSLS-II, designated 28-ID-2-D, and is served by a 1.8 Tesla Damping Wiggler source. This beamline is designed to make use of monochromatic X-ray beam, which is tunable in energy from ~30-70 keV. The most common energies used for operations are 52 and 67 keV. This high energy will be advantageous since it is very penetrative and the diffraction signal will cover a small two-theta region, yielding more diffraction rings in a limited two-theta aperture. We use a Perkin-Elmer (1621 CN3 EHS) flat panel detector, CsI phosphor directly deposited on amorphous silicon, 2048x2048 pixels with 200x200 micrometer pixel size, 15 frames per second readout at 2048x2048, and 30 fps at 1024x1024. We also collect X-radiographic imaging of the sample during experiments by focusing a Point Gray CCD camera on a YAG scintillator.

In the XPD-D endstation, we have installed a 1100-ton hydraulic press designed for high pressure and high temperature multi-anvil experiments. The standard pressure module that will be in use in this press is the DT-25, which is a differential Kawaii-type multi-anvil system that is capable of achieving pressures ranging from the crust through the transition zone to lower mantle pressures. This module is the first of its kind in the world, and will permit new experiments not possible at any other beamline. A more standard D-DIA module is also available for use in this press. Both the DT-25 and the D-DIA have separately actuated top and bottom anvils that are driven by separate differential hydraulic pumps to allow studies involving differential stress and strain and sample deformation mechanisms.

## Scientific Highlights

6BMB has already supported a number of scientific accomplishments and discoveries at the beamline. Our user program, in particular, has significantly improved understanding of flow in the upper mantle by characterizing the rheology of olivine and pyroxene at appropriate pressures and temperatures, characterizing texture development, and studying the effects of changing chemistry including water and iron. The rheology of and texture development in wadsleyite and ringwoodite have also been characterized and an important strength contrast between bridgmanite and (Mg,Fe)O has been discovered. The rheology of other planetary materials such as ilmenite-olivine aggregates and Fe-rich olivine have also been investigated. These studies have implications for mantle dynamics (on Earth as well as for the Moon and Mars), the formation and isolation of geochemical reservoirs and the distribution of seismic anisotropy. Recent studies of other transport properties such as changes in the thermal diffusivity caused by the perovskite to post-perovskite phase transition also have important implications for mantle dynamics. Studies of solid-melt interactions throughout the mantle have implications ranging from the degree to which molten iron in the outer core penetrates into the mantle to the degree to which dynamic partial melting causes seismic wave attenuation. Other studies span a wide range of topics from brittle deformation of quartz to the formation of tissinite in meteorites. Several one-pagers are attached as an appendix.

## Beamline Personnel

Donald J. Weidner – Program P.I. 0% COMPRES / 100% Stony Brook

Matthew L. Whitaker – Co-P.I. & Beamline Scientist 100% COMPRES / 0% Stony Brook

Haiyan Chen – Beamline Scientist 100% COMPRES / 0% Stony Brook

Michael T. Vaughan – Scientific Staff Associate 0% COMPRES / 100% Stony Brook

Kenneth J. Baldwin – Software and Controls Specialist 0% COMPRES / 100% Stony Brook

William R. Huebsch – Electronics Design and Support 25% COMPRES / 75% Stony Brook

Richard S. Triplett – Graduate Student 0% COMPRES / 100% Stony Brook

## Beamline Operations

One shift of beamtime at both NSLS-II and APS is 8 hours. Both 6-BM-B and XPD-D are on a shared beamline where only one endstation can be active at a time, so there is no parasitic or tandem mode availability. Beamtime statistics can be found in the accompanying appendix.

### APS 6-BM-B:

In the 3 cycles of 2016-2017 (2016-3, 2017-1, 2017-2), 6BM-B has 322 shifts, ~52% from APS, of which 295 shifts, or 91.6% were available to users. Over-subscription rate is 127%. COMPRES assigned ~64% of beamtime and APS assigned ~34% of beamtime to users from COMPRES member institutes. Among the 41 proposals received, 87.8% are earth science related proposals. Of the 36 proposals granted beamtime, 86.1% were in earth science. NSF-Earth funded 65% of all proposals granted beamtime.

### 6-BM-B Beamtime Statistics:

Number of beamtime proposals received: 41

Number of beamtime proposals granted beamtime: 36

Total number of shifts requested: 372

Total number of shifts granted: 295

Total number of shifts available: 295

Oversubscription rate (= shifts requested / shifts available): 127%

Number of visits by distinct research groups: See Appendix

Number of unique users, categorized by affiliation and by origin: 42; 35 from USA and 7 from Europe, all from universities.

Total number of person-visits: 99

Number of undergraduate users: 3

Number of graduate student users: 14

Number of visits funded by each funding agency: See Appendix

### NSLS-II XPD-D:

The last year has seen several exciting developments at the XPD-D beamline. Installation and commissioning still continues apace as we work toward completion of the endstation and full opening of the General User program. In addition to the Scientific Commissioning beamtime proposal listed in the statistics below, we have also been granted 24 shifts of Technical Commissioning beamtime in four 2-day blocks. XPD management has been very accommodating in providing valuable beamtime for us to commission and optimize our installation. The four blocks of Technical Commissioning beamtime occurred in April, May, June, and October, 2017. Discussion of the tasks and results of these beamtimes are found under the Beamline Development heading.

At present, there has yet to be a formal Partner User Agreement filed between COMPRES and NSLS-II management. At the request of the NSLS-II management, a new Partner User Proposal was submitted on May 31, 2017 to supersede the terms of the previous PUP that was rendered invalid by the discontinuation of the COMPRES Diamond Anvil Cell program at NSLS-II. The proposal has been reviewed by management, and the process and negotiations are presently ongoing.

### XPD-D Beamtime Statistics:

Number of beamtime proposals received: 1

Number of beamtime proposals granted beamtime: 1

Total number of (8 hour) shifts requested: 6

Total number of (8 hour) shifts granted: 8

Total number of (8 hour) shifts available: 8

Oversubscription rate (= shifts requested / shifts available): 1.00

Number of visits by distinct research groups: 1

Number of unique users, categorized by affiliation (University, Gov’t Lab/agency, Private institution, or Industry) and by origin (USA, Canada, Europe, Asia, Other)

Stony Brook University: 8

NSLS-II: 3

USA: 11

Total number of person-visits: 11

Number of undergraduate users: 0

Number of graduate student users: 1

Number of visits funded by each funding agency: All NSF/COMPRES

## Beamline Performance Metrics

The performance metrics for both 6-BM-B and XPD-D are found in the attached Excel file and included as Appendices 2 and 3.

## Beamline Community Activities

* We have submitted a proposal to COMPRES to hold a Multi-Anvil workshop at NSLS-II in early 2018.
* New technical developments led by Whitaker at 6-BM-B, including the DIASCoPE and planetary impact experiments, have greatly expanded (more than doubled) their user base over the last year.
* Whitaker became co-P.I. of the COMPRES Multi-Anvil Program in 2017.
* Whitaker has been approached by the Journal of Visualized Experiments about preparing a video article regarding DIASCoPE experiments at 6-BM-B.
* Whitaker became a General User at APS 11-ID-B using high energy XRD and PDF.
* Whitaker continued his efforts as a member of the COMPRES Multi-Anvil Cell Assembly project, designing and testing 4 new cell assemblies in 2017.
* Whitaker served as Academic Co-Advisor for H.A.N. Dharmagunawardhane, who obtained his Ph.D. in January, 2017.
* Whitaker has taken on M.L. Sims and M.J. Rucks as Ph.D. students (co-Advisor with T.D. Glotch at Stony Brook University).
* Whitaker participated in the HPCAT Workshop on High Pressure Multi-Grain Crystallography Workshop in October, 2016, and the COMPRES Software Toolkit Workshop in July, 2017.
* Whitaker has been an author on 6 refereed publications (3 published, 3 submitted or in press), 1 extended abstract, and 21 conference abstracts in 2016-2017.
* Chen did not submit professional development activities for this report.

## Beamline Development

This section will focus primarily on beamline development activities related to the XPD-D beamline. For information related to development activities at 6-BM-B, please see the science highlights included in Appendix 5.

### DT25

The DT25 is a larger version of the DT10. These instruments have Kawai-type guideblocks which generally delivers 2 to 5 time more pressure than does the DIA for a similar sample volume. In Kawai-type (6-8) devices eight cubic anvils are used to compress the sample assembly. In the DT10 and DT25 two of the eight cubes which sit along the split-cylinder axis have been replaced by hexagonal cross section anvils. Driving these hexagonal-anvils with secondary differential actuators incorporated into the load frame enables the 6-8 multi-anvil apparatus to be used for controlled strain-rate deformation experiments to high strains. Testing of the design, both with and without synchrotron-X-rays, has demonstrated the DT10 is capable of deforming 1–2 mm long samples to over 55% strain at high temperatures and pressures.

Weidner and Dobson designed the DT system several years ago [*S. A. Hunt et al.*, 2014]. While the DT25 has not been tested, both Weidner’s and Dobson’s programs have had several years of experience with the DT10. Prior to the closing of the NSLS, the DT10 was commissioned on the side station beamline X17B2ss. In the commissioning experiments SiO2 polymorphs quartz, coesite and stishovite were deformed to high strain concurrently with olivine [*Simon A. Hunt et al.*, 2017]. The strengths of the principle phases of SiO2 (quartz, coesite and stishovite) were determined from these experiments. Quartz and coesite deformed by Harper-dorn creep and had very similar strength. Stishovite in contrast deformed by power-law creep or grain-boundary sliding and is significantly stronger than the lower pressure polymorphs. We interpret these results to imply that stishovite is a strong phase in the subducting slab. Moreover, the biggest change in properties between stishovite and the other SiO2 phases is the coordination of silicon in the crystal structure. We conclude that greater strength of stishovite arises from the Si-coordination change. The NSLS was closed before further studies could be undertaken at the NSLS. However, bridgmanite has been deformed under lower mantle conditions to high strains, using an almost identical copy of the DT10 at University College London (London, UK).

The DT25 has 25 mm instead of 10 mm anvils. It will be driven by a 1000 ton press and include two 250 ton differential jacks driving the upper and lower anvils. While we have not used this guideblock yet, we have used the 1000 ton press for several years at the NSLS. We have explored several strategies for transparent cubes. The current preference is to purchase sintered diamond anvil 25 mm cubes that are commercially available for only about 4 times the cost of tungsten carbide. We have used such assemblies with 3 mm truncations for several runs at a 500-ton load in the Stony Brook Sumitomo press with no failures of the sintered diamond anvils to date.

The DT25 can serve as a hydrostatic device as well as a deformation device. Ultrasonic experiments will be possible in this machine as well as the standard phase equilibrium or equation of state study. We anticipate that the DT25 will be the highest pressure multi-anvil uniaxial deformation device. It should compete with the RDA for maximum pressure.

In addition to defining the preferred method for including x-ray transparent anvils (large sintered diamond cubes), we have revised our initial design of the center post and cube. In the DT10, we used a truncated hexagonal post that pushes on the sample from both the top and the bottom. This makes the cell assembly awkward, and as been observed by the Hunt/Dobson group, it is also weakened by the geometry. Instead, we will use a hexagonal post that has three small wedges on the sample end, into which a 25 mm tungsten carbide cube can fit. The final system allows one to use the standard cube assembly that is used in the Kawai system and the uniaxial load will be applied by pushing on the top and bottom cubes. This marks a great improvement over the original design in that all of the sample preparation will be identical to that for the Kawai system with the bonus that the strength will be enhanced from the original DT10 design. We appreciate the input from the Hunt/Dobson group that has shared their experiences. We now have these new anvils in hand.

### XPD-D Development Timeline:

* 01/17/2014 – Original Partner User Proposal submitted
* 05/13/2014 – Partner User Agreement signed by NSLS-II
* 05/22/2014 – Partner User Agreement signed by Weidner
* 10/01/2014 – NSLS facility closes doors
* 12/08/2014 – Transfer of Press, Pumps, Table to NSLS-II
* 12/20/2014 – Transfer of all remaining equipment at X17B2&3 to Stony Brook
* 01/2015 – Segregating and rebuilding operation systems at Stony Brook
* 02/2015 – Informed by COMPRES of potential DAC discontinuation at NSLS-II
* 06/15/2015 – Controls Meeting with NSLS-II Management
* 06/30/2015 – Informed of NSLS-II decision on controls
  + Must conform to NSLS-II standards for controls and operations at Day one
  + Existing VME-based control system from NSLS-I no longer permitted
  + Delta Tau controllers were required for motion control implementation
  + All existing motors needed to be replaced with DT-compatible 2-phase motors
  + Since this decision, our group (Baldwin/Huebsch) has spent **3 Man-Years** on this
* 07/2015-08/2015 – Delta Tau controllers researched and spec’d out
* 08/24-25/2015 – COMPRES Site Visit of NSLS-II
* Early 11/2015 – Informed of COMPRES decision to officially discontinue DAC program
* 11/2015 – Redesign of beamline layout, controls, and operational plans sans DAC
* Early 12/2015 – Delta Tau motor controllers ordered
* Late 01/2016 – Delta Tau controllers and test motors received
* Late 02/2016 – Delta Tau power cables received, because they (naturally) are optional
* Late 02/2016 – Baldwin/Huebsch begin working on Delta Tau controllers and test motors
* Late 03/2016 – Original Partner User Agreement invalidated due to lack of DAC
* 04/27/2016 – Whitaker made official lead XPD liaison
* 04/30/2016 – Contractor meeting for design of differential posts in DT-25
* 05/03/2016 – Integrated into XPD group structure and meeting routines
* 05/05/2016 – Job Assessments and Trainings prescribed to SBU team
* 06/01/2016 – Approval of XPD-D installation work plan
* 06/07/2016 – Testing of anvil materials and configurations for DT-25 begins
* 06/15/2016 – Approval of XPD-D hydraulics work plan
* 07/15/2016 – XPD Controls Engineer leaves BNL
  + This position remained vacant for over six months
  + During this vacuum, no controls work could progress for ALL of XPD and PDF
* 08/10/2016 – Transport of equipment and supplies from SBU to BNL
* 08/30/2016 – General Safety Regulations meeting with NSLS-II
* 09/29/2016 – Final D hutch design and layout approval
* Early 10/2016 – Stop Work Order received from NSLS-II management
  + This stop work order was handed down due to lack of Partner User Agreement
  + We were allowed to resume some work in the new year by Eric Dooryhee
  + In July of 2017, Dooryhee was chastised by NSLS-II for allowing us to continue
  + Technically, stop work order was never lifted, but is not being strictly enforced
  + Final Partner User Agreement must be signed in the near future to avoid problems
* 01/19/2017 – New Controls Engineer appointed for XPD
* 03/20/2017 – Beamline controls system approval
* 03/28-30/2017 – Motor, encoder, and network cables run in and out of D hutch
* 03/31-04/04, 2017 – Termination of motor and encoder cables
* 04/11/2017 – Installation and configuration of managed controls network switch
* 04/12/2017 – First Light in XPD-D hutch
* 04/12-14/2017 – Comprehensive Radiation Survey of D hutch completed
* 04/28/2017 – Receipt of 1-inch sintered diamond cubic anvils for use in DT-25
* 05/08/2017 – Testing of 1-inch sintered diamond cubes begins
* 05/09-10/2017 – Full ray tracing and beam survey of hutch D
* 05/22/2017 – Termination of hutch interior controls network cabling and panel
* 05/22-24/2017 – Technical Commissioning of XPD-D Phase I (details below)
* 05/31/2017 – New Partner User Proposal submitted to NSLS-II
* 06/13/2017 – Hydraulic layout redesigned and approved
* 06/20/2017 – Original contractor for DT-25 post inserts removed from project
* 06/21/2017 – Search for new contractor for DT-25 inserts begins
* 06/29-07/01/2017 – Technical Commissioning of XPD-D Phase II (details below)
* 07/09/2017 – COMPRES Workshop on Software Toolkits for Mineral Physics
* 07/10/2017 – Whitaker presented first diffraction data collected at XPD-D to COMPRES
* 07/12/2017 – Opened dialogue with New Mexico about BNL account creation for equipment purchasing; specifically for the Allen Bradley PLC modules
* 07/20/2017 – Installed and tested Delta Tau motor controllers in XPD-D roof rack
* 07/30/2017 – Aztec Tool Co. contracted for design and fabrication of DT-25 inserts
* 07/31/2017 – Mounting of motors on equipment stages begins
* 08/09/2017 – Meeting to begin set up of IOC controls spreadsheet
* 08/15/2017 – Meeting with BNL tech group to determine final pieces for hydraulics
* 08/22/2017 – Installation and wiring of motor control boxes begins
* 08/29/2017 – Begin process of migrating workstations from Windows to Debian
* 09/01/2017 – Motor IOC controls spreadsheet completed and approved
* 09/01/2017 – DT-25 insert CAD drawings received from Aztec Tool Co.
* 09/07/2017 – Quotes for DT-25 inserts received from Aztec Tool Co.
* 09/12-13/2017 – Final termination of hutch exterior controls network cabling
* 09/13/2017 – Termination of surveillance camera connections in and out of hutch
* 09/26/2017 – Installation and commissioning of security/surveillance camera system
  + We are acting as a pilot program for NSLS-II with this feature
  + Cameras installed inside and outside hutch to view work area and equipment
* 09/26/2017 – Testing of motor controls using Delta Tau native software begins
* 09/27/2017 – Installation of sample imaging camera and YAG scintillator and stages
* 09/29/2017 – Preliminary configuration of installed motors complete
  + Installed and tested motors for camera, YAG, detector table, differential pumps
  + New incident slit motors installed and tested
  + Motors for press pedestal actuation and high pressure hydraulic pump not ready
* 10/02/2017 – Termination of motor brake control cabling
* 10/02-04/2017 – Technical Commissioning of XPD-D Phase III (details below)
* 10/12/2017 – Order for Allen Bradley Modules dispatched
* 10/14/2017 – Aztec tests posts and inserts in DT-25, fit adjustment necessary
* 10/17/2017 – UNM Purchasing denies opening BNL account for equipment purchases
* 10/20/2017 – Installation of main ram hydraulics begins
* 10/26/2017 – Receipt of completed order of Allen Bradley modules
* 10/27/2017 – Allen Bradley modules delivered to NSLS-II Controls Group for assembly
* 11/01/2017 – Main ram hydraulic installation completed
* 11/02/2017 – NSLS-II Safety Approval of main ram hydraulics to 5,000 PSI (550 tons)
* 11/07/2017 – Receipt of brake control templates from controls engineer
* 11/14-16/2017 – Installation, wiring and testing of press pedestal motors
* 11/15/2017 – Receipt of final posts and wedge inserts for DT-25 from Aztec Tool Co.
* 11/15/2017 – Installation of Allen Bradley modules in electronics rack inside D hutch

### XPD-D Development Highlights:

* April 12-14, 2017: First Light and Comprehensive Radiation Survey in the D hutch.
* May 22-24, 2017: Technical Commissioning of XPD-D Phase I
  + A) FULL BEAM FLUX MEASUREMENTS – BEAM ENERGY 52 keV
    - Flux Measurement in C Hutch - 0.899uA :: 2.801x10^12 photons/sec
    - Flux Measurement at front of D Hutch - 0.805uA
    - Flux Measurement upstream of D slits - 0.700uA
    - Flux Measurement downstream of D slits - 0.661uA
    - Flux Measurement at press, unoptimized - 0.666uA
    - Flux Measurement at press, optimized - 0.709uA :: 2.209x10^12 photons/s
  + B) INCIDENT SLIT OPTIMIZATION AND CENTERING
    - 21000 steps = ~3mm vertical
    - 35000 steps = ~7mm horizontal
  + C) FLUX MEASUREMENT WITH CLOSED SLITS
    - Flux Measurement with closed incident slits - 0.000uA
    - ~200x200um beam - 0.003uA :: 9.346x10^9 photons/sec
  + TAKEAWAYS:
    - We lose ~20% of our flux due to scattering in air when comparing the C Hutch sample position to the D hutch sample position.
    - Closing the slits from full beam size (3mm V x 7mm H) to 200x200 m decreases the cross-sectional area of the beam by ~535 times, but we cut the number of photons/sec by only ~236 times. This is consistent with the beam intensity tapering off toward the edges, particularly horizontally.
* June 29-30, 2017: Technical Commissioning of XPD-D Phase II
  + X-ray beam energy of 68 keV
  + All focusing mirrors removed to allow large beam for imaging
  + Flux measurements taken at sample positions in C and D hutches
    - 16% decrease in measured flux from C to D hutch
    - This compares to 20% decrease in measured flux at 52 keV
  + Tested evacuated beam pipe to help background reduction; no significant benefit
  + Optimized beam and imaging size and conditions
  + Introduced lead shielding downstream of incident slits to reduce background
    - More permanent solution will be implemented with final installation
    - Much of the background scattering will be mitigated by the DT-25 or D-DIA pressure modules
  + Collected the first diffraction patterns in the XPD-D hutch
    - Sample standard was Al2O3 in Boron-epoxy cube
    - Several data collection times were tested
    - Large beam spot (300x350 m) – 0.1 sec was sufficient
    - Small beam spot (<100x100 m) – 10 sec was sufficient
  + This data collection was at 68 keV, with low scattering factor
  + Normal operations at 52 keV where flux will be higher
  + Even shorter data collection times could be possible
* October 2-4, 2017: Technical Commissioning of XPD-D Phase III
  + Beam Energy 67 keV
  + Collected image and diffraction data at several different mirror-in conditions
    - Collected data at 50,000 step intervals in mirror bend
      * Maximum bend 350,000 steps
      * Minimum bend 0 steps – no bend
    - Largest beam size was flat mirror with no bend
    - Beam size decreased from 0-300,000 steps in mirror bend
    - Beam size then increased between 300,000 and 350,000
      * This indicates overfocusing of beam in hutch D ~300,000 steps
      * Possibility that higher mirror bends will give larger beam size
      * May still not be able to achieve desired 3mm vertical beam size
      * Plan to test further in December beamtime
  + Mirror moved out of beam path
  + Equipment positions adjusted to account for change in beam height sans mirror
  + Collected image and diffraction data under mirror out conditions
    - Beam not as strong as in June due to extra filters in path
    - Background scatter became significantly stronger with mirror out
  + Image and diffraction data show clear flux density benefit with mirror in
  + Good for diffraction, not good for imaging
  + Future tests will be conducted to see if we can overbend the mirror for imaging

## Planned Development Activities

### 6-BM-B

The planned development activities at 6-BM-B in the 2018-2019 year are centered on necessary hardware and software upgrades. In 2017, we have experienced significant setbacks due to hardware failure as much of our controls systems and computing capabilities are becoming quite aged. In replacing these obsolete components, we expect to see significant gains in performance at the beamline.

### XPD-D Development Timeline

* 11/16/2017 – Final press pedestal motor installation and testing
* 11/16/2017 – Begin working with Allen Bradley PLC modules for signal I/O
* 11/17/2017 – Installation of posts and inserts into DT-25 module
* 11/20/2017 – Delivery of DT-25 and D-DIA modules to NSLS-II
* 11/20/2017 – Installation of DT-25 module and spacer block into press
* 11/21/2017 – Testing implementation of motor brakes via Delta Tau native software
* 11/22/2017 – Charging of hydraulic system with oil and cursory leak test
* 11/27/2017 – Press dummy load in DT-25 to ~100 tons to verify leak checks
* 11/28/2017 – Testing of camera on new Debian architecture and calibration of image size
* 11/29/2017 – Attempt EPICS control of motors and brakes
* 11/30/2017 – Installation and testing of heater power supply
* 11/30/2017 – Final Experimental Safety Review
* 12/01/2017 – Attempt control of press load and heater power using Allen Bradley inputs
* **12/02-04/2017 – FIRST SCIENCE COMMISSIONING EXPERIMENT**
* 01-02/2018 – Full conversion to Debian control computers
* 03/2018 – Full EPICS control of motors and Allen Bradley PLC functions
* **03/2018 – SCIENCE COMMISSIONING BEAMTIME**
* **04/2018 – FIRST USER-ASSISTED COMMISSIONING BEAMTIME**
* 2018-2 Cycle – Science Commissioning and User-Assisted Commissioning continues
* **2018-2 Cycle – GENERAL USER PROGRAM @ XPD-D OPENS**
  + On track to meet **all** deadlines and milestones presented at COMPRES meeting

## Budget

Budget for COMPRES Multi-Anvil Program for 2017-18 and 2018-19

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| Budget Request for 2018-2019 | XPDD/yr 1 | 6BMB/yr 1 | XPDD | 6BMB |
| Budget Year | 2017-2018 | 2017-2018 | 2018-2019 | 2018-2019 |
| BL scientist/MAC, Chen |  | $90,000 |  | $93,000 |
| BL scientist/MAC, Whitaker | $86,000 |  | $93,000 |  |
| Electronics, Huebsch | $25,000 |  | $25,000 |  |
| Fringe Benefits (.41) | $50,505\* | $40,950\* | $48,380 | $38,130 |
|  |  |  |  |  |
| Equipment | $0 | $0 | $0 | $0 |
|  |  |  |  |  |
| Travel | $3,600 | $13,600 | $3,800 | $12,800 |
|  |  |  |  |  |
| Supplies | $21,266 | $21,167 | $22,000 | $31,167\*\* |
|  |  |  |  |  |
| Shop support | $3,139 | $4,082 | $3,139 | $4,082 |
|  |  |  |  |  |
| IDC (.26) - not equipment | $49,273 | $44,148 | $50,782.94 | $43,987 |
|  |  |  |  |  |
| Total | $238,783 | $213,947 | $246,102 | $223,166 |
| Total for both 6BMB and XPD |  | $452,730 |  | $469,268 |

\*This was based on a Fringe rate of 45.5% which was the published Stony Brook rate at the time of the proposal. It is now 41%.

\*\*This reflects an increase of $10,000 over the budget requested in the 6-BM-B beamline proposal because of necessary hardware upgrades described below.

### Budget Justification

The budgets for both the NSLS II (XPD) beamline and the APS (6BMB) beamline are given in separate tables with the final total for both indicated in the second table. The salaries for the first year include support for Whitaker, Chen, and Huebsch. Whitaker (NSLS II) and Chen (APS) are beamline scientists engaged at the two beam sites. Huebsch is an electronics engineer, employed part time, who supports all of the electronic issues for these installations. While employed part time, he virtually works full time and is an expert in dealing with motor drives and electronic controls. Huebsch is definitely the most valuable technical support person involved in the project. The budget includes an 8% increase in Whitaker’s salary reflecting his central role in the NSLS II installation.

The 6BMB travel budget for 2018-2019 includes funds for the beamline scientist, to attend the COMPRES meeting ($800), costs for attending one scientific conference for the beamline scientist ($2000), and support for 10 trips from New York to Chicago for PI and other staff to support the APS operation ($1000 per trip).

The XPD travel budget for 2018-2019 includes funds for one beamline scientist, and one PI to attend the COMPRES meeting ($900 each) plus costs for attending one scientific conference for the beamline scientist ($2000).

Materials and supplies are mainly used for sintered diamond anvils, tungsten carbide anvils, and high pressure cell assemblies. We anticipate that the use of anvils at the NSLS II will increase with time and have built that into our calculations. We have added $10,000 (from the 6BMB proposal to upgrade the computers at 6BMB. We currently use 5 computers that come from X17 and have now been used for several years. We recently had a failure of 2 of these machines, requiring replacement and have been taken from the computers that were purchased for the XPD installation. We need to begin to replace all of the computers in use at APS as the failure of these two almost caused a closing of the beamline for an extended period of time.

In the other category, we include technical support as provided by the synchrotron facility. This support is required for moving heavy objects, machining, or other operation done to the installation. We included between 10 and 20 hours per year as an estimate per facility at an estimated cost of $180.00/hour. We anticipate that our staff can carry out support for our high pressure equipment, but facility support is essential for hutch related operations and for safety control.

We have now spent out virtually all of the COMPRES funds that were held in reserve anticipating installation costs. So we have very little flexibility in addressing unanticipated system failures.

## Appendices

**Appendix 1** – Publication List for 2016-2017: In this Word file.

**Appendix 2** – Beamtime Proposals and Usage: Attached Excel file.

**Appendix 3** – Beamtime Schedules: Attached Excel file.

**Appendix 4** – User Information: Attached Excel file.

**Appendix 5** – User-submitted Science Highlight 1-Pagers: In this Word file.

Appendix 1 – Publications

**2016**

1. Bollinger C. P., Raterron P., Castelnau O., Detrez F., Merkel S., Textures in Deforming Forsterite Aggregates up to 8 GPa and 1673 K, *Phys.Chem. Miner.,* **43,** 409-417, 2016
2. Raterron P., Fraysse G., Girard J., Holyoke C.W. III, Strength of orthoenstatite single crystals at mantle pressure and temperature and comparison with olivine, *Earth and Planet. Sci. Lett.,* **450**, 326-336, 2016
3. Liu Z., Park J., Karato S., Seismological detection of low velocity anomalies surrounding the mantle transition zone in Japan subduction zone, *Geophys. Res. Lett.*, **43**, 2480-2487, 2016.
4. Karato S., 2016. Physical basis of element partitioning: A review, *Am. Mineral.,* **101** (12) 2577-2593, 2016.
5. Masuti S., Barbot S.D., Karato S., Feng L., Banerjee, P., Upper mantle water stratification inferred from observations of the 2012 Indian Ocean earthquake, *Nature,* **538,** 373-377, 2016.
6. Girard J., Amulel, G., Farla R., Karato S., 2016. Shear deformation of bridgmanite and ferropericlase aggregates at lower mantle conditions, *Science,***351**, 144-147, 2016.
7. Olugboji T., Park J., Karato S., Kawakatsu H., Shinohara M., The nature of the lithosphere-asthenosphere boundary in the normal oceanic upper mantle, *Geochem. Geophys. Geosy.,* **17 (4),** 1265-1282, 2016*.*
8. He Q., Liu X., Li B.S., Deng L.W., Liu W., Wang L.P., Thermal equation of state of a natural kyanite up to 8.55 GPa and 1273 K, *Matter and Radiation at Extremes;* **1 (5),** 269-296, 2016.
9. Zhang J.Z., Velisavljevic N., Zhu J.L., Wang L.P., Equation of state and thermodynamic Grüneisen parameter of monoclinic 1,1-diamino-2,2-dinitroethylene, *J. Phys.: Condens. Matter,* **28**, 395402, 2016.
10. Wang P., Wang Y., Wang L.P., Zhang X.Y., Yu, X.H., Zhu J.L., Wang S.M., Qin J.Q, Leinenweber K., Chen H.H., He D.EW, Zhao Y.S., Elastic, magnetic and electronic properties of iridium phosphide Ir2P. *Sci. Rep.,* **6,** 21787, 2016.
11. Wang S.M., Yu X.H, Zhang J.Z., Wang L.P., Leinenweber K., He D.W., and Zhao Y.S., Synthesis, hardness, and electronic properties of stoichiometric VN and CrN, *Crystal Growth & Design,* **16 (1),** 351–358, 2016.
12. Wang Y.J., Liu Z., Khare S.V., Collins S.A., Zhang J.Z., Wang L.P., Zhao Y.S., Thermal equation of state of silicon carbide, *Appl. Phys. Lett.,* **108**, 061906, 2016.
13. Dobson D.P., Hunt S.A.,  Ahmed J., Lord O.T., Wann E.T.H., Santangeli J, Wood, I.G., Vočadlo L., Walker A.M., Mueller H.J., Lathe C., Whitaker M.L., The phase diagram of NiSi under the conditions of small planetary interiors. *Phys. Earth Planet. Inter.* **261,** B 196-206, 2016.
14. Hunt S. A., Walker A.M., Mariani E., In-situ measurement of fabric development rate in CaIrO3, *Phys. Earth Planet. Inter.,* **259**, 91-104, 2016.
15. [Woerner W.R](http://apps.webofknowledge.com/DaisyOneClickSearch.do?product=WOS&search_mode=DaisyOneClickSearch&colName=WOS&SID=2F1HBuRn2EIOZrvzpEa&author_name=Woerner,%20WR&dais_id=2003347902&excludeEventConfig=ExcludeIfFromFullRecPage&cacheurlFromRightClick=no)., [Qian G.R](http://apps.webofknowledge.com/DaisyOneClickSearch.do?product=WOS&search_mode=DaisyOneClickSearch&colName=WOS&SID=2F1HBuRn2EIOZrvzpEa&author_name=Qian,%20GR&dais_id=2003115964&excludeEventConfig=ExcludeIfFromFullRecPage)., Oganov A.R., [Stephens P.W](http://apps.webofknowledge.com/DaisyOneClickSearch.do?product=WOS&search_mode=DaisyOneClickSearch&colName=WOS&SID=2F1HBuRn2EIOZrvzpEa&author_name=Stephens,%20PW&dais_id=2003233735&excludeEventConfig=ExcludeIfFromFullRecPage)., [Dharmagunawardhane H.A.N](http://apps.webofknowledge.com/DaisyOneClickSearch.do?product=WOS&search_mode=DaisyOneClickSearch&colName=WOS&SID=2F1HBuRn2EIOZrvzpEa&author_name=Dharmagunawardhane,%20HAN&dais_id=2002633902&excludeEventConfig=ExcludeIfFromFullRecPage)., [Sinclair A](http://apps.webofknowledge.com/DaisyOneClickSearch.do?product=WOS&search_mode=DaisyOneClickSearch&colName=WOS&SID=2F1HBuRn2EIOZrvzpEa&author_name=Sinclair,%20A&dais_id=2003211147&excludeEventConfig=ExcludeIfFromFullRecPage)., [Parise J.B](http://apps.webofknowledge.com/DaisyOneClickSearch.do?product=WOS&search_mode=DaisyOneClickSearch&colName=WOS&SID=2F1HBuRn2EIOZrvzpEa&author_name=Parise,%20JB&dais_id=2003078278&excludeEventConfig=ExcludeIfFromFullRecPage)., Combined Theoretical and in Situ Scattering Strategies for Optimized Discovery and Recovery of High-Pressure Phases: A Case Study of the GaN-Nb2O5 System, *Inorg. Chem.,* **55,** 3384, 2016.
16. Wu T., Tyson T. A., Chen H.Y., Gao P., Yu T., Chen Z., Liu Z., Ahn K. H., Wang X., Cheong S.-W., Pressure Dependent Structural Changes and Predicted Electrical Polarization in Perovskite RMnO3, *J. Phys. Condens. Matter*, **28**, 056005, 2016.

**2017**

1. Cheung, S. N. C., Weidner, D. J., Li, L., Meredith, P. G., Chen, H., Whitaker, M. L., Chen X., Stress distribution during cold compression of a quartz aggregate using synchrotron X-ray diffraction: observed yielding, damage and grain crushing, *J. Geophys. Res. Solid Earth,* **122 (4)**, 2724-2735, 2017*.*
2. Whitaker M.L., Baldwin K.J., and Huebsch W.R., DIASCoPE: Directly Integrated Acoustic System Combined with Pressure Experiments – A new method for fast acoustic velocity measurements at high pressure, *Rev. Sci. Instrum.,* **88 (3)**, 034901, 2017
3. Farla R., Amulele G., Girard J., Miyajima N., Karato S.I., High-pressure and high-temperature deformation experiments on polycrystalline wadsleyite using the rotational Drickamer apparatus (vol 42, pg 541, 2015), *Phys. Chem. Min.,*  **44 (3)**, 235, 2017
4. Zhang G.N., Mei S.H., Song M.S., Kohlstedt D. L., Diffusion Creep of Enstatite at High Pressures Under Hydrous Conditions, *J. Geophys. Res. Solid Earth,*, **122,** doi:10.1002/2017JB014400, 2017.
5. Kaboli S., Burnley P. C., Xia G., Green II H. W., Pressure dependence of creep in forsterite olivine: comparison of measurements from the D-DIA and Griggs apparatus, *Geophysi. Res. Lett.,* **In Press**, 2017.
6. Kaboli S., Burnley P.C., ECCI, EBSD and EPSC Characterization of Rhombohedral Twinning in Polycrystalline α-Alumina Deformed in the D-DIA Apparatus, *J. Appl. Crystallogr.,* **In Press**, 2017.

**PhD Theses**

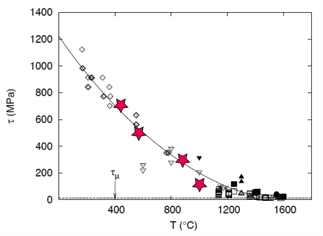
1. Proietti, A. (2016), Rhéologie d’agrégats olivine-orthopyroxène sous haute pression, Universite Toulouse III Paul Sabatier, Toulouse, January.
2. Tercé, N. (2016), Propriétés élastiques des olivines riches en fer, Université Toulouse III Paul Sabatier, Toulouse, 10 November.
3. Zhang, G. (2016), Diffusion Creep of Enstatite at High Pressure under Hydrous Condition, State Key Laboratory of Isotope Geochemistry, Guangzhou Institute of Geochemistry, Guangzhou, China
4. Wang, X. (2016) [Mantle composition and temperature of western North America revealed from in-situ velocity measurements of KLB-1 peridotite](https://search-proquest-com.proxy.library.stonybrook.edu/pqdtlocal1007355/docview/1848270426/657A797B8073480CPQ/1?accountid=14172), Ph. D. Thesis, Stony Brook University
5. Dharmagunawardhane, HAN (2017) Synthesis of oxynitride materials for solar water splitting: investigations with ambient pressure and high pressure synthesis techniques, Ph.D. Thesis, Stony Brook University

### Appendix 5

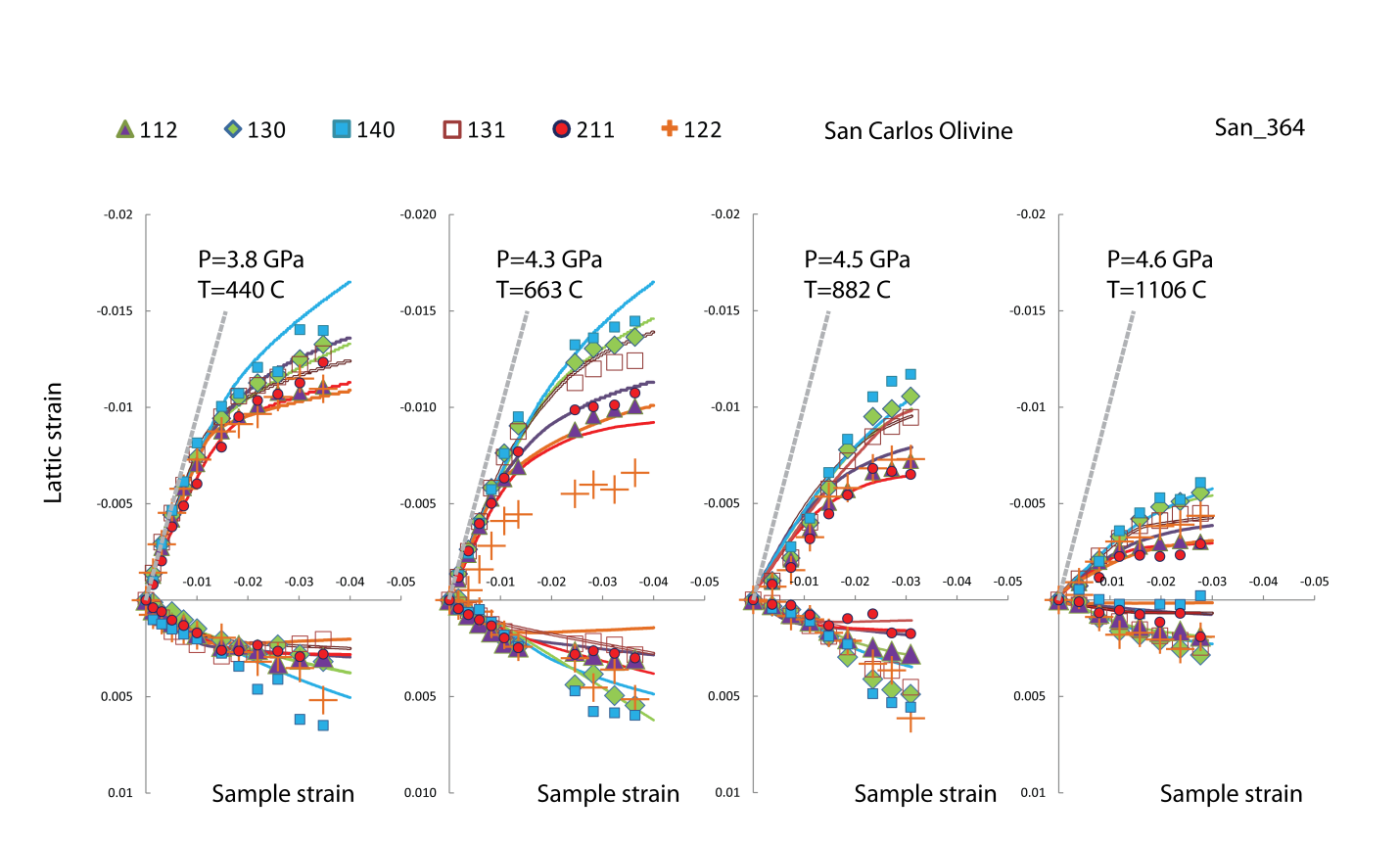
**Using in-situ Diffraction and Elastic Plastic Self-Consistent Models to Quantify Plastic Deformation Mechanisms**

**Pamela C. Burnley and Shirin Kaboli, University of Nevada, Las Vegas**

In-situ diffraction data provides a rich source of information about the individual plastic deformation mechanisms operating in polycrystalline materials as they deform. The challenge is to determine how to best extract this information from the raw diffraction data. We have been using elastic plastic self-consistent (EPSC) models to interpret diffraction data in terms of the deformation mechanisms operating in the sample. We conducted a suite of low strain experiments at ~5 x 10-6/sec at temperatures ranging from 440 C to 1100 C in the D-DIA apparatus at APS 6BMB. For each experiment we fit the diffraction data using EPSC models that included an isotropic deformation mechanism that work hardens rapidly, [001] slip, and kinkband formation. The models closely reproduce the diffraction data (Figure 1) and yield estimates for the CRSS of [001] slip that are comparable to those found in the literature (Figure 2). The isotropic deformation mechanism allows us to model the data’s deviation from the self-consistent value of the Young’s modulus (Figure 1 -grey lines). Although we have not confirmed the exact nature of this deformation mechanism the models provide us with quantification that can be used to compare with proposed mechanisms.



*Figure 1: CRSS for [100] slip in olivine from Durink et al Am. Min.v. 92, 2007. Red stars are values determined in this study from EPSC models*



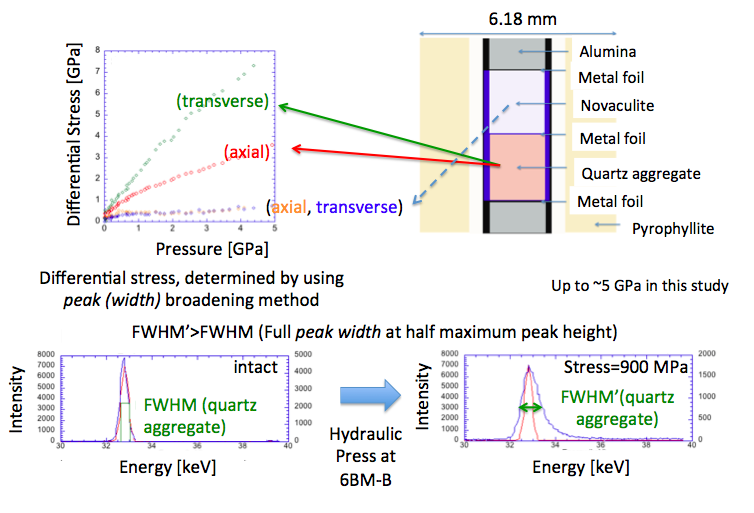
*Figure 2: Lattice strain vs sample strain for deformation experiments conducted on San Carlos Olivine at 5 x 10-6/sec. The solid lines are the EPSC models and the symbols are the diffraction data. The dashed grey line depicts the self-consistent elastic slope.*

**Stress distribution during cold compression of a quartz aggregate using synchrotron X-ray diffraction: Observed yielding, damage and grain crushing [*Cheung et al.*, 2017]**

**Cecilia Cheung, U Wisconsin**

Compaction in porous rock is of fundamental importance to reservoir and geotechnical engineering. Any natural phenomena or artificial geological (e.g., oil and gas extraction) application that reduces pore pressure is likely to cause an increase in the effective stress. Such an increase in effective stress may be sufficient to cause inelastic deformation of the reservoir rock. In geomaterial, stress is likely to concentrate at grain-to-grain contacts and vanish where grains are bounded by open porosity. Therefore, internal stress is likely to vary significantly from point to point in such an aggregate, and hence, it is important to understand and quantify the stress distribution with respect to the externally applied stress. Our samples were probed with synchrotron X-ray diffraction as they were compressed in a multianvil deformation apparatus at ***X17B2*** and ***6BM*** at room temperature from low pressure (tens of megapascal) to pressures of a few gigapascal. Stress was indirectly by using the atomic lattice spacing within grains, as revealed by X-ray diffraction, as a measure of local elastic strain, on samples of a quartz aggregate and a novaculite (as end-members of the spectrum of quartz-rich geo-materials).

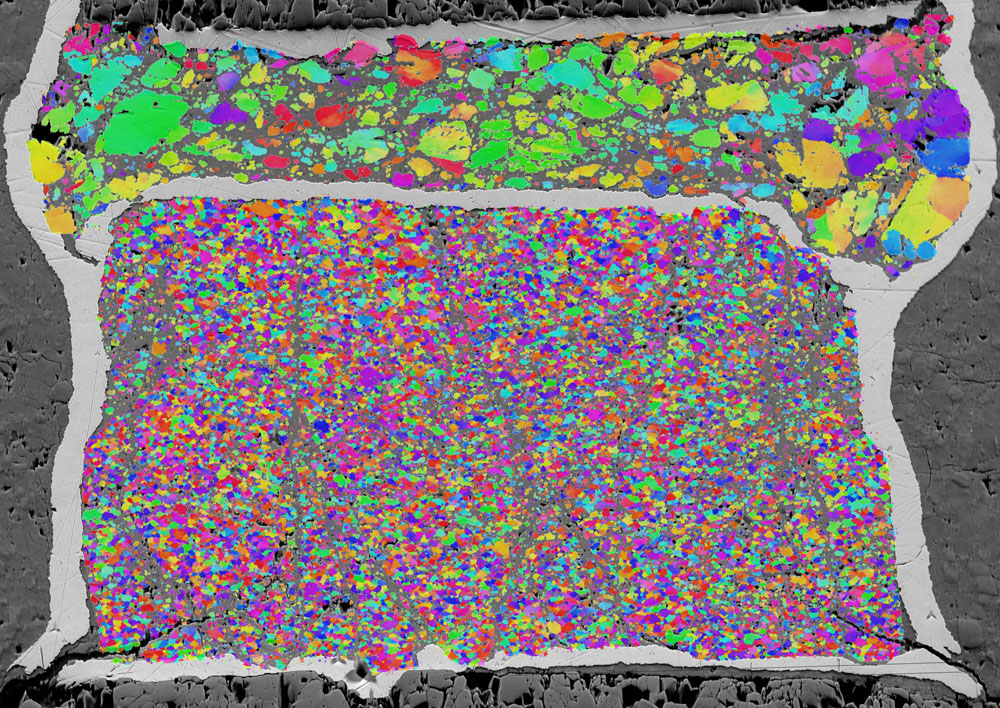
In our quartz aggregate, the diffraction peaks broaden asymmetrically at low pressure (tens of megapascal), suggesting that open pores are still a dominant characteristic of grain boundaries. In contrast, a reference sample of novaculite (a highly dense quartz polycrystal) showed virtually no peak broadening with increasing pressure. In the quartz aggregate, a significant deviation was observed in the pressure-volume curves in the range of P = 400–600 MPa. We suggested that this marks the onset of grain crushing (generally denoted as P\* in the rock mechanic literature), which is commonly reported to occur in sandstones at pressures of this order, in general agreement with a Hertzian analysis of fracturing at grain contacts. In this region, the sample compresses significantly with little increase in pressure. Furthermore, at the time of collapse, we found that the differential microscopic stress is about 1000 MPa. These values are about an order of magnitude lower than those predicted by a model based on Hertzian fracture mechanics.



**The grain-size dependence of yield strength during low-temperature plasticity of olivine: Evidence for weak lithospheric mantle**

Lars N. Hansen, Kathryn M. Kumamoto, Christopher A. Thom, David Wallis, David L. Goldsby, William B. Durham, David L. Kohlstedt

A set of experiments specifically designed to investigate an hypothesized *inverse* relationship between grain size and low-T strength of olivine was initiated with exploratory experiments in the 2017-1 trimester at 6BM-B (APS) and continued as its own GUP for 2017-2. The results strongly signal confirmation of the hypothesis. Concurrent EBSD mapping and slip system models are aimed at providing a consistent physical mechanism for the effect. The results have implications for the strength of the lithospheric mantle during flexure under volcanic or glacial loads, during bending of slabs at subduction zones, and after seismic events in the ductile roots of major fault zones. Furthermore they could explain the inconsistencies among previous studies of lithospheric strength.



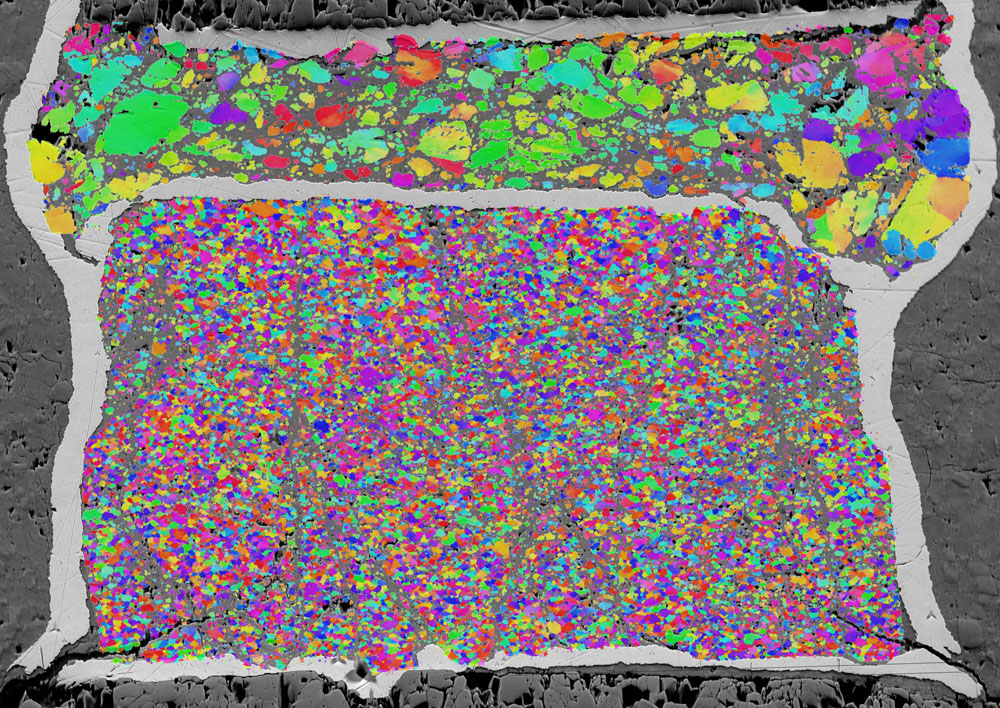


Figure 1. Stress-strain creep curves for stacked olivine samples of contrasting grain size and deformed in the D-DIA at 6BM-B at APS. Symbol shapes refer to (hkl) diffraction condition used to determine stress. Tests were at room T or as indicated. Red refers to the finer grain sample in the stack, black to the coarser. Stresses are generally the same in both samples since they are stacked on top of one another; what differs in fine vs. coarse-grained (red vs black) is strain rate, with the weaker piece strain (almost always black) extending farther along the horizontal axis than the red. Inset shows EBSD map of post-test polished section though the stack (colors indicate crystallographic orientation).

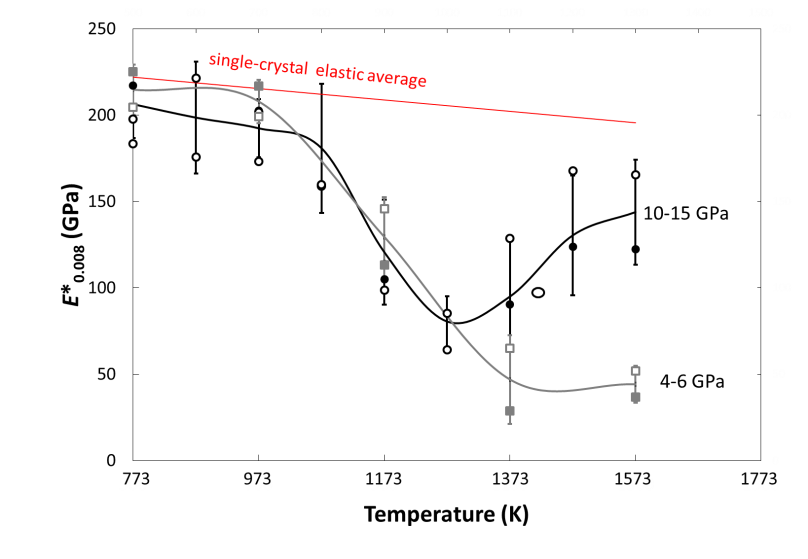
**The effect of sintering pressure on the anelastic properties of pyrope**

**In preparation for PNAS.**

Dobson DP1, Hunt SA1, Schardong L1, Thomson AR1, Ezad IS1, Bailey, E1, Walker AM2, Lord, OT3, Marquardt, K4, Melai, C4, Whitaker, ML5 and Weidner, DJ5

Much of our understanding of the interior of the Earth comes from interpreting the behaviour of seismic waves. Grain-boundaries form a finite volume fraction of the Earth’s mantle but their effects on the seismic properties of the mantle are not fully understood. Grain-boundaries undergo a fundamental change in their properties between ambient and ‘high’ pressure. The changes are attested to by, among other effects, the disappearance of the ‘grain-boundary’ component in electrical conductivity measurements at high pressure and the increase of microhardness with sintering pressure in both pure metals (Edalati & Horita, 2010, Mat. Trans., 51, 1051) and polycrystalline diamond-SiC composites (Osipov et al 2004, Mat. Res., 7, 335). These phenomena imply that grain-boundaries properties become more “lattice-like” with increasing pressure.

To test the potential consequences of this for the Earth, we performed anelasticity measurements on pyrope samples sintered at pressures between 4 and 15 GPa. All recovered samples had a similar grain size of ~2µm but the 15 GPa samples were much lighter in colour than those sintered at lower pressures. All samples were deformed by small sinusoidal strains under identical conditions of ~3 GPa, 500 to 1300°C and at periods between 10 and 1000s. At <900 °C and short periods the effective Young’s modulus is the same as that predicted using the elastic constants of pyrope (Figure 1) and the quality factor (Q) is high. At higher temperatures the samples sintered at low pressure show significant reduction of the effective Young’s modulus and Q, whist the samples sintered > 10 GPa maintain a high Q value



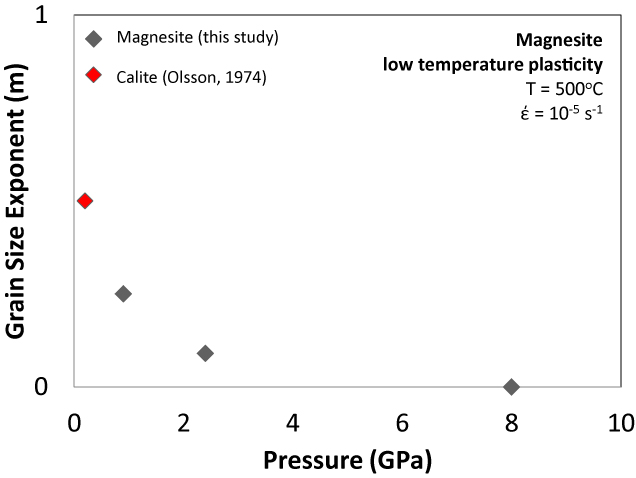
**Figure 1: Effective Young’s modulus (E\*), at 120s period, for the ‘low pressure’ and ‘high pressure’ samples in this study. The effective Young’s modulus is significantly reduced at high temperatures in the low pressure samples.**

These results indicate that grain boundaries in pyrope equilibrated at high pressure can be recovered to ambient conditions but the sintering pressure strongly affects the anelastic behaviour of ceramic materials. We interpret this as a pressure-induced inhibition of the elastically accommodated grain-boundary sliding mechanism due to enhanced strength of grain boundaries created at high sintering pressures.**Grain size and pressure dependence of magnesite aggregate rheology: implications for nucleation of intermediate depth (200-400 km) deep focus earthquakes.**

C. Blasko1,\*, N. Jackson1,\*, C. McDaniel1,\*\*, J. Millard1,\*\*, C. Holyoke1, A. Kronenberg2, P. Raterron3, L. Tokle3, \*\*

1 – University of Akron, 2 – Texas A&M University, 3 – Brown University, \* - undergraduate student, \*\* - graduate student. This study is supported by NSF EAR-1624242 to Holyoke, Kronenberg and Raterron.

In many down-going slabs, such as the Nazca plate beneath Chile, earthquake foci are observed in two depth-related groups separated by an aseismic zone from ~200 to 400 km depth. The mechanisms commonly proposed for the earthquakes above and below the aseismic zones are serpentine dehydration and phase transitions in olivine, respectively. However, in other subducting slabs, such as the Pacific plate beneath Tonga, earthquake foci are observed continuously from the surface to the transition zone (~660 km depth). The mechanism proposed for earthquakes in this zone is the formation of ductile instabilitites due to olivine recrystallization that causes a reduction in grain size and viscosity. However, since all slabs are composed dominantly of olivine, ductile instabilities in olivine should form in all subducting slabs and there should be no aseismic zones in downgoing slabs. Another possible mechanism is strain localization in magnesite, an alteration product in found in some peridotites. Magnesite is stable to depths >700 km and is orders of magnitude weaker than olivine at all pressure and temperature conditions of subducting slabs.

Figure 1 – Grain size dependence of the strength of magnesite decreases with increasing pressure.

In order to determine the grain size and pressure dependence of the strength of magnesite deforming by dislocation creep and low temperature plasticity, we have performed experiments using the D-DIA at beamline 6-BMB at the Advanced Photon Source at Argonne National Laboratory. Experiments were performed at temperatures from 500-900oC, pressures from 3-8 GPa and a strain rate of 10-5/s.

Strengths of magnesite aggregates in experiments performed at conditions where low temperature plasticity mechanisms are dominant increase with increasing pressure. At low pressures (0.9-5 GPa), magnesite aggregates with coarser grain sizes are weaker than those with finer grain sizes. This inverse grain size-strength relationship is consistent with previous results for calcite obtained at low pressure (0.2 GPa). However, this inverse grain size-strength relationship gradually decreases as pressures increase and at 7 GPa, there is no grain size dependence to this deformation mechanism (Fig. 1). These results indicate that throughout much of its stability field the strength of magnesite is insensitive to grain size.

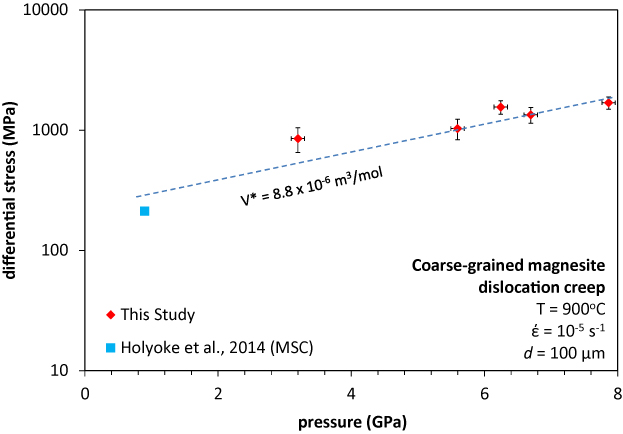


Figure 2 – The pressure dependence of magnesite deforming by dislocation creep (~9\*10-6 m3/mol) is similar to olivine.

Strengths of magnesite aggregates in experiments performed at conditions where dislocation creep is the dominant mechanism increase with increasing pressure, yielding an activation volume (V\*) of 9\*10-6 m3/mol, which is very similar to that of olivine (Fig. 2). These results indicate that the high strength contrast between magnesite and olivine will not be affected by increasing depth and may cause strain localization in magnesite veins or layers in subducting slabs and, possibly, deep focus earthquakes at all depths in subducting slabs.

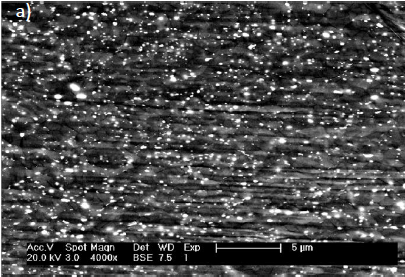
**Studies by the Yale program at NSLS (Karato)**

We use RDA (rotational Drickamer apparatus) combined with the synchrotron x-ray facility at X17B2 at NSLS to study the plastic flow behavior of materials under high-pressure and temperature conditions. We have conducted quantitative deformation experiments on two important minerals in Earth’s transition zone (410 to 660 km depth). The flow laws (stress-strain rate relationship) of these minerals were investigated using the *in-situ* X-ray diffraction and x-ray radiography. These studies have provided constraints on the resistance of these minerals for plastic flow under a broad range of conditions.

In addition, we have pushed the pressure limit of quantitative studies on plastic flow in order to understand the plastic properties of minerals in the lower mantle (660-2890 km). This is the largest portion of the rocky part of this planet. By analyzing the diffracted x-ray from various portions of the sample, we realized that a substantial pressure gradient is present in the sample assembly of RDA. Consequently, we reduced the sample size to conduct deformation experiments at pressures of ~27 GPa and temperature of ~2100 K (both P and T were determined by the equations of state of two materials). Under these conditions, dominant minerals are (Mg,Fe)SiO3 bridgmanite and (Mg,Fe)O. We found that bridgmanite has substantially higher resistance to deformation than (Mg,Fe)O.

SEM micrograph of a deformed bridgmanite + (Mg,Fe)O aggregate. Dark regions are bridgmanite and light grey regions are (Mg,Fe)O. Bright spots are metallic Fe.

Conditions of deformation are P=27 GPa, T=2130 K, strain-rate ~10-5 s-1.

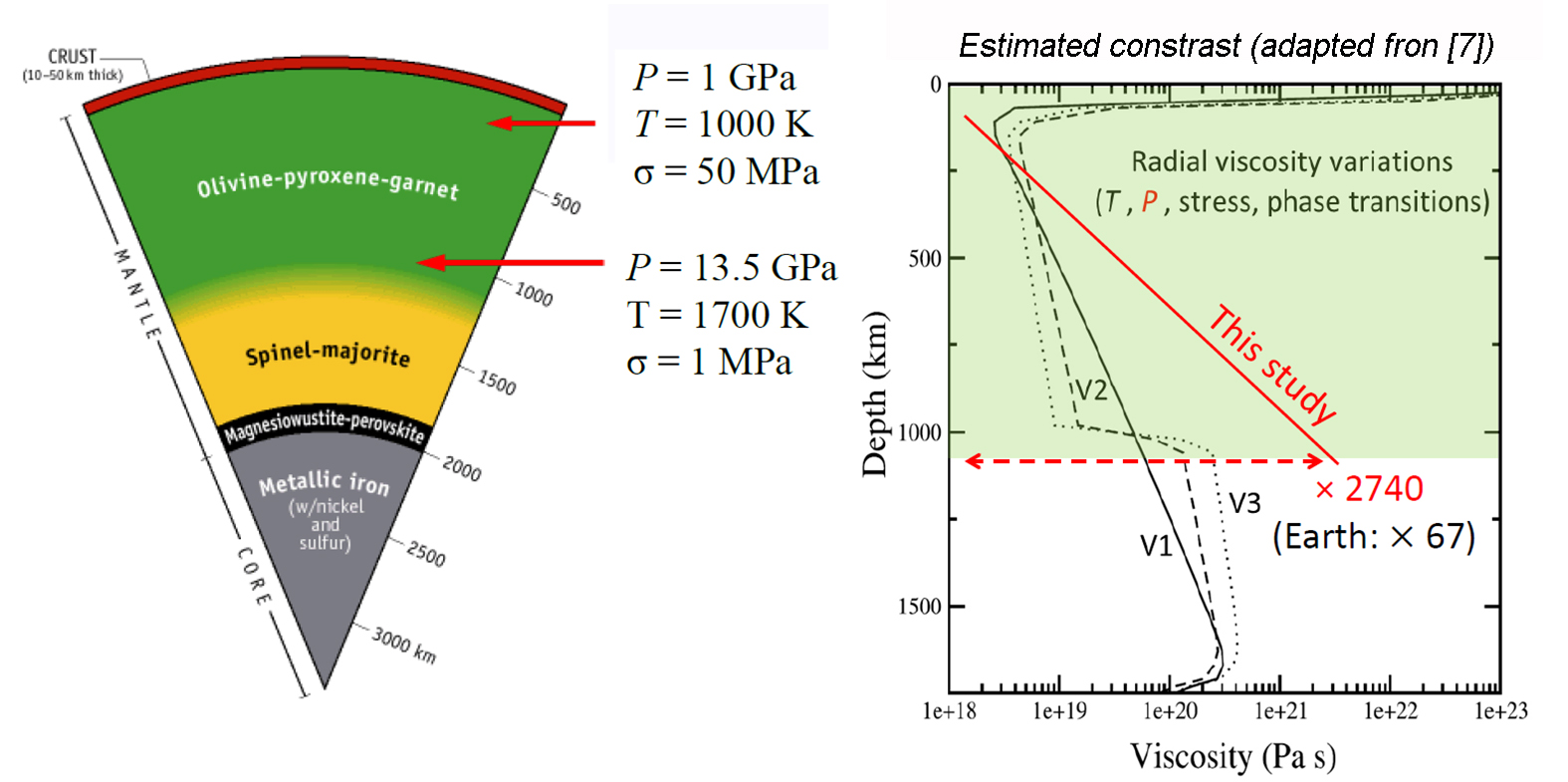


**Effect of Fe content on Olivine Viscosity at High P and Implication for the Martian Mantle**

P. Raterron1,2,3, C. Holyoke4, L. Tokle2, N. Hilairet1, S. Merkel1, G. Hirth2 and D. Weidner3

1UMET, CNRS, Université de Lille, France; 2DEEPS, Brown University, RI ; 3Geosciences, Stony Brook University, NY; 4 Geosciences, Universty of Akron, OH

The upper parts of the mantle of rocky planetary bodies are olivine-rich, with Fe/(Mg+Fe) ratios (Fe#) lower than ~2% for Mercury, up to 25-30% for Mars, and intermediate compositions for the Earth, the Moon and Venus. Olivine represents more than 60 wt.% of the Martian upper mantle [1], where pressure (*P*) and temperature (*T*) reach 13.5 GPa and 1700 K at 1100-km depth. Results from experiments carried out at low pressure (~ 0.3 GPa) indicate that increasing Fe content dramatically decreases olivine viscosity [2]. These data suggest that the Martian upper mantle may be ~10 times less viscous than the Earth’s at the same conditions. Whether such a weakening occurs at the high pressures relevant to Mars interior is unknown. We, thus, carried out deformation experiments in the D-DIA at NSLS X17B@ and APS 6-BM-B synchrotron beamlines on olivine polycrystals with various compositions along the forsterite-fayalite joint. Run *P* and *T* were within 2.5 – 8.0 GPa and 1073 – 1573 K, and strain rate was in the range 0.2 – 14 ×10-5 s-1. Dry specimens with different iron contents, chemically separated from each other by Ni disks, were deformed two by two in order to compare their rheology. Strain rates and the applied stress were measured in situ by X-ray imaging and diffraction, as described elsewhere [3].

The contrast in effective viscosity between Fe-rich olivine and Fe-poor olivine is much lower at high pressure than observed at low pressure (i.e., 300 MPa, [2]). This observation is partly explained by the different strain-rate sensitivities to stress and pressure variations of olivines with different iron contents, which translate in classical power laws into different stress exponents (*n*) and activation volumes (*V\**). By carrying out stress-step and pressure-step experiments, we observed that *n* and *V\** increase with Fe content. A consistent increase of the *n* value with olivine Fe content is also observed at low *P* (a review in [4]). Applying these results to the interior of Mars, with an olivine Fe-content (Fe #) estimated to between 25 and 30 %, and assuming reasonable values for mantle stresses (Figure), we conclude that the viscosity increases with depth in the Martian upper mantle could be up to a factor of 40 times more than estimated in the Earth. Such a strong increase in viscosity may have critical consequences for Mars’ mantle convection mode.

[1] Khan A. & Connolly J.A.D. (2008) *JGR, 113,* E07003. [2] Zhao Y.-H. et al. (2009) *EPSL*, *287,* 229-240. [3] Raterron P. & Merkel S. (2009) *J. Synchr. Rad., 16,* 748-756 ; [4] Bollinger C. et al. (2014) *PEPI*, *228,* 211-219.

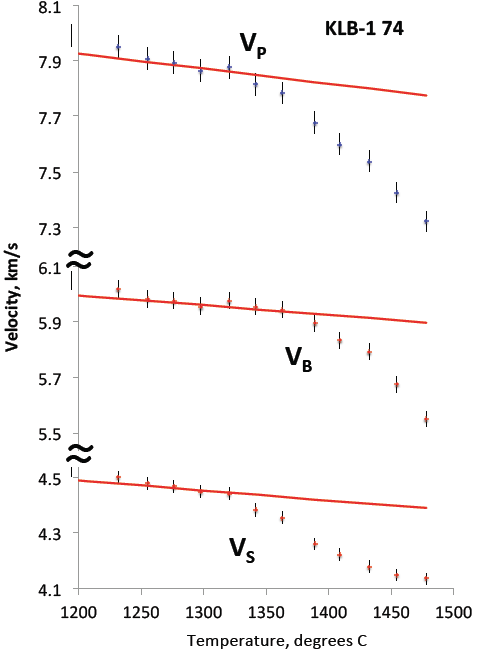
This work is supported by the National Science Foundation (NSF) CSEDI grant EAR-1606793. The NSLS X17-B2 and the APS 6BM-B beamline, respectively was and is supported by the Consortium for Materials Properties Research in Earth Sciences (COMPRES) [NSF EAR 06-49658].

**Ultrasonic Acoustic Velocities During Partial Melting of the Mantle Peridotite, KLB-1 (submitted to JGR, 2017)**

Donald J. Weidner, Li Li, Matthew Whitaker, Richard Triplett

We have measured VP and VS as a function of temperature as KLB-1 begins to melt. The two sound speeds, along with the bulk sound velocity, are illustrated in Figure 1. We find that the values of ∂ lnVS/∂ lnVB were all significantly lower than those calculated by [*Hammond and Humphreys*, 2000] for all melt shapes that they considered implying that the change in bulk modulus was much greater than the classic mixing model would predict. Both our data and the recent data by [*Chantel et al.*, 2016] for partial melts in a system of olivine + basalt are incompatible with the mixing model in this metric, but are compatible with the dynamic melting model in which the stress of the acoustic wave drives the material back and forth between the melt and the solid following the phase equilibrium conditions as proposed by [*Anderson*, 1989; *L Li, Donald J. Weidner*, 2008; *Vaisnys*, 1968]. This model requires sufficiently fast kinetics for this to be effective.

Figure 1. Measured acoustic velocities for KLB-1 as a function of temperature. The longitudinal velocity (VP), shear velocity (VS), and bulk sound velocity (VB) are illustrated as a function of temperature. Error bars of 0.5% includes the estimated uncertainty of sample length. The red lines indicate estimates of the temperature dependence of velocities. The slope of these lines comes from the room-pressure temperature derivative of -0.00036 km/(sec deg) for S waves and -0.00054 km/(sec deg) for P waves for San Carlos olivine calculated from [*Anderson*, 1995]. The validity of this estimate was validated in similar runs that did not encounter partial melting.



Anderson, D. L. (1989), *Theory of the Earth*, 382 pp., Blackwell Scientific Publications.

Chantel, J., G. Manthilake, D. Andrault, D. Novella, T. Yu, and Y. B. Wang (2016), Experimental evidence supports mantle partial melting in the asthenosphere, *Science Advances*, *2*(5), doi:10.1126/sciadv.1600246.

Hammond, W. C., and E. D. Humphreys (2000), Upper mantle seismic wave velocity: Effects of realistic partial melt geometries, *Journal of Geophysical Research-Solid Earth*, *105*(B5), 10975-10986.

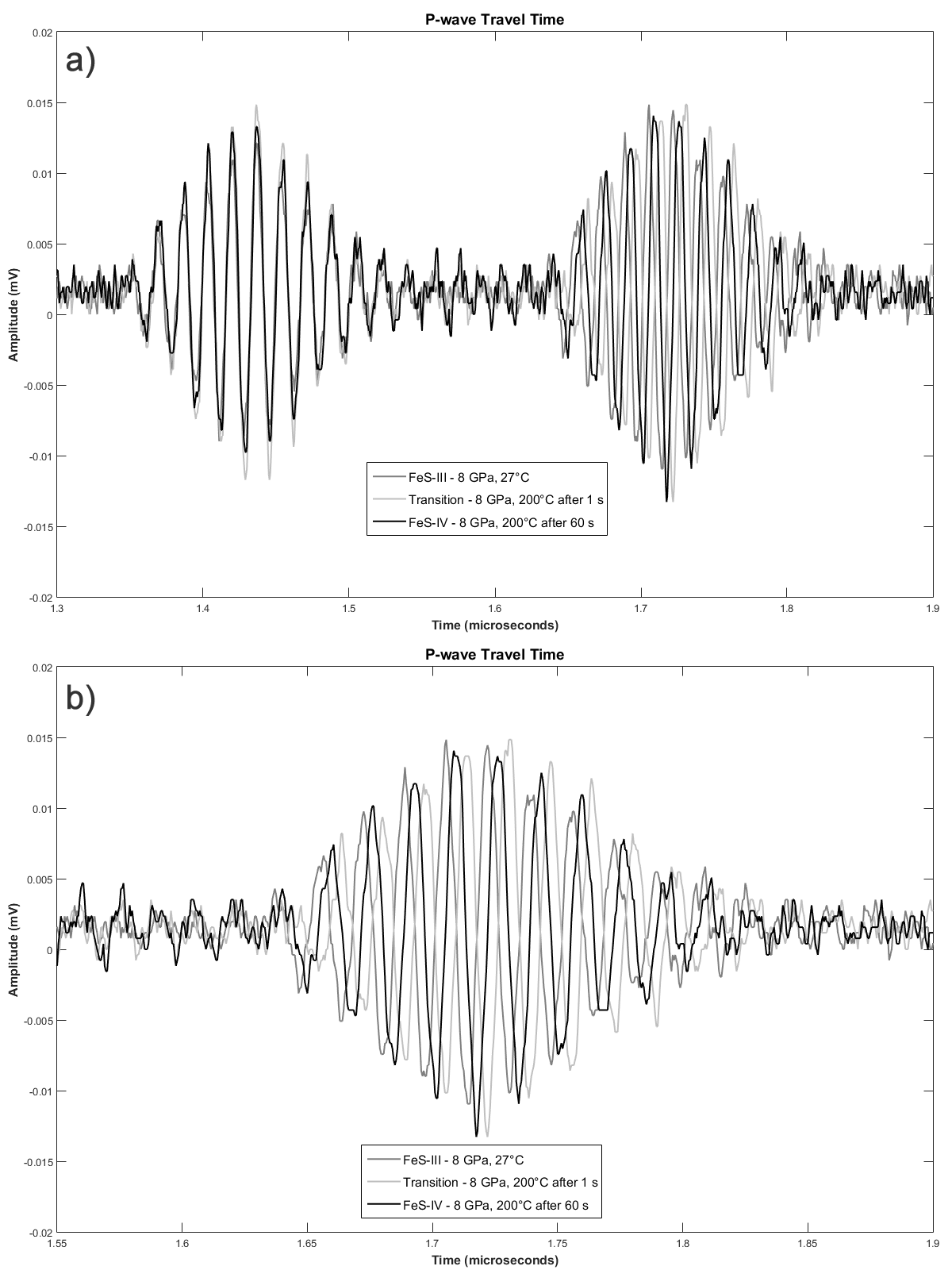
Li, L., Donald J. Weidner (2008), Effect of Phase Transitions on Compressional-Wave Velocites in the Earth's Mantle, *Nature 454* 984.

Vaisnys, J. R. (1968), Propagation of acoustic waves through a system undergoing phase transformations, *Journal of Geophysical Research*, *73*(24), 7675-7683.

**DIASCoPE: Directly Integrated Acoustic System Combined with Pressure Experiments – A new method for fast acoustic velocity measurements at high pressure**

Matthew L. Whitaker1,\*, Kenneth J. Baldwin1, William R. Huebsch1

1Mineral Physics Institute, Stony Brook University, Stony Brook, NY 11794-2100 USA

A new experimental system to measure elastic wave velocities in samples *in situ* under extreme conditions of pressure and temperature in a multi-anvil apparatus has been installed at Beamline 6-BM-B of the Advanced Photon Source at Argonne National Laboratory. This system allows for measurement of acoustic velocities via ultrasonic interferometry, and makes use of the synchrotron beam to measure sample densities via X-ray diffraction and sample lengths using X-radiographic imaging. This system is fully integrated into the automated software controls of the beamline and is capable of collecting robust data on elastic wave travel times in less than one second, which is an improvement of more than one to two orders of magnitude over existing systems. Moreover, this fast data collection time has been shown to have no effect on the obtained travel time results. This allows for more careful study of time-dependent phenomena with tighter snapshots in time of processes that would otherwise be lost or averaged out in other acoustic measurement systems.

The figure to the right shows quite clearly that the P-wave travel times are longer in the FeS-IV phase than in the FeS-III phase. Moreover, the travel times are even longer while this phase transition is taking place than when either phase is the sole stable structure, as is shown by the shift of the light gray signal to the right of the other two. Previously existing systems for ultrasonic velocity measurement at high pressures that take on the order of ~180 seconds to collect data would have easily been able to collect data on the FeS-III (dark gray) and FeS-IV (black) phases. However, their prohibitively long collection times means that the increase in travel time that is observed while the phase transition is actively occurring would be lost and averaged into the longer-term stable signal of the single phase end products.

Whitaker, M.L., Baldwin, K.J., and Huebsch, W.R. (2017) DIASCoPE: Directly Integrated Acoustic System Combined with Pressure Experiments – A new method for fast acoustic velocity measurements at high pressure. Review of Scientific Instruments, 88, 034901. DOI 10.1063/1.4977596