**COMPRES/GSECARS Gas Loading Facility at the APS**

2016 COMPRES Annual Report

November 11, 2016

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## Overview

The COMPES/GSECARS gas loading system at the APS has been a major advance for the U.S. diamond anvil cell community. Prior to the installation of this system in 2008 the use of noble-gas pressure media was restricted to a small number of scientists with access to systems at the Carnegie Institution or the Lawrence Livermore Laboratory. The COMPRES/GSECARS system has led to the improved hydrostatic conditions with Ne or He now being the norm for most synchrotron experiments in this country. This has greatly improved the quality of the measurements being made, and the system is available to the entire community.

The system began operation in February 2008 and has been running with minimal downtime since then. The system works extremely well, with the only significant problems being some failures of the commercial compressor. We have in-house technical support (Guy Macha) to repair such problems, and the mean time to repair has typically been 1 day. We have recently purchased a spare compressor, so that we can rapidly swap it if there is a major problem.

The COMPRES/GSECARS system at the APS is available for use by any member of the COMPRES community, regardless of whether they are performing experiments at GSECARS, at another APS sector, at another synchrotron, or in their home laboratory. The support from COMPRES allows the system to be available for users who cannot afford the time or money to travel to APS, by providing a “mail-in” service. It also allows the system to be available to users who are conducting experiments at APS sectors other than GSECARS. These include users from sectors 3 (inelastic), 4 (magnetism), 16 (HP-CAT), and 32 (imaging), 34 (microdiffraction), and others.

**Scientific Highlights**

The publication list in the appendix lists 74 papers that have been published in 2015 and 2016 using the gas loading system. We present here the abstracts from 3 of these papers.

Zhu Mao, Dawei Fan, Jung-Fu Lin, Jing Yang, Sergey N. Tkachev, Kirill Zhuravlev, Vitali B. Prakapenka, "Elasticity of single-crystal olivine at high pressures and temperatures," Earth Planet. Sci. Lett. 426, 204-215 (2015). DOI: 10.1016/j.epsl.2015.06.045

Elasticity of single-crystal San Carlos olivine has been derived from sound velocity and density measurements at simultaneous high pressure–temperature conditions up to 20 GPa and 900 K using in situ Brillouin spectroscopy and single-crystal X-ray diffraction in externally-heated diamond anvil cells. These experimental results are used to evaluate the combined effect of pressure and temperature on full elastic constants of single-crystal olivine to better understand its velocity profiles and anisotropies in the deep mantle. Analysis of the results shows that the shear moduli display strong concave behaviors as a function of pressure at a given high temperature, while the longitudinal modulus, $C_{11}$, and the off-diagonal moduli, $C_{12}$ and $C_{13}$, exhibit greater temperature dependence at higher pressures than at relatively lower pressures. Using a finite-strain theory and thermal equation of state modeling for a pyrolitic mantle composition along an expected mantle geotherm, our results show that the magnitude of the $V_P$ and $V_S$ jumps at the 410-km depth are 6% and 6.4%, respectively, which are greater than that found in seismic observations, suggesting a mantle olivine content of 40–50 vol%, which is less than what is expected for the pyrolite model. Our modeled velocity profiles for a metastable olivine wedge in the subduction slabs along a representative cold slab geotherm are 6% and 10% lower than those of wadsleyite and ringwoodite, respectively, at corresponding depths of the normal mantle. Our modeled results also show that metastable olivine in the cold slabs could have strong $V_P$ and $V_S$ anisotropies. The maximum $V_P$ anisotropy is estimated to be 19–22% at transition zone depth, whereas the maximum $V_S$ splitting is 13–23% and increases with depth. As a result, the presence of a metastable olivine wedge at the transition zone depth would exhibit a seismic signature of low velocity and strong seismic anisotropy which are consistent with recent seismic observations for various locations of the slabs and can be used as mineral physics constraints for future seismic detections of the metastable olivine wedges in the deep mantle.

Leyla Ismailova, Elena Bykova, Maxim Bykov, Valerio Cerantola, Catherine McCammon, Tiziana Boffa Ballaran, Andrei Bobrov, Ryosuke Sinmyo, Natalia Dubrovinskaia, Konstantin Glazyrin, Hanns-Peter Liermann, Ilya Kupenko, Michael Hanfland, Clemens Prescher, Vitali Prakapenka, Volodymyr Svityk, Leonid Dubrovinsky, "Stability of Fe,Al-bearing bridgmanite in the lower mantle and synthesis of pure Fe-bridgmanite," Sci. Advances 2 (7), e1600427-1-e1600427-8 (2016). DOI: 10.1126/sciadv.1600427

The physical and chemical properties of Earth's mantle, as well as its dynamics and evolution, heavily depend on the phase composition of the region. On the basis of experiments in laser-heated diamond anvil cells, we demonstrate that Fe,Al-bearing bridgmanite (magnesium silicate

perovskite) is stable to pressures over 120 GPa and temperatures above 3000 K. Ferric iron stabilizes Fe-rich bridgmanite such that we were able to synthesize pure iron bridgmanite at pressures between ~45 and 110 GPa. The compressibility of ferric iron–bearing bridgmanite is significantly different from any known bridgmanite, which has direct implications for the interpretation of seismic tomography data.

June K. Wicks, Jennifer M. Jackson, Wolfgang Sturhahn, Kirill K. Zhuravlev, Sergey N. Tkachev, Vitali B. Prakapenka, "Thermal equation of state and stability of (Mg[subscript 0.06]Fe[subscript 0.94])O," Phys. Earth Planet. In. 249, 28-42 (2015). DOI: 10.1016/j.pepi.2015.09.003

We present the pressure-volume-temperature (P-V-T) equation of state of polycrystalline (Mg_(0.06)Fe_(0.94))O (Mw94) determined from laser-heated x-ray diffraction experiments up to 122 GPa and 2100 K, conditions approaching those of the deep mantle. We conducted two sets of experiments, one with an in situ Fe metal oxygen fugacity buffer and one without such a buffer. The internal pressure markers used in these experiments were B2-NaCl and hcp-Fe in the buffered experiment and B2-NaCl in the unbuffered experiment. In the sampled P-T range of the high temperature part of this study, only the B1 structure of Mw94 was observed, indicating that the addition of Mg to FeO stabilizes the B1 phase with respect to the B8 phase at these conditions. Both datasets were fit to a Birch-Murnaghan and Mie-Grüneisen-Debye thermal equation of state using a new open-source fitting routine, also presented here. Analysis of these data sets using the same internal pressure marker shows that the P–V–T data of Mw94 obtained in the unbuffered experiment are well explained by the equation of state parameters determined from the buffered data set. We have also compared the thermal equation of state of Mw94 with that of wüstite and conclude that Mw94 has measurably distinct thermoelastic properties compared with those of wüstite. We use the results obtained in the buffered experiment to determine the density and bulk sound velocity of Mw94 at the base of the mantle and compare these values to geophysical observations of ultralow-velocity zones.

**Personnel**

This COMPRES project partially funds staff scientist to reside at the APS. Dr. Sergey Tkachev began in this position in 2010. Sergey is responsible for loading cells that are sent to the APS by users who do not travel here. He is also responsible for training and assisting all of the on-site users with loading their cells. The other part of Sergey's salary and responsibilities is covered by GSECARS.

Guy Macha is a GSECARS funded technician who provides mechanical support for the system.


**Figure 1.** Dr. Sergey Tkachev with the COMPRES/GSECARS gas loading system

**Operations, Performance Metrics**

The following table summarizes the mail-in service in the past year, since November 1, 2015.

| University | Name | Number of DACs loaded |
|---|---|---|
| Lawrence Berkeley National Laboratory | Camelia Stan + Christine Beaver | 2 |
| University of Nevada Las Vegas | Melanie White + Dean Smith + Ashkan Salamat | 7 |
| Stanford University | Yu Lin | 2 |
| Yale University | Kanani Lee + Kiersten Dawau + Neala Creasy + Sarah Arveson + Jie Deng + Jennifer Gerard | 15 |
| Arizona State University | Dan Shim + Beyongkwan Ko + Huawei Chen + Carole Nisr | 14 |
| University of Utah | Shanti Deemyad | 2 |
| Northwestern University | Steve Jacobsen + Fei Qin + Michelle Wenz + Xiaobing Liu | 6 |
| University of California Santa Cruz | Cara Vennari | 1 |
| University of Hawaii | Przemek Dera | 4 |
| Western University | Sean Shieh + Tianqi Xie + Mauritz van Zyl + Jinbao Wang + Ching Pao Wang | 6 |
| University of Chicago | Yejun Feng + Elizabeth Thompson + Anne Davis + Andrew Campbell | 19 |
| University of California, Berkeley | Shuai Zhang | 2 |
| Jilin University | Xiaoli Huang + Fangfei Li + Bo Han | 4 |
| Brookhaven National Laboratory | Xinguo Hong | 2 |
| Carnegie Institution of Washington | Qingyang Hu + Ross Hrubiak | 5 |
| **TOTAL** | | **91** |

In addition to this mail-in service, there have been 674 diamond anvil cells loaded in this year. 343 of these cells were prepared for experiments at GSECARS, while the remaining 331 were prepared for experiments at other APS sectors, home laboratories and other synchrotrons. The gas loading of all 674 cells were directly assisted by Sergey. Thus on average he is assisting in gas loading of more than 2 cells every working day. Sergey spends more than 60% of his time

assisting such users and providing the mail-in service. Because of the constantly growing usage of the GSECARS/COMPRES gas loading system during the normal APS user operation periods, significant time is required for repairs and preventative maintenance for the valves and commercially built compressor. Sergey and Guy perform these tasks, during the downtime between the APS beamtime cycles.

**Community/Broader Impacts**

The success of the COMPRES/GSECARS system design has led other groups to copy many aspects of it for systems of their own, including HPSync at the APS. In addition four other identical units have been built by the University of Chicago Engineering Center. These have been sold to Jennifer Jackson at Caltech, the Advanced Light Source at the Lawrence Berkeley National Laboratory (where it is available to users of the COMPRES supported high-pressure beamline, 12.2.2), Sandia National Laboratory, and the LNLS synchrotron in Brazil. Sergey has provided valuable expertise and hands-on support during construction for these new gas loading systems. There are expressions of interest for additional systems, both in the US and abroad.

**Planned Activities**

All activities listed in the previous sections will continue during the next COMPRES funding cycle (June 2017 – May 2018).

**Publications**

The publications listed below are those that reported that they used the COMPRES/GSECARS gas loading system in the APS publication database system. This list is certainly incomplete, because it requires users to specify that they used the gas loading system, in addition to which APS beamline they used, when submitting publications to the database system. It also does not include any publications resulting from the mail-in service, since those typically don't use APS beamlines and so would not be entered into the APS publication database and we have no other way of tracking them.

**Publications that acknowledge COMPRES/GSECARS Gas Loading System in the APS Publication Database**
**(2015, 2016)**


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