

IXS at High Pressure and Temperature at 3-ID of the APS

2014 COMPRES Annual Report

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Ercan Alp, Jiyong Zhao, Michael Hu, Ahmet Alatas, Thomas Toellner, and Wenli Bi

Overview

The inelastic x-ray scattering (IXS) at extreme temperature and pressure (between 10 and 3500 K) is a unique capability provided to COMPRES Users through a COMPRES Partner User Proposal at the APS 3-ID-C beamline, as well as supported by General User Proposals.

The capability involves access to two instruments, i) nuclear resonant scattering at 3-ID- B and 3-ID-D, and ii) HERIX-3: High Energy Resolution Inelastic X-Ray Spectrometer at 3-ID-C.

The special capabilities involve

- i) micro focused beam with 10 micron beamsizes at 3-ID-B and 17 micrometer beamsizes at 3-ID-C,
- ii) 1 meV or better resolution monochromators for krypton, iron, tin, europium, and dysprosium Mössbauer isotopes, and a cryogenically cooled high-resolution monochromator at 21.65 keV for the HERIX-3 instrument,
- iii) on-line Ruby pressure calibration system in 3-ID-B,
- iv) panoramic membrane DAC's,
- v) combined inelastic spectrometer/x-ray diffraction capability for single crystal structure and orientation determination inside the DAC,
- vi) combined nuclear resonant inelastic spectrometer with Mossbauer spectroscopy and x-ray diffraction capability for atomic and electronic structure determination, and
- vii) high temperature, double-sided, fiber IR- laser heating capability to reach above 3500 K, and internal resistive heating capability to reach 1000 K. Includes spectroradiometric temperature measurements determined from CCD and FasTeR spectrometers.

In addition, we offer a conventional Mössbauer spectroscopy laboratory with two Mössbauer drives as another resource, one that is used frequently by COMPRES researchers. With the availability of point-sources, spectra can be obtained from high pressure samples inside a DAC.

Scientific Highlights

We will present the highlights based on each of the COMPRES User groups:

1) Jennifer Jackson (Caltech)

This group from Caltech has advanced the field by applying the data extracted from all aspects of nuclear resonant scattering to interesting geophysical problems like determining the first set of extremely high statistical NRIXS data quality to constrain thermoelastic parameters of hcp-iron to 171 GPa, and constraining the temperature of terrestrial planetary cores. Hyperfine interactions like quadrupole splitting and isomer shifts are used to address phenomena related to spin transitions in iron containing minerals. Sound velocity is used to interpret seismic observations. And significant drop in recoil free fraction is exploited to measure melting points of iron and its alloys under pressures. In addition to high-level publications, Jackson's group is involved in installing a fast temperature readout system with 100 Hz readout frequency to facilitate melting studies.

Publications:

Bin Chen, Jennifer M. Jackson, Wolfgang Sturhahn, Dongzhou Zhang, Jiyong Zhao, June K. Wicks, Caitlin A. Murphy, ["Spin crossover equation of state and sound velocities of \(Mg_{0.65}Fe_{0.35}\)O ferropericlasite to 140 GPa,"](#) J. Geophys. Res. **117**, B08208-1-B08208-9 (2012).

Jennifer M. Jackson, Wolfgang Sturhahn, Michael Lerche, Jiyong Zhao, Thomas S. Toellner, E. Ercan Alp,

Stanislav V. Sinogeikin, Jay D. Bass, Caitlin A. Murphy, June K. Wicks, ["Melting of compressed iron by monitoring atomic dynamics,"](#) Earth Planet. Sci. Lett. **362**, 143-150 (2013).

Caitlin A. Murphy, Jennifer M. Jackson, Wolfgang Sturhahn, ["Experimental constraints on the thermodynamics and sound velocities of hcp-Fe to core pressures,"](#) J. Geophys. Res. **118** (5), 1999-2016 (2013).

Daoyuan Sun, Don V. Helmberger, Jennifer M. Jackson, Robert W. Clayton, Dan J. Bower, ["Rolling hills on the core-mantle boundary,"](#) Earth Planet. Sci. Lett. **361**, 333-342 (2013).

June K. Wicks, "Sound Velocities and Equation of State of iron-rich (Mg,Fe)O", Ph.D Thesis. California Institute of Technology (2013).

Dongzhou Zhang, "Applications of nuclear resonant scattering to further our understanding of Earth's interior" Ph.D. Thesis. California Institute of Technology (2014).

Dongzhou Zhang, Jennifer M. Jackson, Bin Chen, Wolfgang Sturhahn, Jiyong Zhao, Jinyuan Yan, Razvan Caracas, ["Elasticity and lattice dynamics of enstatite at high pressure,"](#) J. Geophys. Res. **118** (8), 4071-4082 (2013).

Dongzhou Zhang, Jennifer M. Jackson, Jiyong Zhao, Wolfgang Sturhahn, E. Ercan Alp, Thomas S. Toellner, and Michael Y. Hu "Fast temperature readout system for monitoring atomic dynamics" Rev. Sci. Instr (2014, under review)

2) Jung-Fu (Afu) Lin (U Texas-Austin)

The group from Texas-Austin is very active in using HERIX-3 instrument for measuring the effect of temperature on sound velocities under high pressure. More recently, they are involved in developing the low temperature-high pressure capability for nuclear resonant inelastic x-ray scattering studies, a first in the world. The immediate application of this development is in the study of iron based compounds, and alloys.

Publications:

Jin Liu, Jung-Fu Lin, Ahmet Alatas, Wenli Bi, ["Sound velocities of bcc-Fe and Fe_{0.85}Si_{0.15} alloy at high pressure and temperature,"](#) Phys. Earth Planet. In. **233**, 24-32 (2014).

Zhu Mao, Jung-Fu Lin, Jing Yang, Junjie Wu, Heather C. Watson, Yuming Xiao, Paul Chow, Jiyong Zhao, ["Spin and valence states of iron in Al-bearing silicate glass at high pressures studied by synchrotron Mössbauer and X-ray emission spectroscopy,"](#) Am. Mineral. **99** (2-3), 415-423 (2014).

3) Sang-Heon Dan Shim (Arizona State University)

This group has become more active recently, and accomplished a number of new experiments, involving understanding the oxidation and spin states of iron in pyroclitic and basaltic Mg-Pvs at high pressure in the laser-heated diamond-anvil cell.

Publications:

Chen Gu, Krystle Catalli, Brent Grocholski, Lili Gao, Ercan Alp, Paul Chow, Yuming Xiao, Hyunchae Cynn, William J. Evans, Sang-Heon Shim, ["Electronic structure of iron in magnesium silicate glasses at high pressure,"](#) Geophys. Res. Lett. **39** (24), L24304-1-L24304-5 (2012).

4) Bin Chen

This group from University of Hawaii (previously at COMPTECH) has focused on studying the effect of temperature on the sound velocities of Fe₇C₃ at pressures up to 150 GPa.

PUBLICATION:

Bin Chen, Zeyu Li, Dongzhou Zhang, Jiachao Liu, Michael Y Hu, Jiyong Zhao, Wenli Bi, E. Ercan Alp, Yuming Xiao, Paul Chow, Jackie Li, “Hidden carbon in Earth's inner core revealed by shear softening in dense Fe_7C_3 ”, (Accepted, early edition available on web) PNAS.

5) Nicolas Dauphas

This group has been focused on studies of isotopes fractionation in spinels and glasses to reveal redox conditions in the mantle, and iron - silicon fractionation during basalt generation in the Earth's mantle. This group recently extended their studies to include high pressure, as well as high temperatures.

Publication:

N. Dauphas, M. Roskosz, E.E. Alp, D.R. Neuville, M.Y. Hu, C.K. Sio, F.L.H. Tissot, J. Zhao, L. Tissandier, E. Médard, C. Cordier, [“Magma redox and structural controls on iron isotope variations in Earth's mantle and crust”](#), Earth and Planetary Science Letters, 398, 127–140 (2014)

6) Michael J. Krawczynski (previously postdoc working with Prof. James A. Van Orman at Case Western Reserve University, now Assistant Professor at Washington University in St. Louis)

Dr. Krawczynski has studied the Fe isotope fractionation of FeNi, FeS, FeP, Armalcolite using NRIXS. A manuscript is in preparation.

7) Wenli Bi (UIUC/ANL)

Dr. Bi has been focusing on studies of magnetism in rare earth metals over 1 Mbar pressure at low temperature using synchrotron Mossbauer spectroscopy. A manuscript for publication is in preparation. Dr. Bi is also heavily involved in the development of high pressure and low temperature NRIXS. During testing and commissioning this novel technical capability she has studied the magnetism and lattice dynamics of EuFe_2As_2 under pressure. A manuscript will be submitted soon. Dr. Bi also organizes highly successful workshops related these efforts (see below).

8) Caitlin Murphy

Dr. Murphy from Carnegie Institute of Washington has focused on studies of phonon dispersion relations of single crystal SnO_2 at high pressure using HRIXS. A manuscript is in preparation related to these studies.

Personnel

3-ID beamline is owned and operated by the x-ray science division of the Advanced Photon Source. There are 5 full-time staff, paid by the APS, and a post-doctoral researcher, partially funded by the APS (30 %) and by COMPRES (70 %). In addition, the beamline has full access to APS's technical and engineering support system. The group is part of Inelastic X-Ray scattering group, led by Dr. Thomas Gog, who reports to Dr. Jonathan Lang, the Associate Director of the XSD, and Dr. Linda Young, the Director of XSD. No changes are expected in the coming year to the management structure.

Operations

The beamline operates under the general rules set by the APS. 80 % of the time goes to General User Program. The PUP in place for COMPRES carries a weight of 10 %, or about 1 week in each cycle, with a total of 3 weeks each year. However, this is heavily leveraged because the ideas tested during this time leads to outstanding proposals in the GUP system.

Performance Metrics

No of COMPRES Users: 12 PI's plus their students and postdoctoral associates, and sometimes their collaborators

No of beamtime proposals submitted in the last 12 months: 24

No of papers published in the last 12 months: 7

No of PhD students engaged: ~ 12

No of workshops arranged: 3

No of Grant applications supported by letters: 6

Community/Broader Impacts

Nuclear Resonance Workshops & Symposia

The NRS workshop will be held on Nov. 7-9, 2014 at Argonne National Laboratory. The previous workshop was held in November 2012 with 39 participants. These workshops are organized as part of the COMPRES Education, Outreach and Infrastructure Development programs to promote the application of the state-of-the-art Nuclear Resonant Scattering (NRS) techniques for characterizing the properties of materials under the high P-T conditions of planetary interiors. As of today 33 people from 17 institutions has registered to attend the workshop. The participants are given a hands-on training opportunity to learn data evaluation software like CONUSS and PHOENIX. This year, SciPHON will be introduced first time to users.

We organize the only Mössbauer Spectroscopy Symposium in North America biannually. The previous **North American Mössbauer Spectroscopy Symposium** was held in University of Texas at Austin, Austin, TX, (chaired by J. F. Lin and E. E. Alp of COMPRES) (January 2012) and it was partially supported by COMPRES. The next meeting is scheduled to take place in January 9-10, 2014, Northeastern University, Boston, MA.

Participation in the COMPRES Annual Meeting

We have actively participated the 2014 COMPRES annual meeting at Skamania Lodge, WA. We presented three posters demonstrating our experimental capabilities of HERIX and NRS techniques and their applications in geosciences studies. We also presented one contributed talk to present the development of high pressure and low temperature NRIXS capability.

Development of new capabilities:

i) low temperature-high pressure NRIXS

The development of a novel capability of low temperature and high pressure NRIXS is in progress. We have recently applied and were approved for support from COMPRES Education, Outreach and Infrastructure Development programs. We have a PUP in progress dedicated to this development. We have demonstrated the feasibility using a miniature panoramic DAC and a helium continuous flow cryostat. We are now in process of purchasing a new cryostat to be able to reach temperature of 4 K and making membrane adapter for the miniature DAC to have in-situ pressure tuning capability.

ii) Single crystal orientation inside DAC using an area detector

We have successfully implemented an area detector to orient single crystals inside the DAC, as well as to determine crystal structure under high pressure at the HERIX-3 instrument. Dr. Przemek Dera installed and educated the beamline staff how to use the software. This is now routinely available for the users.

Our staff has delivered many invited talks nationally and internationally at high-pressure related meetings, as well as in general meetings.

Planned Activities

1. Development of low temperature/high-pressure capability, a PUP is in progress
2. Upgrade of the high-heat load monochromator (\$200k, paid by ANL-LDRD)
3. Fast chopper system for Synchrotron Mössbauer Spectroscopy (\$500k, paid by ANL)
4. A new ^{57}Co source (10 mCi, point source) for the Mössbauer Laboratory (paid by ANL-Chemical Sciences)

Budget Request for 2015-2016

Postdoc salary, baseline	\$52,348
Total salaries	\$52,348
Fringe benefits (33.06%)	\$17,306
Total wages and benefits	\$69,655
Postdoc travel (off campus)	\$5,000
Services (conf. reg. Fees; off campus)	\$400
Total direct cost (off campus)	\$75,055
Total indirect cost (off campus, 24%)	\$18,013
Total cost	\$93,068
APS cost sharing	(\$21,151)
Total request from COMPRES	\$71,916

Appendices

Publications

N. Dauphas, M. Roskosz, E.E. Alp, D.R. Neuville, M.Y. Hu, C.K. Sio, F.L.H. Tissot, J. Zhao, L. Tissandier, E. Médard, C. Cordier, [“Magma redox and structural controls on iron isotope variations in Earth's mantle and crust”](#), Earth and Planetary Science Letters, 398, 127–140 (2014)

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