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

2017 COMPRES Annual Report, November 2016 – October 2017

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Overview

The inelastic x-ray scattering (IXS) at extreme temperature (between 10 and 3500 K) and high pressures is a unique capability provided to COMPRES Users through General User Proposals as well as a COMPRES Partner User Program at the APS 3-ID beamlines.

The capability involves access to two types of instruments: i) nuclear resonant scattering at 3-ID-B, 3-ID-D, and 30-ID-B, and ii) HERIX-3 and HERIX-30, the High Energy Resolution Inelastic X-Ray Spectrometers at 3-ID-C, 30-ID-C.

The special capabilities involve

i) Micro-focused beam with 10 microns beam size at 3-ID-B and 17 microns beam size at 3-ID-C, and 30 microns at 30-ID-B and 30-ID-C.
ii) 1 meV or better resolution monochromators for ^{57}Fe, ^{119}Sn, ^{151}Eu, ^{161}Dy, ^{83}Kr Mössbauer isotopes, and cryogenically cooled high-resolution monochromators at 21.65 keV for the HERIX-3 and 23.71 keV for the HERIX-30 instruments.
iii) On-line Ruby system at 3-ID-B and 30-ID-C for pressure measurement and offline Raman system.
iv) Symmetric DACs for synchrotron Mössbauer experiment and screw- and membrane-type panoramic DAC's for nuclear resonant inelastic x-ray scattering experiments and compatible gas loading gearboxes for gas loading at GSECARS
v) Cryogenically cooled cryostat and superconducting magnet (7T) suitable for cooling samples under pressure for magnetism studies.
vi) Combined inelastic spectrometer/x-ray diffraction capability for single-crystal structure and orientation determination inside the DAC at HERIX-3 and HERIX-30 spectrometers.
vii) Combined nuclear resonant inelastic spectrometer with Mössbauer spectroscopy and x-ray diffraction capability for atomic and electronic structure determination, and equation-of-state measurements.
viii) 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 system.

In addition to these synchrotron-based capabilities, we offer an offline conventional Mössbauer spectroscopy (CMS) 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 taken from samples at high pressure inside a DAC or recovered samples after high-pressure experiments.

Scientific Highlights

Sound velocity and density of magnesiowüstites

The effect of Mg/Fe substation on the sound velocities of iron-rich $(Mg_{1-x}Fe_x)O$ was investigated using the NRIXS technique as a function of pressure, approaching those of the lowermost mantle. The systematic cation substitution in the Fe-rich limit has the potential to play an important role in the interpretation of seismic observations of the core-mantle boundary. By determining a relation between sound velocity, density, and composition of (Mg,Fe)O, this study explores potential constraints on the nature of ultralow-velocity zones at the core-mantle boundary (Wicks, *et al.* 2017).

Stability of ferrous-iron-rich bridgmanite under reducing midmantle conditions:

The current understanding of the electronic state of iron in lower mantle minerals leads to a considerable disagreement in bulk sound velocity with seismic measurements if the lower mantle has the same composition as the upper mantle. In modeling studies, the content and oxidation state of Fe in minerals have been assumed to be constant throughout the lower mantle. Combining synchrotron Mössbauer spectroscopy, X-ray diffraction, electron energy loss spectroscopy, and energy-dispersive X-ray spectroscopy, an unexpected change in oxidation state of Fe in bridgemanite, the dominant mineral in the lower mantle, was found. The oxidation state change resolves the discrepancy between laboratory and seismic studies on the chemical composition of the lower mantle showing that the lower mantle has major-element chemistry similar to the upper mantle. The oxidation state change will also lead to a lower Fe content in bridgmanite in the midmantle, whereas the total Fe content remains the same. Such a change can lead to an increase in viscosity at 1,100- to 1,700- km depths, providing a viable mineralogical explanation of the possible viscosity elevation suggested by geophysical studies at the same depth range (Shim *et al.,* 2017).

Iron isotopic fractionation between silicate mantle and metallic core at high pressure:

The +0.1‰ elevated $^{56}Fe/^{54}Fe$ ratio of terrestrial basalts relative to chondrites was proposed to be a fingerprint of core-mantle segregation. However, the extent of iron isotopic fractionation between molten metal and silicate under high-pressure-temperature conditions is poorly known. Based on the mean force constant extracted from NRIXS data, the equilibrium iron isotope fractionation between silicate and iron under core formation conditions in Earth is ~0-0.02‰, which is small relative to the +0.1‰ shift of terrestrial basalts. This result is unaffected by small amounts of nickel and candidate core-forming light elements, as the isotopic shifts associated with such alloying are small. This study suggests that the variability in iron isotopic composition in planetary objects cannot be due to core formation (Liu *et al.,* 2017).

Beamline Personnel

The 3-ID beamline is owned and operated by the X-ray Science Division of the Advanced Photon Source (APS), Argonne National Laboratory. There are 5 full-time staff paid by the APS, and a Senior Spectroscopist researcher (Wenli Bi), jointly funded by COMPRES (60%) and the APS (40%). In addition, the beamline has full access to APS's technical and engineering support system. The group is part of the Inelastic X-Ray and Nuclear Resonant Scattering (IXN) group, led by Dr. Thomas Gog, who reports to Dr. Jonathan Lang, the Director of XSD at the APS.

Beamline Operations

Experiments are performed at the 3-ID and 30-ID beamlines based on general user proposals (GUP's). The oversubscription rate at 3-ID is 2.5. During the period of this report, there were 42 and 14 GUP requests from Earth science and high-pressure research groups for 3-ID and 30-ID, respectively. Of these GUP requests 23 (55 % of the total requests) and 6 (43 %) were granted beam time at Sectors 3-ID and 30-ID, respectively. These percentages are well above the average acceptance rate, which is below 30%. The relatively high success rates for COMPRES proposals are due to the excellent quality of research that these techniques attract from the COMPRES community, and partly due to the close collaboration between the beamline staff and user groups to develop effective proposals that are highly competitive for beam time. During the last year, 18 COMPRES user groups have been allocated beam time. About 22 graduate and 1 undergraduate students, and 16 postdocs participated experiments at 3-ID and 30-ID.

The offline Mössbauer lab provides mail-in service for COMRPES users for beamline related experiments as well as individual sample characterization. It has been in high demand. During the last year over 35 samples from 13 COMPRES user groups have been measured.

The offline Raman system has been used routinely by users from Sector 3 and other beamlines. Since the Raman system was relocated from Sector 13 to Sector 3 and activated in June 2016, 18 research groups have used the system over 50 times for pressure measurements by either ruby fluorescence or diamond Raman edge methods, as well as characterizing samples by Raman spectroscopy.

During the last two years the share of NRS beamtime at 3-ID has gone up from 60 to 80% with the application of the hybrid filling mode in NRS experiments. Furthermore, 10% additional beam time is now allocated for ^{119}Sn experiments at Sector 30, which reduces the burden on Sector 3 and allows more time for COMPRES users. Additionally, due to relocation of the MERIX instruments previously at Sector 30 to Sector 27 the total time for IXS measurements between sector 3 and 30 has gone up from 70% to 90%.

Performance Metrics

In the period of this report 18 groups from geosciences/COMPRES community were allocated beam time at 3-ID and 30-ID. The average time allocated for COMPRES users is ~49% of the total available beam time. Detailed information of the allocation is listed in the Appendix.

Beamline Community Activities

- Organizing the NRS Workshop 2017: CONUSS and Synchrotron Mössbauer Data Analysis, Nov. 16-19, 2017 at the Advanced Photon Source, Argonne National Laboratory. The workshop was jointly funded by COMPRES and APS.

- Organized a focused session titled "Nuclear Resonant and Inelastic X-ray Scattering at High Pressures" at the 26th AIRAPT meeting in Beijing, August 19-23.
- Organized a workshop on High energy resolution Inelastic X-ray Scattering Workshop during the 2017 APS/CNM Users Meeting, APS, ANL, May 9, 2017
- Active participation in the COMPRES annual meeting.
- Organized a workshop on Nuclear Resonant Scattering and Data Analysis, Nov. 11-13, 2016, APS, ANL.
- Year around training of users for beamline experiment operation and data analysis.
- Offered mail-in service to COMPRES users of the Mössbauer lab.
- Beamline staff is heavily involved with beam time proposal writing, diamond anvil cell preparation, sample loading, beamline experimental setup, data collection, data analysis and interpretation, and manuscript writing for users.

Beamline Development

- Upgraded the high-heat-load monochromator from water-cooled diamond to cryogenically cooled Si ($250k, paid by ANL-LDRD). This development increased the throughput of the high-resolution monochromators by a factor of two, and provided additional energy stability for ease of experiments.
- Acquired and commissioned a modern Mössbauer spectrometer (LDRD funds from ANL, $25K).
- Commissioned the high-pressure setup for high-pressure NRS experiments on ^{119}Sn at Sector 30, the strongest nuclear resonant source for Sn worldwide by a factor of ten.

Planned Activities

The work at Sector 3 and Sector 30 has a steady flow of users from the COMPRES community, notably including beamtime requests from new users. The following activities are planned for the next year of the APS and COMPRES funding:

- Workshop on Nuclear Resonant Scattering and Data Analysis, November, 2018.
- Fast chopper system for Synchrotron Mössbauer Spectroscopy ($500k, paid by ANL) : (Strong likelihood of delivery by early 2018)

- **We propose to acquire an online Raman system for in-situ pressure measurements for experiments above 30 GPa and for taking Raman spectra simultaneously on samples. This would be our main new goal to accomplish in the next FY. It will facilitate the work of all COMPRES users who plan to work above 30 GPa and working on sample synthesis under extreme pressure and temperature conditions. The proposed online Raman system will enable diamond Raman peak pressure measurements at pressures where ruby fluorescence is weak, and will allow such measurements without removing the diamond cell from the beamline. This will greatly reduce dead-time that results from off-line Raman measurements, and will therefore enhance the productive time available for NRS measurements.**

- **Below are statements of support from sector 3 frequent users for an online Raman system:**
  - **Thomas Duffy** (Princeton U) - We have a strong need for an on-line pressure measurement system for our on-going project to study the behavior of iron in the perovskite and post-perovskite phases of (Mg,Fe)GeO3. Germanates are well known to be effective analogs for silicates and our work provides important insights into the behavior of Fe in these materials at high pressures. Direct pressure measurement capability by Raman spectroscopy (ruby or diamond edge) would greatly facilitate our experiments by allowing for quick and accurate measurements of pressure, thereby optimizing the use of scarce beamtime.
  - **Jung-Fu Lin** (UT-Austin) - I support the request for an online Raman/ruby system at Sector 3. The new system will allow in situ pressure determination using ruby fluorescence and Raman. The Raman spectra can also be used to identify phases. This is a key system for high-pressure research.
  - **Suki Dorfman** (Michigan State U.) - Online Raman would be a great addition to the sector 3 facility. In-situ pressure measurement is important for maximizing efficiency at beamtime, but the current ruby system is not applicable to the pressure conditions of my group's research on mineralogy of Earth's lower mantle. Furthermore, online pressure measurement with diamond Raman is helpful for our measurements of nuclear resonance in materials that may react with ruby.
  - **Jackie Li** (U. of Michigan) - As a user of the Mössbauer and NRIXS facilities at Sector 3 of the Advanced Photon Source, Argonne National Laboratory, I support the initiative to acquire an online Raman system to enable efficient in situ measurements of pressures above 30 GPa in experiments using diamond anvil cells.
  - **Yang Ding** (HPSTAR) - The online Raman system is a key facility to high-pressure capable beamline, especially the beamline like sector 3, which has a high demanding of using Raman system by high pressure users with many experiments usually going above 1 Mbar.
  - **Anat Shahar** (CIW)- It would be extremely beneficial to have an online Raman system at Sector 3. It would save time and be much more efficient if we could make in situ pressure measurements instead of having to offline or borrow instrumentation from other beam lines. Having an online system would mean that we would not need to remove our samples from the system and be able to monitor the pressure throughout, as opposed to only the beginning and the end.

Several years ago we instituted the use of an on-line ruby system for pressure measurements, and the use of membrane cells. The intent was to be able to measure pressure and increase pressure without the time-consuming task of removing the diamond cell from the beamline for pressure measurement and for changing pressure, which also involves removing the APD detectors in the case of NRIXS experiments. Reinstalling a DAC on the beamline requires lengthy realignment of the sample as well as the APD detectors which also wastes precious beamtime. Installing an on-line Raman system is the logical next step in increasing the efficient utilization of available beamtime for NRS measurements.

The total budget breakdown for an online Raman system is as follows:

The estimated cost of a complete system is approximately $75 K. Quotes for the detector and spectrometer are attached.

Detector (PIX-100BR) with LF software from Princeton Instruments: $35, 725

Spectrometer and 3 ruled gratings. Princeton Instruments: SP-2560-19-NS-NP-MS-NS-PS-PN-UJ-G1). $20,570

Laser: Possible vendors are Gem, Ventus, Torus, SpectraPhysics. ~$15,000

Optics: Objectives, filters, fiber optics, stage, ~$4,000

We are asking COMPRES to provide $56,295 for the detector and spectrometer. APS will contribute the ~$19,000 needed to complete the system and install it online.

Budget Request for June 2018 – May 2019

We request partial support (3.5 months) for a scientist (Dr. Wenli Bi) to work at Sectors 3, 30 and the offline Mössbauer lab. Dr. Bi will be: 1) developing new high pressure capabilities at Sector 3 and 30 beamlines and Mössbauer lab; 2) working with the COMPRES community in developing competitive proposals for beam time; 3) assisting COMPRES users of Sector 3 and 30 during their beam time; 4) performing on-line analysis of their results (which is often formidable for even experienced users); 5) education and outreach through organizing workshops; 6) assisting users of the conventional Mössbauer spectrometer; 7) writing instruction manuals for the complex software used in analyzing results; 8) maintaining the offline Raman system. Dr. Bi will be a primary contact for the COMPRES community with Sectors 3, 30 and the offline CMS lab.

	2017-2018 Current FY	**2018-2019 New Request**
Salary, Wenli Bi (3.5 months)	46,007	23,200
Fringe Ben.	20,452	8,830
Sal.+Fringe	66,459	32,029
Travel, Domestic	3,950	4,000
Travel, Foreign	2,000	
Equipment*		56,295*
Mat. & Supp.	1,265	1,559
Conf. Regist.	900	900
Tot. Dir. Cost	74,574	94,783
MTDC (w/o equipment)	74,574	38,488
IDC	19,389	10,007
Total	93,963	104,790

* Equipment funds will be retained at COMPRES Central and will not be part of the subaward to Univ. of Illinois.

Requested funds for subaward to U Illinois: $48,495
Requested Equipment Funds Retained by COMPRES: $56,295
APS-Argonne Matching funds: $46,122 (4 months + benefits + IDC)

Budget Justification and Notes

Personnel: For FY 2018-2019 we request only 3.5 months of an annual salary of $79,541 for the Research Spectroscopist, Dr. Wenli Bi, instead of 8 months as would be usual. Under a no-cost extension for COMPRES-III, we have been using unexpended funds accumulated during the 2012-2017 COMPRES-III period to pay some of Dr. Bi's current-year salary. This has left unexpended funds in the current FY 2017-2018 account, which we plan to offset by a lower salary request for FY 2018-2019. The APS-ANL match toward Dr. Bi's 2018-2019 salary will not be affected and remains at 4 months of Dr. Bi's 12-month salary, benefits and indirect costs. The off-campus indirect cost rate of 26% applies.
Fringe Benefits are calculated at the rate of 38.06%.

Domestic Travel: The PI requests funds to make 3 trips of 2 days each ($750 @ $250 per trip) each year to Argonne to confer with staff and manage the project.

Funds are requested for the Research Spectroscopist and the PI to attend the COMPRES Annual Meeting ($750/person/year).
Travel funds of $1750 are requested for the Wenli Bi to attend one domestic scientific conference (e.g., Fall AGU), and/or COMPRES workshops.

Materials & Supplies: We request $1559 for miscellaneous materials and supplies for experiments by synchrotron and off-line Mossbauer users and the Research Spectroscopist, including reagents, rhenium and other metal foils for gaskets, diamond anvil seats, ^{57}Fe metal to make samples for Mossbauer experiments, custom sample holders, etc. Dr. Wenli Bi is encouraged as a COMPRES research scientist to pursue experiments for her career development, and is expected to do experiments leading to the development of new techniques for high pressure at her beamline.

Other - Conference Registration Fees: We request $200/year to cover the costs of the registration fee for the PI for the COMPRES Annual Meeting. In addition, $700/year is requested to cover the costs of registering for conferences for the Spectroscopist at APS Sector 3 (e.g., Fall AGU or other similar conference, and the COMPRES Annual Meeting).

Appendices

I. List of publications

1. Finkelstein, G. J., Jackson, J. M., Sturhahn W., Zhang, D., Alp, E. E., Toellner, T. S., Single-crystal equations of state of magnesiowüstite at high pressures, *Am. Mineral.* **102**, 1709, 2017.
2. Solomatova, N. V., Jackson J. M., Sturhahn, W., Rossman, G. R., and Roskosz, M., The electronic environment of ferrous iron in rhyolitic and basaltic glasses at high pressure, *Journal of Geophysical Research: Solid Earth*, in press., 2017.
3. Liu, J., Dauphas, N., Roskosz, M., Hu, M. Y., Yang, H., Bi, W., Zhao, J., Alp, E. E., Hu, J. Y., Lin, J-F., Iron isotopic fractionation between silicate mantle and metallic core at high pressure. *Nat. Commun.* **8**, 14377, 2017.
4. Wicks, J. K., Jackson, J. M., Sturhahn, W., Zhang, D., Sound velocity and density of magnesiowüstites: Implications for ultralow-velocity zone topography, *Geophys. Res. Lett.* **44** (5), 2148, 2017.
5. Solomatova., N. V., Iron-bearing Oxides, Silicate Glasses and Carbonates at Lower Mantle Pressures, **Ph.D.-Thesis**, California Institute of Technology, 2017.
6. Shim, S.-H., Grocholski, B., Ye, Y., Alp, E. E., Xu, S., Morgan, D., Meng Y., and Prakapenka, V. B., Stability of ferrous-iron bridgmanite under reducing midmantle conditions, *PNAS*, **114**, 6468, 2017.
7. Dauphas, N., John, S. G., Rouxel, O., Iron Isotope Systematics. *Review in Mineralogy & Geochemistry*, **82**, 415-510, 2017.

8. Thompson, E. C., Davis, A. H., Bi, W., Alp, E. E., Zhang, D., Greenberg, E., Prakapenka, V. B., and Campbell, A. J., High-pressure geophysical properties of fcc phase FeH_x, **submitted.**
9. Yang, H., Lin, J-F., Hu, M. Y., Bi, W., Zhao, J., Alp, E. E., Roskosz, M., Dauphas, N., Iron isotopic heterogeneity in Earth's lower mantle, **submitted.**
10. Klein, R. A., Walsh, J. P. S., Clarke, S. M., Bi, W., Alp, E. E., Jacobsen, S. D., Freedman, D. E., An S=1/2 Kagomé lattice via pressure-induced spin-crossover in jarosite, **submitted.**
11. Liu, J., Dorfman, S., Zhu, F., Li, J., Wang, Y., Zhang, D., Xiao, Y., Bi, W., Alp, E. E., Toward mapping redox states of iron in the lower mantle, **submitted.**
12. Girard, J., Karato, S., Alp, E. E., Bi, W., Limited depth range of a metallic-Fe-bearing layer and its implication for melting in the lower mantle, **submitted.**
13. Zhao, J. Y., Bi, W., Sinogeikin, S., Hu, M., Alp, E. E., Lin, J. F., Jin, C. Q., A compact membrane-driven diamond anvil cell and cryostat system for nuclear resonant scattering at high pressure and low temperature, **submitted.**
14. Chen, B., Lai, X., Li, J., Liu, J., Zhao, J., Bi, W., Alp, E. E., Hu, M. Y., Xiao, Y., Experimental contraints on the sound velocities of cementite Fe_3C to core pressures, **submitted.**

II. List of proposals granted beamtime and usage

Please find the attached excel file titled "3ID_Users_Statistics_2017".

III. The beamtime schedule for the last year's cycles

Please refer to the attached excel file titled "3ID_beam_schedule_2017".

IV. List of users in the Mössbauer lab from geosciences and high pressure physics Community.

PI	Institution	PI	Institution
Thomas Duffy	Princeton Univ.	Andrew Conelius	UNLV
Anat Shahar	CIW	James Walsh	Northwestern U.
Dave Mao	HPSTAR	Ercan Alp	Argonne National Lab
Shun-ichiro Karato	Yale Univ.	Dam Shim	Arizona State University
Jennifer Jackson	Caltech	Kanani Lee	Yale Univ.
Susannah Dorfman	Michigan State Univ.	Wendy Mao	Stanford U.
Jung-Fu Lin	Univ. of Texas at Austin		

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Customer Details:

Contact Name	Ercan Alp
Account Name	Argonne National Laboratory
Bill To	Advanced Photon Source 9700 Cass Avenue Argonne, Illinois 60439 United States
Phone	(630) 252-4775
Email	alp@anl.gov

Princeton Instruments Details:

Created Date	10/30/2017
Expiration Date	12/29/2017
Sales Inquiries	Randy Rieger rrieger@princetoninstruments.com + 1 609-587-9797

Quote Number 20263

Line Number	Product	Product Description	Price	Quantity	Total Price
1	PIX-100BR	Princeton Instruments' PIXIS:100BR Digital CCD Camera System Permanent Vacuum Guarantee • Proprietary CCD36-00 scientific grade 1, back-illuminated deep depletion • 1340 x 100 pixels, 20 x 20 μm size (26.8 mm x 2mm image area) • Thermoelectric Peltier cooled (-80°C) with forced air • Dual speed read out, 16-bit, 2 MHz and 100 kHz • USB 2.0 interface with 5 meter USB cable	$ 31,250.00	1.00	$ 31,250.00
2	LF	LightField Acquisition software with built-in Math Engine • Full acquisition support for Princeton Instruments cameras and spectrometers • Built-in math engine for real time and post-acquisition analysis • Improved user experience • Integrated LabView and Matlab support - samples provided • Supports IntelliCal - accurate and easy intensity and wavelength spectral calibration (light sources sold separately) • Free 1-yr maintenance upgrades	$ 4,475.00	1.00	$ 4,475.00
Grand Total					$ 35,725.00

All prices in USD
Sales tax and shipping charges will be added where appropriate
Payment Terms: Net 30

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Princeton Instruments Details:

Created Date	11/3/2017
Expiration Date	2/28/2018
Sales Inquiries	Randy Rieger rrieger@princetoninstruments.com + 1 609-587-9797

Quote Number 20297

Line Number	Product	Product Description	Price	Quantity	Total Price
1	HRS-300MS	SpectraPro HRS 300mm Focal Length Dual Exit Port Spectrograph and Monochromator with 90 and 180 degree optical paths Includes: • Exclusive IntelliCal wavelength/intensity calibration system • Exclusive ResXtreme™ spectral deconvolution software • Side entrance slit • Side exit slit • End array port with adapter for PI array detectors • Motorized exit port selection mirror. Accepts 68 x 68 mm (i1-xxx-xxx) or 68 x 84 mm (i2-xxx-xxx) gratings, purchased separately ResXtreme and IntelliCal require LightField software for operation, purchased separately	$ 17,495.00	1.00	$ 17,495.00
2	i1-060-750-P	Ruled Grating, 68x68mm, 600 G/mm with 750nm blaze wavelength	$ 1,050.00	1.00	$ 1,050.00
3	i1-120-500-P	Ruled Grating, 68x68mm, 1200 G/mm with 500nm blaze wavelength	$ 1,000.00	1.00	$ 1,000.00
4	i1-120-750-P	Ruled Grating, 68x68mm, 1200 G/mm with 750nm blaze wavelength	$ 1,025.00	1.00	$ 1,025.00
Grand Total					$ 20,570.00

All prices in USD
Sales tax and shipping charges will be added where appropriate
Payment Terms: Net 30