

# **COMPRES Annual Report**

Nov. 1, 2016 – Nov. 30, 2017

Wenli Bi

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I have been working at sector 3, APS since Nov., 2011, first as a joint Postdoctoral Research Associate with COMPRES (Nov. 2011- Jan. 2015), then as a Visiting Spectroscopist since Feb. 2015- May 2017, and recently as a Senior Spectroscopist since June 2017, supervised by Prof. Jay Bass (UIUC) and Dr. Ercan Alp (APS, ANL). My responsibilities include assisting users with experiments involving Nuclear Resonant and Momentum Resolved Inelastic X-ray Scattering at sector 3 and 30 (NRS and IXS), development of instrumentation and techniques for high-pressure Earth Sciences applications of IXS and Mössbauer spectroscopy, being the interface between users in the COMPRES community and the beamlines, working with members of the COMPRES community to develop competitive proposals for beamtime, and organizing workshops on NRS and IXS techniques.

This position is supported approximately 60% by COMRPES and ~40% by the APS.

## **Activities**

During the period of this report I have been working at the sector 3 and 30 beamlines as well as the offline conventional Mössbauer Lab, primarily assisting users from the COMPRES, with some involvement in projects of non-COMPRES users, carrying out experiments, characterizing samples, organizing NRS workshops and conducting my own research.

I have helped users with beamtime proposal writing, beamline experimental setup, diamond anvil cell preparation, sample loading, data collection, and online/offline data analysis. I also have helped users at sector 3 to install CONUSS and PHOENIX software on their computers and trained new users on how to analyze data. For experiments at the Mössbauer lab I have helped run measurements and analyzed data of various samples from users at ambient conditions as well as at high pressure or low temperature conditions.

Sector 3 has acquired and activated an offline Raman system that was previously located at GSECARS. I have been maintaining the system and trained users with system operation and data collection. The offline Raman system has been used routinely by users from Sector 3 and other beamlines. During the last year 18 research groups have used the system over 50 times for pressure measurements from either ruby fluorescence and/or diamond Raman edge, as well and taking Raman spectra from samples.

I maintain the glovebox and EDM at sector 3, help users with sample loadings from mostly sector 3 and also sector 30, and gas loading of pressure media in DAC's at GSECARS.

### **List of scientific projects I am involved with.**

The specific tasks I perform with users varies greatly, depending on the nature of the project and the experience of users. My contributions include training users in the use of diamond cells and other high pressure equipment, loading samples and inert gas pressure media, instruction on how to operate the beamline and collect data, data collection myself, analysis of results using the CONUSS and PHOENIX software packages, instruction on data analysis, measurement of pressure and collection of Raman spectra, and helping to write manuscripts. Again, the amount of direct participation, for example sample prep, data collection, analysis, and instruction on all the above, varies widely from user to user. All experiments involved a certain amount of beamline setup prior to the experiments, which cannot be done by even experienced users.

A list of projects I directly participated in are as follows:

#### **1. At sector 3, sector 30 and other beamlines**

- (1) Revisiting Birch's law: high-temperature sound velocities of  $\text{Fe}_7\text{C}_3$  and  $\text{Fe}_3\text{C}$  up to core pressures (GUP-48635, Bin Chen's group from U. Hawaii).
- (2) Valence electron states of iron in Ca-bearing bridgmanite (GUP-52033, Dan Shim's group from ASU).
- (3) Phonon density of states and sound velocities of  $\text{FeO}_2$  in the lower mantle (GUP-50312, Wendy Mao's group from Stanford U).
- (4) Sound velocities and spin state of Fe-bearing delta- $\text{AlOOH}$ : implications for the deep water cycle (GUP-51689, Jennifer Jackson's group from Caltech).
- (5) High-resolution synchrotron Mössbauer spectroscopy of iron-rich  $(\text{Mg,Fe})\text{O}$  at high-pressures (GUP-50316, Jennifer Jackson's group from Caltech).
- (6) Characterizing  $(\text{MgFe})\text{GeO}_3$  samples under pressure to determine the spin state in perovskite and post-perovskite phases (Thomas Duffy's group, contributed beamtime). Carried out experiment during 1.5 shifts of contributed beamtime and analyzed data. Visited Prof. Duffy's group at Princeton U. and worked on SMS data analysis on this system with group members, June Wicks and Sally Tracey.
- (7) Melting of solid krypton at high pressure (GUP-46595, PI: Dongzhou Zhang, U of Hawaii).
- (8) The effect of pressure on the iron isotope fractionation of silicate glass (GUP-54964), Anat Shahar from CIW).
- (9) Studies of bcc-hcp transition via synchrotron Mössbauer spectroscopy at high

- pressure and low temperature (collaboration with Jennifer Jackson, Caltech).
- (10) Electronic properties of hydrogen compounds of iron at very high pressures (GUP-53609, Viktor Struzhkin, CIW).
  - (11) Spin and valence state of  $\text{FeO}_2$  and  $\text{FeO}_2\text{H}_x$  (collaboration with Wendy Mao's group, Stanford U).
  - (12) Studies of pressure-induced spin crossover in jarosite (collaboration with D. Freedman and S. Jacobsen from Northwestern U.).
  - (13) Exploration of lanthanoid induced magnetic anisotropy in  $\text{Fe}_2\text{Dy}_2$  single molecular magnets (GUP-48137, Volker Schunemann, University of Kaiserslautern).
  - (14) Resonant X-ray scattering reveals possible disappearance of magnetic order under hydrostatic pressure in the Kitaev candidate  $\gamma\text{-Li}_2\text{IrO}_3$  (PI: Nicholas Breznay, UC Berkeley).
  - (15) Magnetic order under high pressure in the Fe-based superconductor  $\text{FeSe}$  (GUP- 48670, PI: Karunakar Kothapalli, Ames Lab).
  - (16) Studies of phonon anomaly under pressure using NRIXS at sector 30 (collaboration with Olivier Delaire from Duke U.).
  - (17) Studies of magnetism in Dy by synchrotron Mössbauer spectroscopy at extreme Pressures (self-PI).
  - (18) Studies of pressure-induced phonon anomaly in As single crystal (Raphael Hermann, ORNL).
  - (19) Pressure-induced electronic transitions of  $\text{GaTa}_4\text{Se}_8$  using RIXS at sector 27 (Jungho Kim).
  - (20) Studies of pressure-induced structural transitions of  $\text{CuMnSb}$  (collaboration with Prof. James Schilling's group at WashU).
  - (21) Analyzed high pressure SMS data from Afu Lin's group on bridgmanite and ferropericlase.
  - (22) Mentored Dr. Philipp Materne, a new postdoc at sector 3. Provided instruction on high pressure and low temperature techniques and experimental setup, and analysis of SMS data in CONUSS. Collaborated with Dr. Materne on a project on the magnetism in iron-pnictides at high pressure and low temperature conditions (GUP-53291).
  - (23) Inelastic X-ray scattering of  $\text{CaLa}_{11}\text{2}$ : evolution of phonon modes at pressure-induced Bi-critical point (GUP-52291, S. Haravifard's group from Duke U.).
  - (24) Mapping of iron speciation and phase transformations in Ultrahard Chiton Teeth using cryogenic Mössbauer microscopy (GUP-55167, PI: Linus Stegbauer, Northwestern U).

2. In the offline Mössbauer lab, we have two independently running Mössbauer spectrometers. Recently a modern Mössbauer spectrometer has been acquired and commissioned (LDRD funds from the APS, ANL). I have taken measurements on samples to support beamline projects, as well as independent experiments requested

by researchers across the country. During last year 35 samples from 13 COMPRES user groups have been characterized in the lab.

### **Beamtime Proposals I have involved in writing:**

- (1) Synchrotron Mössbauer Spectroscopy (SMS) Studies of the Magnetic State in Dy under Extreme Pressure (GUP-40395, self-PI)
- (2) Nuclear resonant scattering study of spin state transitions of Fe in (Mg,Fe)GeO<sub>3</sub> post-perovskite (GUP-53956, PI: June Wicks, Princeton U)
- (3) Nanoscopic coexistence of magnetism and superconductivity in BaFe<sub>2</sub>As<sub>2</sub> as a function of pressure and temperature (GUP-53291, PI: Philipp Materne, APS)
- (4) High pressure studies of magnetism and valence in novel EuAFe<sub>4</sub>As<sub>4</sub> (A=Cs, Rb) superconductors using synchrotron Mossbauer spectroscopy (GUP-53839, self-PI).
- (5) Electronic phase diagram of LaFeAsO under pressure - coexistence of magnetism and superconductivity or phase separation? (GUP-57027, PI: Philipp Materne, APS)
- (6) Determination of relaxation times of Dysprosium containing single molecular magnets via <sup>161</sup>Dy NFS (GUP-55230, PI: Volker Schunemann, University of Kaiserslautern).
- (7) <sup>151</sup>Eu synchrotron Mossbauer spectroscopy study of the temperature and pressure dependence of intermediate valence and magnetic ordering in Ca<sub>1-x</sub>Eu<sub>x</sub>Co<sub>2</sub>As<sub>2</sub> (GUP-54626, Michael Shatruk, Florida State U.).
- (8) Sn-partial phonon DOS across pressure-induced phase transition in anharmonic SnSe (GUP-52093, PI: Olivier Delaire, Duke U.)

### **Education, Outreach, Workshop and Community service:**

- (1) Organized and chaired the NRS Workshop 2017: CONUSS and Synchrotron Mössbauer Data Analysis, Nov. 16-19, 2017 at the Advanced Photon Source, Argonne National Laboratory. 28 people from 4 countries and 16 institutions participated the workshop.
- (2) Convened and chaired the focused session (FS10) titled “Nuclear Resonant and Inelastic X-ray Scattering at High Pressures” at the 26<sup>th</sup> AIRAPT meeting in Beijing, August 19-23, 2017.
- (3) Actively participated in the COMPRES 2017 annual meeting.
- (4) Carried out mail-in service to 13 COMPRES user groups of the Mössbauer lab.
- (5) Involved with beamtime proposal writing, diamond anvil cell preparation, sample loading, beamline experimental setup, data collection, data analysis and interpretation, and manuscript writing for users. Year around training of users for beamline experiment operation and data analysis.

### **Invited and Contributed presentations**

*Magnetism of Eu and Dy under extreme pressures*, lecture talk at the NRS Workshop 2017: CONUSS and Synchrotron Mössbauer Data Analysis, APS, ANL, Nov. 2017.

*Studies of pressure-induced magnetic and valence transitions in  $\text{EuFe}_2\text{As}_2$  via synchrotron Mössbauer spectroscopy (SMS)*, invited talk at AIRAPT meeting, Beijing, August 2017.

*Pressure-induced transitions using  $^{57}\text{Fe}$ ,  $^{119}\text{Sn}$ ,  $^{151}\text{Eu}$  and  $^{161}\text{Dy}$  Nuclear Resonant Scattering*, poster presentation at the COMPRES 2017 annual meeting, New Mexico, July 2017.

*Report on nuclear resonant scattering workshop in 2016*, talk at the COMRPES 2017 annual meeting, New Mexico, July 2017.

*Studies of Magnetism under Pressure via Synchrotron Mössbauer Spectroscopy in  $^{57}\text{Fe}$ ,  $^{151}\text{Eu}$  and  $^{161}\text{Dy}$* , talk at Prof. Thomas Duffy's group meeting, Princeton U., New Jersey, May 2017.

*Studies of Phase Transitions in  $\text{EuFe}_2\text{As}_2$  by  $^{57}\text{Fe}$  and  $^{151}\text{Eu}$  Nuclear Resonant Scattering under hydrostatic pressures*, poster presentation and poster slam talk at APS/CNM Users Meeting, May 2017.

*Studies of magnetism in dysprosium under extreme pressures*, contributed talk at the American Physical Society March Meeting, New Orleans, Louisiana, March 2017.

## List of recent publications

Song, J., **Bi, W.**, Haskel, D. & Schilling, J. S., Evidence for strong enhancement of the magnetic ordering temperature of trivalent Nd metal under extreme pressure, (2017). *Phys. Rev. B* **95**, 205138.

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, (2017). *Nat. Commun.* **8**, 14377.

Breznay, N.P., Ruiz, A., Frano, A., **Bi, W.**, Birgeneau, R.J. , Haskel, D. , and Analytis, J.G., *Phys. Rev. B* **96**, 20402 (2017).

Choi, Y. Jiang, X., **Bi, W.**, Lapa, P., Chouhan, R. K., Padudyal, D., Varga, T., Popov., D., Cui, J., Haskel, D., and Jiang, J. S., Element-revolved magnetism across the temperature- and pressure-induced spin reorientation in MnBi, *Phys. Rev. B* **94**, 184433, 2016.

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, **accepted**, *Rev. Sci. Instrum.*

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<sub>x</sub>, **submitted**.

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**.

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**.

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**.

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**.

Chen, B., Lai, X., Li, J., Liu, J., Zhao, J., **Bi, W.**, Alp, E. E., Hu, M. Y., Xiao, Y., Experimental constraints on the sound velocities of cementite Fe<sub>3</sub>C to core pressures, **submitted**.

Materne, P., **Bi, W.**, Goltz, T., Spehling, J., Alp, E. E., Zhao, J., Hu, M. Y., Zhang, D., Jesche, A., Geibel, C., Kappenberger, R., Wurmehl, S., Büchner, B., and Klauss, H-H., Equivalence of hydrostatic and chemical pressure: suppression of the magnetic order in CeFeAsO, **submitted**.

Fei, Y., Iqbal, M., Kong, S., Xue, Z., Mcfadden, C., Guillet, J., Doerr, L., Alp, E., **Bi, W.**, Lu, Y., Ranganath, P., Javier, K., Ahmadian, M., Ellison, C., Johnston, K., Aqueous Superparamagnetic Magnetite Dispersions with Ultra-High Initial Magnetic Susceptibilities, **submitted**.