**Annual Report of Year 2014-2015**

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), and recently as a Visiting Spectroscopist from Feb. 2015, supervised by Dr. Prof. Jay Bass (UIUC) and Dr. Ercan Alp (ANL). My responsibilities include assisting users with experiments involving Nuclear Resonant and Momentum Resolved Inelastic X-ray Scattering at sector 3 (NRS and IXS), the development of instrumentation and techniques for high-pressure Earth Sciences applications of IXS and Mössbauer spectroscopy, being the interface between potential 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 70% supported by COMRPES and 30% supported by the APS. I have been spending about 70% of my time on COMPRES-related activities and 30% on other research activities.

**Activities**

From October 2014, I have been working at the sector 3 beamlines and at the conventional Mössbauer spectroscopy Lab affiliated to the beamline, assisting users from both COMPRES and non-COMPRES community carrying out experiments, characterizing samples, 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 analysis. I also have helped users at sector 3 to install CONUSS and PHOENIX softwares on their computers and trained new users on how to analyze data. For experiments at conventional Mössbauer lab I have helped run experiments and analyzed data of various samples from users at ambient conditions as well as at high pressure or low temperature conditions.

I have been leading the development of the new capability of high pressure and low temperature (HPLT) nuclear resonant inelastic x-ray scattering (NRIXS) at sector 3. We have developed a miniature panoramic DAC, a cryostat with liquid helium flow further down to the DAC holder, and an efficient gas-membrane driven system to tune pressure in-situ. I also designed a gas loading adapter for this mini-panoramic DAC to be able to load gas pressure medium at GSECARS. The new setup has been integrated, commissioned and used by three research groups.

I also helped Dr. Jiyong Zhao to maintain the glovebox at sector 3. I helped users with sample loadings inside the glovebox and gas loading at GSECARS in panoramic DACs.

**Here is a list of scientific projects I have involved:**

1. At sector 3 and other beamlines

1. Development of High-Pressure and Low-Temperature Nuclear Resonant Scattering Capability for Geoscience and Condensed Matter Physics Applications at 3-ID (PUP-38969). Drafted the PUP. We were allocated 6 days of beamtime in each cycle for two years of period. Lead the project and commissioned the recently developed setup for high pressure and low temperature NRIXS experiments.
2. Exploitation of the hybrid-filling mode of the APS to map out the pressure-temperature phase diagram of iron metal (GUP-45174, self-PI). This project is aimed to explore the APS’s hybrid filling mode for applications in mineral physics. The extraordinary long time window of 1 micro-second following a super-bunch of 16 mA (corresponding to 10 decay half-life for 57Fe nucleus) in the hybrid mode allows more accurate determination of valence, spin, magnetic state, and phase mixtures. We apply this feature to map the temperature dependence of the alfa (bcc)-to-epsilone (hcp) phase transition under pressure .
3. Pressure-induced spin and magnetic transitions in Fe3S4. Collaboration with John Tse. Prepared DAC, loaded sample, set up beamline, took beamtime, collected and analyzed data.
4. Mössbauer microscopic investigation of disproportionation of ferrous ions into metallic iron and ferric ions under pressure (GUP-43134). Collaboration with Prof. Shun-ichiro Karato’s group. Helped with beamline setup. Took beamtime, collected and evaluated data.
5. The high pressure phonon density of states of fcc iron hydride (GUP-40453). Collaboration with Prof. Andrew Campbell’s group from U. of Chicago. Helped with DAC preparation. Focused x-ray beam, took beamtime, collected and evaluated data.
6. Effect of temperature on the sound velocities of Fe7C3 at pressures up to 150 GPa (GUP-39186, PI: B. Chen). Helped with laser heating optics alignment, x-ray monochromation and focusing, and data reduction. Trained the students how to analyzed data.
7. Studies of isotope fractionation in basalts under pressure GUP-40663 (Jung-fu Lin’s group). Helped with beamline setup, x-ray focusing.
8. High Pressure Studies of Spin, Magnetism, Valence, and Lattice dynamics in EuFe2As2 using Nuclear Resonant Scattering (GUP-32955, self-PI). In the course of this project, we also commission and test the newly developed cryostat and membrane-driven DAC system. A manuscript is in preparation.
9. Studies of magnetism and valence in Dy metal by X-ray near edge absorption spectroscopy, x-ray magnetic circular dichroism and synchrotron Mössbauer spectroscopy at Extreme Pressures (GUP-40395, GUP-43631, self-PI). Recently XANES studies at L3 edge in Dy were carried up successfully up to 114 GPa and found anomalous changes of electronic structure across structural phase transitions. Magnetism of Dy was also studied up to 104 GPa using synchrotron Mössbauer spectroscopy. A manuscript is in preparation.
10. Phonon density of states of ferropnictide superconductors at high pressures and low temperatures by nuclear resonant inelastic X-ray scattering (GUP-37536, Jung-Fu Lin's group). X-ray monochromation, beam focusing, DAC preparation, sample and Ne gas pressure medium loading, cryostat setup. Took beamtime, collected and analyzed data. Trained new students.
11. The role of phonon softening in the pressure-induced amorphization process of the tin-halide molecular crystals SnI4 and SnBr4 (PI: Michael Hu, APS). Prepared DAC, loaded sample, took beamtime and evaluated data.
12. Temperature and redox effects on the iron phonon density of states of silicate glasses and olivine (GUP-42947, PI: N. Dauphas, U of Chicago). Helped with x-ray monochromation and focusing.
13. Characterizing MgFeSiO3 samples under pressure to determine the spin state in perovskite and post-perovskite phases (Thomas Duffy’s group). Took beamtime, collected and analyzed data.
14. Phonon softening in SnSe under pressure (PI: Olivier Delaire, Oak Ridge National Lab). Prepared DAC, loaded samples and applied pressures during HERIXS beamtime at sector 30.
15. Studies of spin reorientation and structural phase transitions in MnBi (collaboration with Jun Cui from Pacific Northwest National Lab and Jidong Jiang from ANL). Prepared DAC and loaded samples. Took beamtime and helped with data analysis.
16. Pressure induced magnetic transitions in EuTiO3 (PI: Philip Ryan, APS). Prepared DAC and loaded sample.

2. At Mössbauer lab, there are two independently running Mӧssbauer spectrometers. I run samples to support beamline projects, as well as independent experiments as requested by researchers around the country.

(1) Studies of spin transitions in GeMgGeO3 under pressure. Samples from

Thomas Duffy, Princeton University.

(2) Spin and magnetic transition in greigite (Fe3S4) under pressure, collaboration with John Tse, [University of Saskatchewan](https://www.usask.ca/)

(3) Characterization of Li-ion battery materials from Chemical Science and Engineering, ANL. Collected and analyzed data.

(4) Characterization of FeSiO3 single crystals from Przemyslaw Dera, U. of Hawaii

1. Bridgmanite and (Mg, Fe)O from Shun-ichiro Karato, Yale University.
2. GdFe0.6Si2 from Daniel https://mail.anl.gov/owa/14.3.248.2/themes/resources/clear1x1.gifhttps://mail.anl.gov/owa/14.3.248.2/themes/resources/clear1x1.gifFredrickson, U. Wisconsin Madison.

(7) iron oxide nanoparticle from  Christopher J Ellison, U. Texas at Austin

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

1. Spin state of transitions of Fe in (Mg,Fe)GeO3 using synchrotron Mössbauer spectroscopy (GUP-46440)
2. The high pressure phonon density of states of fcc iron hydride (GUP-40453)
3. Mössbauer microscopic investigation of disproportionation of ferrous ions into metallic iron and ferric ions under pressure (GUP-43134)
4. Exploitation of the hybrid filling mode of the APS to map out the pressure-temperature phase diagram of iron metal (GUP-45174, self-PI)
5. Development of High-Pressure and Low-Temperature Nuclear Resonant Scattering Capability for Geoscience and Condensed Matter Physics Applications at 3-ID (PUP-38969)
6. Synchrotron Mössbauer Spectroscopy (SMS) Studies of the Magnetic State in Dy under Extreme Pressure (GUP-40395, self-PI)
7. Studies of Magnetism in Dy under Extreme Pressure by X-ray Magnetic Circular Dichroism (XMCD) (GUP-43631)
8. Investigation of Two-Dimensional Spin Liquid at Magnetic Insulator / Magnetic Topological Insulator Interface Using Synchrotron Mössbauer Spectroscopy (GUP-44482)
9. Lattice dynamice study of pressure-induced amorphization in the tin-halide molecular crystals (GUP-40200)
10. Single Crystal X-ray Diffraction in EuFe2As2 under High Pressure (GUP-40428, self-PI)

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

1. Organized and chaired the Nuclear Resonant Scattering Workshop on Nov. 7-9, 2014 at the APS, ANL. The workshop was organized as part of the OMPRES Education, Outreach and Infrastructure Development Programs to promote the application of the state-of-art Nuclear Resonant Scattering techniques for characterizing the properties of materials under the high P-T conditions of planetary interiors. Introductory lectures were offered by invited speakers and sector 3 beamline staff. Softwares CONUSS, PHOENIX and SciPhon for data evaluation were introduced and hands-on trainings were offered in the use of softwares. New features of the softwares and recent experiment capability developments were covered. The workshop attracted 41 participants from 16 institutions and 4 countries with various backgrounds of geosciences, physics, chemistry, material science and biophysics.
2. Helped users to install CONUSS and PHOENIX software packages on their computers; trained new users for data analysis
3. Lead the instrument development project titled “High pressure Low temperature Nuclear Resonant Scattering for Studying Properties of Planetary Materials in Extreme Environments”. The project is supported by the Infrastructure Development programs of COMPRES.
4. Lead the PUP-38969 titled Development of High-Pressure and Low-Temperature Nuclear Resonant Scattering Capability for Geoscience and Condensed Matter Physics Applications at 3-ID.

**Invited and Contributed talks**

*Development of Nuclear Resonant Inelastic X-ray Scattering at High Pressure and   
Low Temperature*. Invited talk at International Workshop on Nuclear Resonance Scattering of Synchrotron Radiation, Sep. 21, 2015, DESY, Germany

*Magnetism of Europium under Extreme Pressures*, contributed talk, ICAME meeting, Sep. 17, 2015, Hamburg, Germany.

*Nuclear Resonant Scattering at High Pressure and Low Temperature.* Invited talk at APS High Pressure Special Interest Group Meeting, August 6, 2015, APS.

*New Design of Cryostat and Membrane-driven Diamond Anvil Cell for Nuclear Resonant Inelastic X-ray Scattering (NRIXS) at High Pressure and Low Temperature.* Contributed talkat COMPRES annual meeting, July 7, 2015, Colorado Springs, Co.

*Unusual Magnetism of Europium under Extreme Pressures.* Invited talk at 8th North American Mössbauer symposium, Jan. 9, 2015, Boston, MA.

*Nuclear Resonant Scattering Under High Pressure.* Invited talk at Lawrence University, WI., May 22, 2014.

**List of recent publications**

**W. Bi**, J. Zhao, J. Lin, Q. Jia, M.Y. Hu, C. Jin, R. Ferry, W. Yang, V. Struzhkin, and E.E. Alp, *Nuclear Resonant Inelastic X-ray Scattering at High Pressure and Low Temperature,* J. Synchrotron Radiat. **22**, 760 (2015).

Mathieu Roskosz, Corliss K.I. Sio, Nicolas Dauphas, **Wenli Bi,** François L.H. Tissot, Michael Y. Hu, Jiyong Zhao, Esen E. Alp, *Spinel–olivine–pyroxene equilibrium iron isotopic fractionation and applications to natural peridotites*, Geochim. Cosmochim. Acta **169**, 184-199 (2015).

S.R. Bruno, C.K. Blakely, J.B. Clapham, J.D. Davis, W. Bi, E.E. Alp, and V. V. Poltavets, J. *Synthesis and electrochemical properties of novel LiFeTiO4 and Li2FeTiO4 polymorphs with the CaFe2O4-type structures,* Power Sources **273**, 396 (2015).

H. Liu, J.S. Tse, M.Y. Hu, **W. Bi**, J. Zhao, E.E. Alp, M. Pasternak, R.D. Taylor, and J.C. Lashley, *Mechanisms for pressure-induced crystal-crystal transition, amorphization, and devitrification of SnI4,* J. Chem. Phys. **143**, 164508 (2015).

**W. Bi,** J. Lim, G. Fabbris, J. Zhao, D. Haskel, E. E. Alp, and J. S. Schilling, *Possible quantum criticality in compressed elemental europium,* submitted for publication

Jennifer Girard, Shun-ichiro Karato, Esen E. Alpand **Wenli Bi**, *Limited depth range of a metallic-Fe-bearing layer and its implication for melting in the lower mantle,* submitted for publication.

Jamie Y. C. Chen, Lianna Dang, Hanfeng Liang, **Wenli Bi**, James B. Gerken, Song Jin, E. Ercan Alp, Shannon S. Stahl, *Operando Analysis of NiFe- and Fe-Oxide Electrocatalysts for Water Oxidation: Detection of Fe+4 by Mössbauer Spectroscopy,* submitted for publication

Chunyu Li, Zhenhai Yu, Michael Y. Hu, Jiyong Zhao, **Wenli Bi**, Jinggeng Zhao, Wei Wu, Jianlin Luo, Nanlin Wang, Hao Yan, Haozhe Liu & Esen E. Alp, *In situ high-pressure synchrotron* *Mössbauer and X-ray diffraction studies:* *exploring the structure-related* *valence fluctuating in EuNi2P2*, submitted for publication.

Xiujuan Jiang, N. A. Zarkevich, **Wenli Bi**, Yongseong Choi, Pavel Lapa, Dmitry Popov, Daniel Haskel, D. D. Johnson, Jun Cui, and J. S. Jiang, *Pressure-Induced Structural Transition in MnBi Powders,* submitted for publication

Bin Chen, Zeyu Li, Dongzhou Zhang, Jiachao Liu, Michael Y. Hu, Jiyong Zhao, **Wenli Bi**, E. Ercan Alp, Yuming Xiao, Paul Chow, and Jie Li, *Hidden carbon in Earth’s inner core revealed by shear softening in dense Fe7C3*, PNAS (2014)

S. R. Bruno, C. K. Blakely, J. B. Clapham, J. D. Davis, **W. Bi**, E. E. Alp, and V. Poltavets, *Synthesis and Electrochemical Properties of Novel LiFeTiO4 and Li2FeTiO4 Polymorphs with the CaFe2O4-type Structures,* Journal of Power Sources, 273, 296 (2014)

A. B. Hadler, V. J. Yannello, **W. Bi,** E. E. Alp and D. C. Fredrickson, *Gd13Fe10C13 and its Oxycarbide: Tracing Multiple Bonding and Oxygen Substitution in Carbometalates to the 18 electron rule,* JACS, 136, 12073 (2014)

M. K. Karunananda, E. E. Alp, **W. Bi**, S. Chattoadhyay, T. Shibata, and N. P. Mankad, *Experimental Determination of Metal-Metal Redox Cooperativity and Electronic Structure in Catalytically Active Cu-Fe and Zn-Fe Heterobimetallic Complexes*, Dalton Transactions, 43, 13661 (2014)

Jin Liu, Jung-Fu Lin, Ahmet Alatas, and **Wenli Bi,** *Sound Velocities of bcc-Fe and Fe0.85Si0.15 Alloy at High Pressure and Temperature,* Earth and Planetary Science Letters, 233, 24 (2014)

C. K. Blakely, J. D. Davis, S. R. Bruno, M. Zhu, X. Ke, **W. Bi**, E. E. Alp, and V. V. Poltavets, *Multistep Synthesis of the SrFeO2F Perovskite Oxyfluoride via the SrFeO2 Infinite-layer Intermediate,* Journal of Fluorine Chemistry, 159, 814 (2014)