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

2015 COMPRES Annual Report

November 2014 – October 2015

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**Overview**

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 beamlines, as well as supporting COMPRES users through 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 (momentum resolved IXS from acoustic phonons).

The special capabilities involve

1. Micro-focused beam with 10 micron beamsize at 3-ID-B and 17 micrometer beamsize at 3-ID-C,
2. 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,
3. On-line Ruby pressure calibration system in 3-ID-B,
4. Panoramic membrane DAC’s,
5. Combined inelastic spectrometer/x-ray diffraction capability for single crystal structure and orientation determination inside the DAC,
6. Combined nuclear resonant inelastic spectrometer with Mossbauer spectroscopy and x-ray diffraction capability for atomic and electronic structure determination and equation of state,.
7. 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 off-line Mössbauer spectroscopy laboratory with two Mössbauer drives as another resource that is used frequently by COMPRES researchers. With the availability of point-sources, spectra can be obtained from high pressure samples inside a DAC. This is the only Mossbauer facility that is open for use by the COMPRES community. The COMPRES staff member, Dr. Wenli Bi, runs experiments and provides interpretation of Mossbauer experiments. This facility is commonly used as a mail-in service.

**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 important geophysical problems such as 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 such as quadruple 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. Changes in the recoil free fraction of spectra have been exploited to measure the melting points of iron and Fe alloys under pressures. In addition to high-profile publications, Jackson’s group has been involved in the installation and commissioning of a fast temperature readout system with 100 Hz readout frequency to facilitate melting studies.

**Publications:**

Dongzhou Zhang, Jennifer M. Jackson, Jiyong Zhao, Wolfgang Sturhahn, E. Ercan Alp, Thomas S. Toellner, Michael Y. Hu, ["Fast temperature spectrometer for samples under extreme conditions,"](http://dx.doi.org/10.1063/1.4905431) Rev. Sci. Instrum. **86** (1), 013105-1-013105-10 (2015). DOI: 10.1063/1.4905431

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

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[subscript 0.65]Fe[subscript 0.35])O ferropericlase to 140 GPa,"](http://www.agu.org/pubs/crossref/2012/2012JB009162.shtml) 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,"](http://dx.doi.org/10.1016/j.epsl.2012.11.048) 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,"](http://onlinelibrary.wiley.com/doi/10.1002/jgrb.50166/abstract) 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,"](http://dx.doi.org/10.1016/j.epsl.2012.10.027) 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, Jennifer M. Jackson, Bin Chen, Wolfgang Sturhahn, Jiyong Zhao, Jinyuan Yan, Razvan Caracas, ["Elasticity and lattice dynamics of enstatite at high pressure,"](http://onlinelibrary.wiley.com/doi/10.1002/jgrb.50303/abstract) J. Geophys. Res. **118** (8), 4071-4082 (2013).

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

The group from Texas-Austin is very active in using HERIX and NRS instruments 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:**

Jung-Fu Lin, Junjie Wu, Jie Zhu, Zhu Mao, Ayman H. Said, Bogdan M. Leu, Jinguang Cheng, Yoshiya Uwatoko, Changqing Jin, Jianshi Zhou, ["Abnormal Elastic and Vibrational Behaviors of Magnetite at High Pressures,"](http://www.nature.com/srep/2014/140904/srep06282/full/srep06282.html) Sci. Rep. **4**, 6282-1-6282-6 (2014).

Jin Liu, Jung-Fu Lin, Ahmet Alatas, Wenli Bi, "Sound velocities of bcc-Fe and Fe0.85Si 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,"](http://ammin.geoscienceworld.org/content/99/2-3/415.abstract?related-urls=yes&legid=gsammin;99/2-3/415) 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 pyrolitic and basaltic Mg-Pvs at high pressure in the laser-heated diamond-anvil cell.

**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, Jie Li, ["Hidden carbon in Earth's inner core revealed by shear softening in dense Fe[subscript 7]C[subscript 3],"](http://www.pnas.org/content/111/50/17755) Proc. Natl. Acad. Sci. USA **111** (50), 17755-17758 (2014).

**5) Nicolas Dauphas** (U. of Chicago)

# 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.

# Publications:

M. Blanchard, N. Dauphas, M.Y. Hu, M. Roskosz, E.E. Alp, D.C. Golden, C.K. Sio, F.L.H. Tissot, J. Zhao, L. Gao, R.V. Morris, M. Fornace, A. Floris, M. Lazzeri, E. Balan, ["Reduced partition function ratios of iron and oxygen in goethite,"](http://dx.doi.org/10.1016/j.gca.2014.12.006) Geochim. Cosmochim. Acta **151**, 19-33 (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,"](http://dx.doi.org/10.1016/j.gca.2015.07.035) Geochim. Cosmochim. Acta **169**, 184-199 (2015). DOI: 10.1016/j.gca.2015.07.035

[N. Dauphas](http://www.sciencedirect.com/science/article/pii/S0012821X14002738), [M. Roskosz](http://www.sciencedirect.com/science/article/pii/S0012821X14002738), [E.E. Alp](http://www.sciencedirect.com/science/article/pii/S0012821X14002738), [D.R. Neuville](http://www.sciencedirect.com/science/article/pii/S0012821X14002738), [M.Y. Hu](http://www.sciencedirect.com/science/article/pii/S0012821X14002738), [C.K. Sio](http://www.sciencedirect.com/science/article/pii/S0012821X14002738), [F.L.H. Tissot](http://www.sciencedirect.com/science/article/pii/S0012821X14002738), [J. Zhao](http://www.sciencedirect.com/science/article/pii/S0012821X14002738), [L. Tissandier](http://www.sciencedirect.com/science/article/pii/S0012821X14002738), [E. Médard](http://www.sciencedirect.com/science/article/pii/S0012821X14002738), [C. Cordier](http://www.sciencedirect.com/science/article/pii/S0012821X14002738),  **“Magma** [redox and structural controls on iron isotope variations in Earth's mantle and crust](http://www.sciencedirect.com/science/article/pii/S0012821X14002738)**”,** Earth and Planetary Science Letters, 398, 127–140 (2014)

# 6) Andrew Campbell (U. of Chicago)

This group focused on studying the thermodynamic and vibrational parameters of iron hydride (FeHx) under high pressure and temperature.

**7) Shun-ichiro Karato** (Yale University)

This group has been using both synchrotron and conventional Mössbauer spectroscopy to study the metallic iron concentration in bridgmanite and discovered a substantial amount of metallic iron in a narrow depth range in Earth’s lower mantle. A manuscript was submitted for publication.

**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 was recently submitted to Phys. Rev. Lett reporting her recent results of magnetism of europium under 1 Mbar pressure. Dr. Bi is also leading 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 EuFe2As2 under pressure and magnetism of dysprosium under 1 Mbar pressure. Manuscripts will be submitted soon reporting these results. Dr. Bi also organizes highly successful workshops related these efforts (see below), as well as runs the conventional Mössbauer Lab for sample characterization at ambient as well as high pressure conditions.

**Publication: authored and co-authored by Dr. Wenli Bi:**

Wenli Bi, Jiyong Zhao, Jung-Fu Lin, Quanjie Jia, Michael Y. Hu, Changqing Jin, Richard Ferry, Wenge Yang, Viktor Struzhkin, E. Ercan Alp, ["Nuclear resonant inelastic X-ray scattering at high pressure and low temperature,"](http://journals.iucr.org/s/issues/2015/03/00/hf5283/index.html) J. Synchrotron Rad. **22** (3), 760-765 (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,"](http://dx.doi.org/10.1016/j.gca.2015.07.035) Geochim. Cosmochim. Acta **169**, 184-199 (2015). DOI: 10.1016/j.gca.2015.07.035

Bin Chen, Zeyu Li, Dongzhou Zhang, Jiachao Liu, Michael Y. Hu, Jiyong Zhao, Wenli Bi, E. Ercan Alp, Yuming Xiao, Paul Chow, Jie Li, ["Hidden carbon in Earth's inner core revealed by shear softening in dense Fe[subscript 7]C[subscript 3],"](http://www.pnas.org/content/111/50/17755) Proc. Natl. Acad. Sci. USA **111** (50), 17755-17758 (2014).

Jin Liu, Jung-Fu Lin, Ahmet Alatas, Wenli Bi, ["Sound velocities of bcc-Fe and Fe[subscript 0.85]Si[subscript 0.15] alloy at high pressure and temperature,"](http://dx.doi.org/10.1016/j.pepi.2014.05.008) Phys. Earth Planet. In. **233**, 24-32 (2014).

W. Bi, J. Lim, G. Fabbris,J. Zhao, D. Haskel, E. E. Alp,M. Y. Hu, P. Chow,Y. Xiao, W. Xu,and J. S. Schilling

“Possible quantum criticality in compressed elemental europium”, submitted to Phys. Rev. Lett., Nov. 3, 2015

**Invited and Contributed talks by Dr. Wenli Bi**

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 talk at 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.

**8) Caitlin Murphy**

Dr. Murphy from Carnegie Institute of Washington focused on studies of phonon dispersion relations of single crystal SnO2 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 Visiting Spectroscopist 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.

**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: 15 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: 10

No of PhD students engaged: ~ 12

No of workshops/symposium arranged: 3

No of Grant applications supported by letters: 6

**Community/Broader Impacts**

**Nuclear Resonance Scattering Workshop**

The NRS workshop was held on Nov. 7-9, 2014 at Argonne National Laboratory. Wenli Bi chaired the workshop. The workshop is 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. In this workshop 41 participants from 16 institutions attended the workshop. The participants were given introductory lectures and hands-on training opportunity to learn data evaluation software like CONUSS and PHOENIX. SciPHON was also introduced first time to users to analyze the NRIXS data.

**Mössbauer Symposium**

We organize the only Mössbauer Spectroscopy Symposium in North America biannually. The **8th North American Mössbauer Symposium** was held at Northeastern University in Boston on January 8 and 9, 2015. and it was partially supported by COMPRES.

**High-energy Resolution Inelastic X‐ray Scattering Workshop**

This workshop was held during 2015 APS/CNM Users Meeting on May 12 at the APS. Recent HERIXS beamline instrument developments were addressed. And recent research results in diverse areas including geoscience were presented.

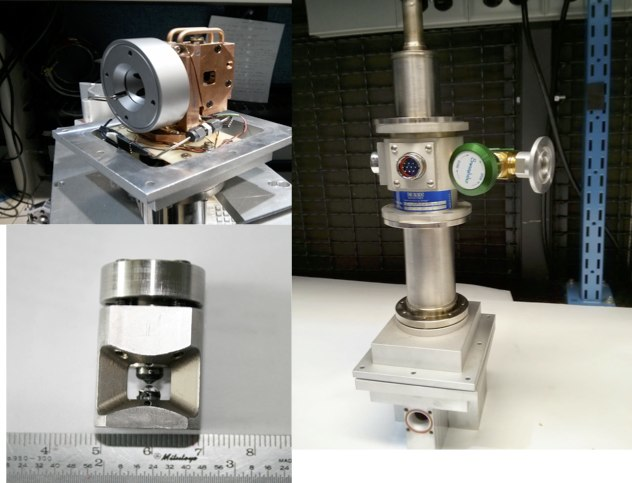
**Participation in the COMPRES Annual Meeting**

We have actively participated the 2015 COMPRES annual meeting at Colorado Springs, Co. We presented two contributed talks to present the development of high pressure and low temperature NRIXS capability, and **high pressure synchrotron 57Fe Mossbauer Spectroscopy in Hybrid Mode of the APS: Iron under pressure.**

**Development of new capabilities:**

**i) Low temperature-high pressure NRIXS**

The development of a novel capability of low temperature and high pressure NRIXS is completed. This technical development is under support of COMRPES Education, Outreach and Infrastructure Development programs. We have a PUP in progress dedicated to this development and commission. We have developed a miniature panoramic DAC, a novel design cryostat with liquid Helium flowed further down to the DAC holder to achieve temperatures on the sample, and a gas membrane-driven system to be capable of tuning the pressure in situ. The cryostat and the gas membrane-driven system have been integrated and commissioned. Lowest temperature of 9 K and highest pressure of 104 GPa was achieved with this setup. Pictures of the cryostat, the membrane system and the DAC are shown below. Three research groups have used the new system this year.



**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 and has been used by 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. Commission 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)

**Budget Request for 2015-2016**

Visiting Spectroscopist salary, baseline $69,000

Total salaries $69,000

Fringe benefits (44.77%) $30,891

Total wages and benefits $99,891

Travel $5,000

Services (conf. reg. Fees) $800

Supplies and shop services $750

Total direct cost (off campus) $106,341

Total indirect cost (off campus, 24%) $25,522

Total cost $131,863

APS cost sharing ($38,000)

Total request from COMPRES $93,963

**Budget Justification**

Budget Period: June 2016-May 2017

**Personnel**: Support is requested for Dr. Wenli Bi, Research Spectroscopist for the COMPRES IXS and Mossbauer spectroscopy Facilities project at Sector 3 of the Advanced Photon Source (APS), Argonne National Laboratory. The 2016-2017 salary for the Dr. Bi is $69,000/year. The workplace for Dr. W Bi is 100% time off campus at APS.

**Fringe Benefits** are calculated at the rate approved by the Federal Government. Fringe benefits for the proposed senior personnel and other academic employees are at the rate of 44.77%.

**Travel**: Funds are requested for the Research Spectroscopist and the two PI’s (Bass, Alp) to attend the COMPRES Annual Meeting ($750 each). The main cost is for transportation (airfare, car rental). The PI requests funds to make 4 trips of 2 days each ($1000 @ $250 per trip) to Argonne to confer with staff and manage the project. Domestic travel funds ($1750) are requested for the Wenli Bi, the Research Spectroscopist located at Argonne, to attend one domestic scientific conference (e.g., Fall AGU), and COMPRES workshops.

**Materials & Supplies**: We request $750 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, 57Fe metal to make samples for Mossbauer experiments, custom sample holders, etc. Dr. Wenli Bi is encouraged as a COMPRES research scientists 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:** $800 is requested to cover the costs of registering for conferences for the Spectroscopist at APS Sector 3 (e.g., Fall AGU and the COMPRES Annual Meeting), and for the registration fee of the PI for the COMPRES Annual Meeting. Budget figures are estimated from historical costs and included in the budget category “other”.

**Indirect Costs**: The indirect cost rate applied has been negotiated and approved by the Office of Naval Research, on July 28, 2015. The indirect rate is 24.0% for off-campus costs for the Research Spectroscopist salary and travel, and 60% for on-campus costs related to PI travel. This is charged on Modified Total Direct Cost (MTDC).

**Publications**

Wenli Bi, Jiyong Zhao, Jung-Fu Lin, Quanjie Jia, Michael Y. Hu, Changqing Jin, Richard Ferry, Wenge Yang, Viktor Struzhkin, E. Ercan Alp, ["Nuclear resonant inelastic X-ray scattering at high pressure and low temperature,"](http://journals.iucr.org/s/issues/2015/03/00/hf5283/index.html) J. Synchrotron Rad. **22** (3), 760-765 (2015).

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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,"](http://ammin.geoscienceworld.org/content/99/2-3/415.abstract?related-urls=yes&legid=gsammin;99/2-3/415) Am. Mineral. **99** (2-3), 415-423 (2014).

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W. Bi, J. Lim, G. Fabbris,J. Zhao, D. Haskel, E. E. Alp,M. Y. Hu, P. Chow,Y. Xiao, W. Xu,and J. S. Schilling

“Possible quantum criticality in compressed elemental europium”, submitted to Phys. Rev. Lett., Nov. 3, 2015

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.

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.