**Beamline scientist annual report for the year of 2015**

**Name:** Dongzhou Zhang

**Position:** Partnership for extreme crystallography beamline scientist (APS 13-BM-C)

**Length of time at current position:** 10 Months, since January 2015.

**Brief job description:** The PX^2 program is a collaboration between the University of Hawaii and the GeoSoilEnviroCARS beamline at the APS, and is fully supported by COMPRES. My job is to design, build, and maintain the state of the art instrumentation for the PX^2 project at Argonne National laboratory, and support user program in high-pressure science at the PX^2 facility.

**Activities:**

My activities in the year of 2015 are majorly divided into two categories: beamline development and user support. PX^2 started general user program for diamond anvil cell experiments in the 2015-1 run. In each run cycle (11 weeks of beamtime + 5 weeks of shutdown time), approximately 5.5 weeks of beamtime are used for non-high-pressure surface diffraction experiments, with the remaining 5.5 weeks available for COMPRES diamond anvil cell (DAC) users. PX^2 is still in a phase of active commissioning (new horizontal focusing mirror has just been installed in September 2015, and the laser spectroscopy system is scheduled for installation in early 2016), and we have been using about 1 week, out of the available 5.5 weeks of DAC beamtime for commissioning activities. Commissioning, and instrument development and improvement activities that do not require X-ray beam are also carried out during shutdown periods.

*Beamline development*

- Optical camera with zooming: In high pressure diamond anvil cell research, the sizes of the samples are usually very small. Therefore, a high precision sample viewing camera with zooming capability is important to locate sample and to effectively carry out measurements. I installed an optical camera system with motorized zooming tube. This camera is dedicated to the PX^2 program. The translating, focusing and zooming capabilities of the camera are all motorized, and can be operated remotely. This system has been appreciated by the users.

- Resistive heating and temperature readout setup: Contemporary research on Earth-related materials requires not only high pressure, but also high temperature, and resistive-heated diamond anvil cell has been broadly used in recent mineralogy experiments. I developed and installed a remotely-controllable resistive-heating and temperature reading setup in the experimental station 13-BM-C. A 1000-W power supply is used to power the heaters in the DACs, and a digital multimeter is used to read temperatures from thermal couples. The setup is capable of heating the samples up to 1000 C. A water-cooling system has been installed to stabilize the sample and the diffractometer.

- New X-ray focusing K-B mirror: K-B mirror is critical for diffraction studies using high pressure diamond anvil cells, because it focuses X-ray photons to the tiny sample. In September a new horizontal K-B mirror has been commissioned. In comparison with the old, smaller K-B mirror, this new K-B mirror provides about twice as much X-ray flux, and features significant improvements in both spatial resolution and stability.

- Membrane pressure controller: Diamond anvil cells mounted in membrane pressure controller allows pressure to be changed remotely, and is therefore more convenient for user operations. The gas pipes for the membrane pressure controller have been installed in the experimental hutch, and the pressure controller is ready for operation.

- Diffractometer: All of our experiments are carried out with the Newport 6-axes heavy duty diffractometer in the experimental station 13-BM-C. After 10 years of usage, the diffractometer showed some wearing. I helped GSECARS staffs Peter Eng and Joanne Stubbs to diagnose (quantitative measurements of the sphere of confusion with laser autocollimator system) and realign the diffractometer, and replaced several parts. I developed a custom Python code to calculate the sphere of confusion of the rotation axes of the diffractometer.

- User interface and website: I wrote an MEDM user interface for the DAC users. I developed a new website for PX^2 program (<http://gsecars.uchicago.edu/page/partnership-extreme-crystallography-px2>). All the manuals for data collection and analysis can be found on the website.

- Instrument design: I designed and assembled the parts that are needed for high pressure single crystal diffraction experiments in 13-BM-C, including the adapter for the goniometer, the sample holders for room temperature and high temperature experiments, and the water cooling system. All the designs are saved as SoildWorks 3-D models, and are compatible with the GSECARS SoildWorks repository.

*Experimental method development*

- Thermal diffuse scattering: The thermal diffuse scattering is a novel way to determine sample’s elasticity at high pressures. At PX^2, we used two different instrument configurations for TDS experiments: the standard configuration with CCD detector, and a custom configuration, featuring background reducing flight-path and PILATUS 100K detector.

- Single crystal and powder diffraction data collection utilizing two detector rotation circles: In order to solve and refine sample’s crystal structure at high pressures, the number of total diffraction peaks is a critical parameter. The six-circle kappa geometry diffractometer used by the PX^2 program features two detector rotation circles: 2-theta and nu. We utilized the detector rotation in both circles in our new experimental approach. This new approach significantly increased the total number of diffraction peaks collected in the experiment, and hence improved the data quality collected in our beamline.

*User support*

- Single crystal diffraction: PX^2 features high pressure single crystal diffraction capabilities using diamond anvil cells. In the past year, we hosted the following single crystal diffraction users: Liuxiang Yang (Carnegie Institution of Washington, diffraction study on carbon and Fe-alloys); Greg Finkelstein (California Institute of Technology, diffraction study on magnesiowustitie); Yi Hu (University of Hawaii at Manoa, diffraction study on pyroxene); Hannah Shelton (University of Hawaii at Manoa, diffraction study on water-containing minerals); Xiaojing Lai (University of Hawaii at Manoa, diffraction study on iron carbide); Fei Qin (Peking University, China and Northwestern University, diffraction study on humite); Su-Ying Chien (HPSTAR, China, stress-strain relationship on mantle minerals); Gang Liu (Carnegie Institution of Washington, single crystal diffraction on piezo-electric material); Wenge Yang (Carnegie Institution of Washington, diffraction study on quasicrystal).

- Powder diffraction: PX^2 is capable of collecting powder diffraction data with extended 2-theta range and very low background. In run2015-1, we hosted Rachel Morrison from California Institute of Technology, and who measured isothermal equation of state of Fe-Ni alloy up to 1 Mbar. This study set the pressure record in our program thus far.

- Thermal diffuse scattering: In collaboration with Jin Zhang (University of Hawaii at Manoa/COMPTECH) and Ruqing Xu (Argonne National Lab), thermal diffuse scattering data on different materials, such as silicon and olivine, have been measured at the PX^2 beamline.

*Crystallographic Training*

- Fundamentals of single crystal diffraction and structure determination: In June 2015 I participated in the American Crystallographic Association Summer School. The two-week long summer school focused on crystal structure determination with X-ray single crystal and powder diffraction. I learned many details on the X-ray diffraction data collection and crystal structure determination. Part of the knowledge I learned, and the lecture notes I collected from the summer school have been used to train our users.

- User training in data collection during experiments: I work extensively with Prof. P. Dera from the University of Hawaii at Manoa in learning and developing single crystal diffraction data collection software and crystallographic computational tools. I wrote the manual of basic operations in the PX^2 beamline for high pressure DAC users, and the manual of data collection software. I trained all the first-time users to collect data properly in our beamline.

- User training in data analysis and post-experiment user data evaluation: PX^2 is a brand new instrument for DAC studies and since it is very different from typical high pressure DAC installations, majority of users who come to PX^2 for the first time require extensive training in the data collection procedures and data analysis process. Very often my help in the data evaluation continues long after the experiment is over, particularly in cases when data shows interesting phenomena or is of lower quality (e.g. twinning, crystal breakage after phase transition, etc.)

- Visit to Bruker AXS headquarters and training in variable-temperature single crystal data collection on diamond inclusions. We hope that this technique will become available to PX^2 users in near future.

*Outreach and Conference presentations*

- In July 2015, I attended the COMPRES Annual Meeting in Colorado Springs. I was invited to give a talk about the development of PX^2 program. In addition, I gave a poster to show the progress of the beamline development.

- I am invited to give a talk in the 2015 AGU Fall Meeting in San Francisco. I am also presenting a poster about the development of the PX^2 program. Besides, I am going to receive the 2015 MRP Graduate Research Award.

- In May 2015, I visited the University of Hawaii at Manoa, and gave a talk at the Hawaii Institute of Geophysics and Planetology. My affiliate faculty status at HIGP was approved recently.