MS18.04.06 LOW TEMPERATURE STUDY—NEW RESULTS AND FUTURE PROSPECT. Kazuhiko Tsuji, Department of Physics, Keio University, 3-14-1, Hiyoshi, Yokohama 223, Japan

An apparatus for X-ray diffraction measurements at high pressures and low temperatures using synchrotron radiation was constructed on a bending-beamline at the Photon Factory, KEK. It consists of a diamond anvil cell(DAC) in a cryostat, a two-dimensional detector of imaging plate, and an in-situ pressure measuring system of ruby fluorescence method.

Low temperature X-ray diffraction measurements under pressure are important for studies of kinetics of pressure-induced phase transitions, magnetic and dielectric phase transitions etc. In the experiments, however, there are some difficulties: (1) A position of the sample moves due to the thermal contraction with temperature. (2) For optical measurements of ruby fluorescence, a distance from the sample to a objective lens is long because of presence of thermal shields and optical window in the cryostat. (3) Size of the sample in DAC is small. Therefore, a special design for x-ray and optical measurements is needed.

X-ray from the bending magnet of BL-18C is monochromatized by Si(111) crystals and is focused by two mirrors of Pt-coated quartz. It impinges a sample between anvils. Diffracted x-ray goes through a window on the cryostat and is detected by the cylindrical or flat imaging plate with a positional resolution of 100 micrometers. By integrating the intensity over a wide area along Debye ring, more precise data can be obtained.

The DAC in the cryostat is cooled using a He refrigerator. Pressure in the sample can be changed by controlling the He gas pressure in a diaphragm which pushes a piston in the DAC. For pressure measurements, ruby fluorescence excited by an Ar ion laser through an optical fiber is collected by two lenses and is focused onto the end of a light fiber whose other end terminates at the entrance slit of a spectrometer. A CCD detector is used for a rapid data acquisition.

Studies on the temperature dependence of pressure-induced phase transitions including amorphizations for several semiconductors are in progress. Performance of the apparatus and new results will be reported.

MS18.04.07 AN EXTERNALLY-HEATED HIGH-TEMPERATURE DIAMOND-ANVIL CELL. Y. Fei and H. K. Mao, Geophysical Laboratory, Carnegie Institution of Washington, 5251 Broad Branch Road, N.W., Washington, DC 20015.

We have developed an externally heated high-temperature diamond-anvil cell that is capable of achieving pressures greater than 125 GPa at temperatures up to 1100 K. The cell was accordingly modified from the Mao-Bell type cell to optimize pressure generation at high temperature. The major modifications include (1) inconel piston-cylinder, (2) thermally insulating the piston-cylinder from the lever arm body, and (3) a double- ring alignment system on the piston to freely retract and advance the piston at high temperature. High temperatures were achieved by a large sleeve-shaped platinum-wire heater fitted around the protruding portion of the piston-cylinder and a small molybdenum-wire heater around the diamond anvils. Combined with synchrotron x-ray diffraction techniques, we can now obtain isothermal compression data for various materials at high temperatures, and in-situ measurements of phase transitions at high pressure and temperature. Experiments on MgO, FeO, and FeS were conducted under hydrostatic or quasi-hydrostatic conditions, using neon as a pressure transmitting medium.

PS18.04.08 LARGE-VOLUME, HIGH-PRESSURE FACILITY AT GSECARS, ADVANCED PHOTON SOURCE. Yanbin Wang, Mark Rivers, Steve Sutton, and Tom Duffy Center for Advanced Radiation Sources, The University of Chicago, 5640 South Ellis Ave., Chicago, IL 60637.

A large-volume, high pressure facility for in-situ X-ray studies is being built at the GeoSoilEnviroCARS (GSECARS) at the Advanced Photon Source (APS). A DIAtype, cubic anvil apparatus will be installed at the bending magnet beam line (13BM-D). This apparatus consists of a 250 ton hydraulic press and six anvils each with a square anvil tip, and has a proven record of being capable of producing high- quality powder diffraction data to pressures up to 15 GPa and temperatures to 1800 K. The 13BM beam line provides energy- dispersive diffraction to energies in excess of 80 keV, as well as monochromatic diffraction with useful energies between 20 and 40 keV. A 1200 ton press will be installed at the insertion device beam line (13-ID-D), with interchangeable tooling that ranges from a large DIA compressing 8 cubic anvils (a 6/8 system) to a conventional split- cylinder multi-anvil configuration making use of large WC anvils. With sintered diamond anvils, pressures and temperatures in excess of 30 GPa and 2000 K, respectively, can be generated with the two-stage, 6/8 system. Deformation experiments are possible by introducing a second jack that independently pushes the top DIA anvil, producing a triaxial stress field with strains up to 10 - 20%. The insertion beam line is also capable of providing both energy dispersive and monochromatic X-rays. This facility will provide opportunities for a wide range of research such as crystallography, equation of state, phase equilibrium, and deformation of materials under simultaneous high pressure and temperature conditions.

PS18.04.09 ADVANCES IN SINGLE-CRYSTAL HIGH-PRESSURE DIFFRACTION. G. Herrmannsdörfer, R.J. Angel, D.R. Allan and R.M. Miletich, Bayerisches Geoinstitut, Universitat Bayreuth, D-95440 Bayreuth, Germany

We have designed a new diamond anvil cell for X-ray diffraction that is suitable for determining both equations of state and structures of single crystals at high pressures. The cell design incorporates the best features of previous cell designs: massive supports to eliminate elastic deformation of the cell under load, widely spaced guide pins to ensure the maintenance of alignment, and diamond mounts that allow precise alignment of the diamonds. Tests of the cell with 600μm culet diamonds have generated sample pressures in excess of 22 GPa on single-crystal samples.

In combination with the new diamond cell design we have developed the use of a large Huber 4-circle diffractometer specifically designed to optimise precision in cellparameter measurement. The large size of the diffractometer leads to intrinsically better collimation than is available on smaller designs, and hence greater resolution in peak positions. In combination with the development of peak centring algorithms and the stability of the new DAC design, we can routinely achieve precision in unitcell volume measurements of better than 1 part in 10,000 at high pressures. This allows us to determine equation of state parameters to much higher precision than previously achieved; typical precisions for materials with bulk moduli of ~80 GPa are 0.1% in K, and ±0.3 in K'. These more precise measurements have also revealed the existence of previously unsuspected changes in the compression mechanisms of complex structures at high pressures.