

PS18.04.10 THE KIEL-BERLIN-CELL: NEW DEVELOPMENTS AND RESULTS. W. Depmeier¹, K. Knorr², B. Annighöfer¹, ¹Institute for Mineralogy, University of Kiel, Kiel, Germany; ²BENSC, Hahn-Meitner-Institute, Berlin, Germany

High pressure - high temperature crystal structure investigations play an important role in various fields of solid state sciences, including geosciences and studies of phase transitions. In the last few years we have constructed a treatable high pressure cell of the piston-cylinder type for neutron powder diffraction. This "Kiel-Berlin-Cell" runs successfully at the BERII reactor of BENSC, Hahn-Meitner-Institute, Berlin. Actual operating data are simultaneously 600 K and 17 kbar with a sample volume of 500 mm³. The zero-scattering Ti - Zr alloy used for the pressure cylinder allows Rietveld-refineable diffractograms to be collected. Some recent results will be reported and discussed.

In order to extend the accessible range of temperatures and pressures a new cell has been designed. We hope to attain at least 35 kbar and 773 K with this new "KielBerlin-Cell II". The cell is currently being built.

PS18.04.11 THE DETERMINATION OF THE SEEBECK COEFFICIENTS OF SOME COMMON THERMOCOUPLE WIRES USING EPITHERMAL NEUTRON RESONANCE BROADENING. S. M. Clark, R. L. Jones, R. te-Lindert, D. Walker¹, M. C. Johnson¹, P. Fowler², CCLRC, Daresbury Laboratory, Warrington, WA4 4AD, UK, ¹Lamont-Doherty Geological Obs., Columbia University, Palisades, New York, 10964, USA, ²H. H. Wills Phys. Lab., University of Bristol, Bristol, BS8 1TL, UK

Thermocouples provide the only means of determining temperature in large volume high pressure cells. The effect of temperature on thermocouple emfs was not previously known above 3.5 GPa. We have determined the effect of pressure on thermocouple emfs up to 1.2 GPa and 1700°C using epithermal neutron resonance broadening as an in-situ pyrometric technique. A Walker high pressure cell with a 10,000 kN hydraulic ram was used for the generation of high temperatures and pressures. Data were collected on the TEST station of the ISIS spallation neutron source. Details of the experimental technique and data analysis procedures will be given together with details of the emf corrections.

PS18.04.12 THE MELTING TEMPERATURE AND COMPRESSIBILITY OF ALPHA SILICON NITRIDE UP TO 35 GPa Y. Cerenius, Institute of Earth Sciences, Uppsala University, Sweden

The melting temperature of Silicon Nitride as a function of pressure was investigated by laser-heated diamond-anvil-cell (DAC) experiments. The system consists of a stabilized cw 33W Nd:YAG laser that heats a Rhenium foil situated in the middle of the sample. The thermal radiation from the sample was collected by an imaging spectrograph together with an area CCD-detector. The spectra were fitted by a Planck's function in the range between 600 and 900 nm to obtain the temperature. The correlation between the laser-power and the temperature, that is change in the slope of the sample temperature versus laser power, together with direct observation of the heated spot for convecting movements gives the melting criterion. Pressure was determined by the Ruby fluorescence method.

The melting temperature was found to vary from 2200±100 K to 3600±200 K in the pressure range between 3.5 and 35 GPa.

The bulk modulus was determined with in-situ energy dispersive X-ray diffraction in the DAC using synchrotron radiation as the X-ray source. Pressure was determined by using Sodium Chloride as an internal standard.

The cell parameter data of the Silicon Nitride was fitted to Birch-Murnaghan equation of state keeping the pressure derivative B₀ fixed at certain values in the interval between 2 and 8. The best fit was obtained for B₀ = 7 resulting in a bulk modulus (B₀) of 350±25 GPa.

PS18.04.13 DESIGN OF A COMPACT HIGH-PRESSURE X-RAY DIFFRACTION CELL M.Kriechbaum, M.Steinhardt, K.Pressl, P.Laggner, Institute of Biophysics and X-Ray Structure Research, Austrian Academy of Sciences, A-8010 Graz, Austria.

A compact X-ray sample cell capable of measuring diffraction patterns within a maximal angular range of 30° at hydrostatic pressures of up to 3 kbar - using water as the pressure transmitting liquid - has been developed. The sample thickness can be 1.5 mm with a volume of approximately 1 mm³ completely irradiated by pin-hole collimated (1.0 mm diameter) X-rays. Additionally, the temperature in the pressure cell can be regulated by external Peltier devices in the range from -10°C to 80°C. The large accessible angular range in the reciprocal space makes the cell well suited for scattering/diffraction measurements in the small- and wide-angle region of samples like solid polymers, liquid-crystalline probes and biological model-membrane systems. Thus, information on the short and long range order can be obtained simultaneously, i.e. lattice and chain packing parameters in lipid bilayers. Particularly, barotropic and thermotropic phase transitions of liquid crystalline systems at constant temperature or pressure, respectively, and their p-T phase diagrams can be studied. Results of these measurements obtained with laboratory X-ray sources as well as at the synchrotron source DESY in Hamburg, Germany, will be presented.

Currently, this system is being adapted for the use of time-resolved measurements of dynamic processes (pressure-jump relaxation experiments) with a targeted time resolution of diffraction patterns in the millisecond range and integrated into the sample stage environment of the SAXS beamline at the synchrotron source ELETTRA, Trieste, Italy.