Reactions in the system Fe-H-O at high pressures and temperatures

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Abstract – Knowledge of phase relations in the system Fe-H-O is important for understanding oxidation – reduction reactions between the mantle and core. If the reaction: $2Fe+H_2O = FeO + FeH_2$ proceeds to the right, oxygen is pumped into the mantle and the core becomes hydrogenated, whereas the opposite is the case if the reaction proceeds to the left. Such reactions may be important in the formation of the seismically imaged D" layer at the core-mantle boundary. We attempted to investigate this reaction at ultra-high pressures and temperatures in the diamond anvil cell by using a combination of laser-heating and in situ X-ray diffraction at at beamline BL10XU. The idea was to heat Fe metal embedded in a hydrogen-oxygen bearing pressure medium, to detect the formation of FeO by X-ray diffraction, and perhaps to determine a change in the unit cell volume of Fe if hydrogen was included in its structure. The goal was to see if the reaction was sensitive to pressure, especially at conditions approaching the core mantle boundary.

Experimental – A fine powder of ultra-pure Fe metal with a grain size of approximately 1 micron in diameter was precompressed into a metal foil about 10 microns thick and 50 microns in diameter. The metal foil was placed into a pressure containing H and O. Several materials were tried, including aluminum hydroxide, magnesium hydroxide, and lizardite (a natural serpentine mineral). Although we were able to laser heat with this sample configuration, we found that it was extremely difficult to maintain a stable heating for more than tens of seconds at a time, even with a laser feedback system in operation. The reason for this difficulty was determined to be the release of water from the pressure medium upon heating above about 1500 K. The water moved around the pressure medium rapidly, causing inhomogeneous heating and reaction with Fe. Water and/or Fe (probably both), also moved to the surface of the diamond anvils causing surface damage including pits and cracks. It is likely that the pressure medium contained to much water, so we tried an anhydrous medium (aluminosilicate glass) or CeCl, and added hydrogen by placing the open DAC in a mixture of Argon + 1% H. This combination produced much more uniform and stable heating of the sample.

Several experiments were performed at pressures from 50 to 60 GPa and at temperatures of about 2000 K. The samples were heated by scanning the laser across the entire sample for up to 5 minutes at a time. After heating, the sample was mapped using in situ x-ray diffraction. In the experiments no FeO was found, indicating that the reaction between Fe and H_2O was not occurring at these conditions. Also, no change in the unit cell volume could be documented within experimental uncertainty. More work is need to determine the phase relations between Fe and H including 1) finding the best sample configuration, including the most suitable pressure medium and method for introducing H into the cell. 2) longer more stable heating over a range of higher pressures.

Structural and Electrical Study in Li under Low Temperature and High Pressure

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X-ray structural study was performed up to 42 GPa in lithium at low temperature. A new structure was found by ESRF group above 39 GPa last year, however within this beam-time period we could not re-examine and find a new structural phase. At the previous beam-time we succeeded to suppress the chemical reactivity of lithium by treating at low temperature lower than 50 K, however a critical problem remained; the sample chamber shrank and the sample lithium went out away above 30 GPa. This high mobility was often observed with a several kind of gasket of stainless steel, 310S, Be-Cu alloy and rhenium. We used a "pit-anvil" at this time to keep the sample in the chamber. A pit was made by ultra-violet laser (248nm) by 50-micron in diameter and 7-micron in depth on the pressure surface of the diamond-anvil. The detail will be described elsewhere.

The pressure of DAC in the cryostat was remote-controlled by helium-gas pressure. After cooled down to 50 K, the data was recorded with the imaging plate (IP). In both experiment rhenium gasket was used. The first run reached 35.5 GPa and in the following compression the chamber shrank. At the second run the pressure reached up to 42 GPa and the diamond was broken at around 45 GPa. The thickness of the sample was twice of the previous run by using the pit-anvil, however the obtained diffraction pattern from the lithium was obtained with spots with one from used gasket and no considerable change in the pressure dependence. The mobility of lithium under pressure and low-temperature itself enough interest aspect which should be investigated in future.