ESRF

Experiment title: Equation of state and phase transition behaviour in metal carbides, hydrides, and close-packed structures in 100 GPa pressure range.

Experiment number:

HC-447

Beamline:

BL3 (ID9)

Date of Experiment:
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9

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Report:

The work we report here was motivated by geophysical needs to investigate the the candidacy of hydrogen and carbon in the Earth's Core. Basically the Outer Core is believed to consist of Fe-Ni alloy with lighter components. The aim of this experiment was to study structure stability of the Ni-H and Fe-C systems, as well as to determine the equation of state for nickel hydride.

Ni-H. Under ambient pressure nickel hydride is thermodynamically unstable. The sample was synthesized *in situ* from nickel powder and liquid hydrogen under high pressure, Two experimental runs were made at beamline BL3 (ID9) operating in monochromatic mode. The image plate was used to collect 2-dimensional diffraction patterns. Both runs were carried out at room temperature in diamond-anvil cell. Pressure was determined by ruby fluorescence method.

In the 1st run the powder diffraction data up to 50 GPa were obtained from the sample with $30\mu m$ in size confined in gasket chamber $70\mu m$ in diameter and $50\mu m$, thick. No structure change from the original fcc lattice was observed up to the maximum pressure (Fig.1, a) (we are sensitive to only Ni sublattice). Pressure-volume data obtained were fitted by Vinet *at al.* equation of state (EOS) with K_0 = 183 GPa, K_0' = 3.56, and V_0 = 7.914 cm^3/mole.

In the 2nd run a sample with size of $\sim 10\mu m$ was confined into a $\sim 20\mu m$ chamber made in a Re gasket. An 20 x $20\mu m$ vertically focused incident beam was used. Only the (200) peak of fcc lattice was observed. All others are hidden in the background diffraction pattern of the gasket (Fig. 1, b). At 123 GPa a (111) peak has become visible (the compressibility of NiH_x is higher than that of rhenium). The specific volumes for NiH_x obtained from (200) peak as well as from both (111) and (200) peaks for the last F'- *V* datum point fit well to the *P-V* curve calculated from the data to 50 GPa (Fig. [nihpv]).

Powder diffraction on H_2. Concomitantly with the studies of nickel hydride in the 1st run we observed for the first time a powder diffraction pattern from molecular hydrogen. In our experiment (101) powder diffraction peak appeared at 10 GPa and remained visible up to the highest pressure achieved in the run, that is at 50 GPa (Fig. 1, a) (see preliminary partial report). This experiment demonstrates that polycristalline sample of molecular hydrogen maybe obtained in diamond-anvil cell by pressurization of hydrogen together with a metal powder. The intensity of the x-ray beam is sufficient to obtain diffraction pattern with ~7:1 signal-to-nose ratio. Such work opens up the possibility that phase transitions in solid hydrogen can be studied directly with powder techniques.

Re-H. Background diffraction patterns from rhenium gasket observed in second run exhibit a duplication of most of peaks (Fig. 1, b). After fitting by pseudo-Voigt profiles the peak positions of the upper angle set are in consistent with the EOS of Re [1]. The peaks related to the lower angle set are matching each other in hcp lattice and can be attributed to ReH_x, which is known to be of the same hcp structure as Re is, and its lattice is slightly expanded due to saturation with hydrogen. One can conclude that no structure changes is observed in rhenium hydride up to 123 GPa. Also a room temperature equation of state for rhenium hydride is determined.

Fe_3C. Iron carbide Fe_3C (cohenite) is thermodynamically unstable under ambient conditions, but it has a metastable phase which is orthorhombic with *Prima* space group. In our study we used a natural sample extracted from a IA iron meteorite *Canyon Diablo* (provided courtesy V.F. Buchwald, Technical University of Denmark). The sample was confined into the chamber of 140 μm diameter and 35 μm thick, made in Re gasket, Helium was used as a pressure-transmitting medium. Six 2-dimensional angular-dispersive diffraction patterns (images) at the pressures between 2.8 and 21 GPa have been obtained at room temperature. From 25 to 30 diffraction peaks at different pressures were observed. The precise analysis accounting for admixing phases as well as possible decomposition of cohenite onto carbon and iron requires further work. However higher resolution data seems to be needed to resolve the structure.

[1] Y. K. Vohra *at al.*, *Phys. Rev. B* *36,9790-9792 (1987)*

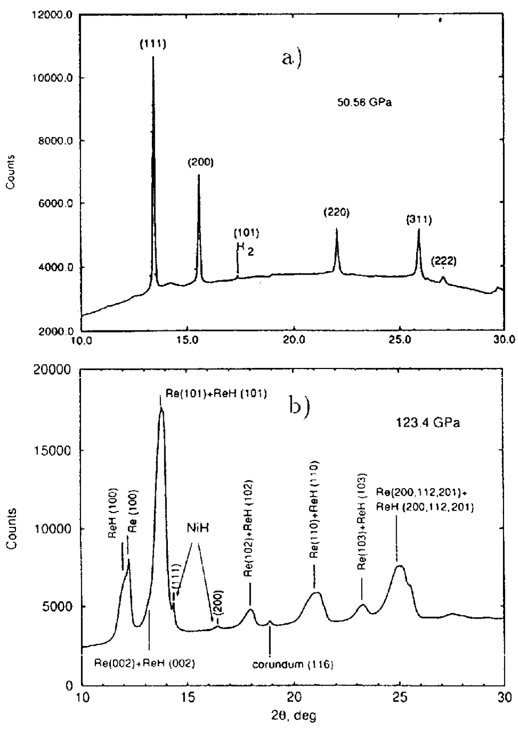

Figure 1: a) Diffraction patterns of NiH from the 1st run. No structure changes were found. The (101) peak of a powder diffraction pattern from molecular hydrogen is also observed.
b) Diffraction pattern at 123 GPa

Figure 2: Equation of state (EOS) of NiH Crosses - 1st run, circles - 2nd run. Solid curve is the Vinet *at* al. EOS fit to the data of the 1st run.