ES-RF

Experiment title: Equation-of-state and phase-transition behaviour in metal hydrides and deuterides in the 100 GPa pressure range		**Experiment number:** HS136
Beamline: ID30	**Date of Experiment:** from: A:28/11/96; B:27/3/97 to: A:29/11/96; B:30/3/9;	Date of Report: 29/8/97
Shifts: 9	**Local contact(s):** M. Kunz & D. Häusermann	Received at ESRF: 4 SEP 1997

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Report:

We were originally scheduled single-bunch mode in order to test beam focusing at ID30. The flux was therefore significantly lower than normally required for high-pressure DAC experiments above 50 GPa. The original intention in 11/96 was to run monochromatic. Problems in mirror operation after lengthy set up prevented these experiments going ahead as planned. We were able, due to rapid response of BL staff, to run in energy-dispersive mode and succeeded in obtaining data on AlH_3 and AlD_3 to up to 50 GPa. We confirmed two objectives: (1) Repeat of AlD_3 in excess He to correct for (apparently) sensitive dependence of the EOS obtained in a previous study to onset of nonhydrostatic conditions and (2) compression of AlH_3 in a solid H_2 medium to verify no additional exchange or deviation of atomic H in metal Al host with pressure. We obtained a constant resolvable difference in volume between the two isotopes as a function of pressure and accurate measurements of the H volume.

Due to the earlier beam problems, we returned in 3/97 to continue experiments in monochromatic mode. We obtained pressures above 100 GPa on the Ni (Fig. l), Fe, and Re - H (Fig. 2) systems and high resolution data on low symmetry Fe_3C to 50 GPa under hydrostatic conditions (Fig. 3). Beam size had been measured at ESRF before arrival at $\leq$0.010 mm. We found that strongly interfering diffraction was obtained from the gasket confining the sample. Nonetheless, early results were sufficient to provide some indication of the pressure dependence of the H atom volume in metals under pressure: Ni appears in accord with a d-band electronic model, but Re does not. Further work on d metals from other Groups in the periodic table will help understand these effects.

It appears that the technique to focus and/or ***measure monochromatic*** beam sizes in the 0.010 mm range are not yet effective at ESRF. Unlike single-crystal techniques, it is essential in powder studies of low-2 systems to exclude gasket diffraction as intensities from the sample are significantly lower. Further, there is a distinction here between use of a gasket as the sample itself (as has been reported at ESRF (ID9)) and the more stringent use of a gasket to confine a different sample which means that all extraneous diffraction must be excluded. In focusing geometry, beam collimation (a useful solution in white-beam ED experiments), may or may to be effective if the aperture is not located close to the sample and is usually prevented by the geometry of the DAC. We plan continuation of proposals as technical developments and solutions resulting from these experiments take place at ESRF.

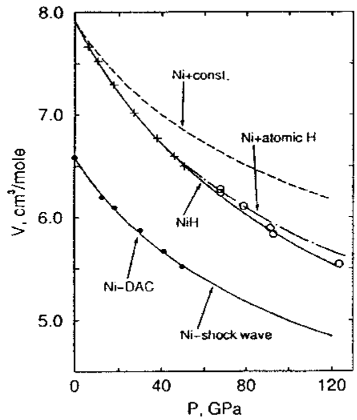

Figure 1: Equation of state of NiH_x and Ni. Crosses and open circles denote. the experimental points from the 1st and the 2nd runs respectively. Solid circles are the data obtained on pure Ni in the DAC. The difference between tie specific volumes of NiH_x and Ni at ambient pressure (ΔV_0 = 1.326 cm^3/mol) corresponds m a hydrogen concentration x in the range $0.7 > x < 1.2$.

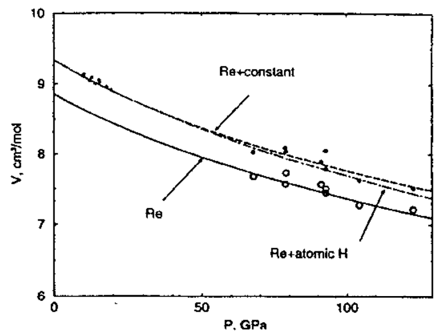

Figure 2: Equation of state of Re and ReH_x. Open circles a the experimental points for Re. Solid circles are for ReH Diamonds are plotted from and the dashed line is the EO for $ReH_{0.38}$ from Badding *et al.*, 1995. where hydrogen w found to be "incompressible" to 20 GPa The solid line fit the EOS of Re from Vohra *et* al.. 1987. The dash-dot line the EOS obtained by summing the volume of Re with that atomic H from Taguchi *et* al,. 1994 (reduced to x = 0.38).

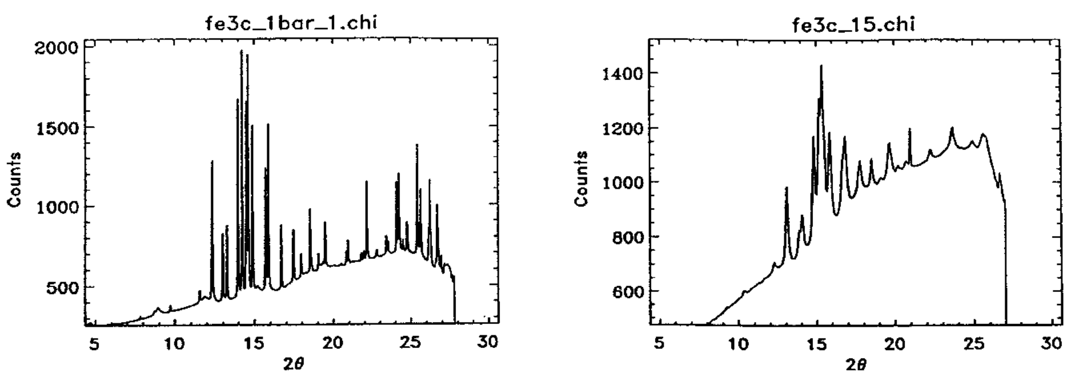

Figure 3: Comparison of integrated image-plate spectra obtained from Fe_3C at 1 bar and at 50 GPa under quasihydrostatic conditions with He as pressure-transmitting medium. No phase transition is observed in this pressure range, although peak widths and resolution have decreased. One goal of future studies will be to examine the evolution of low-symmetry structures at high pressures and the effects of strain broadening on the powder pattern.