ESRF	**Experiment title:** Equation of state, phase transitions, and structures in metal hydrides and deuterides in the 100 GPa pressure range.	**Experiment number:** HS489
Beamline: ID9/BL3	**Date of experiment:** from: 16/4/98 to: 19/4/98	**Date of report:** 1/11/99
Shifts: 9	**Local contact(s):** M. Hanfland	*Received at ESRF:*

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Report:

The objectives of the experiments included: (i) test of 2D, image-plate, powder diffraction with two (new) designs of a diamond-anvil cell (DAC) and (ii) examination of phase transition behaviour in metal hydrides, ZrD_2 and AlH_3. One other material – an oxynitride perovskite ($NdTiO_2N$) – was compressed for analysis by Rietveld techniques.

The DAC's included a beryllium anvil support cell and a traditional Mao-Bell piston-cylinder design modified with the tungsten carbide support flat replaced by a diamond composite (PCD) plate, 5mm thick. The PCD plate was provided in 1991 by A.E. Ringwood of ANU. It is sintered diamond with non-metal (SiC) binders for relatively low x-ray absorption. Fig. 1 illustrates this cell design where the flat PCD insert supported the single crystal diamond anvil directly up to a maximum pressure in this experiment of 120 GPa (see below). Calibration and pre-beam trials suggest approximately 30% transmission at 0.4 Å in rough agreement with other, recent (multianvil) experiments [1]. The EOS of ZrD_2 was observed with this DAC and revealed a continuous structural distortion to a bcc Zr sublattice (bct c/a=1) near 125 GPa from the approaching coalescence of the fct-(111) and fct-(200) reflections of the ϵ phase. The change is reversible and the 1-bar pattern of ϵ-ZrD_2 was recovered on release of pressure (Fig. 2). The beryllium cell produced 2D data sets on the oxy-nitride perovskite (synthesized at Oxford in the Department of Chemistry by Dr S.J. Clarke). LeBail extractions and subsequent GSAS refinement produce accurate lattice parameters (Fig. 3) and indicate that, although the cell and Ti sublattice are metrically cubic above ≈8 GPa, the symmetry remains orthorhombic to 26 GPa determined

from the presence of weak reflections. A reversible transition in AlH_3 was observed near 50 GPa (Fig. 4), possibly associated with an insulator-metal transition in this region and photochemically assisted by (visible) laser irradiation that occurred during ruby pressure measurement. The transition is marked by many weak diffraction peaks that may be associated with shift of the H atom locations and is reversible on pressure cycling near 50 GPa.

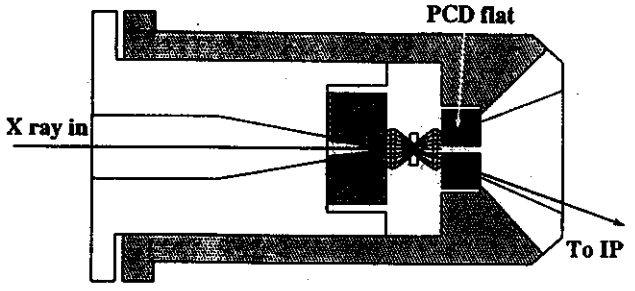
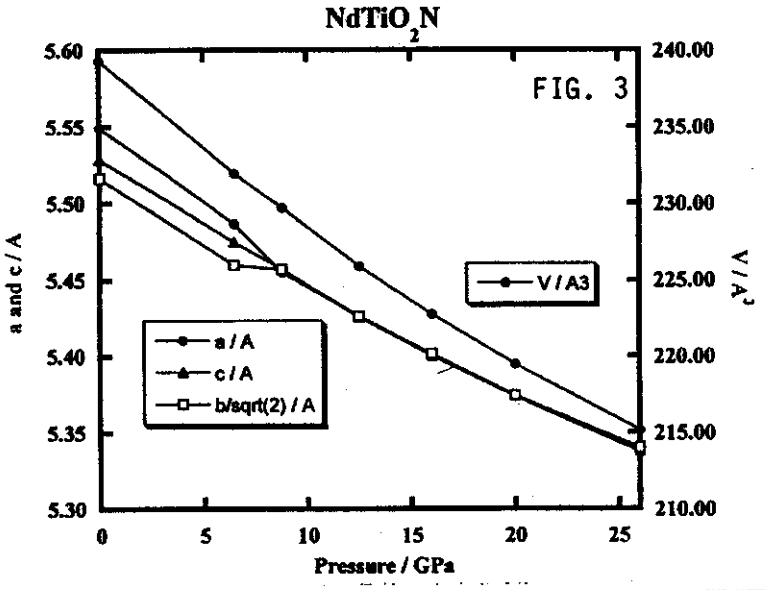


Figure 1: 2D diffraction geometry with PCD anvil support



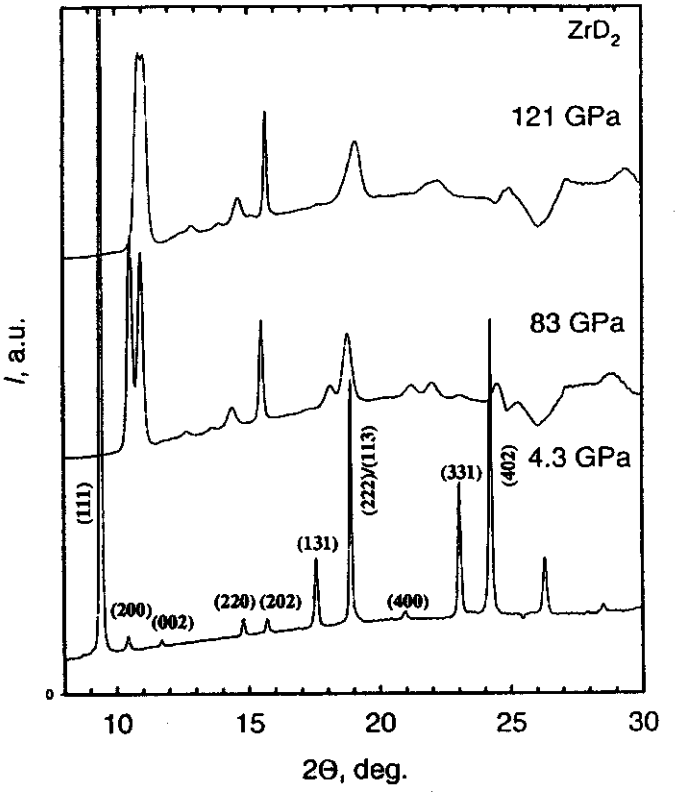
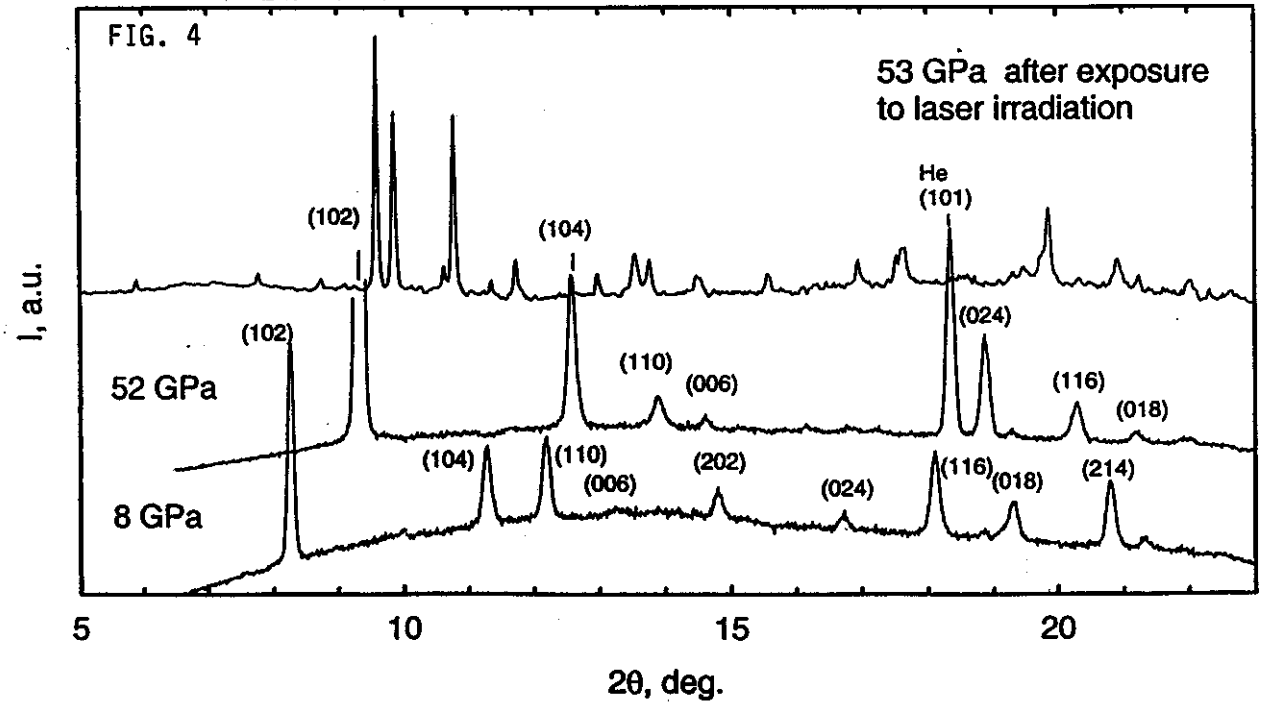


FIG. 2



[1] T. Irifune, W. Utsumi, T. Yagi, *Proc. Japan. Acad.*, **68**, 161 (1992).