# X-ray Diffraction Experiments of Solid Deuterium at High Pressures and Low Temperature

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Solid hydrogen exhibits three crystalline phases mainly based on the difference in the orientation of molecules in the unit cell. Phase I (low pressure phase) has a hcp structure with freely rotating molecules in the unit cell. The crystal structures of phase II (low temperature phase) and phase III (high pressure phase) have not been clarified yet. The transition boundary between phase I and phase II is reported to exhibit a strong isotope effect and it shifts to lower pressure for deuterium. The boundary has positive slope in P-T phase diagram and solid deuterium transforms into phase II at about 28 GPa and 15 K. Moreover, it is pointed out that for n-$D_2$ phase II' exsists in the narrow pressure range between phase I and phase II. In the previous experiment we have observed that deuterium molecules in phase II are on the hcp lattice points and the c/a ratio changes discontinuously at 27.8 GPa.

A Merrill-Bassett diamond anvil cell (MBC) was used for high pressue generation. The top surface of the diamonds was 0.4 mm in diameter. To fill deuterium into the sample hole in a metal gasket (U-700) with a hole diameter of 160 mm and a thickness of 50 mm, the MBC was placed in a high-pressure vessel of a gas-loading system in which deuterium gas of 180 MPa was introduced. High-pressure powder x-ray experiments at low temperature of 15 K were carried out using a monochromatic SR source and cryosystem on the BL10XU station at SPring-8.

In this experiment our purpose is to examine how the lattice parameters of solid n-$D_2$ change under pressure between phase I and phase II. The upper anvil, however, was broken at pressure below 28 GPa.



# Study on the Formation of Rhenium-hydride by X-ray Diffraction Experiment

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## Introduction

Most of transition metals incorporate hydrogen without structural changes under high pressure. Hydrogen dissolved in a metal causes expansion of the lattice. The differences in the unit cell volumes of metal hydride and pure metal can be used to estimated the stoichiometry of the hydride. It is known for rhenium hydride that the unit cell volume is expanded by 2.19 Å$^3$ per one hydrogen atom.

## Experimental

Powdered Re was loaded together with hydrogen at 0.18 GPa at room temperature in a diamond anvil cell. The molar ratio of hydrogen to rhenium was estimated to be larger than 5. Pressures were measured with ruby fluorescence method. High-pressure powder x-ray experiments were carried out using a monochromatic SR source (l = 0.6211 Å). Powder patterns were obtained by an angle dispersive method with an image plate detector.

## Results

Figure 1 shows the diffraction profiles at selected pressures. Sharp diffraction lines were obtained because the pressures remained quasi-hydrostatic due to the presence of excess hydrogen. The hcp structure remained up to the highest pressure of 18 GPa. Figure 2 shows the volume of rhenium hydride as a function of pressure. Closed triangles and open circles denote data collected on the pressure increasing and decreasing cycle, respectively. In the figure, dotted line represents the EOS of pure rhenium metal. The volume of rhenium hydride increases to the pressure of about 10 GPa. The behavior is attributed to the increase of incorporated hydrogen with increasing pressure. H/Re content is estimated about 0.35 in atomic ratio above 10 GPa.

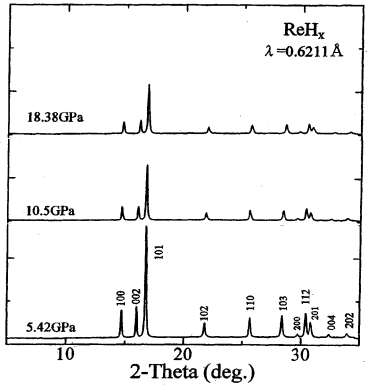


Figure 1. X-ray diffraction profiles of rhenium hydride.

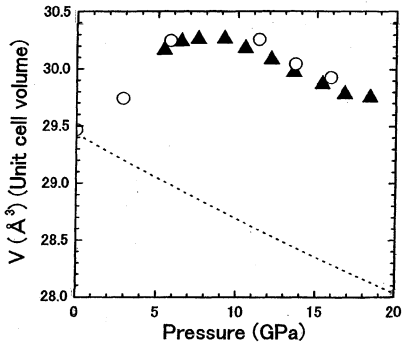


Figure 2. Unit cell volume of rhenium hydride as a functon of pressure. Dotted line represents the EOS of pure Re metal.