ESRF	**Experiment title:** EQUATION OF STATE AND CRYSTAL STRUCTURE OF NEARLY STOICHIOMETRIC WÜSTITE AND OF HIGH MAGNETITE	**Experiment number:** CH - 169
Beamline: ID9 - BL3	**Date of Experiment:** from: 02.04.96 to: 06.04.96	**Date of Report:** 27.02.97
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Report: To determine the phase relations in the Fe-O system at high pressure and temperature is challenging because of the complexity of one of the phases in this system; wustite . At atmospheric pressure wustite, $Fe_{(1-x)}O$, forms eutectoidally at 843 K, where it crystallises in a highly defective form of the NaCl-type structure. $Fe_{(1-x)}O$ transforms from the cubic NaCl-type structure to a rhombohedral structure at 16 GPa at 300 K[1]. A NiAs-type structure is observed at 90 GPa and 600 K[2]. The bulk modulus of wustite has earlier been determined both by static and dynamic compression methods and values in the range from - 140 to 180 GPa have been reported. The variation of the bulk modulus with degree of non-stoichiometry is uncertain and a study of an iron-rich ($Fe_{0.99}O$) and iron-deficient ($Fe_{0.92}O$) sample has been undertaken. Additionally, Fe_3O_4 have been investigated. At pressures around 25 GPa, Fe_3O_4 transforms from the cubic inverse-spine1 structure to a high - pressure structure (= h - Fe_3O_4)[3]. It has been suggested that the high - pressure phase is monoclinic,[3] but the structure is still unresolved.

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[1] Fei, Y., (in press) *Geochemical Soc. Special Publication The Roger G. Burns Memorial Volume* (1995)
[2] Fei, Y. and Mao, H. K., *Science, 266, 1678* (1994)
[3] Mao, H. K., Takashi, T., Bassett, W. A., Kinsland, G. L. and Merrill, L.,
J. Geophys. Res., 79, 1165 (1974)
[4] Finger, L. W., Hazen, R. M. and Hofmeister, A. M., *Phys. Chem. Minerals,* 13,215 (1986)

High-Pressure X-ray diffraction experiments were carried out using monochromatic radiation, $\lambda \sim 0.48$ A, at BL3. The diffraction data were collected by a two dimensional image-plate detector. High pressure was generated using membrane diamond anvil cells (MDAC). The diamond anvils had a top surface diameter of 320 pm. The sample, small ruby grains and liquid nitrogen was loaded into a ~ 150 pm diameter hole of a stainless steel gasket. The pressure was varied in the range from 0 to 40 GPa at room-temperature and determined by using the pressure induced shift of the ruby R_1 luminescent line.

The phase transition of $Fe_{(1-x)}O$ from the cubic to the rombohedral structure was seen in the X-ray diffraction patterns as a splitting of the 111 and 220 diffraction peaks (fig. 1). A broadening of the diffraction peaks is seen below the transition pressure. No corresponding broadening of the diffraction peaks is observed in the X-ray diffraction patterns of Fe_3O_4. Figure 2 shows the X-ray diffraction patterns of Fe_3O_4 at pressures below and above the transition. The p-V data of the cubic $Fe_{(1-x)}O$ are plotted in figure 3. The volume of $Fe_{(1-x)}O$ was as a first approach calculated using only the 200 diffraction peak. Our results indicate that the bulk modulus of wustite is composition independent. Our p-V data of 1 - Fe_3O_4 are plotted together with results from earlier investigation (fig. 4).