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FINAL REPORT

High-pressure experimental data on planetary materials are critical in developing planetary models and in addressing otherwise insoluble problems of the internal structure of the major planets. Progress at this laboratory in the last five years has been particularly marked. Maximum static pressure of 550 GPa was achieved. For the first time, x-ray diffraction of solidified gases (Ne, Xe) and ices (H₂O) were obtained at pressures above one megabar [Hemley *et al.*, 1987b,c; Jephcoat *et al.*, 1987b], single-crystal diffraction of ultra-light elements (H₂, He) were detected up to 25 GPa [Mao *et al.*, 1988a,b], pressures over 200 GPa at 77 K were reached in solid hydrogen, including the discovery of a phase transformation in the molecular solid [Hemley and Mao, 1988]. Advances in instrumentation and new measurements performed during 1983-1988 are summarized below. This research was supported by the four-year NASA grant NAGW-214.

Instrumentation

High Pressure Technology. Major break-through in high-pressure technology has occurred as a result of improvements in the diamond cell design. The record of maximum static pressure has increased from 172 GPa before the starting of the present project to 300 GPa in 1984 [Bell *et al.*], to 550 GPa in 1986 [Xu *et al.*]. The maximum pressure in hydrogen has increased from 60 GPa to 147 GPa in 1985 [Mao *et al.*, 1985b], and 250 GPa in 1988 [Hemley and Mao]. The ruby pressure scale has been calibrated to 80 GPa under hydrostatic condition [Mao *et al.*, 1986] and 180 GPa under nonhydrostatic pressure [Bell *et al.*, 1986a].

Synchrotron X-ray Diffraction. X-ray diffraction is the most definitive method for determining density and crystal structures, i.e. the fundamental parameters for modelling at high pressures. Using techniques based on the diamond-anvil cell, we have developed both single-crystal and polycrystalline diffraction methods using white radiation synchrotron and EDX (energy dispersive x-ray) detectors. Incident and diffracted beams are collimated down to a size of 5-10 μm , which is necessary for ultra-high pressure experiments. X-ray induced ruby fluorescence was measured simultaneously with the diffraction for pressure calibration. We have also developed an angular dispersive technique which employs a focusing monochromatic synchrotron beam for high-precision x-ray diffraction at high pressures and moderate temperatures (up to 900 K with an external heater). These techniques have been tested successfully at beamlines that have a range of energies, brilliance, and divergence characteristics. Beamlines at the National Synchrotron Light Source (NSLS), the Cornell High Energy Synchrotron Source (CHESS), and the Stanford Synchrotron Radiation Laboratory (SSRL) have been used by our group.

The experience gained in these experiments has been used for constructing a new state-of-the-art superconducting wiggler beamline (X17) at NSLS. The beamline, which is scheduled to begin operation in January 1989, is dedicated full-time to the high-pressure diamond-anvil cell community. We are a member of the Insertion Device Team (IDT) in charge of building permanent equipment funded by other IDT members and by NSF.

With the reduction of set-up time at the permanently equipped beamline, we anticipate an increase of 100% in operating efficiency over previous experiments.

Optical Spectroscopy. As summarized by *Hemley et al.* [1987] and *Hemley and Porter* [1988], laser Raman and fluorescence spectroscopy have proven to be invaluable for studies at ultra-high pressures. In the last two months, we have redesigned our spectroscopic system for routine simultaneous study of Raman, fluorescence, transmission, and reflectance spectroscopies at pressures above 200 GPa and at temperatures from 77 to 800 K.

Infrared Spectroscopy. Fourier Transform Infrared (FTIR) spectroscopy has been coupled with high-pressure diamond-cell technique [*Mao et al.*, 1983b]. Pressure-induced infrared spectra of hydrogen were obtained up to 54 GPa.

Materials Studied

Hydrogen. Hydrogen, the most abundant element in the solar system, has the lowest possible x-ray scattering efficiency of all materials, and is invisible in conventional x-ray diffraction studies. X-ray diffraction of single-crystal hydrogen above 10 GPa became feasible only in the last year with the development of new synchrotron x-ray techniques [*Mao et al.*, 1988a]. Normal hydrogen was found to remain as a single crystal with hexagonal-close-packed (hcp) structure at 300 K from 5.4 to 26.5 GPa. The lattice parameter ratio, c/a , had the ideal value of 1.633 at 5.4 GPa, but decreased with pressure to 1.594 at 26.5 GPa, suggesting the possibility of partial orientational ordering. The pressure-volume EOS was in good agreement with the newly improved 5 K isotherm of *van Straaten and Silvera* [1988].

The maximum static pressure achieved in hydrogen (or any gas) before 1988 was 147 GPa [*Mao et al.*, 1985]. With improvements in diamond-anvil techniques, this year we achieved pressures in the 250 GPa range in hydrogen. Raman spectra of the intramolecular stretching mode in solid H_2 were obtained at 77 K up to the maximum pressures. A phase transition was observed near 150 GPa at low temperature. The spectroscopic data are consistent with the transition of hcp to the orientationally ordered Pa3 structure [*Hemley and Mao*, 1988].

Helium. X-ray diffraction of helium is almost as difficult to obtain as that of hydrogen. Recently, single-crystal x-ray diffraction of helium at 300 K from 15.6 to 23.3 GPa was obtained with synchrotron techniques, and the structure was also found to be hcp [*Mao et al.*, 1988b]. However, unlike the results for hydrogen, the finding was unexpected because helium was predicted by theory to have the face-centered-cubic (fcc) or body-centered-cubic (bcc) structure [*Ross and Young*, 1986; *Loubeyre*, 1987]. The c/a ratio of helium remained constant at the ideal value of 1.633 at all pressures. The EOS of helium is considerably softer than that calculated from lattice dynamics using accurate pair potentials for helium [*Aziz et al.*, 1987] but is in good agreement with results obtained using empirical potentials that were fit to shock-wave data at high temperature [*Ross and Young*, 1986]. The results indicate that many-body forces significantly soften the EOS at high densities.

Neon. Below 15.0 GPa, solid neon crystallizes at 300 K in the diamond cell as single crystals, while above that pressure the solid starts to break down into polycrystalline aggregates. The polycrystalline x-ray diffraction of neon has been measured with synchrotron radiation to 110 GPa at 300 K [Hemley *et al.*, 1987c] and 22.0 GPa at 600 K [Mao *et al.*, 1988c]. Over this pressure and temperature range, neon remains an insulator with fcc structure. The P - V EOS of neon agrees with pair-potential predictions at low pressures; however, at higher pressures the pair-potential EOS without many-body terms becomes too stiff [Aziz *et al.*, 1983]. The theoretical EOS based on band-structure calculations shows much better agreement with the data [Boettger, 1986].

Argon X-ray powder diffraction studies of fcc argon have been done to 80 GPa at 300 K. The results are in excellent agreement with exponential-six potential derived from shock wave data on liquid argon to 40 GPa and 16800 K [Ross *et al.*, 1986].

Xenon. Xenon has been studied up to 137 GPa at 300 K by synchrotron x-ray diffraction techniques [Jephcoat *et al.*, 1987b]. The low pressure fcc phase of xenon transformed to an intermediate close-packed phase above 14 GPa, and eventually transformed to the predicted hcp structure above 75 GPa. The measured EOS of xenon was used later to help constrain the metallization pressure of xenon [Goettel *et al.*, 1988; R. Reichlin., pers. comm.].

N₂. Raman, absorption, and x-ray diffraction measurements have been performed on solid N₂ at high pressures. The Raman spectra reveal a sequence of structural transitions in the molecular form to pressures of 180 GPa [Bell *et al.*, 1986b]. Transitions occurring below 65 GPa have been confirmed by energy-dispersive x-ray diffraction using synchrotron radiation [Jephcoat *et al.*, 1988a]. Optical measurements demonstrate the solid becomes nearly opaque by 180 GPa. This result indicates that compression causes large perturbations in the electronic structure of the molecular solid, although there is currently no evidence for pressure-induced molecular dissociation [McMahan and LeSar, 1986].

H₂O. Solid H₂O has been compressed in a diamond cell to 128 GPa at 300 K, and has been studied with synchrotron x-ray EDX techniques [Hemley *et al.*, 1987b] and with Raman spectroscopy. The x-ray diffraction data and Raman spectra are consistent with the conclusion that the structure of bcc oxygen sublattice of ice VII persists to the highest pressures of these measurements. The experimental EOS of ice is considerably softer than that predicted by statistical electron [Zharkov and Trubitsyn, 1978] and pair-potential models [Grimsditch *et al.*, 1984].

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