X-ray Diffraction Measurements for Expanded Fluid Selenium

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A great deal of effort has been made to study the semiconductor-metal (SC-M) transition of fluid Se which occurs in the supercritical region near the liquid-gas critical point (the critical data of Se [1]: Tc=1615 ℃, pc=385 bar, dc=1.85 g/cm^3). To understand the SC-M transition it is very important to investigate how the atomic arrangement is changed when fluid Se is expanded. Recently x-ray diffraction measurements using an in-house x-ray source were made for expanded fluid Se, giving information on the first neighbour coordination [2]. In this report the first experiments of the x-ray diffraction measurements using synchrotron radiation, extending from the liquid to the dense vapor region, are described.

We have performed energy-dispersive x-ray diffraction measurements for expanded fluid Se using the spectrometer installed at the BL-04B1 in SPring-8. The storage ring was operated at 8 GeV with 20 mA. White x-rays were generated through the bending magnet ranging energy up to 150 keV. The incident x-ray beam was collimated down to 0.2×0.2 mm^2 using the horizontal and vertical slits located at the upper stream. The beam was directly introduced into the high pressure vessel through the Be window and the transmitted beam was blocked using a lead stopper outside the vessel. As a result the background noise due to the secondary x-rays in the hutch was substantially reduced. It took several days to set up the high pressure vessel and to adjust the incident x-ray beam into the exact sample position in the vessel. After this procedure the good experimental condition was achieved.

The fluid Se was contained in the specially designed sapphire cell being transparent to x-rays and resistant to chemical corrosion by fluid Se [2]. The sample thickness was 200 μm in the experiments. The outgoing x-rays scattered from the sample pass through the Be windows and the intensity is detected at the fixed 2θ of 5, 10, 20 and 33 deg. using Ge solid state detector. We obtained the x-ray scattering spectra of the empty sapphire cell before loading the sample. The x-ray diffraction measurements were carried out at constant pressure of around 800 bar in the temperature ranges from 300 ℃ to 1500 ℃. The high pressure apparatus permits the measurements up to 1700℃ and 2000 bar. The spectra of the dense vapor was measured at 1650℃ and 379 bar. The density ranges from 3.9 to 0.9 g/cm^3.

After the data analysis [2], the interference function, $S(k)$, of fluid Se at each temperature and pressure are obtained. The pair distribution function, $g(r)$ are also obtained from Fourier transform of the $S(k)$. Further analysis is now in progress.

[1] S. Hosokawa, T. Kuboi and K. Tamura, Ber. Bunsenges. Phys. Chem. **101** (1997) 120.
[2] K. Tamura and S. Hosokawa, Ber. Bunsenges. Phys. Chem. **96** (1992) 681.