High Temperature

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1 . Introduction

The BL04B1 is a bending magnet beam line. This beam line has no monochromators and white x-ray will be supplied to the experimental stations. Two scientific subgroups, high pressure mineral physics group and high temperature group, are building their experimental hutches on this beam line. The high temperature experimental station will be built in the downstream hutch of this beam. Continuous white x-rays (10-200keV) will be used for the experiments.

2. Research Subjects

The aim of high temperature group is to investigate the structural properties of disordered materials under high temperature. The main topic at the present is the structural studies for expanded fluid metals and semiconductors. When liquid metals are heated and pressure is applied to prevent boiling, significant density decreases can be achieved. When temperature is elevated at low pressure, the first-order phase transition from liquid to gas occurs accompanied by a large volume change. At higher pressure) the liquid can be heated to much higher temperature and lower density. The volume change on vaporization decreases with increasing pressure, and disappears at the critical point (C.P.). At the pressure higher than this critical pressure) the volume of expanded fluid can be changed continuously in a wide range by heating (Fig.1). The structure of these expanded fluids, such as Hg and Se, will be studied in a wide density range by the x-ray diffraction measurements. In order to investigate density fluctuations and cluster formations near the critical point, we are planning to install a monochromator for the measurements of small angle x-ray scattering as well as XAFS.

The second target is to investigate the partial structures in multi-component liquids b means of XAFS and AXS at high

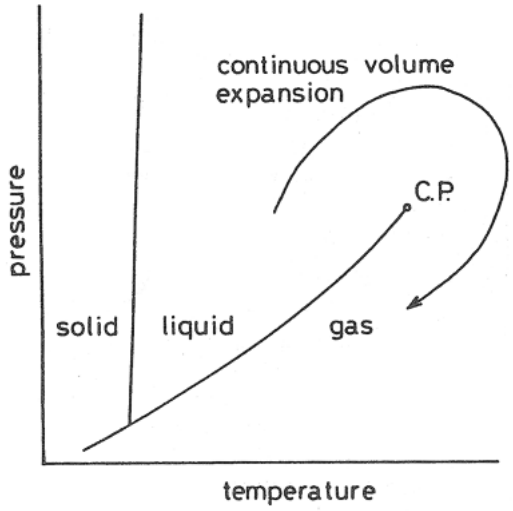

Fig.1 Schematic phase diagram on a pressure-temperature plane.

temperatures. To understand the properties of multi-component systems, the information on the partial structure factors needed, A full set of partial structure factors may provide us details of a local structure in a multi-component disordered system. This would enable us to understand some relations between local atomic structures and physical and chemical properties in disordered materials.

3. Experimental facilities

This station is composed of a high pressure and high temperature generation system and an energy dispersive x-ray diffractometer. The former system includes a compressor, a high pressure vessel, a thermocontroller, and a chiller. Since helium high pressure gas is used as pressure medium, all these facilities will be placed in small rooms surrounded by the protection wall built inside the hutch (Fig. 2). The pressurized gas is transferred from the compressor to the vessel through a high pressure tube. This high pressure vessel permits x-ray diffraction measurements at high temperature and pressure up to 1650 and 2000kg/cm2 (Fig. 3) [1] . The vessel has Be windows for the incident and scattered x-ray beam. The high pressure vessel is mounted on a horizontal goniometer which is fixed on a XZ stage (Fig. 4). The goniometer and the stage are controlled using a personal computer from

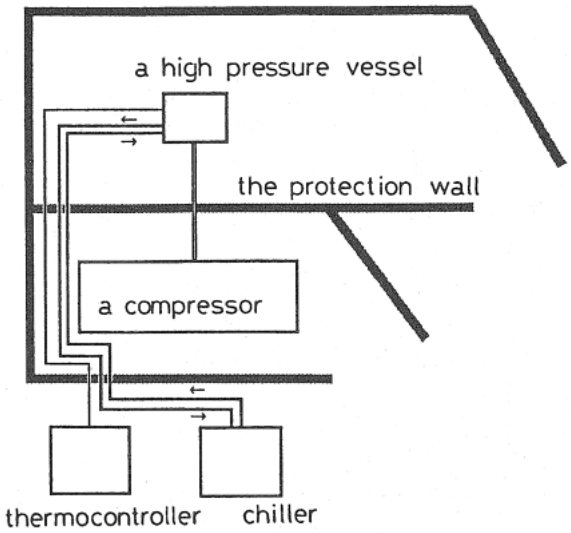

Fig.2 Schematic layout inside the hutch.

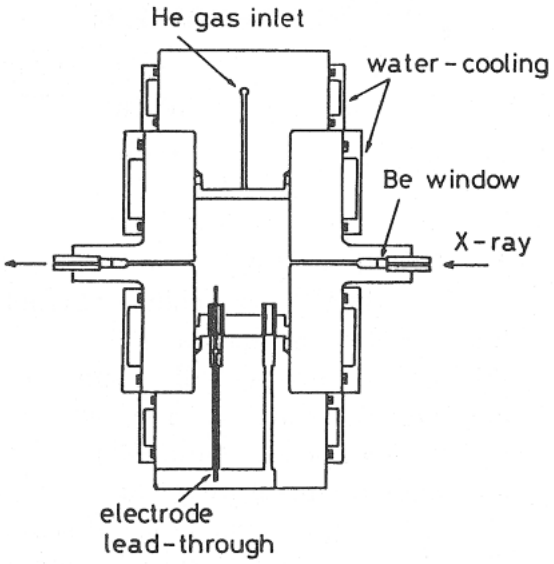

Fig.3 Side view of the high pressure vessel

outside of the hutch. While x-ray beam collimated with a incident slit is irradiated to supercritical fluid and the intensity of scattered x-rays is measured with a solid state detector. The supercritical fluid must be contained in a cell made of special material being transparent to x-rays and resistant to chemical corrosion by the fluid at high temperature. A single-crystal sapphire cell was developed for the purpose, the construction of which is illustrated in Fig. 5(a). The details around the fluid sample are also shown in Fig. 5(b) on an enlarged scale. The thickness of the closed ends of the sapphire tubes is 150m to reduce the absorption by the cell. In measuring x-ray diffraction from the fluid sample contained in the sapphire cell, the problem is that the strong Bragg diffraction from the sapphire may disturb the obtained x-ray spectrum. This interference is overcome by adjusting crystalline axis of the sapphire in the cell construction.

References

[1] K. Tamura and S. Hosokawa, J. Non-Cryst. Solids, **150** (1992) 29-34.

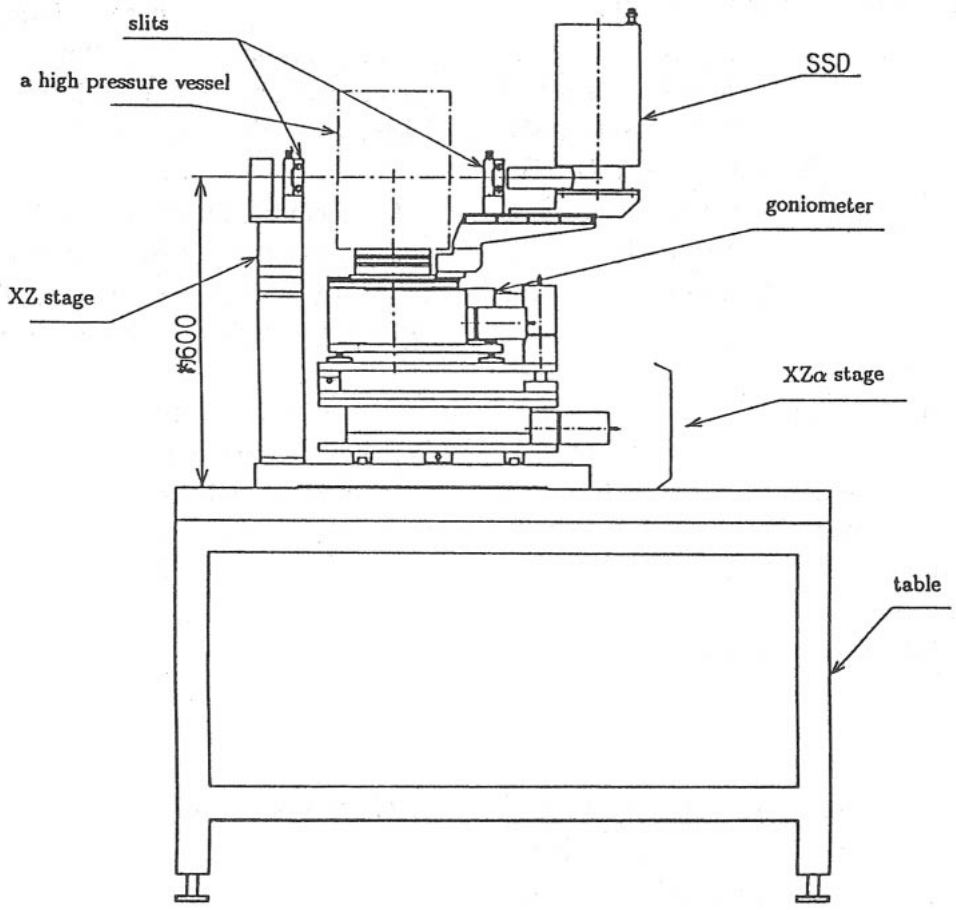

Fig.4 Schematic diagram of energy-dispersive x-ray diffractometer

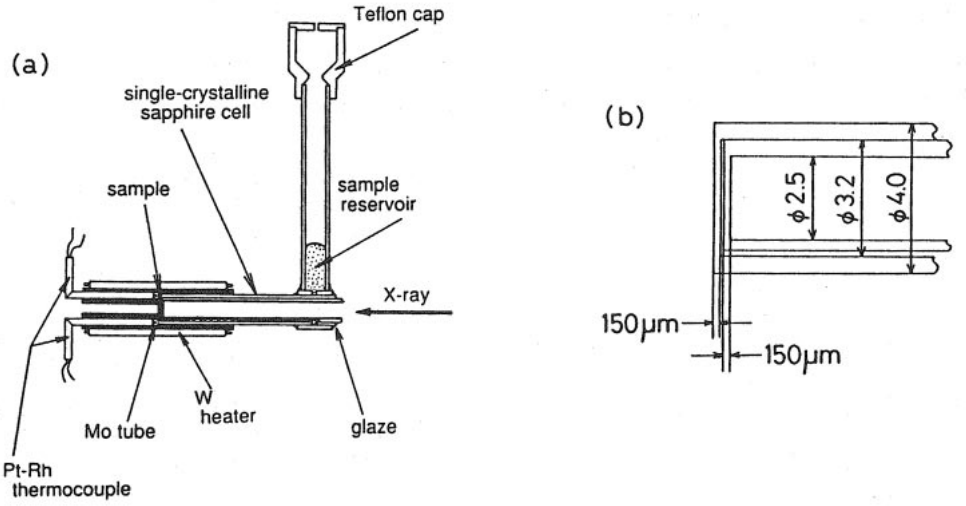

Fig.5 (a) The construction of a single-crystal sapphire cell. (b) The details around the fluid sample.