# Structural Properties of Extremely Dense Materials Subgroup

**Nozomu HAMAYA**

## 1. Introduction

Recent advance in high-pressure technique has enabled us to reach a pressure of a few hundreds gigapascals with a diamond anvil cell. At such an high pressure, materials are highly compressed and exhibit novel physical and chemical properties. The appearnace of metallic state in molecular crystals is a good example. In general, crystal structure changes successively as the pressure is increased. Therefore, it is most fundamental to determine high-pressure crystal structures in order to discuss physical and chemical properties.

One of two tandem stations at BL10XU was designed and constructed for high-pressure powder x-ray diffraction experiment and XAFS spectroscopy. This station has been open for users since October in 1997.

## 2.Light source and optical elements

An in-vacuum linear undulator (U032V) installed at BL10XU has been operating properly during user time. So far only the third harmonic was used. Powder diffraction miasurements were carried out using a monochromatic x-ray with a photon energy of 27 keV, which was obtained through a rotated-inclined double-crystal monochromator. A 35 keV x-ray, the maximum energy tunable with the Si (111) plane, was used for the density measurement of liquid metal at high pressure and high temperature.

In the early atage of operation, we were aware of three types of instability of the incident beam intensity. One was found to be caused by continuous movement of the beam position. The second instability was a discrete change in intensity by about 20%, which seemed to be strongly correlated to the change in undulator-gap installed at other beamlines. The third was relatively high frequency fluctuations of the intensity by ± 10%. Those instabilities have been analyzed by JASRI groups and will be improved soon.

Detailed characterization of the beam quality is in progress. A linear Bragg-Fresnel Lens (BFL) of Si (111) crystal was already developed and tested by the JASRI optics group. This BFL will be checked at BL10XU in March, 1998.

## 3.Diffractometer

Mechanical adjustment of the diffractometer was completed in June, 1997. In the first user time in October, optical elements on the diffractometer were aligned with the monochromatized x-ray without any serious trouble. Some diffraction patterns were measured at ambient condition in a last few days. In the second user time, first high-pressure diffraction measurment using a diamond anvil cell (DAC) was carried out after procedures for determining the wavelength and the film distance were established.

Figure 1 shows the diffraction pattern of α-quartz recorded on an imaging plate (IP) with a wavelength of 0.4512 Å and a film distance of 240.89 mm. A full width at the falf maximum of reflections was estimated to be 0.07-0.08 degrees in 2θ, which may be regarded as the instrumental resolution width. Figrue 2 illustrates the diffraction pattern of sulfur compressed to 83 GPa in a DAC. The incident x-ray with a wavelength of 0.4512 Å was collimated to 50μm square and an exposure time was 15 min. Preliminary analysis of this pattern indicates that the intensity of reflections measured at BL10XU is several times figher than that measured at BL18C at the Photon Factory.

It was confirmed that a Paris-Edinburgh press could be mounted on the diffractometer. High-temperature and high-pressure study can be carried out using the PE press at BL10XU.

A program for controlling an adjustable beam aperture and X-Y-Z stages composing the deffractometer has been developed by K. Mori of Okayama University of Science and will be in routine operation very soon.

## 4.Pressure measurement system

A ruby-fluorescence technique is used to measure pressure in a sample chamber in a DAC. A He-Cd laser and a spectrometer are placed outside the station hutch. The laser light is led to a sample in the DAC mounted on the diffractometer through optical fiber and lenses. Excited ruby fluorescence is transmitted through the same optical path and finally reflected by a half mirror to the spectrometer. This system was proven to work properly. We are planning to install another "off-line" pressure measurement system in a preparation room.

## 5.Computer network

Only a HP workstation was available for the last half year. This was used to set measurement parameters such as an exposure time, to move a beam stopper, to select an attenuator, to process the data recorded on IP and to analyze diffraction patterns. This workstation is equipped with a 2 GB hard disk, which was found to be fully occupied by the data collected in a few days. Backup of the data on DAT consumes much of time and the DAT is not an appropriate medium for personal computers.

In order to improve the present situation, we are planning to construct the following computer network in the next fiscal year. Minimal local network consists of the HP workstation and three PCs which are connected to each orher with 100 Mbps Ethernet. Linux operation system is installed on two PCs. One is used as a file server with 32 GB hard disks and the other is used to analyze IP data with a program coded by H. Fujihisa of National Institute of Materials and Chemical Research. The third PC is operated under Windows NT and equipped with CD-ROM writer as a data backup medium and other media.

## 6.Accessories

A cryostat of He-closed-circuit type in combination with a He-gas driven DAC will be available to users in coming July.

A loading device for a box-type DAC and a microscope was placed in a preparation room.

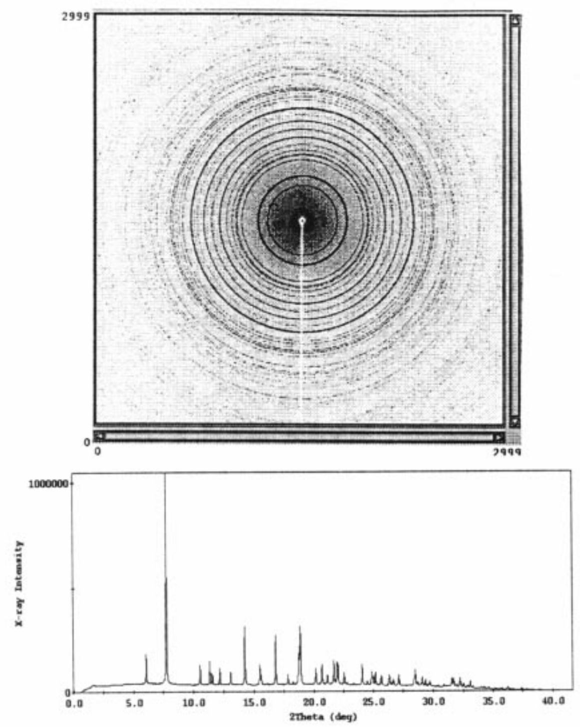


Figure 1. Diffraction pattern of $\alpha$-quartz at ambient condition.

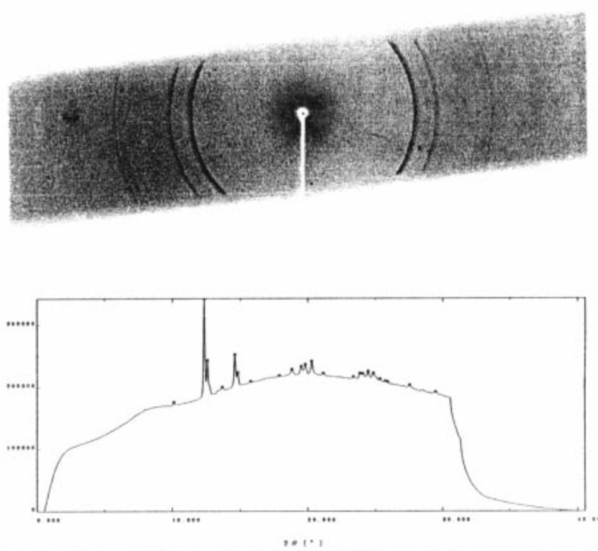

Figure 2. Diffraction pattern of sulfur at 83 GPa.