Structural Properties of Extremely Dense Materials

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Introduction

The objective of this group is to study novel structural properties of condensed matter at ultra-high pressures with powder x-ray diffraction and XAFS techniques. For this purpose, one of two stations at BL10XU is assigned for dedicated use. We have designed a diffractometer and It is being assembled by Rigaku Co. Ltd. Details of the diffractometer are described below. The beamline and station is scheduled to open for users in October, 1997.

Light source and optical elements

An in-vacuum linear undulator (U032V) is installed at the low-P section at BL10XU. Hard x-rays with a photon energy between 20 and 60 keV can be obtained by the use of either the third or the fifth harmonics of undulator radiation. This radiation is monochromatized by a rotated-inclined double-crystal monochromator and then focused by a linear Bragg-Fresnel Lens (BFL) of Si(111) crystal placed at a distance of 56m from the light source. At the BFL position, an incident beam size Is estimated to be O.3 mm in the vertical direction. As the vertical acceptance of the BFL is 0.14 mm, a photon flux received by the BFL is estimated to be 2.2×10^{12} photons/s in 0.1% bw for an x-ray of 40 keV. The beam is shaped by slits in the horizontal direction and focused in the vertical direction into about ten microns at the sample 1.2m apart. Taking account of the reflective efficiency of 40% of the BFL, we may obtain a flux of 1.8×10^{10} photons/s in 0.1% bw on the sample surface of $100\mu m^2$. The angular divergence of the focused beam is about 0.1 mrad in both the vertical and horizontal directions.

Diffractometer

An experimental station hutch has dimensions of 6 m in length along the x-ray beam, 5m in width and 3m in height. In this space, a diffractometer shown in Fig.1 is installed. The diffractometer consists of a base stage, a 2-axis goniometer for the BFL, and a flat bed arm on which optical elements, an XYZ stage for the DAC and an imaging plate detector are mounted.

The base stage has a wide flat surface of 3 x 2 m^2 on which all components are arranged. The stage can be adjusted to the incident x-ray beam by using a remotely-controlled translation mechanism.

The BFL is mounted on the 2-axis goniometer and placed at the most upstream side on the base stage. The 2.7-m-long, 0.2-m-wide flat bed arm is a sort of a long 2θ arm. This can be rotated around the axis of the BFL goniometer. This mechanism enables us to bring the sample in the focused-beam position whatever photon-energy the incident x-ray has. An adjustable beam aperture, a shutter and attenuator unit, an ion-chamber beam monitor, a pinhole collimator and an X-Y-Z stage for high pressure apparatus all are arranged on the flat bed arm. Each optical component is mounted on a positioner which can be operated remotely.

The DAC is assumed to be a primary high-pressure generation device. A cryostat and other small devices such as a Paris-Edinburgh press also can mounted on the X-Y-Z stage. This stage can be slid on the flat bed arm so that the sample is set at the focal point of the focused beam.

We chose Rigaku R-AXIS4 as a detector for powder x-ray diffraction measurement. This detector carries four 30 cm x 30 cm imaging plates (IP) on a rotating belt and has a capability of collecting a diffraction pattern on one IP while the readout of the data already recorded on another IP is proceeded simultaneously. This mechanism allow us to make rapid acquisition of diffraction patterns. The camera length can be varied in a range from 150 mm to 450 mm. For XAFS measurement, the IP detector is replaced by an ion-chamber or a pin photodiode detector.

Other types of high-pressure x-ray measurements can be made using the present diffractometer. For example, if users wish to conduct a scattering measurement using a

scintillation counter (SC), the X-Y-Z can be replaced by a goniometer on which the SC is equipped. Also possible Is to mount an x-ray CCD camera detector which we are planning to introduce in near future.

A personal computer Is used to control the 2-axis goniometer and all positioners. A diffraction image recorded on IP Is processed and analyzed by an X-window workstation connected to the network.

Accessories

A ruby-fluorescence pressure measurement system is set up. It is not necessary to dismount the DAC from the diffractometer for pressure measurement. An Ar-laser and a spectrometer are placed outside the station hutch. The laser light is led to the sample in the DAC mounted on the diffractometer through optical fiber and lenses. Excited ruby fluorescence is transmitted through the same optical path and is finally reflected by a half mirror to the spectrometer.

A cryostat will be prepared for high-pressure low-temperature experiment. A design for it is in progress.

Other facilities

Sample preparation can be made in a laboratory room near the station. A couple of microscopes and some tools are available in this room. In near future, some computers for data analysis and for network communication will be installed.

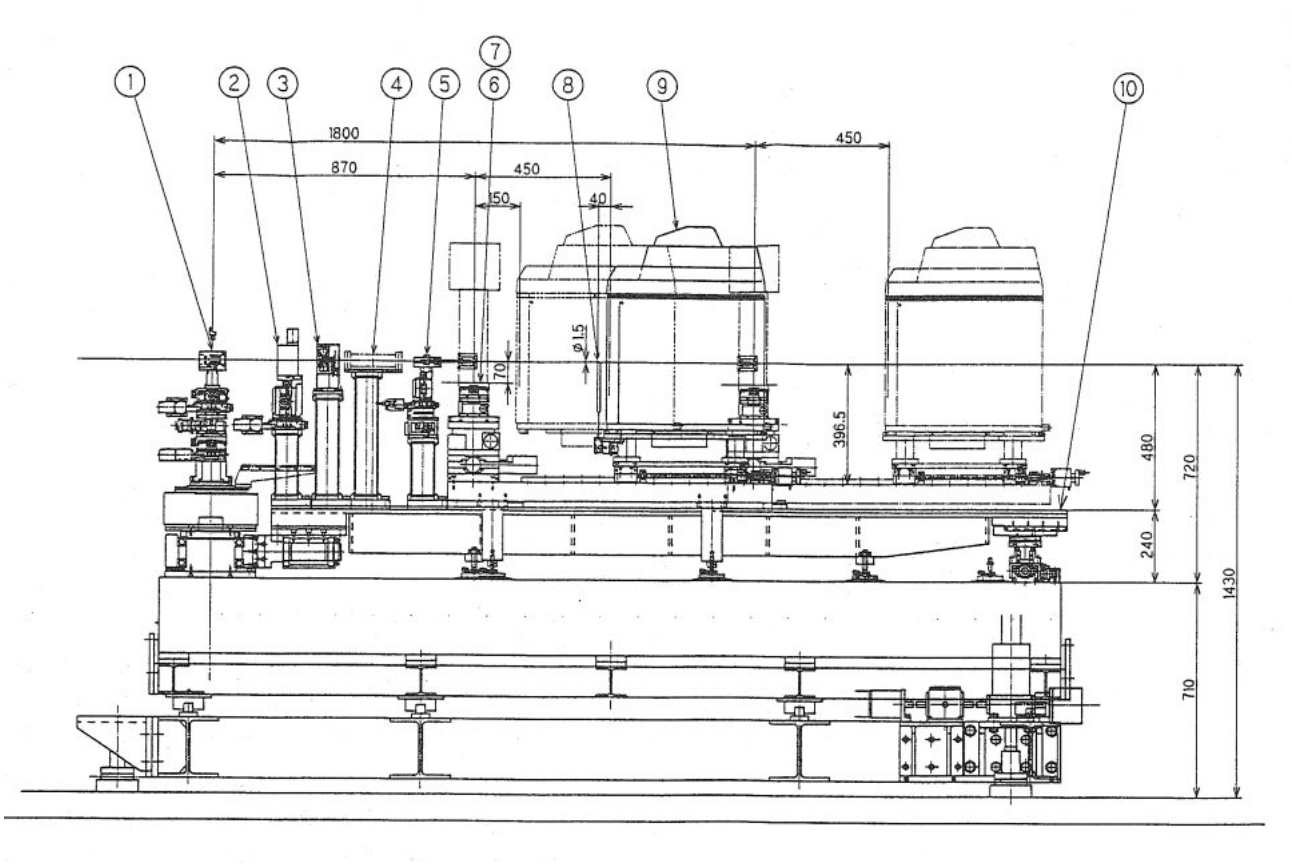

Figure 1 . Side view of a diffractometer for high-pressure x-ray measurement. (1) Bragg-Fresnel Lens, (2) variable aperture, (3) shutter and attenuator unit, (4) ion chamber, (5) pinhole collimator, (6)(7) X-Y-Z stage for high-pressure apparatus, (8) direct-beam stopper,(9) R-AXIS4, (10) flat bed arm.