BL04B1
(High Temperature and High Pressure Research)

1. Introduction

BL04B1, which is a bending magnet beamline, is mainly employed for energy dispersive X-ray diffraction measurements and X-ray radiography observations under high-temperature and high-pressure conditions using white X-rays. The X-rays emitted from the bending magnet are directly introduced into the experimental hutch and white X-rays with a wide energy range are used in experiments. It is also equipped with a compact Si(111) double-crystal spectrometer, which makes it possible to perform angle-dispersive X-ray diffraction measurements and X-ray radiographic observations using monochromatic X-rays at 30–60 keV.

The beamline has two experimental hutches in series, each has a large, high-pressure press with a maximum load of 1500-tons installed. From upstream of the X-ray beams, the SPEED-1500 Kawai–type high-pressure press (DIA-type press, optical hutch 2) and the SPEED-Mk.II Kawai–type high-pressure press (D-DIA–type press, optical hutch 3) are installed. SPEED-Mk.II can conduct high-pressure deformation experiments using D-RAM (differential ram moving independently of the main ram) in addition to high-pressure, high-temperature experiments of 30 GPa and 2000K or higher using sintered diamond anvils.

In FY2018, the AE measurement system was developed, a time division ultrasonic velocity measurement system was introduced, sequential automatic stage control software was developed, etc.

2. Introduction of the AE measurement system

In BL04B1, AE (Acoustic Emission) measurement experiments were carried out by combining a D-DIA–type high-pressure deformation press and an ultrasonic measurement system. Several oscilloscopes are linked to obtain six-channel acoustic wave signals and a preamplifier for each AE signal is connected. Thus, ultrasonic velocity measurements and AE measurements are switched manually. Since this system uses several oscilloscopes, the synchronization between the channels is not sufficient, which deteriorates the positioning accuracy in AE measurements. The manual switching interrupts the measurements.

To improve the synchronization, an 8-channel oscilloscope (MSO58; Tektronix) was introduced, enabling the use of 6-8–type high-pressure cell as well as the conventional 6-6–type high-pressure cell for AE experiments (This system was introduced by Kyushu University).

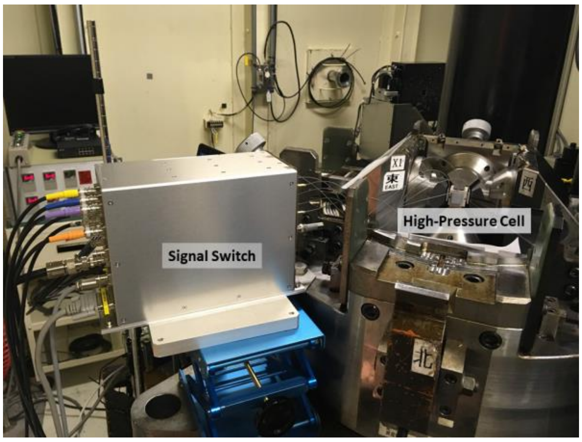

Fig. 1. AE measurement system.

A switching device was developed for high-frequency circuits in cooperation with Thamway (Fig. 1). In this system, ultrasonic velocity and AE measurements can be switched instantaneously by a TTL signal. The generation status of AE can be monitored, and the elastic modulus of the sample can be measured simultaneously. Moreover, a remote control (20 dB / 40 dB) can change the amplification factor of the preamplifier, and the experiment can be carried out without entering the experiment hutch.

3. Development of time-resolved ultrasonic velocity measurement system.

Ultrasonic echo, X-ray absorption image, and X-ray diffraction (XRD) data are required for ultrasonic velocity measurements. To obtain these data, it is necessary not only to change the width of the incident X-ray slit between X-ray radiography and XRD measurements, but also to operate each system using specialized software. Currently, the time-resolved measurements are operated manually. Because the operation speed of the slit is slow, it is difficult to perform a single time-resolved measurement in several minutes or less.

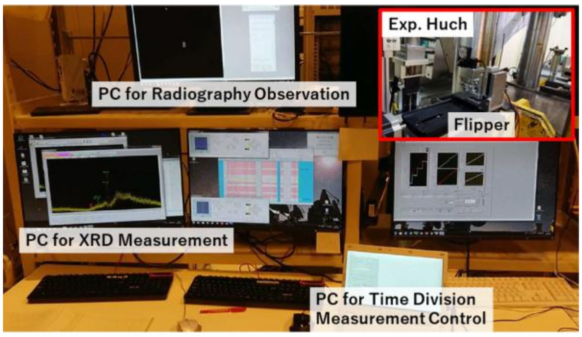

Fig. 2. Time-resolved ultrasonic measurement system.

In FY2018, a new slit system with a fixed width was introduced for XRD (Fig. 2). This slit can be alternately inserted and removed by an electric flipper, enabling a fast switching (within a few seconds) between radiographic observations and XRD. In addition, integrated control software was developed for sequential operations of each software for radiography and XRD. “HiPic” manufactured by Hamamatsu Photonics is used to acquire X-ray images. Remote acquisition of X-ray images was realized using RemoteEx Client software. The remote-control function was added on the software for the XRD measurements. The software was developed using LabVIEW, and the multifunction I/O (N9403) device manufactured by National Instrument was used to generate the TTL signal required for flipper control.

Using this system, a nonlinear change in the elastic wave velocity of an amorphous sample was successfully obtained at continuously increasing temperatures. In the future, this system will be incorporated into all user experiments. This system should enable time-resolved measurements of the elastic wave velocity, which have yet to be performed.

4. Automatic stage sequential control software

In high-temperature and high-pressure experiments, XRD measurements and radiographic observations should be performed as quickly as possible since the sample environment is unstable. During such experiments, however, the user must complete many operations such as controlling the high-pressure press, heating the instrument, and adjusting the sample position. Integrated control software was developed for a new measurement method such as mapping measurements. The newly developed control software can move the stages to pre-set positions automatically and change the width of the incident slit (Fig. 3).

Fig. 3. Automatic stage sequential control software.

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