JAERI BL14B1

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1. Introduction

BL14B1 is a JAERI Material Science I beamline for experiments on various kinds of structural studies, particularly in the field of high-pressure/high-temperature science and surface/interface structural studies. The construction of the beamline was completed in October 1997. It has been used for scientific studies from March 1998.

This report summarizes the optics and experimental stations of BL14B1.

2. Outline of Beamline

The light source of BL14B1 is a bending magnet. The basic concept of the optics is adopted in the SPring-8 standard bending magnet beamlines, for example, BL01B1 and BL02B1. Therefore, the main optics contains two bent plane mirrors and a double crystal monochromator with a sagittal focusing system. One mirror is located upstream and the other downstream from the monochromator. The specifications of the optical elements are listed in Table 1.

This optics can be removed from the direct beam to carry out experiments with the white X-ray beam. We have two experimental hutches at BL14B1. Experiments using the white X-ray beam are permitted in the upstream experimental hutch (white X-ray experimental hutch) having higher radiation-shielding ability than the downstream one (monochromatic X-ray experimental hutch). Accordingly, there are three cases of experiments at BL14B1 as follows.

Case1: White X-ray experiments in the white X-ray experimental hutch.
Case2: Monochromatic X-ray experiments in the white X-ray experimental hutch.
Case3: Monochromatic X-ray experiments in the monochromatic X-ray experimental hutch.

Beamline arrangements are different in each case as shown in Fig.1.

3. Scientific Program

3.1. Structural Studies under High Pressure

In the upper stream experimental hutch, where there can be either white or monochromatic x-rays, research on the structures of materials under high pressure is being conducted. Under high-pressure and high-temperature conditions, many materials may have different structures than at ambient conditions, so physical properties such as electric resistivity and magnetic susceptibility can vary greatly. Structural information of such materials by in situ observation is fundamental to understanding these properties. To obtain this information, a new multi-anvil type high-pressure apparatus system for in situ x-ray study has been installed on this beamline.

Figure 2 is a schematic drawing of the

Table 1. Specifications of optical components

Distance from source	Optical element	Function
35.1 m	First mirror (Si body, rhodium coated)	Higher harmonics elimination, Vertical collimating
36.8 m	Double crystal monochromator Si(111),(311),(511): interchangeable in vacuum	Monochromatization, Horizontal focusing
40.2 m	Second mirror (quartz body, rhodium coated)	Vertical focusing

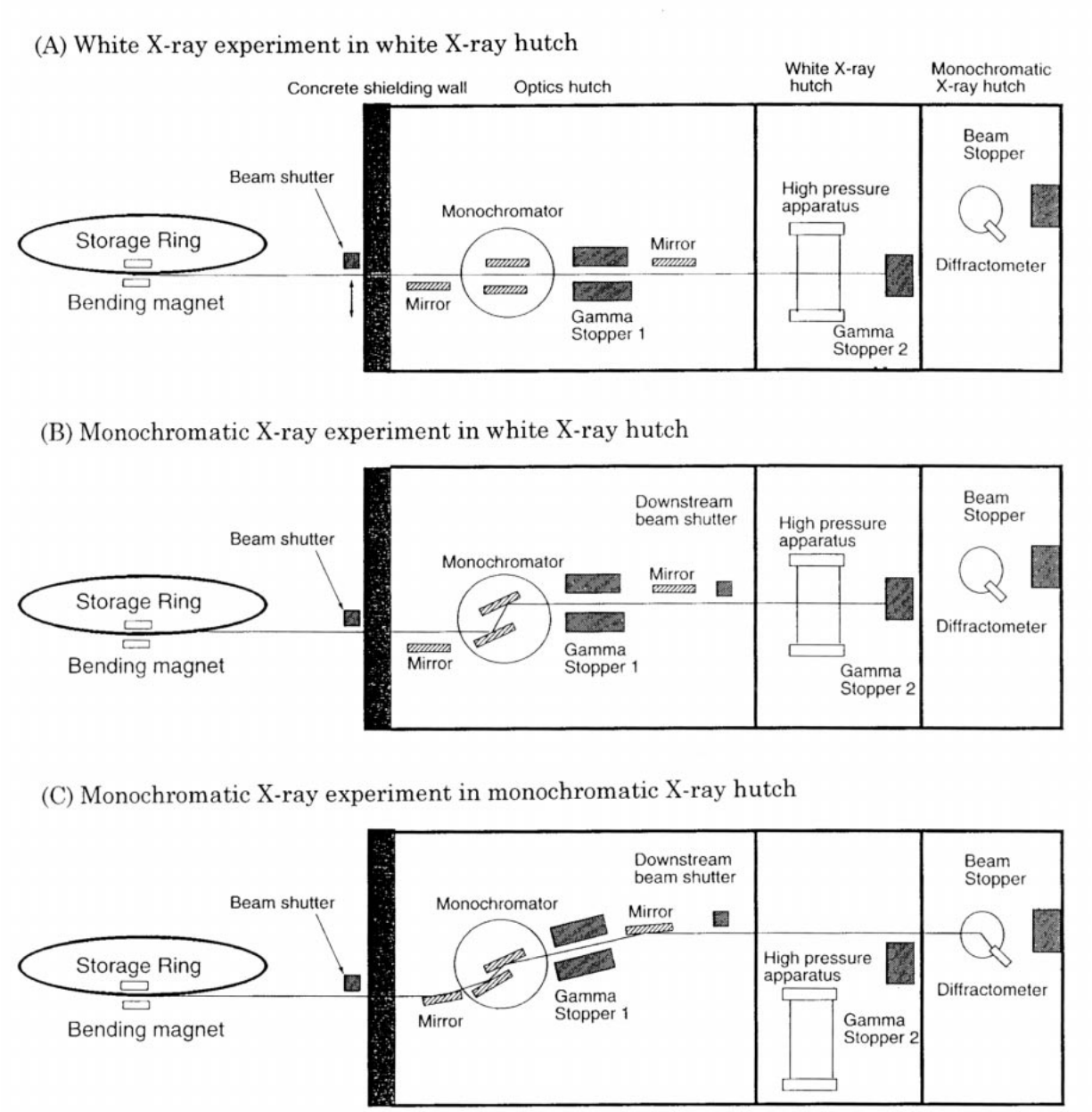

Fig. 1 Schematic drawing of beamline arrangements of BL14B1 depending on experimental requirements

system installed in the hutch. The main part of its high-pressure generation device is a DIA type multi-anvil apparatus placed on a 180ton uniaxial hydraulic press. Six cubic anvils are mounted on the guide block and a cubic-shaped pressure medium is compressed by these anvils isotropically. Using this apparatus, high-pressures and high-temperatures up to 15GPa and 1500K can be generated. This system is characterized by its optics. In order to carry out an angle dispersive powder diffraction experiment as well as an energy dispersive diffraction study, it has two-dimensional detectors instead of a Ge-SSD. Two concentric vertical goniometers are placed near the high-pressure apparatus. An SSD for the energy dispersive diffraction is mounted on the outer goniometer. On the inner goniometer, a multi-channel collimator (solar slits) is mounted. This multi-channel collimator is used to reduce the background noise signals from the sample's surrounding materials such as the pressure medium for the angle

dispersive experiments using an Imaging-Plate or a CCD detector. The center slit of the multi-channel collimator is also used as a single collimator in the energy dispersive experimental mode. Also, an x-ray absorption experiment under high pressure can be carried out with a monochromatized beam.

3.2. Surface/Interface Structural Studies

The diffraction experiment with an X-ray beam from the third generation synchrotron radiation source requires a multi-axis diffractometer with accuracy matching to the small beam size and angular divergence. The sample located on the center of the diffractometer should be kept in position when it is oriented by the diffractometer. In practice, however, assembly errors cause extra translations of the sample, which are included within a sphere when the axes of the diffractometer are moved over the whole of their ranges. This sphere is called the sphere of confusion (SOC) and defines the spatial accuracy of the diffractometer. A diffractometer with a SOC less than the beam size is needed for the diffraction experiment at Spring-8.

A new κ-type multi-axis diffractometer has been designed to satisfy the above condition and installed in the monochromatic X-ray experimental hutch of BL14B1. This diffractometer is used for structural studies on liquid/solid interfaces, solid/solid interfaces, glass, single crystals and so on. Figure 3 shows a schematic of the diffractometer. It has four axes for orienting the sample (ϕ, κ, ω, ψ), two axes for positioning the detector (θ, η) and two axes for setting the analyzer crystal (ωa, θa). This design differs from that of the conventional four-circle diffractometer in having a κ-axis that facilitates the out-of-plane positioning of the detector and a κ-axis that makes an angle of 50 degrees with respect to the ω-axis. These peculiarities effectively reduce the SOC while realizing the degrees of freedom needed for the diffraction experiments.

For surface and interface diffraction

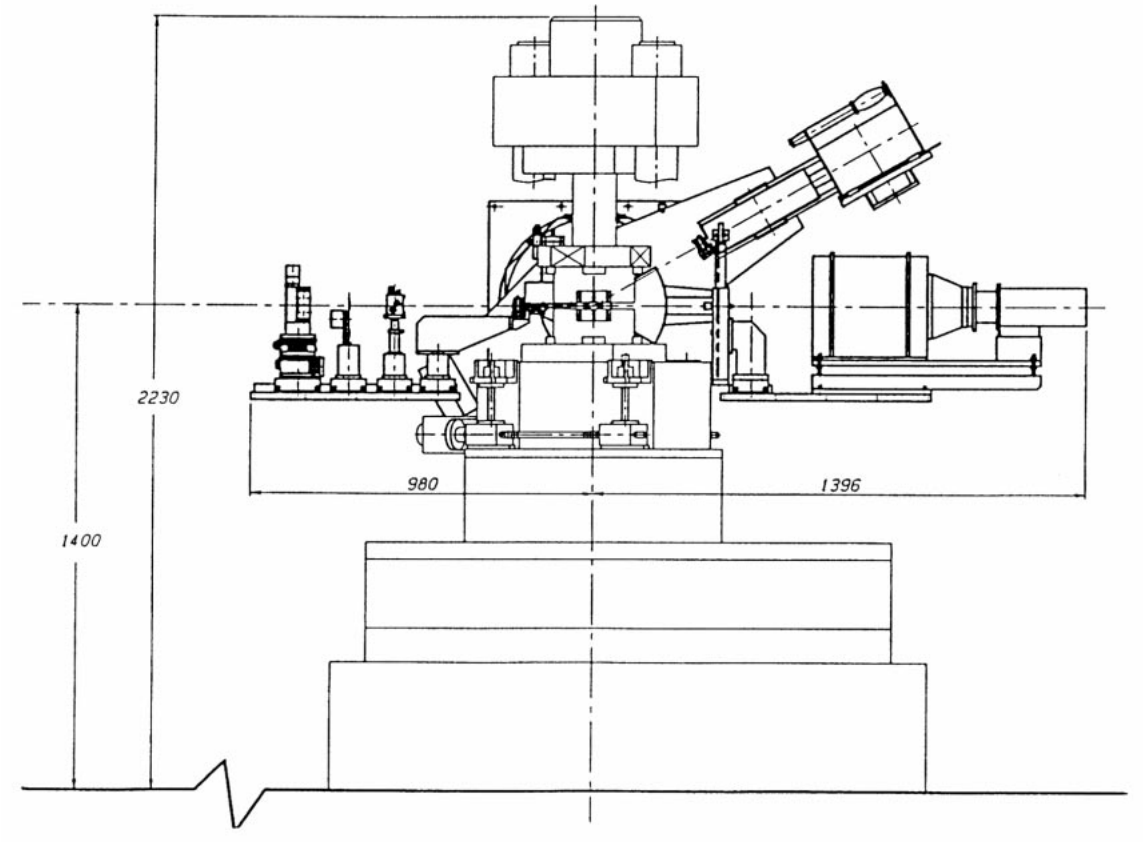

Fig. 2 Schematic drawing of multi-anvil type pressure apparatus system

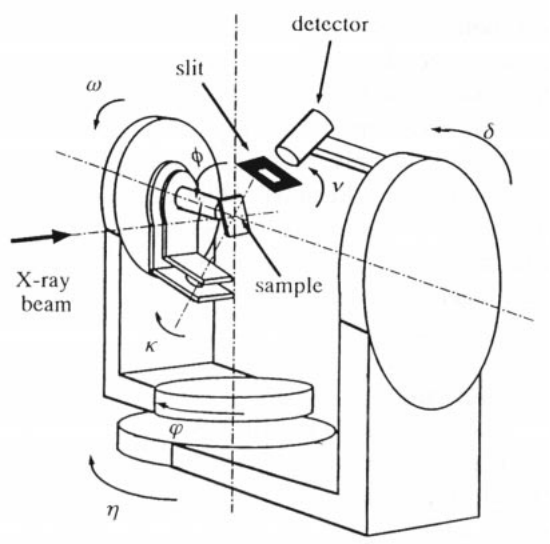

Fig. 3 Schematic diagram of κ -type multi-axis diffractometer

studies, this diffractometer is equipped with another axis (ν) for azimuthal rotation of the detector slit [1]. This novel axis allows accurate measurement of the surface structure factor even in the case of large perpendicular momentum transfer.

A preliminary experiment on a liquid/solid interface has been performed. The cyclic voltammetry study suggests that the Au(111) electrode surface in 0.1M sulfuric acid exhibits a transition between 1×1 and $23\times\sqrt{3}$ phases, depending on the potential E. The $p\times\sqrt{3}$ reconstruction gives an additional reflection at $(1+\delta/\sqrt{3},\ \delta/\sqrt{3},$ l) around (1 0 l), where (h k l) is represented by a hexagonal coordinate system, a*=1/3a(2,2,-4), b*=1/3a(-2,4,-2), c*=1/3a(1,1,1) and a=4.078 Å. For p=23, δ =0.038 is expected. Figure 4(a) and (b) shows the measured X-ray scattering profile for applied potentials E=0.15V and E=0.6V vs. Ag/AgCl/KCl, respectively. The wavelength used is 0.819Å. The perpendicular component of the momentum transfer is l=0.6. Besides the 1×1 peak at δ =0, an additional peak at δ =0.038 was observed when E=0.15V, whereas only a 1×1 peak was observed when E=0.6V. This result shows that the $23\times\sqrt{3}$ structure is formed in sulfuric acid and that the phase transition to

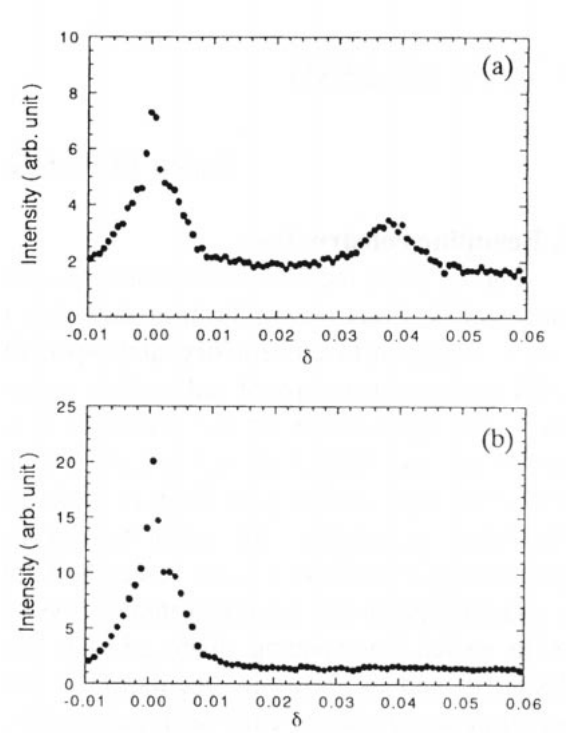

Fig. 4 X-ray scattering profiles of Au(111) electrode surface in 0.1M sulfuric acid around (1 0 0.6) at potentials of (a) 0.15V vs. Ag/AgCl/KCl and (b) 0.60V vs. Ag/AgCl/KCl

the 1×1 phase occurs at a potential between 0.15V and 0.60V.

References

[1] M. Takahashi and J. Mizuki, J. Synchrotron Rad., to be published.

Commissioning Members

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