BL46XU
Engineering Science Research III

1.Introduction

BL46XU is an industrial applications beamline with an undulator light source dedicated to promoting the utilization of synchrotron radiation by industry. A multi-axis X-ray diffractometer is installed in the first experimental hutch (EH1). In addition, X-ray imaging and micro-focus X-ray diffraction are available in the open space of EH1. The second experimental hutch (EH2) has two hard X-ray photoelectron spectroscopy (HAXPES) systems. In FY 2019, an X-ray mirror system, which provides a grazing incident X-ray beam on thin films grown on surfaces of liquid, was installed for the multi-axis X-ray diffractometry. For HAXPES, a neutralizer for charge compensation of insulator samples irradiating with high-energy electrons (up to 20 keV) was installed.

2. Optics and performance

The light source is a standard in-vacuum undulator at SPring-8 and the optics adopt a liquid nitrogen–cooled Si (111) double-crystal monochromator. The tunable energy range is 4.5–37.5 keV. To eliminate harmonics, two Rh-coated mirrors (70-cm length, horizontal reflection direction) are placed in the most downstream part of the optics hutch. The mirrors can be bent for horizontal light focus. A Si (111) channel-cut monochromator is placed between the monochromator and the mirrors to achieve incident X-rays with fine energy resolution. Figure 1 shows the beamline layout of BL46XU.

3. New equipment and developments

3.1 Grazing incidence X-ray diffraction for the air/liquid interface

The multi-axis diffractometer is widely used by industrial researchers. In FY2019, the system to measure grazing incidence X-ray diffraction (GIXD) on thin films on surfaces of a liquid was installed in this diffractometer. This system was developed at BL19B2 in FY2018 to meet the demand for crystal structural analysis of Langmuir films grown on liquid surfaces in the field of air/liquid interface chemistry [1]. In experiments using this system at BL19B2, each GIXD profile measurement required more than 1 hour because X-ray diffraction from a Langmuir film was significantly weak. Therefore, this measurement

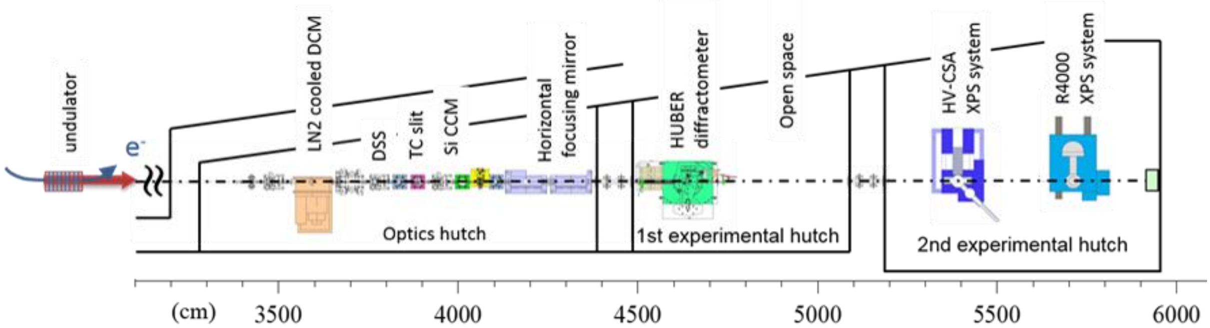

Fig. 1. Beamline layout of BL46XU.

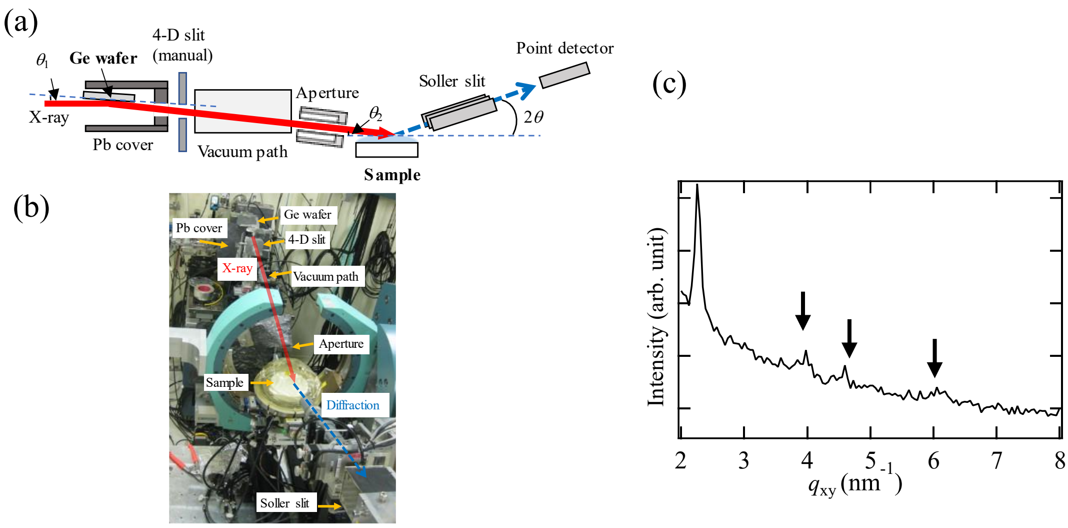

Fig. 2. GIXD measurement setup for air/liquid interface. (a) Schematic illustration, (b) photograph, and (c) GIXD profile from a Langmuir film of 1,3,5-tris-(4-carboxyphenyl)-benzene grown on surface of water.

system was installed in the undulator beamline, BL46XU, to improve the detected signal of GIXD from Langmuir films.

Figures 2(a) and (b) show a schematic illustration and a photograph of the GIXD measurement setup, respectively. Monochromatic X-rays are guided downward by tilting a Ge wafer mirror by angle (θ_1). The incidence angle to the sample (θ_2) can be controlled by θ_1. A four-dimensional slit system and a guard aperture are adopted downstream of the Ge wafer mirror to remove the X-ray scattering from this mirror. This apparatus can be remotely controlled by combining with auto x and z stages. Figure 2(c) shows the GIXD profile from a Langmuir film of 1,3,5-tris-(4-carboxyphenyl)-benzene grown on water. The time required to measure a GIXD profile from Langmuir films is successfully reduced to about 15 minutes.

3.2 Neutralizer with an electron energy of up to 20 keV for charge compensation in HAXPES

The HAXPES measurement system equipped with an electron energy analyzer is available to industrial researchers. It is a powerful tool to directly explore the electronic structure deep inside a material. For example, the system is applicable to electrode/dielectric interfaces buried in gate stack structures, which are not accessible by conventional XPS [2]. However, HAXPES measurements for insulator samples have suffered from a charging effect due to the small shift and small change in the shape in the photoelectron spectra. In soft X-ray photoelectron spectroscopy (SX-PES), the charging effect is generally compensated by supplying a flood of low-energy electrons with an electron gun. Because the probing depth of HAPXES is much larger than SX-PES, a low-energy electron flood gun is insufficient to compensate for the charge at the deeper region from surface of samples. Consequently, a new neutralizer electron gun with

EGF-3104 (Kimball Physics) was installed in FY2019. This new gun can irradiate samples with high-energy electrons (available energy is up to 20 keV) .

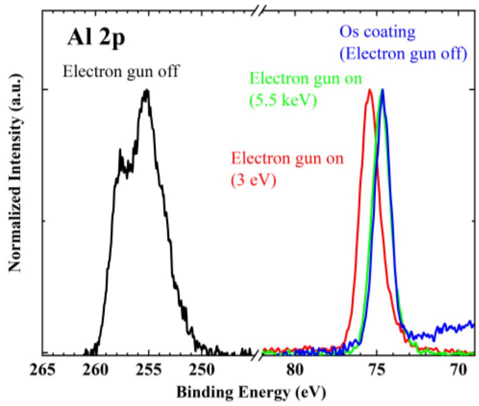

Fig. 3. Al 2*p* spectra of an Al_2O_3 single crystal when the neutralizer is off (black), the neutralizer is on with an electron energy of 3 eV (red), the neutralizer is on with an electron energy of 5.5 keV (green), and the spectrum of an Os-coated sample when the neutralizer is off (blue).

The effectiveness of this neutralizer was tested by measuring a polished Al_2O_3 single crystal. Figure 3 shows the Al 2p spectra of an Al_2O_3 single crystal with and without the neutralizer (electron energy of 3 eV or 5.5 keV). As a reference, the spectrum for Al_2O_3 sample treated by an osmium (Os) coating, which is an effective method to achieve charge compensation [3]. When the neutralizer is off, the Al 2*p* peak shifted to a higher binding energy due to charging the sample positively. When the neutralizer irradiated electrons with an energy of 5.5 keV, the peak position of Al 2*p* was almost the same as that of the Os-coated sample. On the other hand, when irradiating with an electron energy of 3 eV, the peak position shifted to a higher energy than that of the Os-coated sample. It is inferred that an electron energy of 3 eV is insufficient to discharge the Al_2O_3. These results suggest that an electron beam of several keV is suitable for charge compensation in HAXPES measurements [4].

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