BL09XU
(Nuclear Resonant Scattering)

1. Introduction

BL09XU, which is a nuclear resonant scattering beamline, is an X-ray beamline with a 32-mm-period standard linear undulator and a liquid-nitrogen cooled monochromator [1]. Intense X-rays between 5 keV and 80 keV are obtained. X-rays below 38 keV are first monochromatized by double Si 111 reflections, while X-rays above 38 keV are monochromatized by Si double 333 reflections. Numerous fields use this beamline for nuclear resonant scattering (NRS) and hard X-ray photoelectron spectroscopy (HAXPES).

2. Nuclear resonant scattering

NRS is a resonant scattering using the transition between the ground and excited states in nuclei. Its energy width is typically μeV to neV, which is much narrower than the atomic electron level of meV to eV. Techniques currently conducted in the beamline using NRS are follows:

(1) Synchrotron Mössbauer spectroscopy

This technique investigates local electronic states through hyperfine interactions. It is useful to study samples under high pressure and thin films due to the much smaller beam size than that obtained using a radioactive source. It is also used for isotopes that do not have an adequate radioactive source.

(2) Nuclear inelastic scattering (NIS)

This technique visualizes atomic vibrations as phonon excitations. It provides complimentary information to inelastic neutron scattering and inelastic X-ray scattering because the partial phonon density of states that specifies the atom can be observed by NIS.

(3) Quasi-elastic scattering (QES)

QES using time-domain NRS measures the dynamical structure factor in the (q, ω) space, which corresponds to the dynamics in soft matter.

(4) Nuclear excitation for nuclear physics

In FY2018, we developed a spectrometer for the QES using time-domain NRS and one-dimensional (1D) focusing lenses for total reflection experiments. We tested the cooled analyzer for energy-domain synchrotron Mössbauer spectroscopy.

2-1. Spectrometer for QES using time-domain NRS

The QES using time-domain NRS was intensively developed [2]. A new spectrometer with improved specifications was constructed in collaboration with Dr. Saito of Kyoto University. The spectrometer is composed of a high-resolution monochromator (HRM), cryostat, and APD detectors. The HRM is designed to have higher resonant flux by replacing the resolution of 3.5 meV with that of 6 meV. This is because the resonant flux is more important than the resolution considering the current prompt and delayed counting rates. Si 440 and 10 6 4 reflections are adopted in the nested-type instead of the original 511 and 975 reflections. Figure 1 shows the fabricated HRM with high-precision rotation mechanics using a piezo actuator. A delayed intensity is sometimes more important than the q-resolution. Therefore, the outer diameter of the cryostat is designed to be as short as possible to set the APD detector as close as possible to a sample in the cryostat. To cover the sequential q-range, the APD detectors are designed to have a shorter dead space to set two APD detectors nearby.

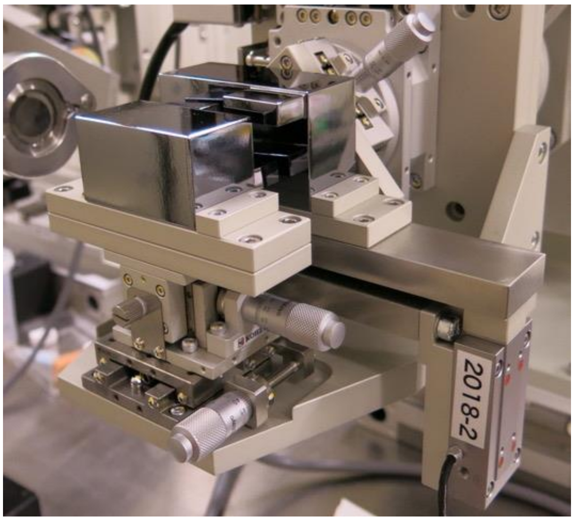

Fig. 1. HRM developed for the QES using time-domain NRS.

2-2. One-dimensional focusing lenses for Fe-57 and Sm-149

As reported in the SPring-8·SACLA annual report FY2017, there are two-dimensional (2D) focusing lenses for Fe-57, Sm-149, and Th-229. Time-domain synchrotron Mössbauer spectroscopy using the total reflections to investigate thin films is a popular technique at BL09XU. To reduce the intensity loss due to 2D focusing, 1D focusing lenses are prepared for Fe-57 (14.4 keV) and Sm-149 (22.5 keV). Those for Sm-149 are also used for Eu-151 (21.5 keV) and Sn-119 (23.9 keV) with different focusing points. These lenses are mounted in the same holder as the 2D focusing lenses. The measured throughput for Fe-57 was 81% and the focused beam size was 200 μm in the vertical direction. This value is larger than the 110 μm obtained by the 2D focusing lenses for Fe-57.

2-3. Analyzer for energy domain Mössbauer spectroscopy with a closed-cycle cryostat

Energy domain Mössbauer spectroscopy does not require high-current single bunches. In FY2017, an analyzer with a liquid He flow-type cryostat was prepared for isotopes with higher energies. However, due to a recent supply problem for liquid He in Japan, an analyzer that does not use liquid He is required for the smooth user experiments.

Because an energy scan is realized by the Doppler shift of the reference sample connected to the transducer in the analyzer, it is quite sensitive to vibrations. A vibration-damped analyzer with a closed-cycle cryostat was tested under collaboration with Prof. Kobayashi of the University of Hyogo (Fig. 2). This system was used in Sm-149 Mössbauer spectroscopy. Sm_2O_3 and $^{149}SmB_6$ were used as a sample on the beam and a reference sample in the analyzer, respectively. The energy spectra were measured at 10 K and 30 K with pumping and between 10 K and 40 K without pumping. The obtained line widths of these spectra are the same within statistical error. This result shows that this analyzer can be used for Mössbauer spectroscopy of Ni-61, Yb-174, etc.

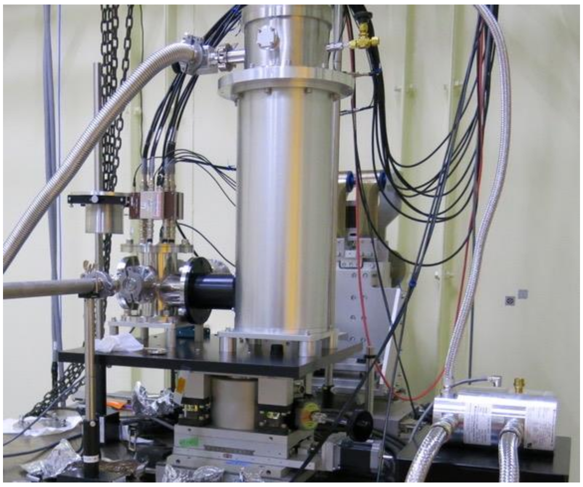

Fig. 2. Analyzer for energy domain Mössbauer spectroscopy with a closed-cycle cryostat.

3. Hard X-ray photoelectron spectroscopy (HAXPES)

The HAXPES station at BL09XU was opened for public use in FY2014. The advantages over BL47XU are its high flux and energy tunability [3]. The high-flux micro-focus beam with the size of about 5 (vertical) µm × 13 (horizontal) µm is achieved by the long length of about 1 m of the K-B focusing mirror. Its intensity is 30 times higher in the photoelectron detection efficiency than that at BL47XU. The high-flux beam allows spectra with high energy resolution ΔE of about 100 meV to be acquired and a diamond phase retarder to be used. Hence, magnetic circular dichroism of HAXPES spectra can be measured by changing the beam polarization, which means that, for example, spintronic materials can be investigated.

An energy tunable system was developed to realize resonant HAXPES (r-HAXPES) measurements in collaboration with the Partner User (PU) members led by Dr. Kojiro Mimura of the Osaka Prefecture University in FY2017. This project was based on the PU program of SPring-8 and titled, “Construction of composite measurement technology of resonant hard X-ray photoemission and X-ray absorption spectroscopies, for elucidating quantum critical phenomena of strongly correlated electron system”. Selective utilization of Si 331 and Si 333 channel-cut monochromators (CCM) can measure r-HAXPES spectra with ΔE<300 meV in the incident photon energy range of 5–10 eV.

Recently, the sample positioning time had to be shortened, especially for studies on strongly correlated electron systems. In addition, there were requests from r-HAXPES users to expand the hν range and enhance the beam flux. In FY2018, we developed an observation system of the sample coaxial to the incident X-ray beam using a reflecting mirror and introduced a new high-resolution CCM.

3-1. Coaxial observation system using a reflecting mirror

For strongly correlated electron systems, users often try to cleave or fracture samples *in situ* to measure clean surfaces. However, this often results in imperfect cleavage with poorly cleaved areas. In such cases, well cleaved areas must be identified from the imperfectly cleaved surfaces. Thus, sample positioning is very time-consuming work. To overcome this problem, we developed a sample observation system coaxial to the incident X-ray beam using a reflecting mirror, which is applicable to ultrahigh-vacuum (UHV) conditions (Fig. 3(a)). The system is mostly the same as that introduced at BL47XU in FY2017 [4]. This system greatly simplifies identifying the target position. In the positioning procedure, first, the target position is identified visually using the system. Next, the sample position is searched to maximize the photoelectron intensity by scanning the sample along the beam direction. This system drastically shortens the searching time required and increases the effective beam time.

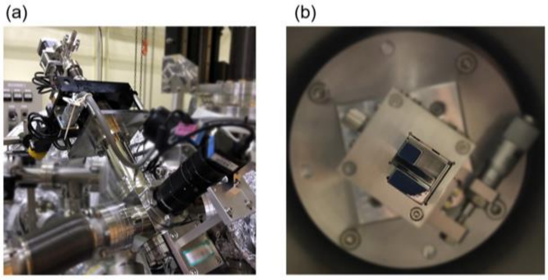

Fig. 3. Picture of (a) coaxial observation system and (b) Si 311 CCM.

3-2. New CCM to expand energy-region and high-flux measurements in r-HAXPES

We introduced a Si 311 CCM and adjusted it in the energy range of 4.91–12 keV with support by the PU program. The CCM has a narrow gap width of 3 mm to reduce the variation of the beam height during photon energy scanning (Fig. 3 (b)). The Si 311 CCM increases the beam flux more than 10 times compared with the Si 333 one. Moreover, ΔE is smaller than 300 meV in the energy range of <7 keV. This Si 311 CCM is effective for powder and low-concentration samples, especially in the photocatalyst field where users are increasing.

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