BL35XU High-Resolution Inelastic X-Ray Scattering

1. Overview

BL35XU investigates dynamics in materials using inelastic X-ray scattering. In FY2020, the beam time was allocated to non-resonant high-resolution inelastic X-ray scattering (IXS) measurements, where a Si backscattering monochromator is utilized in accordance with a high-order reflection. IXS energy resolutions are around 1.5 meV (hv =21.747 keV, using the Si(11 11 11) reflection) and 3 meV (hv = 17.794 keV, using the Si(9 9 9) reflection).

Versatile measurements are conducted for both crystalline and disordered materials. The cylindrical mirror provides a beam size of less than 80 μ m × 80 μ m (FWHM), which greatly benefits measurements for small samples with a size of less than 1 mm³. Furthermore, owing to the additional focusing system using the KB mirror, a smaller beam size of less than 20 μ m \times 20 μ m (FWHM) is available, which allows measurements under extreme conditions such as high temperature with high pressure (in a diamond anvil cell). The targeted scientific fields are also wide, since this method directly observes the atomic dynamics and phonons in materials, including atomic dynamics in liquids and glasses, electron-phonon correlation in correlated materials, elastic constants with high pressure, which are often correlated to seismology, and phonon lifetimes of thermoelectric materials.

The beam time at BL35XU was considerably limited in FY2020. The beamline was not operated in May and most of April owing to the COVID-19 pandemic. Furthermore, for the preparation of the transfer of the nuclear resonant scattering (NRS) station, originally at BL09XU, some facilities at BL35XU were upgraded and/or modified in June, January, February, and March. In these periods, the beamline was not utilized for IXS measurements. As a result, the total available beam time for users is about half of the typical amount. Moreover, as with other beamlines, some overseas users canceled the beam time or performed their experiments remotely because of COVID-19-related travel restrictions.

2. Soller slit

Thin-film measurements require a small incident angle (α), leading to a long footprint of X-ray at the sample surface. The footprint length L is given by L $= b_{\nu}/\sin \alpha$, where b_{ν} is the beam size. For example, in the case of $b_v = 0.05$ mm and $\alpha = 0.1$ deg, L becomes around 30 mm, which is too long for the original optical design. As seen in Figs. 1(c)-1(f) (black lines), bulk Si measurements with a small incident angle (α ~0.1 deg ~2 mrad) yielded some artificial features from the neighboring analyzers. Figure 1(f) shows the spectrum at Analyzer 5 of the 4×3 array (Fig. 1(a)), which includes some contribution from the neighboring Analyzer 6 (Fig. 1(e), the energy position is indicated by the green bar). Similarly, the spectrum at Analyzer 8 (Fig. 1(c)) is contaminated with the spectral contribution from Analyzer 7 (Fig. 1(d), the energy position of Analyzer 7 is indicated by the orange bar). Note that these momentum transfers mainly enhance the longitudinal acoustic phonon modes, and there should be no other phonons nearby.

To eliminate these artificial contributions by



Fig. 1. Effects of Soller slit installation. (a) Analyzer array of BL35XU, (b) side view of Soller slit, and (c)–(f) IXS spectra of bulk Si with small incident angle (α~0.1 deg.) at Analyzers 5–8 (A5–A8) in (a). Q indicates the momentum transfer of Si at Analyzers 5–8. Black lines are the spectra at the original configuration without the Soller slit. Red lines are the spectra after the Soller slit installation.

reducing the effective footprint L seen by the analyzers, a Soller slit was placed just after the sample position (Fig. 1(b)). Red lines in Figs. 1(c)–1(f) indicate the spectra after the Soller slit was installed, clearly showing that the artificial contribution is suppressed. One notes that a related system was previously installed at BL43LXU^[1].

3. Feedback system of optics

The liquid-N₂-cooled double-crystal monochromator is located just after the front end, and this monochromator predominantly affects the stability of the entire optical system. In recent years, the monochromator sometimes becomes unstable suddenly, and the beam might be lost at the sample position. To compensate for the beam shift owing to the instability of the monochromator, a positionsensitive ion chamber was introduced 40 m downstream from the monochromator.

This position-sensitive ion chamber detects the vertical position and flux simultaneously. With the ion chamber, a simple feedback system was established. As seen in Fig. 2, when the beam moves more than $\sim 12 \,\mu m$ from the starting position, one of the monochromator crystals is automatically adjusted to obtain the original (starting) beam position. This system, now in regular use at the beamline, improves the stability of the beam position during the user experiments.



Fig. 2. Vertical X-ray beam position (40 m downstream from the liquid-N₂-cooled monochromator) during the experiment. Green arrows indicate that the automatic feedback system worked.

4. Remote experiments

To avoid many cancellations of the user beam time due to the COVID-19 pandemic, BL35XU began to allow remote experiments without the user group present on site. Practically, this can be complex, as BL35XU accommodates a broad range of user setups. For example, experiments performed at low momentum transfer (such as studies of liquids) and those in complex environments require additional attentions. Those lead to a large burden on limited Hence, remote operation is only accepted staff. for occasional experiments with simpler setups/requirements. Even then, care is needed, especially for new users, as clear understanding of the BL operation and how to effectively use the instruments can be very difficult if the user is not present.

Practically, remote experiments are

performed using a Google document (as a log book) and a BL35XU cloud server (for data transfer). The users suggest the experimental plan via the Google document and obtain the results from the cloud server through sftp connection. Beamline staff members write what they performed in the Google document and upload the results to the cloud server.

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Reference:

[1] Baron, A. Q. R. Ishikawa, D. Fukui, H. & Nakajima, Y. (2019). *AIP Conf. Proc.* 2054, 20002.