A high-efficiency X-ray emission spectrometer of SPring-8 BL39XU beamline

X-ray spectroscopy is a useful tool for studying the element-selective electronic states of functional materials. In particular, X-ray emission spectroscopy (XES) can obtain detailed information about electronic states, such as valence, spin, and coordination states [1-3]. However, an accurate XES spectrum of trace elements may take a long time to acquire because their emission signals are generally weak. To overcome this problem, a high-efficiency XES spectrometer equipped with multiple analyzer crystals has been developed at synchrotron radiation facilities worldwide. Such an XES spectrometer was desired for the public beamline of SPring-8. Thus, a new high-efficiency XES spectrometer was installed at SPring-8 **BL39XU** for the main purpose of high-energy resolution X-ray absorption spectroscopy.

The specifications of the spectrometer are summarized in Table 1. The sample, analyzer crystals, and detector are located on the Rowland circle of 820 mm diameter, as shown in Fig. 1(a). The spectrometer consists of three towers each with an assembly of five analyzer crystals, so that a maximum of fifteen analyzer crystals can be mounted on the spectrometer (Fig. 1 (b)). The X-ray path for the emission X-rays, including the analyzer crystals, is vacuumized, except at the sample position corresponding to the X-ray emission point and at the detector position corresponding to the focal point. The vacuum chamber provides the emission intensity that is the collection of the emission intensities of 15 analyzer crystals and suppresses X-ray absorption through air and window materials (Kapton film) so that the efficiency of XES measurements is improved.

It is important to be able to freely select target elements and/or fluorescence (emission) lines for X-ray spectroscopy. Figure 2 shows the types of analyzer crystal and the emission energy range available for the XES spectrometer. Emitted X-rays with a wide energy range of 4.4–16 keV are available with little energy loss, so that important elements of the functional materials, such as 3*d*/5*d* transition metals, 4*f* lanthanide elements, and 5*f* actinide elements, are covered. The energy resolution of the spectrometer is estimated to be 1×10^{-4} in an incident photon energy range of 4.9–18 keV. Currently, 15 analyzer crystals of Ge (111) plane and those of Ge (220) plane can be used for XES measurement. For the other analyzers, mostly five crystals can be used for the measurements. To adjust the emission X-ray energy, an automatic alignment system for the

Table 1. Specification of the X-ray emission spectrometer installed at BL39XU.

Emission energy range (keV)	4.41 – 16.0
Energy resolution (eV)	0.5 – 1.5 ($\Delta E/E = 10^{-4}$)
Rowland radius of spectrometer (mm)	820
Available Bragg angle (degrees)	70 – 83
Motion range of analyzer crystal (degrees)	75 ± 10 (horizontal, θ_B) / 0 ± 10 (vertical, χ)
Scattering angle (degrees)	30 – 150
Photon flux @ sample (phs/s)	10^{11} – 10^{13} (horizontal, vertical, and circular polarizations)
Beam size @ sample (μm)	300 (vertical) × 120 (horizontal), or 2 (vertical) × 9 (horizontal)*
Detectors	PILATUS 100K (sensor: Si, pixel size: 172 μm), PiXirad-2 (sensor: CdTe, pixel size: 60 μm (honeycomb array)), SOPHIAS-L (sensor: Si, pixel size: 30 μm)
Sample environment	Pulse-tube type cryostat (3 – 300 K), Helium-flowing type cryostat (10 – 330 K), Permanent magnet (1.15 T in gap 10 mm), High-pressure cell (Diamond-Anvil-Cell, A.P.–50 GPa)

* using Kirkpatric-Baez mirror

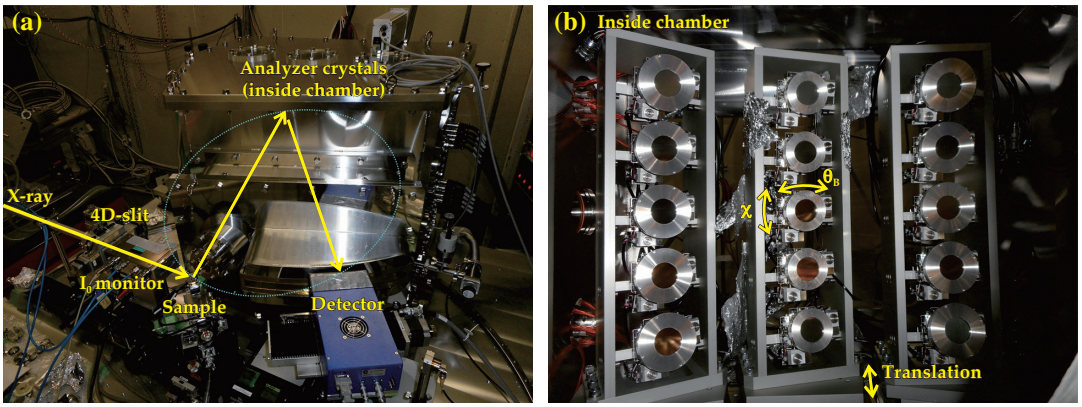

Fig. 1. **(a)** New vacuum chamber of the X-ray emission spectrometer with 15 analyzer crystals. The emission point (sample) and focal point (detector) are located outside the chamber. **(b)** Three towers, each of which can mount a set of five analyzer crystals, are installed inside the chamber.

analyzer crystals is currently under development. This system provides a collection of emitted X-rays from the sample to the same focal point by adjusting the five analyzer crystals in each tower. The system will allow users to change the analyzer crystals in accordance with the target elements/emission lines on their own.

The number of users of XES spectroscopy has gradually increased, and about 50% of beamtime is occupied. The research fields have expanded to strongly correlated electron systems, chemical analysis, and earth/environment science. This wider range of research field necessitates a certain degree of freedom in selecting elements and/or emission lines. We also have a plan to amplify different types of analyzer crystal to facilitate the widening of the X-ray energy range.

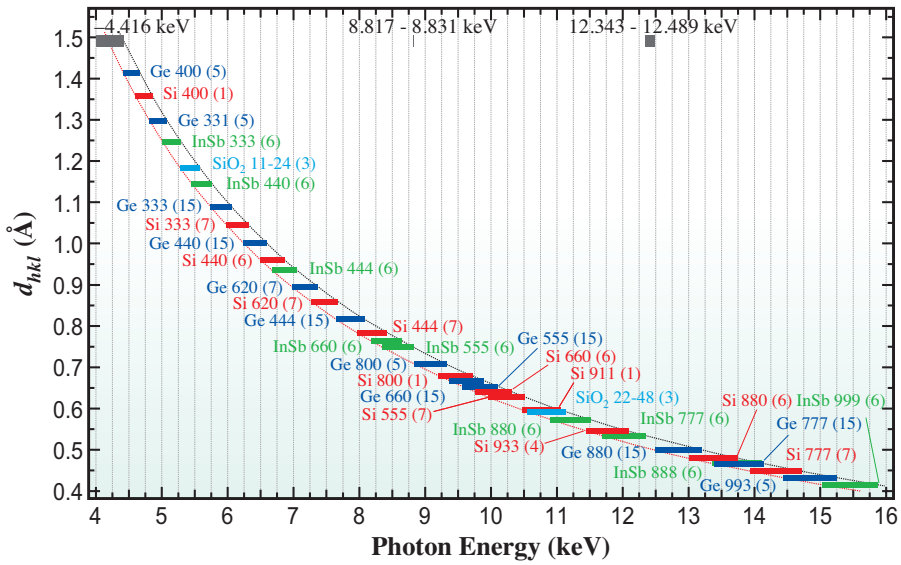

Fig. 2. Analyzer crystals available for the XES spectrometer. The number in parentheses ( ) represents the quantity of crystals. Black and red dotted lines denote the conditions of Bragg angles of 70 and 83 degrees, respectively. Gray thick lines denote the unavailable energy range.

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