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# X-ray beam properties available at the nuclear resonant scattering beamline at SPring-8

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**Abstract**
High-resolution monochromators for various nuclei have been developed at the nuclear resonant scattering beamline at SPring-8. In particular, monochromators for $^{57}$Fe are prepared with three different resolutions. The focusing optics of a Kirkpatrick-Baez mirror and compound refractive lenses have been installed in recent years. The available X-ray beam properties of the energy resolution and beam size at the transition energies of Mössbauer nuclei are summarized in this report.

**Keywords** Nuclear resonant scattering · Synchrotron radiation · High-resolution monochromator · Kirkpatrick-Baez mirror · Compound refractive lens

## 1 Introduction

Various fields of science, including fundamental physics, material science, and biochemical science, have been studied at the nuclear resonant scattering (NRS) beamline BL09XU at SPring-8 since 1997. BL09XU is the only NRS beamline open for public users at SPring-8. Experiments have been conducted using various techniques regarding NRS, such as nuclear excitation, synchrotron Mössbauer spectroscopy, nuclear inelastic scattering, and quasi-elastic scattering using time-domain NRS [1]. The beamline has an undulator with a 32 mm period in the storage ring and a liquid-nitrogen-cooled monochromator comprising Si 111 double crystals in the optics hutch [2, 3]. The beamtime has been partly shared with HAXPES (hard X-ray photoemission spectroscopy) since 2014.

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The energy resolution is one of the most important beam properties for NRS experiments. The energy resolution determines the resolution of the spectra in the case of nuclear inelastic scattering experiments, and the detectors can be prevented from saturation due to a high resolution in the case of synchrotron Mössbauer spectroscopy. Sample damage in soft materials can also be reduced by a high resolution in the case of quasi-elastic scattering experiments using time-domain NRS.

The beam size is another important property for NRS experiments. In particular, a micro-focused beam is suitable for a tiny sample at high pressure and a thin film in total reflection. Mössbauer microspectroscopy can also be performed using a small beam. The source size of synchrotron radiation in third-generation facilities and the beam divergence are much smaller than those of a conventional radioactive source. Therefore, the beam size of synchrotron radiation at the sample position is originally smaller without focusing optics and much smaller with focusing optics in general.

At the NRS beamline at SPring-8, high-resolution monochromators for various nuclei have been developed for years, and the focusing optics have been installed in recent years. The available X-ray beam properties of the energy resolution and beam size at the transition energies of Mössbauer nuclei are summarized in this report.

## 2 Energy resolution

In general, a higher energy resolution can be achieved using reflections with a higher Bragg angle by a perfect crystal. The energy resolutions for various Mössbauer isotopes available at the NRS beamline are listed in Table 1. The resolutions in the table are measured values. All the reflections in the high-resolution monochromators (HRMs) are performed after the first high-heat-load double Si 111 reflections of two Si crystals. We have eleven HRMs for eight isotopes. Because $^{57}$Fe is the most popular isotope in NRS experiments, HRMs with three different resolutions of 0.8 meV, 2.5 meV and 3.5 meV are prepared.

The angular width at a finite energy or the energy width at a finite angle in Bragg reflection by the perfect single crystal, strongly depends on the X-ray energy. Therefore the different kinds of reflection geometry are used depending on the X-ray energy and the needed energy resolution. The listed HRMs are classified in the following four different type: (1) Two

**Table 1** The energy resolutions available for various Mössbauer isotopes at the NRS beamline at SPring-8. The resolutions listed are measured values. All the reflections in the HRMs are conducted after the first high-heat-load double Si 111 reflections. We have three different resolutions for $^{57}$Fe

| Isotope | Energy (keV) | Reflections | Resolution (meV) |
|---|---|---|---|
| $^{181}$Ta | 6.21 | Si311 - Si511 - Si511 | 10.5 |
| $^{57}$Fe | 14.41 | Ge331 – Si975 – Si975 | 0.8 |
| | 14.41 | Si511 – Si975 (nested) | 2.5 |
| | 14.41 | Si511 – Si975 (nested) | 3.5 |
| $^{151}$Eu | 21.54 | Si422 - Si12 12 8 (nested) | 1.7 |
| $^{149}$Sm | 22.51 | Si422 – Si16 8 8 (nested) | 1.6 |
| $^{119}$Sn | 23.87 | Si440 – Si12 12 12 (nested) | 1.6 |
| $^{40}$K | 29.83 | Si660 (channel-cut)– Si22 14 0 | 2.6 |
| $^{125}$Te | 35.49 | $\alpha$-$Al_2O_3$ 9 1–10 68* | 1.7 |
| $^{121}$Sb | 37.13 | Si333 (channel-cut) – Si 4 4 32 | 1.7 |

*in Miller-Bravais notation

asymmetric reflections (2) Nested two channel-cuts (3) Cryo-cooled Si with channel-cut (4) Sapphire backscattering. An medium resolution monochromator is used for the isotopes with energies above 38 keV beside these four types HRMs.

Asymmetric reflections are used to obtain higher resolutions in $^{181}$Ta and $^{57}$Fe 0.8 meV HRMs [4, 5]. Because the energy width obtained by the symmetric reflection is wider in the lower X-ray energy region. Two Si 511 asymmetric reflections are used in $^{181}$Ta. The asymmetric reflection of Ge 331 and the different asymmetric factors between the two Si 975 reflections are used to obtain a higher throughput in $^{57}$Fe 0.8 meV HRM (see the detail in [6]). The flux obtained after the $^{57}$Fe 0.8 meV HRM is $2.8 \times 10^9$ cps.

Nested type HRMs consisting of two channel-cut crystals are used for $^{57}$Fe 2.5 meV, $^{57}$Fe 3.5 meV, $^{151}$Eu, $^{149}$Sm and $^{119}$Sn to obtain high resolutions and accept wider angular divergences [7]. A quick change in the resolution between 2.5 meV and 3.5 meV is realized by monolithic fabrication of the second channel-cut Si 975 crystal for $^{57}$Fe [5]. The flux obtained after the $^{57}$Fe 2.5 meV and 3.5 meV HRMs are $1.2 \times 10^{10}$ cps and $2.1 \times 10^{10}$ cps respectively.

The high-index reflections of Si 22 14 0 and Si 4 4 32 are used in $^{40}$K and $^{121}$Sb HRMs, respectively [5]. Cryo-cooling of the crystal by liquid nitrogen is adopted to suppress the decrease in the reflectivity due to the Deby-Waller factor. The reflections by the channel-cut Si crystals are used to restrict the angular divergences to obtain higher resolutions before the high-index reflections. In contrast with the asymmetric reflections in the nested type HRMs, the channel-cut Si crystals reduce the resonant intensity by the restriction.

An $\alpha$-$Al_2O_3$ 9 1–10 68 reflection in a backscattering geometry is used for $^{125}$Te [8]. The output energy is controlled by the temperature of the sapphire crystal. The cryo-cooled Si type HRMs for $^{40}$K and $^{121}$Sb could be replaced by the sapphire backscattering type HRMs in the future [9].

The flux produced by the undulator at the NRS beamline is high enough, up to approximately 100 keV, for some NRS experiments. The incident angle of the X-rays to the Si 111 crystal is mechanically limited above 3 degrees. Therefore, an X-ray below 38 keV is first monochromatized by double Si 111 reflections, and an X-ray above 38 keV is monochromatized by Si double 333 reflections. As shown in Fig. 1, the Si 220 reflections by the channel-cut crystal after the Si 333 reflections are arranged in (+, −, −, +) for the energy and time-domain Mössbauer spectroscopy for the isotopes with energies above approximately 60 keV, such as $^{61}$Ni, $^{193}$Ir, and $^{174}$Yb. A medium resolution is achieved by this arrangement. For example, a resolution of 1.4 eV in FWHM is obtained in the calculation for a 67.41 keV X-ray, which is the transition energy of $^{61}$Ni assuming a Gaussian vertical divergence of 2 arcsec in FWHM. In contrast, a resolution of 7.4 eV is obtained in the calculation without Si 220 reflections. The Si 220 reflection is also served to eliminate X-rays with one-third of the energy allowed in the first Si 111 reflections.

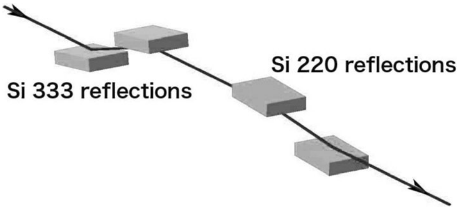

**Fig. 1** A Schematic drawing of the medium-resolution monochromator for the isotopes with energies above approximately 60 keV, such as $^{61}$Ni, $^{193}$Ir, and $^{174}$Yb. Si 333 reflections in the beamline high-heat-load monochromator and Si 220 reflections in the channel-cut crystal are arranged in (+, −, −, +)

## 3 Beam size

The source size at the NRS beamline is 744 μm (horizontal) × 12 μm (vertical) in FWHM. The front-end slit is placed 29 m downstream from the source. The aperture of the slit is limited to less than 1.5 mm (H) × 0.65 mm (V) to reduce the heat load to the first monochromator, which is located in the optics hutch 37.5 m away from the source. We have two experimental hutches downstream of the optics hutch [3]. The measured beam size at 14.41 keV is 1.4 mm (H) × 0.5 mm (V) in FWHM at the 1st experimental hutch without focusing optics.

The Kirkpatrick-Baez (K-B) mirror for the HAXPES experiments is placed in the 2nd experimental hutch. Although the mirror is optimized for the X-ray energy below 10 keV, it can also be used for NRS experiments on isotopes with lower energies such as $^{57}$Fe with the reduced reflectivity. The obtained beam size by the K-B mirror is 10.8 μm (H) × 4.2 μm (V) at 14.41 keV. The measured reflectivity is 44%. NRS experiments of earth science are conducted using this small beam.

Polymer compound refractive lenses (CRLs) made by the Karlsruhe Institute of Technology are used for on-line two-dimensional focusing [10]. Three CRLs suitable for the NRS experiments of $^{57}$Fe, $^{149}$Sm and $^{229}$Th are prepared. The CRLs are designed assuming the distances between the source and the lens are infinite in vertical and 49 m in horizontal. The numbers of CRLs for vertical and horizontal focussing are 13 and 16 for $^{57}$Fe, 33 and 42 for $^{149}$Sm, 52 and 67 for $^{229}$Th respectively. The corresponding energies are 14.41 keV, 22.51 keV and 29.19 keV, respectively. The CRLs for $^{149}$Sm can also be used for $^{151}$Eu and $^{119}$Sn with a slightly different focusing position or a slightly larger beam size. The CRLs are placed in the 1st experimental hutch, and a sample is placed in the 2nd experimental hutch. The distance from the CRLs to the centre of the sample table is 12.8 m. The horizonal and vertical beam sizes measured by changing the distances from the CRLs for $^{229}$Th are shown in Fig. 2. A beam size of 149 μm (H) × 41 μm (V) is obtained at the sample position. A beam size of 155 μm (H) × 110 μm (V) is obtained using the CRLs for $^{57}$Fe. The minimum vertical beam sizes are obtained at approximately 14.5 m from the lens for $^{229}$Th and 17 m for $^{57}$Fe. The vertical beam size will be improved by inserting the CRLs before the HRMs, making the vertical divergence smaller. The measured throughputs in the CRLs are 67% for $^{229}$Th and 65% for $^{57}$Fe.

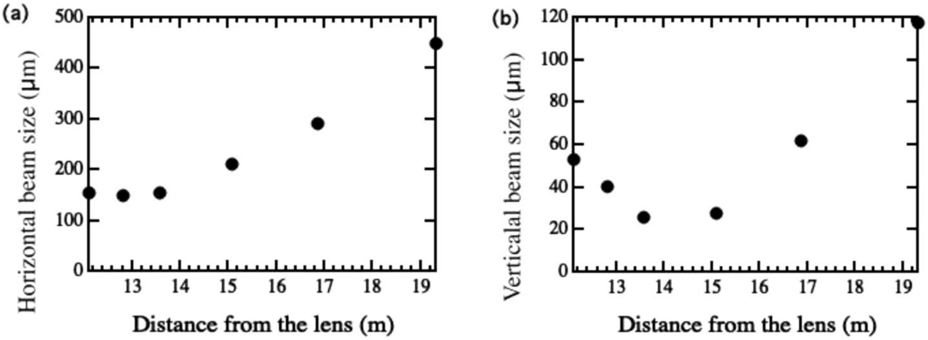

**Fig. 2** Position dependence of the focusing beam sizes (**a**) in the horizontal and (**b**) in vertical directions. The X-ray energy is 29.19 keV, which is the transition energy of $^{229}$Th. The centre of the sample table is 12.8 m away from the CRLs. A beam size of 149 μm (H) × 41 μm (V) is obtained at the sample position

## 4 Summary

High-resolution monochromators for various nuclei have been developed at the NRS beamline at SPring-8. In particular, the monochromators for $^{57}$Fe are prepared with three different resolutions of 0.8 meV, 2.5 meV and 3.5 meV. The focusing optics of a K-B mirror and CRLs have been installed in recent years. A beam size of 10.8 μm (H) × 4.2 μm (V) is obtained by the K-B mirror for $^{57}$Fe. The available X-ray beam properties of the energy resolution and beam size at the transition energies of Mössbauer nuclei are summarized in this report.

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