The Tender Energy Spectroscopy Beamline at SSRF

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Abstract

The tender energy spectroscopy beamline (BL16U1) is a phase-II beamline project at the Shanghai Synchrotron Radiation Facility (SSRF). The design and performance of the tender energy spectroscopy beamline at SSRF are described in this paper. Based on a 26 mm-period in vacuum undulator (IVU) source, the beamline is to give an operable energy range between 2.1 and 16 keV, covering the K-edges of those elements from P to Rb and the L3-edges of those elements from Zr to Bi. The principal optical elements of the beamline consist of a toroidal mirror, a liquid-nitrogen cooled double-crystal monochromator, a high harmonic rejection mirror and two pairs of Kirkpatrick–Baez (KB) mirrors. Three end-stations, including the non-focusing, microprobe and sub-microprobe end-stations, are installed on the beamline. X-ray fluorescence (XRF), X-ray absorption spectroscopy (XAS) including X-ray absorption near-edge structure (XANES) and extended X-ray absorption fine-structure (EXAFS), have been achieved under vacuum or He atmosphere at the non-focusing end-station with a spot size of ~ 670$\times$710$\mu m2. Based on two KB mirrors systems, micro-X-ray fluorescence (\mu XRF) mapping studies and micro-X-ray absorption near-edge structure (\mu XANES) will be operated with a spot size of nearly 3.3\times1.3\mu m2 at the microprobe end-station, and with a smaller spot size of 0.5\times$0.25 m2 at the sub-microprobe end-station. Up to now, the non-focusing end-station of the BL16U1 is officially opened to users in Jan. 2024. The microprobe and sub-microprobe end-stations will open to users in the near future. This paper describes the characteristics, short-term technical developments and a few of the early experimental results of this new beamline.

Full Text

Preamble

The Tender Energy Spectroscopy Beamline at SSRF

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The tender energy spectroscopy beamline (BL16U1) is a phase-II beamline project at the Shanghai Synchrotron Radiation Facility (SSRF). This paper describes the design and performance of the tender energy spectroscopy beamline at SSRF. Based on a 26 mm-period in-vacuum undulator (IVU) source, the beamline provides an operable energy range between 2.1 and 16 keV, covering the K-edges of elements from P to Rb and the L_3-edges of elements from Zr to Bi. The principal optical elements consist of a toroidal mirror, a liquid-nitrogen cooled double-crystal monochromator, a high harmonic rejection mirror, and two pairs of Kirkpatrick–Baez (KB) mirrors. Three end-stations are installed on the beamline: a non-focusing end-station, a microprobe end-station, and a sub-microprobe end-station. X-ray fluorescence (XRF) and X-ray absorption spectroscopy (XAS), including X-ray absorption near-edge structure (XANES) and extended X-ray absorption fine-structure (EXAFS), have been achieved under vacuum or He atmosphere at the non-focusing end-station with a spot size of 670×710 µm^2. Based on two KB mirror systems, micro-X-ray fluorescence (µXRF) mapping studies and micro-X-ray absorption near-edge structure (µXANES) will be operated with a spot size of nearly 3.3×1.3 µm^2 at the microprobe end-station, and with a smaller spot size of 0.5×0.25 µm^2 at the sub-microprobe end-station. As of now, the non-focusing end-station of BL16U1 was officially opened to users in January 2024, while the microprobe and sub-microprobe end-stations will open to users in the near future.

This paper describes the characteristics, short-term technical developments, and several early experimental results from this new beamline.

Keywords: Tender energy X-ray spectroscopy, X-ray fluorescence, SSRF, X-ray absorption spectroscopy (XAS), Microprobe

Introduction

As the first third-generation synchrotron radiation light source in mainland China, the Shanghai Synchrotron Radiation Facility is equipped with a storage ring energy of 3.5 GeV, a circumference of 432 m, and an emittance of approximately 3.9 nm rad [**?**]. SSRF opened to users in 2009 with seven Phase-I beamlines [**?**], and over the next few years, six additional beamlines were built as part of the Follow-up Beamline Program (FBP). Within the framework of

the SSRF Phase-II Beamline Project (2016) [**?**, **?**], 16 new beamlines and more than 30 end-stations have been constructed, extending the photon energy to previously uncovered regions such as the tender X-ray region (BL16U1), the super-hard X-ray region [**?**], and the low-energy gamma-ray region [**?**].

XAS techniques, including XANES and EXAFS, have been recognized as efficient and comprehensive analytical tools for probing the electronic and local atomic structure of metals and elements due to their advantages of element selectivity, valence state identification, and characterization of local atomic structure. To date, XAS platforms at SSRF spanning from soft X-ray to hard X-ray include the soft X-ray spectromicroscopy beamline (BL08U1A, STXM, 250–2000 eV, [**?**]), the X-ray absorption fine structure beamline (BL14W1, XAFS, 4.5–50 keV, [**?**]), the hard X-ray micro-focusing beamline (BL15U1, 5–20 keV, [**?**]), and the hard X-ray spectroscopy beamline (BL11B, 5–30 keV, [**?**]), among others.

Thanks to the SSRF Phase-II Beamline Project, the tender-energy spectroscopy beamline (BL16U1) is the only beamline designed to fill the tender photon energy gap at SSRF. The tender energy range of 2–5 keV, between the energy ranges of soft and hard X-rays, covers the K-edges of elements such as phosphorus (P), sulfur (S), chlorine (Cl), potassium (K), calcium (Ca), and titanium (Ti), which are important in soil and environmental sciences \cite{11–17}, geologic and cosmologic materials \cite{18–20}, life sciences \cite{21–23}, catalysis, and archaeology [**?**, **?**]. This tender energy range also covers the L-edges of Mo to I, which are important for novel materials [**?**], mineral resources [**?**], environmental contaminants, and biological toxins [**?**]. Several beamlines worldwide focus on the tender X-ray energy region, including Diamond-I18 (2–20.7 keV) [**?**], SLS-PhoenixI (0.8–8 keV) [**?**], CLS–SXRMB (1.7–10 keV) [**?**], ESRF-ID21 (2–10 keV) [**?**], 8-BM at NSLS-II (2–5.5 keV) [**?**], BL27SU at SPring8 (2.1–3.3 keV) [**?**], 4B7A at BSRF (1.75–6.0 keV) [**?**], and TBS 32A at NSRRC [**?**]. Among all these beamlines, XAS and XRF imaging with microprobe are the main research methods.

Taking advantage of the high brightness of SSRF, the BL16U1 beamline is designed to cover the X-ray energy range of 2.1–16 keV using a U26 in-vacuum undulator (IVU). In addition to the tender X-ray energy range, the beamline also covers most transition metals and non-metallic elements of interest in energy, catalysis, and other areas, such as titanium (Ti), nickel (Ni), iron (Fe), gold (Au), platinum (Pt), and palladium (Pd). Based on a toroidal mirror, a liquid-nitrogen cooled double-crystal monochromator, and a high harmonic rejection mirror, XAS can be obtained at the non-focusing end-station with a spot size of 670×710 μm^2. Samples can be operated under vacuum (lower than 1 mbar), but if samples are aqueous, helium gas is purged into the vessel and no vacuum is used. Based on two pairs of KB mirrors, XANES and XRF mapping will be operated at the microprobe end-station with a spot size of nearly 3.3×1.3 μm^2, and at the sub-microprobe end-station with a smaller spot size of 0.5×0.25 μm^2. The BL16U1 beamline construction was completed in July 2023, and the non-focusing end-station has been officially opened to users since January

2024. The microprobe and sub-microprobe end-stations will open to users in the near future. The beamline design, its short-term technical developments, and several early experimental results are described in this paper.

II. Beamline Design

Specific optimizations of the beamline design have been conducted to meet the requirements for flux and focusing. An undulator source is used to achieve high flux density in small spot sizes for microprobe XRF imaging. High-angular-range monochromator design is needed for the low critical energy of 2.1 keV. Harmonic rejection mirrors with different incident angles are used for different energy ranges, and different coatings are required to avoid absorption edges from the mirror coatings. Depending on the properties of user samples, either vacuum or He atmosphere can be provided.

A. Light Source

The upstream section of a 12 m long canted long straight section in SSRF is selected as the light source for the tender energy spectroscopy beamline, while the downstream section (3.06 m long) is used for the fast X-ray imaging beamline (BL16U2) [**?**, **?**]. A U26 in-vacuum undulator (IVU) with 3.2 m length, 26 mm period, and 6 mm minimum gap was finally chosen as the light source. Detailed information for the undulator of the BL16U1 beamline is shown in Table 1 . The maximum magnetic field strength exceeds 1.02 T with a total power of over 7.7 kW. By tuning its gap from 6 to 15 mm, 1st–7th harmonics and X-ray energy ranges between 2.1–16 keV can be generated. For the IVU design in SSRF, taper mode is used for EXAFS detection, which means the two out-of-vacuum girders are tilted. In BL16U1, with a maximum gap taper adjustment range of 0.5 mm, which means a reproducible mechanical gap difference between exit gap and entrance gap ($±$0.5 mm [**?**]), EXAFS above 5 keV can be obtained.

B. Beamline Optics

The main optical layout of the beamline is shown in Fig. 1 [Figure 1: see original paper]. A toroidal mirror, a liquid-nitrogen cooled double-crystal monochromator, a high harmonic rejection mirror, and two pairs of Kirkpatrick–Baez mirrors are installed on the beamline. Details on all beamline mirrors are listed in Table 2 . The layout is similar to that of the hard X-ray micro-focusing beamline (BL15U1) at SSRF [**?**] and the microfocus spectroscopy beamline (I18) at Diamond Light Source [**?**]. A horizontally deflecting toroidal mirror (FMB Oxford) achieved by mechanically bending a sagittal cylindrical mirror is placed 35 m from the source. A set of water-cooled slits (Slit1, Fig. 1), located 26 m from the source, are used to define the incoming beam on the toroidal mirror. Considering the effective length, reflectivity, and heat load, the toroidal mirror is water-cooled and operates at a grazing incidence angle of 3.5 mrad with an active area of 800 mm. Rh coating on a single-crystal Si substrate is used for energies

above 8 keV, and Si coating is used for photon energies below 8 keV. The two coatings can be switched by an in-vacuum translation mechanism. Using the toroidal mirror, the beam in the vertical plane is collimated and the influence of vertical source divergence is removed, making the energy resolution primarily a function of the bandpass of the crystals used in the monochromator. In the horizontal plane, the beam is focused using a mechanically elliptical bend onto the secondary source, which is placed 48 m away from the light source where the secondary slits (MS1 in Fig. 1, 10 m after the monochromator) are installed. The secondary source is used for the horizontal focusing optics of the KB mirrors after the monochromator.

Owing to the high-power density of the undulator, the monochromator is installed after the toroidal mirror. A fixed-exit double-crystal monochromator (DCM, TOYAMA) is located about 38 m away from the light source. Photon energies between 2.1–16 keV with resolution below 1.64×10^{-4} (ΔE/E@2.5 keV) can be obtained with Si(111) crystal sets. The Si(220) crystal is applied for better energy resolution with photon energies between 3.35–16 keV. The crystals are translated by an in-vacuum translation mechanism. Owing to the high power density of the undulator source, the first and second crystals are indirectly cooled with liquid nitrogen. The fixed beam exit is maintained by translating the second crystal vertically, with a final height difference of 25 mm. To cover the required energy range, the monochromator has a high angular range of 0–75°. To maintain alignment of the first and second crystal lattice planes over this angular range, two coarse motors ($\pm$\$12 mrad and $\pm 8 mrad) are used for roll and pitch coarse adjustment, and two piezo actuators ($\pm$\$0.2 mrad) are also used for fine adjustment of the roll and pitch.

Two sets of monochromatic four-knife slits without water cooling are installed downstream of the monochromator. The first monochromatic four-knife slit (MS1, Fig. 1), located 10 m away from the monochromator, serves as the secondary source for the focusing optics in the horizontal direction, with a slit size of 350×1400 µm^2 (h×v). Another monochromatic four-knife slit (MS2, Fig. 1), located 4 m away from MS1, is used to limit the irradiation range of the beam on the KB mirrors, with a slit size of 1400×1600 µm^2 (h×v). The slit position is fixed but the slit width can be controlled via a parallelogram mechanism.

A harmonic rejection mirror (HRM, TOYAMA) is placed at 51 m from the source. A pair of horizontally reflecting flat silicon mirrors is used for rejection of higher harmonics. The mirrors have three stripes of chrome (Cr), silicon (Si), and rhodium (Rh), which are translated vertically in vacuum. The Cr reflector can be used for 2.05–3.5 keV with a grazing incidence angle of 10 mrad. The Si reflector can be used for 3.5–7.5 keV with a grazing incidence angle of 3.5 mrad. The Rh reflector can be used for 7.5–13 keV with a grazing incidence angle of 3.5 mrad. The grazing incidence angle is regulated by two horizontal vacuum motors installed upstream and downstream of the mirror. Besides the three coatings that reflect the X-ray beam, the mirrors can be moved out of the

beam by in-vacuum translation to allow the incoming X-ray to pass through without being reflected.

III. Experimental Station

Aimed at XAS and XRF microprobe imaging between 2.1–16 keV, three end-stations are installed at the BL16U1 beamline: the non-focusing end-station, the microprobe end-station, and the sub-microprobe end-station, focused by two sets of KB mirrors. The schematic layout of the three end-stations is shown in Fig. 2 [Figure 2: see original paper]. The specifications of energy range, energy resolution, flux, and spot size at different end-stations are listed in Table 3 .

The non-focusing end-station is placed after the harmonic rejection mirror, about 53 m away from the source. X-ray fluorescence (XRF) and X-ray absorption spectroscopy (XAS), including X-ray absorption near-edge structure (XANES) and extended X-ray absorption fine-structure (EXAFS), can be achieved with a spot size of 670 × 710 μm^2. After the non-focusing end-station, two sets of K-B systems (motors from CINEL, mirrors from JTEC) are used as microprobe and sub-microprobe tools to focus the secondary source to a spot with micron size (Sample 2) and a spot with sub-micron size (Sample 3) in two different vacuum vessels, as shown in Fig. 2. Two vacuum valves (V1 and V2 in Fig. 2) are installed downstream of the non-focusing end-station and are used when He atmosphere is employed in the non-focusing end-station. A liquid in-situ end-station will be installed in the future by removing the vacuum tube between V1 and V2, and a Be window will be installed after valve V1 to maintain the vacuum of the non-focusing vessel.

The photon flux and energy resolution of the beamline are obtained at the non-focusing end-station. Fig. 3(a) [Figure 3: see original paper] shows the photon flux measured at (I1) in the non-focusing end-station. The designed spot size (full width at half maximum, FWHM) at this station is 670 × 710 μm^2. The photon flux of the beamline at this station is above 2.0 × 10^{12} photons/s for energies between 2.15 to 13 keV, and between 1.5 × 10^{12} to 5.0 × 10^{11} photons/s for energies between 14 to 16 keV. We believe this is not yet the optimal status of our beamline, and better flux values should be obtained with longer use. Fig. 3(b) shows the rocking curve at 2.5 keV using a Si(111) single crystal. The DCM energy was set to 2.5 keV and a Si(111) single crystal was placed after the non-focusing end-station and rotated in vacuum around 52.2669°, with a photodiode (AXUV300C) used to measure the diffracted photon flux. The FWHM (Δ) of the rocking curve at 2.5 keV is 212 μrad, yielding an energy resolution of 1.64 × 10^{-4} calculated by Δ /tan , where is the diffraction angle of Si(111) at 2.5 keV (52.2669°).

Micro-X-ray fluorescence (μXRF), micro-X-ray fluorescence mapping, and micro-X-ray absorption near-edge structure (μXANES) can be obtained at the KB and SKB microprobe end-stations in the near future. Details of the

KB and SKB mirrors are listed in Table 2 . For each set of KB mirrors, fixed-surface-shape KB mirrors are used. The mirror substrates are made of silicon and coated with Ni, Si, and Rh stripes. The coating stripes are translated by an in-vacuum translation mechanism. A vertically focusing mirror (VFM) and a horizontally focusing mirror (HFM) are aligned behind each other in orthogonal planes, with incident angles of 4 mrad and 4.7 mrad for VFM and HFM mirrors, respectively.

XRF detection and partial fluorescence yield (PFY) detection of the sample are performed using a photodiode installed next to the SDD to measure the total fluorescence yield (TFY) of the sample. Total electron yield (TEY) mode is also used to measure the sample current. The schematic of the three detection modes is shown in Fig. 2 [Figure 2: see original paper].

A. Non-focusing End-station

The non-focusing end-station is housed in a vacuum vessel allowing operation in vacuum (1–10^{-6} mbar) or He atmosphere. No load-lock system is used for sample replacement in the non-focusing end-station. Usually, only the dry pump is turned on, and a vacuum of 1 mbar is sufficient for the non-focusing end-station. He gas is purged into the vessel when water is present in the samples and no vacuum is used. The dry pump and turbo pump (Pfeiffer, HiPace 700) are turned on when high vacuum and KB systems are used, requiring 20–30 minutes for vacuum venting and sample replacement.

Fig. 4 [Figure 4: see original paper] shows a photograph of the non-focusing end-station. A set of translation (X-Z) and rotation (R) motors (VACGEN) are used to adjust the sample position in the vacuum vessel. The sample holder is 9 cm in total length with a YAG crystal on the top to assist with beam location (inset in Fig. 4). Samples are typically smeared onto carbon or kapton tapes or pressed into disks, with 6–9 samples placed on the sample holder at a time. By indirect cooling with liquid nitrogen, samples in the non-focusing end-station can be operated in vacuum under cryogenic conditions down to 120 K. A graphene carbon window (Ketek, 900 nm thickness and 10 mm diameter) separates the vacuum of the non-focusing vessel from the beamline.

Four photodiodes (AXUV300C) are installed at the four corners of a 5 mm hole to measure the fluorescence after a thin Al film with 2 µm thickness, which is used as the incident beam intensity (I_0). Due to space constraints on the beamline, the I_0 detector is placed before the graphene carbon window. Before the I_0 detector, several Al foils with different thicknesses (25–500 µm) are used as attenuators. A photodiode (AXUV300C) is mounted after the sample in the vacuum vessel to measure the transmitted beam intensity (I_1). The I_1 photodiode can be moved out of the beamline by in-vacuum translation when the KB microprobe end-station is used. A three-channel silicon drift diode (SDD, RaySpec) with a collimated active area of 150 mm^2 is installed perpendicularly to the beamline for XRF detection and partial fluorescence yield (PFY) detection of

the sample.

Several XAS results obtained at the non-focusing end-station are shown in Fig. 5 [Figure 5: see original paper]. Different XAS detection modes are used according to the morphology, conductivity, and absorption edge of samples. For elements with absorption edges above 5 keV, TEY, TFY, PFY, and transmission modes are used for XAS detection depending on morphology and concentration. For elements with absorption edges below 5 keV, TEY, TFY, and PFY modes are used. For PFY mode with low concentration and transmission mode with high concentration, samples should be pressed into disks with proper thickness. For TEY and TFY modes, samples are typically smeared onto carbon or kapton tapes. The I_0 and I_1 photodiodes in Fig. 4 are used for transmission mode.

The P K-edge XANES of KH_2PO_4 obtained by TEY mode is shown in Fig. 5(a). The P K-edge XANES is very similar to that obtained at ESRF-ID21 [**?**]. The maximum of the "white line" (s→p electronic transition) of the P K-edge of KH_2PO_4 is corrected to 2152.8 eV according to ID21 [**?**]. The Sr K-edge XANES of $C_4H_6O_4Sr$ obtained by transmission mode is shown in Fig. 5(b), and the spectrum is similar to the XANES spectrum of $SrCO_3$ in [**?**]. These test results demonstrate that the photon energy range of the beamline covers the design energy range between 2.1 and 16 keV. During testing, each energy integration time was one second with different undulator gaps. The undulator taper was set to 0.45 mm and the beam current was 220 mA.

The S K-edge XANES of $CaSO_4$ obtained by TFY, TEY, and PFY modes are shown in Fig. 5(c). The S K-edge XANES is very similar to that obtained at ESRF-ID21 [**?**]. The maximum of the "white line" (s→p electronic transition) of the S K-edge of $CaSO_4$ is corrected to 2482.5 eV according to ID21 [**?**]. High-purity $CaSO_4$ powder and $CaSO_4$ powder diluted by LiF to mass concentrations of 2.5% and 0.5% were used as samples. The $CaSO_4$ powder was smeared evenly onto kapton or carbon tapes with very thin thickness. High-purity $CaSO_4$ powders measured by TEY and TFY modes are shown in Fig. 5(c). Due to self-absorption of fluorescence, the fluorescence spectral signal intensity of TFY (red) is much lower than that of TEY mode (blue) for high-purity samples. In Fig. 5(c), $CaSO_4$ with 0.5% concentration was measured by PFY mode (green) and $CaSO_4$ with 2.5% concentration was measured by TFY mode (purple). The order of normalized maximum values is 100% TEY mode, 0.5% PFY mode, 2.5% TFY mode, and 100% TFY mode, respectively. Typically, TEY is used for samples with high concentration, TFY for samples with concentrations between 1% and 5%, and PFY for samples with concentrations less than 1% [**?**]. $CaSO_4$ with 0.5% concentration measured by TFY mode with a very close working distance between sample and TFY photodiode (10 mm distance) produced a less smooth spectrum. Thus, for samples with low concentration (<1%), PFY mode is recommended.

The results of K-edge XAFS of Ni standard foil obtained by transmission mode are shown in Fig. 5(d). The I_0 and I_1 photodiodes in Fig. 3 were used for transmission mode. The EXAFS k^2 data and Fourier transform (FT) spectra of the

Ni standard foil K-edge XAFS spectrum are shown in Figs. 5(e) and 5(f). For energy calibration, the energy and Bragg angle of the DCM are reset according to the first derivative spectrum of Ni foil from Exafs Materials [**?**]. After energy calibration, the EXAFS k^2 data and Fourier transform (FT) spectra of the Ni standard foil K-edge XAFS spectrum can be compared to those obtained at X18B at the National Synchrotron Light Source [**?**].

The K-edge XAFS of Ti standard foil obtained by transmission mode is shown in Fig. 5(g). The I_0 and I_1 photodiodes in Fig. 4 were also used for transmission mode. For comparison, the K-edge XAFS spectrum of TiO_2-nano (rutile) powder diluted with LiF to a mass concentration of 3% was also tested by TFY mode, as shown in Fig. 5(g). The energy was calibrated according to the spectrum of Ti foil from Exafs Materials. The EXAFS k^2 data and Fourier transform (FT) spectra of Ti standard foil and TiO_2-nano (rutile) are shown in Figs. 5(h) and 5(i). The EXAFS k^2 data and Fourier transform (FT) spectra of the Ti standard foil K-edge XAFS spectrum can be compared to those obtained at TPS 44A at Taiwan Photon Source [**?**]. The EXAFS and Fourier transform (FT) spectra of TiO_2-nano (rutile) are similar to those obtained at the synchrotron laboratory HASYLAB/DESY, Hamburg [**?**].

These figures demonstrate that BL16U1 can collect XAS spectra across the entire target photon energy range of 2.1–16 keV. For the tender energy range of 2–4 keV, XANES spectra for phosphorus (P), sulfur (S), chlorine (Cl), potassium (K), calcium (Ca), and other elements are typically collected by TEY, TFY, and PFY modes. For energies above 4 keV, XAFS spectra are typically collected by transmission, TEY, TFY, and PFY modes. Although ion chambers are mainly used for synchrotron spectroscopy beamlines worldwide, our results show that photodiodes can also be used for XANES and XAFS spectra. The only drawback of photodiodes is the diffraction peaks resulting from the crystalline nature of the photodiodes [**?**], which can be removed by the "deglitch" function in Athena software.

The non-focusing end-station has been in operation for more than one year since the final acceptance test in July 2023. To date, this station has served more than 85 users with a total user time of 2086 hours. Important achievements have been made in many fields, particularly in Co oxidation reactions [**?**], semi-hydrogenation of propylene [**?**], and flexible aqueous batteries [**?**]. This end-station is currently officially open to users.

B. Microprobe and Sub-microprobe End-stations

Aimed at materials analysis at the microscopic scale, microprobe end-stations have been constructed at synchrotron facilities worldwide in recent years, as summarized in Table 4 . Spot sizes of 2.1×2.5 μm^2 (h × v) on Diamond I18 [**?**], 2.5×2.5 μm^2 (h × v) on SLS PHOENIX I [**?**], 0.7×0.35 μm^2 (h × v), or even smaller than 180 nm on ESRF ID21 [**?**], have been achieved using undulator sources. Using a bending magnet source, the spot size on the SXRMB beamline

at CLS is nearly 10 µm [**?**], and the spot size on the TES beamline at NSLS-II can be tuned from 2–25 µm [**?**]. For these microprobe beamlines, KB mirrors are used to focus the beam, and micro-X-ray fluorescence, micro-EXAFS, and micro-X-ray diffraction are the main methods employed.

Here we focus on micro-X-ray fluorescence and micro-XANES techniques. With the use of a multi-channel silicon drift diode (SDD) detector, one can map elemental distributions and correlations on the micrometer scale. With micro-XANES scans, chemical speciation of elements can be obtained by recording XANES spectra of selected sample spots with grain sizes on the order of micrometers. Micro-X-ray fluorescence and micro-XANES can also be performed on the BL16U1 beamline at the microprobe and sub-microprobe end-stations using two sets of KB-mirror systems. These end-stations are installed after the non-focusing end-station, with two sets of KB mirrors placed in two different vacuum vessels, as shown in Fig. 6 [Figure 6: see original paper]. The vacuum of the two KB systems is maintained below 5$×10^{-7}$ mbar using ion pumps, and two sets of load-lock systems are used for sample transfer.

1. Microprobe End-station Using one pair of fixed-surface-shape KB mirrors, the focal spot of the microprobe end-station is located about 56.85 m from the source. The mirror substrates are made of silicon and coated with 6 mm-wide Ni, Si, and Rh stripes. The coating stripes are translated by an in-vacuum translation mechanism according to the energy. A vertically focusing mirror (VFM) and a horizontally focusing mirror (HFM) are aligned behind each other in orthogonal planes, with incident angles of 4 mrad and 4.7 mrad for VFM and HFM mirrors, respectively. Details of the KB mirrors are listed in Table 2 . For the first set of KB mirrors, the focal spot is located 600 mm and 245 mm from the center of VFM and HFM mirrors, respectively, giving a standard working distance of 75 mm from the end of the HFM mirror to the sample focal plane. A photograph of the KB mirrors and sample stages is shown in Fig. 7(a) [Figure 7: see original paper]. The mirrors and sample holder are installed in the same vacuum vessel without any vacuum window separating the KB mirrors from the samples. A four-axis sample stage (Micronix) is used for sample positioning, as shown in Fig. 7(b). There is a 45° angle between the sample horizontal motion and the beam. The XYZ stages have a scanning precision of 200 nm. A photodiode (AXUV300C) is mounted after the sample in the vacuum vessel to measure the transmitted beam intensity (I_1).

To measure the focal spot size of the KB system, knife-edge scans using a 50 µm gold wire were performed, similar to those done by Ando et al. [**?**]. The profile was measured by scanning a 50 µm gold wire through the beam, with the intensity of the transmitted beam recorded by the photodiode (I_1) behind the gold wire. The smallest full width at half maximum (FWHM) spot size obtained at 10 keV is 4.59 × 1.22 μm^2 (h × v), as shown in Figs. 7(c) and 7(d). Since there is a 45° angle between the sample horizontal motion and the beam (Fig. 7(b)), the horizontal FWHM spot size is obtained by multiplying the

Gaussian fitting result by sin(45°). Thus, the smallest FWHM of the horizontal spot size at 10 keV is 3.25 µm. Considering the motor resolution, the focal spot size of the KB system should be 3.3×1.3 µm^2 (h × v). The photon flux at this station can be recorded by the photodiode (I_1). The highest current recorded by I_1 is 3.5$×10^{-4}$ A@10 keV (Figs. 7(c) and 7(d)), giving a photon flux above 2.48×10^{12} photons/s@10 keV.

Using the same "I_0" mentioned for the non-focusing end-station, µXANES spectra and µXAS detection can be performed in the KB vessel. A four-channel SDD (Vortex, Hitachi USA) with a collimated active area of 200 mm^2 is installed perpendicularly to the beamline for µXRF and PFY detection. Micro-XRF mapping can also be executed in the KB vessel. Due to the windowless design, micro X-ray fluorescence (µXRF) and micro X-ray absorption near-edge structure (µXANES) can only be achieved under vacuum at the microprobe end-station. Figs. 7(e) and 7(f) show the XRF mapping and XANES of a Cu net (GILDER G200-C3). The scan range is 200×200 µm^2 with a step size of 5 µm.

2. Sub-microprobe End-station After the microprobe end-station, a pair of smaller KB (SKB) mirrors is employed to focus the beam to sub-micron spot sizes. When the X-ray is focused by the SKB system, the KB mirrors and photodiode in the microprobe end-station are moved out of the beam by in-vacuum translation. Similar to the KB system in the microprobe end-station, fixed-surface-shape SKB mirrors with Ni, Si, and Rh stripes are used in the SKB system, with coating stripes translated by an in-vacuum translation mechanism. Details of the SKB mirrors are listed in Table 2 . For the SKB mirrors, the focal spot is located 230 mm and 90 mm from the center of VFM and HFM mirrors, respectively, giving a standard working distance of 60 mm from the end of the HFM mirror to the sample focal plane.

Design drawings of the SKB mirrors and sample stages are shown in Figs. 8(a) and 8(b) [Figure 8: see original paper]. Unlike the KB system, the mirrors and sample holder are installed in different vacuum vessels, separated by a Be window (8 µm thickness and 9.2 mm diameter). Compared to the KB system, the SKB system has lower flux and smaller spot size. In-situ measurements under various conditions can be performed at this station. A four-axis sample stage (Micronix) is used for sample positioning, with a 45° angle between the sample horizontal motion and the beam. The XYZ stages have a scanning precision of 50 nm. A photodiode (AXUV300C) is mounted after the sample in the vacuum vessel to measure the transmitted beam intensity (I_1).

With the same incident angles for VFM and HFM mirrors, a spot size of 0.67×0.21 µm^2 can be obtained at 2.5 keV using this SKB system, as shown in Figs. 8(c) and 8(d). A 45° angle between the sample horizontal motion and the beam is also used in the SKB sample stages (Fig. 8(b)). The horizontal FWHM spot size is obtained by multiplying the Gaussian fitting result by sin(45°). Thus, the smallest FWHM of the horizontal spot size at 2.5 keV is 0.47 µm. Considering

the motor resolution, the focal spot size of the SKB system should be 0.5 × 0.25 μm^2 (h × v). The photon flux at this station can be recorded by the photodiode (I_1). The highest current recorded by I_1 is 7.5$×10^{-6}$ A@2.5 keV (Figs. 8(c) and 8(d)), giving a photon flux above 7 × 10^{10} photons/s@2.5 keV.

μXAS and μXANES detection can also be performed in the SKB vessel. A one-channel SDD (Vortex, Hitachi USA) with a collimated active area of 50 mm^2 is installed perpendicularly to the beamline for μXRF and PFY detection. Unlike the microprobe end-station, a Be window (8 μm thickness) is used to separate the vacuum of the mirrors from the samples. Thus, micro X-ray fluorescence (μXRF) and micro X-ray absorption near-edge structure (μXANES) under vacuum or He atmosphere can be achieved at the sub-microprobe end-station.

IV. Summary

The tender energy spectroscopy beamline at SSRF has been completely constructed and opened to users in January 2024. Photon energies between 2.1–16 keV with resolutions below 1.64 × 10^{-4} (ΔE/E@2.5 keV) have been obtained at the beamline. XAS spectra obtained by transmission, PFY, TEY, and TFY modes have been made available to users with a spot size of 670 × 710 μm^2 under vacuum or He atmosphere.

Based on two sets of Kirkpatrick–Baez mirror systems, a spot size of nearly 3.3 × 1.3 μm^2 with a photon flux of 2.48 × 10^{12} photons/s@10 keV and a smaller spot size of 0.5 × 0.25 μm^2 with a photon flux of 7 × 10^{10} photons/s@2.5 keV have been obtained at the microprobe and sub-microprobe end-stations. Micro X-ray fluorescence (μXRF) and micro X-ray absorption near-edge structure (μXANES) will be opened to users in the near future.

Note: Figure translations are in progress. See original paper for figures.