Design and Commission of a Superconducting Wiggler X-ray Beamline for Advanced Materials Investigation at the National Synchrotron Radiation Research Center

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The x-ray beamline BL07A, fascinating over the use of the x-rays generated from a 3.1 Tesla, in-achromatic superconducting wiggler at the NSRRC, is designed for x- ray scattering and x-ray absorption spectroscopy for advanced materials researches in the photon energy range 5–23 keV. The beamline optics is optimized over the sequence of the collimating mirror, the double crystal monochromator, the focusing mirror and the higher harmonic rejection mirror. The beamline exhibits substantially identical performances with the ray-tracing simulation, of photon flux 10^{13} photons/s with energy resolution $\Delta E/E \sim 2 \times 10^{-4}$, focal size 250 μm $\times$ 550 μm (vertical $\times$ horizontal, FWHM), and higher-harmonic rejection ratio $\sim 10^{-4}$. Three experimental stations, namely, a diffraction/scattering station equipped with eight-circle diffractometer, an EXAFS station, and a micron beam station, all are located in the experimental hutch, upon which, a wide variety of sample environments, including temperature, pressure, electric field, and magnetic field, greatly extend the capabilities of detection in the extreme and non- ambient conditions.

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I. INTRODUCTION

Advanced experimental equipment and techniques have greatly enhanced the capability in preparing and characterizing materials of extremely high quality. Among them, the synchrotron radiation x-ray techniques can provide many unique opportunities for performing critical experiments to address the stiff needs in both materials characterization and fundamental studies for these new scientific and technological issues.

The beamline 07A at the National Synchrotron Radiation Research Center (NSRRC) is designed to provide modern x-ray techniques, namely the x-ray scattering and x-ray absorption spectroscopy for advanced materials studies. The beamline fully utilizes the brilliant hard x-rays generated from a 3.1 T in-achromatic superconducting wiggler (IASW60), giving rise to a continuous spectrum with critical energy of 4.64 keV. The beamline optics consists of one collimating mirror (CM), one double crystal monochromator (DCM),

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one focusing mirror (FM) and one higher harmonic rejection mirror (HHRM). The photon flux at sample position is measured in the level of 10^{13} photons/s, with energy resolution $\Delta E/E = 1.9 \times 10^{-4}$, and spot size 250 μm $\times$ 550 μm (vertical $\times$ horizontal).

Currently, three end stations are installed, namely an x-ray absorption spectroscopy station for extended x-ray fine structure spectroscopy (EXAFS), an eight circle diffractometer for conventional and magnetic x-ray scattering, and a micron beam station with smallest spot of 10 microns for scanning x-ray fluorescent spectroscopy. The sample environments can be controlled under high pressure ($<$ 100 GPa), low temperature ($>$ 10 K), high magnetic field (4 T), polarization (diamond phase plate) etc. In 2012, a new powder x-ray diffraction setup will be installed.

The beamline was constructed under the collaboration between the NSRRC and the Associated University Team led by National Tsing Hua University and Tamkang University. The construction started in 2007 and completed in 2009. This beamline was opened to carry out the experiments of user proposals since 2011.

II. LIGHT SOURCE

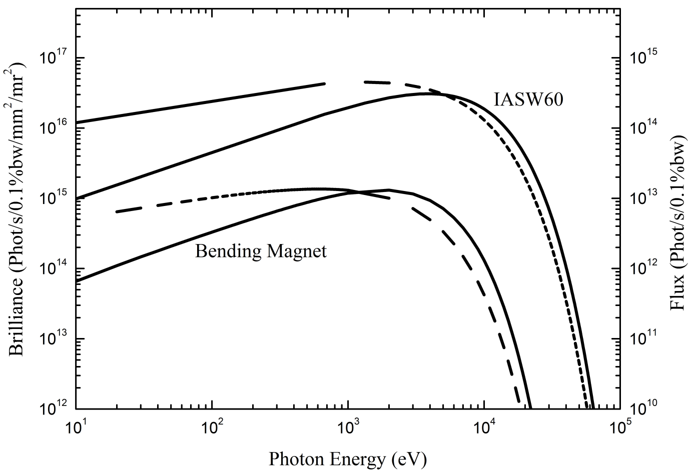

FIG. 1: Comparisons of the spectral brilliance (solid lines) and flux (dash lines) of the IASW60 and bending magnet light sources at the NSRRC.

The light source for this beamline is generated from a 14-period superconducting wiggler [1]. With magnetic period length of 61 cm, the total length of the wiggler is less than 1 meter that can be fitted into the in-achromatic section between two adjourn bending magnets in the storage ring. As a first estimate [2], the installation of the 14-period wiggler has an intrinsic critical energy E_c of 4.64 keV which is higher than 2.14 keV from the bending magnet. Additionally, an estimate of 28 times of incoherent add-up photon flux is obtained. Taking 2 mrad span of the central radiation cone, the source spectra is calculated by software SHADOW, as depicted in Fig. 1. The spectra, in energy range of $5 \sim 23$ keV, exhibit a brilliance of $10^{15} - 10^{16}$ photons/(s×mrad^2×mm^2×0.1%bw) and photon flux $10^{13} - 10^{14}$ photons/(s×0.1%bw). The spectral of the bending magnet source is also shown in Fig. 1 for comparison. The characteristic parameters for the wiggler are tabulated also in TABLE I.

TABLE I: Characteristics of Light Source (IASW60)

Magnetic Field	3.5 T
Number of Periods	14
Magnetic Period	6.1 cm
Deflection Parameter K= 0.934	17.7
Source Size (at 15 keV)	370 μm×160 μm (H×V)
Source Divergence (at 15 keV)	6.86 mrad×0.25 mrad (H×V)
Total radiation angle	±6 mrad [68% of flux @ 15 keV: ±3 mrad]
Photon Flux @ 15 keV (400mA)	4.7×10^{13} photons/(0.1%bw× mrad)
Beam Brightness @ 15 keV (400mA)	9×10^{15} photons/(0.1% bw×mm^2×mrad^2)
Total Power @ 400 mA	2.7 kW

III. BEAMLINE

The beamline consists of four major optical components, starting from upstream, the tangential-cylindrical CM, DCM, the toroidal FM and the plane HHRM. The beamline layout is depicted in Fig. 2.

The water-cooled silicon CM provides functions of beam collimation, heat load reduction and higher-ordered harmonic (HOH) suppression. The incident angle is 3.5 mrad. The mirror is bent to a radius of 4.8 km by a cylindrical roller bender. The half part of the CM surface is coated with Pt (25nm) and Rh (5nm) to extend the usable energy up to 23 keV, while the rest half part of the CM is left uncoated. In order to suppress the HOH contamination, the CM is operated at bared silicon surface for photon energy $5 \sim 8$ keV and operated at Pt/Rh coated surface for $8 \sim 23$ keV.

The DCM is equipped with a pair of silicon (111) crystals. With the help of CM, the DCM diffracts the nearly parallel incident beam with a Darwin-width-limit energy resolution, in the present case, $\Delta E/E \sim 1.4 \times 10^{-4}$. The adapted single-cam design assures the parallelism of the two diffractive crystals, measured within 10 arc second in $5 \sim 23$ keV. A water-cooled piping with flow rate of 2 liter/min is running underneath the first crystal.

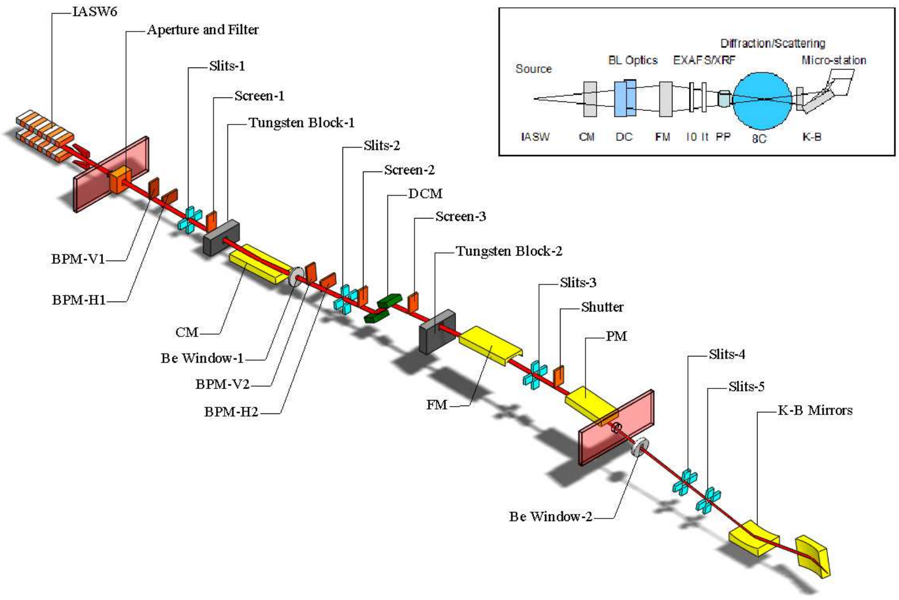

FIG. 2: Optical layout of the beamline. The layout of experimental stations is shown in the inlet.

The FM, with its toroidal shape, focuses the monochromatic beam both in vertical and horizontal direction. It is positioned in beamline in such a way that the distance ratio between the source and image is roughly 2:1, giving rise to a minimal spherical aberration at the sample position [3]. The mirror surface is fully coated with Pt (25nm) and Rh (5nm) to provide sufficient HOH suppression for most measurements.

The plane HHRM with similar surface coating configuration with CM provides more HOH suppression in the cases that the HOH are stringent. The combination and degree of HOH suppression is tabulated in TABLE II.

Several beamline components for beam shape conditioning and diagnostic, including, four-jaw slits, screen and photon-beam-position monitor (PBPM) is shown in Fig. 2.

The expected beamline performance was confirmed by ray tracing simulation SHADOW. With horizontal acceptance of 2 mrad and ring current at 360 mA, the simulated photon flux is shown in Fig. 3. Several factors cause the spectral flux drop have been taken into consideration, including the direct absorption by Be windows, spatially filtered by slits, photons with incidence angle higher than critical angle absorbed by mirrors, spectral bandwidth narrowed by DCM, etc. Also shown in Fig. 3 is the ultimate flux after a pair of Kirkpatrick-Baez (K-B) mirrors for micron focusing.

TABLE II: Estimated third-order harmonic suppression ratio for various mirror combinations at the end station of EXAFS (E1), diffractometer (E2) and focusing station (E3)

Energy (keV)	Source Flux (photons/s)	End Station Flux (photons/s)	Flux Ratio (15 keV/5 keV)
5 – 8 keV: Si surface of CM			
5	5.82×10^{14}	3.87×10^{12} (E1) 4.61×10^{12} (E2) 2.58×10^{11} (E3)	6.82×10^{-4} (E1) 6.03×10^{-4} (E2) 5.78×10^{-4} (E3)
15	9.87×10^{13}	2.75×10^{9} (E1) 2.78×10^{9} (E2) 1.49×10^{8} (E3)	
Energy (keV)	Source Flux (photons/s)	End Station Flux (photons/s)	Flux Ratio (24 keV/8 keV)
8 – 23 keV: Rh/Pt (5/25 nm thickness) surface of CM			
8	3.56×10^{14}	7.26×10^{12} (E1) 8.42×10^{12} (E2) 5.09×10^{11} (E3)	5.96×10^{-4} (E1) 5.26×10^{-4} (E2) 1.68×10^{-4} (E3)
24	1.71×10^{13}	4.33×10^{9} (E1) 4.43×10^{9} (E2) 8.57×10^{7} (E3)	

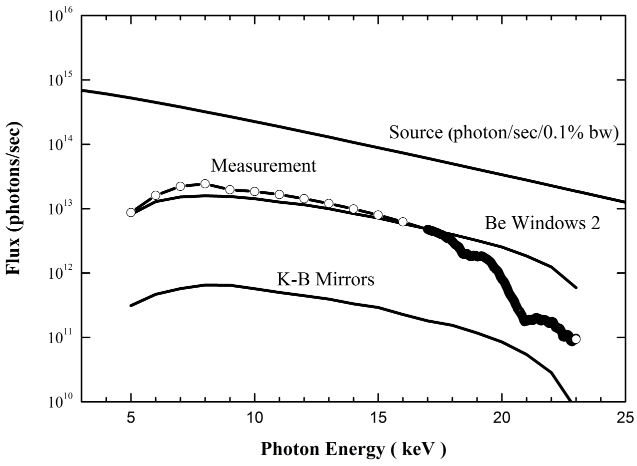

FIG. 3: Comparison between the measured flux (circle-line) at the center of the diffractometer, and the calculated ones (lines) of the source, after Be-window 2 and after the K-B mirror.

IV. END-STATIONS

The experimental station is designed to provide as feasible as possible for several x-ray techniques. In the present stage, there are three end stations, starting from upstream, namely an x-ray absorption spectroscopy station for extended x-ray fine structure spectroscopy (EXAFS), an eight circle diffractometer for conventional and magnetic x-ray scattering, and micron beam station (Fig. 2). The beamline optics focuses the beam at the center of the diffractometer. Despite that the three end stations can be operated independently; they can also be served as a complementary part to each other. For example, the EXAFS station can be served as a beam flux as well as beam position monitor for the diffractometer. The focal position at the diffractometer center is also served for those experiments requiring stringent focal spot such as high pressure X-ray absorption fine structure. In this case, an anvil diamond cell is positioned at the center of diffractometer. The focal spot is served as the virtual source for the downstream K-B mirror pair of the micron beam station.

The EXAFS station is equipped with three ionization gas chambers and a Lytle detector for transmission type as well as fluorescence type measurements, respectively. The nominal position of sample stage is placed 1.5 meters upstream to the center of diffractometer. In the transmission mode, the three ionization gas chambers are served to measure the incident flux (I_0), transmitting flux (I_t) after samples and transmitting flux (I_f) after a standard reference foil. Under the circumstance of dilute samples, the Lytle detector is employed for measuring fluorescent signals. When combined with a low temperature cryostat the station is able to perform EXAFS at low temperatures. The high pressure (HP) x-ray absorption spectroscopy can be conducted by rearranging the station in the way that the anvil diamond cell is placed at the center of diffractometer. Only the transmission mode is adoptable for HP-EXAFS.

The diffractometer is stationed at the center of the experimental hutch, coincident with the focal spot of the beamline. The diffractometer comprises eight rotational axes, among which, four, two and two axes are assigned for rotating the sample, detector and crystal analyzer, respectively [4]. Depending on experimental purpose, the users can choose to perform diffraction/scattering in vertical or horizontal geometry. The crystal analyzer can be replaced by a polarization stage for those experiments that with polarization dependence, for example, the magnetic scattering on the charge and spin modulations in iron-based oxides. The polarization of the incident beam can be altered by a diamond phase plate operating in Bragg geometry [5]. A cryostat with three precise orthogonal translational stages is mounted to cool down the sample temperature to 7K. The anomalous x-ray scattering can be conducted by tuning the energy of incident x-rays across the absorption edge of specific elements. The fluorescence signal is detected by an energy dispersive detector pointing perpendicular to the incident beam.

The micro-focusing station comprises a pair of platinum-coated mirrors posed in Kirzpatrick-Baez geometry in the way that the upstream mirror focuses the beam vertically while the downstream mirror focuses the beam horizontally. The demagnification ratios of the mirrors are 7.3 and 15.7 in vertical and horizontal directions, respectively. The mirror curvatures were measured by in-house long trace profiler [6] and corrected by benders before being installed into the station. The beamline focal spot at the center of diffractometer is treated as the virtual source for the K-B mirrors. The beamline slits 1 (Fig. 2) is employed to close and suppress the penumbra due to the finite source size and the mixture of phase space caused by toroidal FM. The sample is mounted on a stage with three orthogonal translational movements and two rotational axes. An L-shape beam stopper is placed between the sample and the K-B mirror to prevent the light leakage from the edge of mirror. The scattering x-ray from the beam stopper is monitored by a scintillation counter (Bicron) for calibrating the incident flux. An ionization gas chamber and a Lytle detector are used to detect the transmission and fluorescence signals, respectively.

V. COMMISSION OF BEAMLINE OPTICS

The total photon flux delivered by the beamline was measured at various x-ray energies by a gas ionization chamber filled with pure nitrogen gas and positioned at the center of eight-circle diffractometer. Fig. 3 shows the comparison among the light source, the calculated flux after Be window 2 and the measured flux. It is noticed that in the low energy region below 18 keV, the measured flux is well matched to the calculated one in the level of $10^{13} \sim 10^{12}$ photons/s. Above 18 keV, the measured flux is yet lower than the calculated one and shows oscillating feature due to the finite coating thickness on CM. One can calculate the coating thickness to be roughly 30 nm by applying conventional x-ray reflectivity formula. The earlier energy drop- off than the designed 22.5 keV is expected due to the adaption of toroidal FM over which the photons with incident angles larger than the designed 3.5 mrad can exist near the outer part of the toroidal mirror.

The energy resolution of DCM was measured by rocking the downstream crystal of DCM while keeping the upstream fixed. The measured energy resolution is a resultant result of the source size, the divergence after the CM, and the intrinsic Darwin width after the crystal of DCM. After divided by $\sqrt{2}$ for deconvoluting the effect from double crystals, the full-width-half-maximum (FWHM) of the measured rocking angle was applied to determine the energy resolution by the $\Delta E/E = (\Delta\theta)_{FWHM} \cot\theta_B$, where θ_B is the Bragg angle of the crystal been used. A well match between the measured energy resolution and the calculated one is shown in Fig. 4 at various energies. An abrupt increase at 8 keV is artificial due to the slightly mechanical off- alignment introduced by the change between two different coating parts of the CM mirror during the experiment.

Fig. 5 shows the measured spot size at the center of diffractometer at various energies. The spot size was measured by scanning a pair of sharp tungsten knifes independently in vertical and horizontal directions. The surface roughness of the knife is less than 5 μm verified by optical measurements. Minor adjustment to re-align the major optical components has been done at various energies before measurement. The spot size is about 250 μm and 550 μm (vertical$\times$horizontal, FWHM), well matched to simulation results. The measured maximum drift of the spot position from 5 keV to 23 keV are (not shown in the figure) 5 μm and 15 μm in vertical and horizontal directions, respectively.

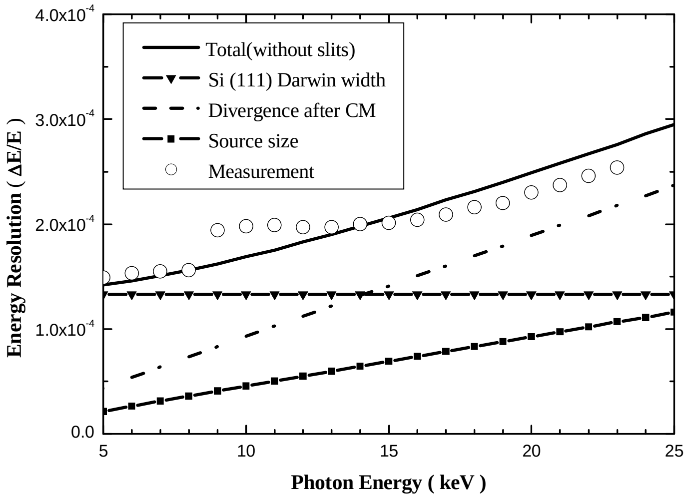

FIG. 4: The measured (circles) and calculated (lines) energy resolution of the beamline.

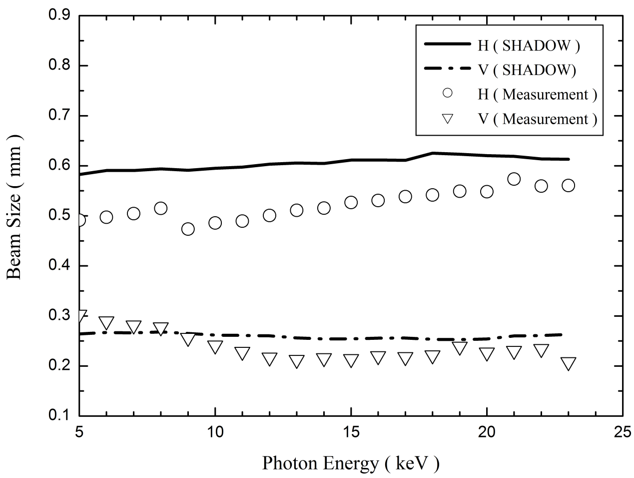

FIG. 5: The measure (circles and triangles) focal spots and the calculated ones (lines) at the center of the diffractometer.

VI. CONCLUSION

A high flux and high energy resolution hard x-ray beamline was built at NSRRC. The beamline optics is designed to fulfill a wide range need in various energy ranges and research fields. The beamline performance has been well verified. The focal spot is measured in the level of 250 μm $\times$ 550 μm (vertical *times* horizontal), in the energy range 5–23 keV with flux level 10^{13} photons/s. Three end stations are built for x-ray scattering, x- ray absorption spectroscopy and micron applications. Several non-ambient sample environments, including high pressure, low temperature, high magnetic field, have been added up step by step to the end stations. This beamline is commissioned under the task force of the Associated University Team. The beamline is opened for users to carry out their proposals since 2011.

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