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DESIGN OF MULTIPLE EXPERIMENTAL MODELS FOR PINK SAXS STATION

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Abstract

Pink small angle X-ray scattering station is dedicated to performing scattering related experiments. A classical in air planar undulator is adopted as the beam source. The fundamental radiation can be adjusted within 8-12 keV through altering the magnetic gaps. Monochromatic beam and pink beam can be switched through moving in and out of the monochromator. Three set diamond compound refractive lenses with different curvatures are employed to focus the 12 keV monochromatic beam to achieve different focusing modes. With the help of a flexible vacuum detector tube, varies experimental models could be carried out easily.

INTRODUCTION

Small angle X-ray scattering (SAXS) is a powerful technique for studying nano materials [1]. As for numerous scattering experiments, different experimental demands are proposed by users. For example, as monochromatic beam is not necessary for some SAXS measurements [2], they prefer higher beam flux to shorten exposure time and to carry out higher time resolved scattering experiments at the expense of sacrificing energy resolution and beam size. Conversely, some researchers hope to carry out fine experiments with higher energy resolution and small beam size. In order to accommodate these seemingly contradictory needs of diverse users, a muti-functional SAXS station is under construction at HEPS.

HEPS [3] (high energy photon source), which is a 6 GeV synchrotron radiation facility with low emittance, provide perfect conditions for meeting these requirements. The high flux pink beam, which is from the fundamental radiation of the undulator, will be used directly after reflected by a pure silicon reflector to perform high time-resolution experiments. Monochromatic beam, which is obtained by a horizontal double Si(111) crystal monochromator, also can be used alternately to perform high energy resolution experiments. With the help of flexible monochromator, focusing element and a SAXS tube, the main parameters of SAXS station can be adjusted conveniently, which are reflected in the following aspects. First, the pink beam and monochromatic beam can be switched through moving in and out a horizontal double crystal monochromator. Second, the incident beam energy can be altered through adjusting the gaps of undulator at the range of 8-12 keV with the help of a monochromator. Third, for the commonly used 12 keV monochromatic beam, four types of focusing modes can be changed through changing on-line diamond CRLs. Four, the different range of scattering angle can be altered easily by the help of a flexible tube. This design can meet the vast majority needs of users. The main specifications of the SAXS beamline is shown in Table 1.

The available experimental techniques include single SAXS, WAXS, USAXS, SAXS-CT, ASAXS and combined SAXS/WAXS/USAXS, etc. The measuring mode includes transmission and grazing incidence, static and dynamic (in situ, time resolved) measurements. The time resolution lies in microseconds to seconds based on different sample environments and detectors. Some sample environmental devices, including in-situ heating, in-situ growing, in-situ tension, will be equipped in our station.

Table 1: Main Specifications of the SAXS Beamline at HEPS

Pink beam	
Energy range	8-12 keV
Flux at sample	~10^{15} ph/s
Beam size @sample	500 µm × 500 µm
Energy resolution	1.5 %
Scattering angle	0.001°~50°
Monochromatic beam	
Energy range	8-30 keV
Flux at sample	~10^{13} ph/s
Beam size @sample	300 µm × 300 µm 14 µm × 6 µm;
Energy resolution	~2×10^{-4}

DESIGN

Overall Description

The basic idea of design, sketched in Fig. 1, is that the monochromator and the focusing devices (CRL) can be moved in and out of the beamline. Without monochromator and CRLs, the quasi monochromatic beam from the fundamental radiation of the undulator also can be directly collimated to measure the sample. A set of three-slits is used to reduce the scattering background, which are not drawn in the diagram.

We specify the beam source as the starting position (0 m). The front-end of the beamline is about 32 m long, which is mainly used to provide radiation protection and heat reduction to the downstream devices. The beamline starts from the front-end ratchet wall exit (32 m from the source) and ends at 49.9 m, which is located in the first optical

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experimental hutch (FOE). The beamline's major function includes beam deflection, beam monochromatizating, beam focusing, collimation, monitoring, vacuum maintenance and bremsstrahlung stop. In the following, we only describe the main elements, including undulator, deflector, monochromator, CRLs and vacuum SAXS tube.

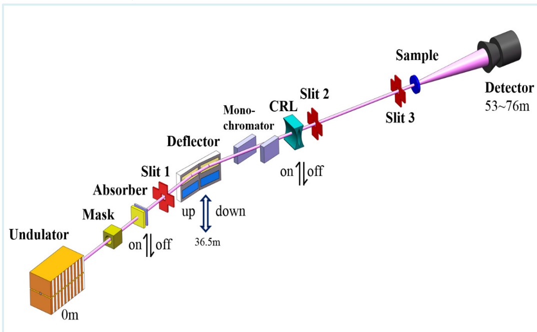

Figure 1: Main composition of pink SAXS beamline.

Insertion Device (Undulator)

In order to fully utilize the high energy and low emittance of HEPS, a typical planar in-air undulator (IAU25) undulator [4] is chosen as the light source, whose fundamental radiation could be directly used as the pink beam. The undulator has been optimized to provide fundamental radiation energy at the range of 8 ~ 12 keV with magnets gap of 11.29 ~ 17.40 mm for high flux application. The source spot has 43.7 µm × 17.5 µm FWHM size and 9.6 µrad × 9.1 µrad FWHM diverfence at 12 keV with 0.1% BW, which will be our commonly used energy in the future for users. The maximum angular flux density is 6.2E18 phs/s/mrad2/0.1% BW.

Deflector

To use the fundamental radiation of the source, the higher harmonics must to be suppressed. A horizontally deflecting system is installed firstly at the downstream of the source. The deflection system is employed to achieve the following functions: (1) Deflect the synchrotron radiation for deviation from the direct bremsstrahlung radiation. (2) Suppress the higher harmonic. (3) Reduce the thermal load for the downstream devices. In order to improve the beam stability and further suppress the higher harmonic, a set of twin separate flat single-crystal silicon mirrors (sketched in Fig. 1) are accepted. As the two mirrors are arranged with a fixed 5 mrad angle, the angle between incident beam and exit beam is fixed at 10 mrad, which is not changeable with the rotation. As the cut-off energy of pure silicon reflection is about 14 keV when the grazing angle is about 2.5 mrad, the higher harmonics from the 2nd will be suppressed efficiently. The specification of deflection double mirror is shown in Table 2.

Higher energy X-ray also be needed to measure some metal samples. In order to obtain X-ray beam higher than 12 keV, the metal Pt would be evaporated with a width of 5 mm on the upper side of the deflectors as showed in Fig. 1. At the grazing angle of 2.5 mrad, the cut-off energy of Pt reflection can be increased to 30 keV, which covers the 1st, 2nd and 3rd harmonics. The cut-off energy can be altered by vertically raising and lowering the deflection mirrors. Additionally, it should be noted that the monochromator must be used when the Pt film is employed as deflector.

Table 2: Specification of Deflection Double Mirror

Specification	**Value**
Mirror material	Sigle-crystal silicon
Reflective surface	Polished Si, Evaporated Pt
Dimension	200 × 50 × 50 mm^3
Surface error	≤ 0.3urad
Surface roughness	≤ 3Å
Angle between two mirrors	5 mrad

Monochromator

Following the deflecting system, a normal horizontal double-crystal Si(111) monochromator is equipped. The specification of monochromator is illustrated in Table 3. Pink beam and monochromatic beam can be switched through moving the first crystal in or out of the beam path. The working energy range is between 7 to 30 keV, which is corresponding to the cut-off energy of Pt reflection at the grazing angle of 2.5 mrad. The horizontal offset between the pink beam path and the monochromatic beam path is only 15 mm. Thus, the two different beam paths can be compatible in the same tube easily.

Table 3: Specification of Monochromator

Specification	**Value**
crystal	Si(111)
Energy range	7 ~ 30 keV
Deflecting direction	horizontal
Minimum energy step	0.1 eV
Exit offset	15 mm
Cooling method	liquid nitrogen

Compound Refractive Lenses (CRL)

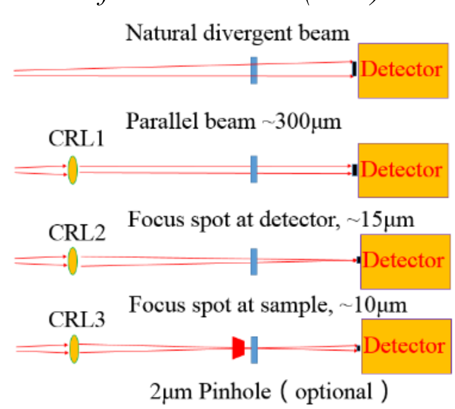

Figure 2: Four type focusing modes (only for the12 keV monochromatic beam).

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As for the commonly used 12 keV monochromatic X-ray, three single diamond CRLs are employed to obtain different focusing modes. The CRLs are mounted and controlled by a homemade tranfocator, which is placed at the downstream of monochromator. Without any CRL, we can use the natural divergent beam and large beam spot to carry out the normal SAXS measurements. The larger beam spot can improve the SAXS statistics. By using the first set CRL, we can obtain parallel beam with circle shape of about 300 μm diameter. As the divergence angle is basically zero, the parallel beam is conveniently for the functional extension. By using the second set CRL, the incident beam is focused at the detector (about 15 μm). Then we can obtain the best angle resolution with a smaller scattering angle and lower scattering background. By using the third set CRL, the beam can be focused at sample position with the size of about 10 μm × 6 μm. Then we can carry out some high spatial resolution experiments and use small sample holders, such as DAC, capillary cell, etc. To carry out higher spatial resolution SAXS measurements, a scatterless pinehole with pore size of 2 μm is being considered as future upgrade plan. The schematic diagram of focusing modes is shown as Fig. 2.

Vacuum SAXS Tube and Detectors

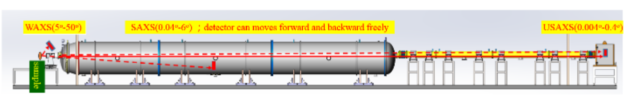

Figure3: The schematic diagram of the vacuum SAXS tube and the detectors.

A 23 m long versatile SAXS tube is shown in Fig. 3. In our station, the sample is fixed at the 53 m from the source. Three vacuums compatible Eiger2 detectors will be installed along the tube. The WAXS detector is suspended diagonally above the sample to collect about −5°~ 50° scattering signals. The SAXS detector, which is used to collect scattering angle between 0. 04° and 6°, is installed in the front large tube with a diameter of 1.5 m and a length of 14 m. The detector can move freely within the tube according to experimental requirements. The USAXS detector, which is used to collect 0.001°~ 0.1° scattering signals, is placed at the end of tube. The vacuum degree of the tube is less than 1.0 Pa. In addition to normal individual measurements, the three detectors can work simultaneously to collect the whole large angle range from 0.001° to 50°. Two kinds of beamstop used for transmission mode and grazing incidence mode respectively, are installed in front of the SAXS and USAXS detectors.

CONCLUSION

As a user facility, to meet the different demands is our goal of effort. Beside normal monochromatic SAXS experiments, we also achieve abnormal high photon flux to perform high time resolved scattering experiments at the expense of energy resolution on our station. The beam energy, photon flux, beam sizes and SDD (sample to detector distance) also can be altered conveniently according to the actual demands of users. Various experimental techniques, including SAXS, WAXS, USAXS, ASAXS, GiSAXS and combined SAXS/WAXS/USAXS, etc., can be performed on our station. In the near future, some home-made in-situ sample environmental devices will be equipped.

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