RESEARCH ARTICLE | JANUARY 15 2019

# Design of new sub-micron protein crystallography beamline at SSRF

Bo Sun; Yuzhu Wang; Ke Liu; Qisheng Wang; Jianhua He



*AIP Conf. Proc.* 2054, 060028 (2019)
https://doi.org/10.1063/1.5084659



# Design of New Sub-micron Protein Crystallography Beamline at SSRF

Bo Sun [1,a)], Yuzhu Wang [1], Ke Liu [1], Qisheng Wang [1], and Jianhua He [1]

[1]*Shanghai Institute of Applied Physics, Chinese Academy of Sciences, Shanghai 201204, China*

[a)] *Corresponding author: sunbo@sinap.ac.cn*

**Abstract.** In Phase-II project at Shanghai Synchrotron Radiation Facility (SSRF), a sub-micron protein crystallography beamline is designed to achieve the focused beam size from 0.5 μm to 20μm. The high harmonics in the spectrum of a cryogenic permanent magnet undulator (CPMU) is adopted to provide X-rays in 5~25keV. A $LN_2$ cooled double crystal monochromator (DCM) for high energy resolution mode and a double multilayer monochromator (DMM) for high photon flux mode will be used to monochromatize the beam while two-stage focusing optics will be selected to focus the beam. A pair of pre-focusing KB mirrors will generate a secondary source while a second pair of KB mirrors will image the secondary source to a half micron size beam at the sample position. The experimental end station will provide high-resolution native data collection with experimental phasing method (SAD/MAD), in-situ 96-well plate data collection, raster strategy for tiny crystals, and serial crystallography method.



## INTRODUCTION

Many important proteins, such as the membrane proteins including G-protein coupled receptors (GPCRs), form notoriously small, weakly diffracting, and radiation-sensitive crystals. These crystals cannot be studied on conventional crystallography (MX) beamlines, e.g. several operational beamlines at SSRF. When we look at the parameters of the current MX beamlines at SSRF, shown in Table 1, there is a gap between these beamlines and the requirement of the micro-crystallography, which is keen to match the size of tiny crystals with micron or sub-micron beam [1, 2].

**TABLE 1.** Parameters of current MX beamlines at SSRF

| Parameters | BL17U1 | BL17B | BL18U | BL19U1 |
|---|---|---|---|---|
| Energy Range(keV) | 5~18 | 5~20 | 5~18 | 7~15 |
| Focused beam size ($\mu m^2$) | 67×23 | 150×180 | 10×7 | 130×80 |
| Flux at sample position(phs/s) | $6\times10^{12}$ | $3\times10^{11}$ | $7\times10^{11}$ | $1\times10^{12}$ |
| Beam divergence($mrad^2$) | 0.3×0.1 | 1.5×0.2 | 0.7×0.3 | 0.1×0.1 |

We tried to use collimator or further focus the beam by compound refractive lens (CRL) to obtain small beam [3], but the intensity is too low to for some membrane crystals' diffraction, which are sometimes the cutting-edge research from the user community. In fact, the users want smaller and higher intensity beam for MX [4]. Thus we proposed to build a new sub-micron MX beamline, which is named as membrane protein crystallography beamline (MPX) in Phase-II project.

*Proceedings of the 13th International Conference on Synchrotron Radiation Instrumentation – SRI2018*
AIP Conf. Proc. 2054, 060028-1–060028-4; https://doi.org/10.1063/1.5084659
Published by AIP Publishing. 978-0-7354-1782-3/$30.00

# OPTICAL DESIGN

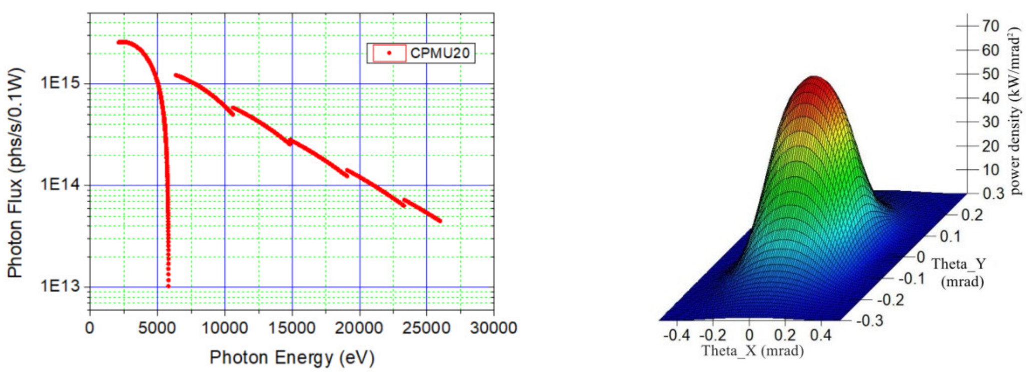
After the investigation and related evaluation, the designed specifications of these beamline are listed in the Table 2. Because there are DCM and DMM in the beamline, the different performance of energy resolution and flux are labeled.

**TABLE 2.** Parameters of the new MPX beamlines

| Parameters | MPX |
|---|---|
| Energy Range (keV) | 5~25 |
| Energy Resolution | $2\times10^{-4}$ (DCM) |
| Minimum Focused beam size ($\mu m^2$) | 1×0.5 |
| Flux at sample position (phs/s) | $1.6\times10^{12}$ (DMM) |
| | $1.6\times10^{11}$ (DCM) |
| Beam divergence($mrad^2$) | 2.5×1.0 |

To offer the higher flux beam at the high energy (25 keV), a long CPMU undulator is chosen as the source of this beamline. There are 160 periods in the 3.2 meters CPMU which could provide ≧1T peak magnetic field. The development of this undulator is done by SSRF mechanical and magnet group.

The CPMU20 have a good tuning curve, shown in Figure 1(a). There is the energy gap between 5.8 keV to 6.36 keV, which is acceptable in MX experiment. With this high performance undulator, we obtain the higher flux with high heat load. The max power is 7.44 kW, and max power density is 75.4 kW/mrad$^2$, shown in Figure 1(b).




(a) (b)

**FIGURE 1.** The tuning curve (a) and power density (b) of CPMU20

After the carefully calculation, at 12 keV, the CPMU20 could provide 3 times photon flux than the same length in-vacuum undulator (IVU20). At the high energy part, 25 keV, it could provide 6 times photon flux than the IVU20. High energy (short wavelength) will enhance the signal-noise ratio and the quality of diffraction data [5, 6]. That is one of the reasons to change the undulator from IVU to CPMU.

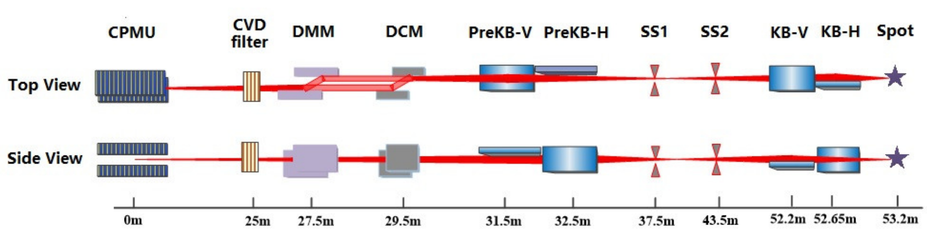



**FIGURE 2.** The schematic beamline optics

Figure 2 shows the schematic beamline optics. Because the spot size range is from 0.5 µm to 20µm, two sets of demagnification ratios are designed to focus the beam to 1 µm or 10 µm. The other spot size could be obtained by changing the size of SS.

DCM is selected for high energy resolution usage mode, and DMM is selected for high photon flux usage mode. In each mode either DCM or DMM can be moved into the beam while the other moves out. Water-cooled slits and CVD filters are set before the DMM, which will absorb a large amount of the heat load. However, the density of heat power absorbed by the first multilayer/crystal is still very high. Both the DMM and DCM will be cooled with circulating liquid nitrogen.

To enhance the stability performance of the monochromators, H-axis crystals positioning system is selected. DCM and DMM have the same beam off-set, which is 15 mm. This small beam offset will be helpful to reduce the crystal length and movement distance of DMM.

A pair of pre-focusing KB mirrors are used to generate the secondary source, where the slits SS (SS1 and SS2) are positioned. The pre-focusing KB mirrors are elliptical shape bended from flat mirrors. Thinking about the energy range 5~25 keV, with careful calculation and comparison, the mirrors will be coated by 50 Å rhodium (Rh) and 450 Å iridium (Ir), which is shown with green curve Rh-Ir in Figure 3.

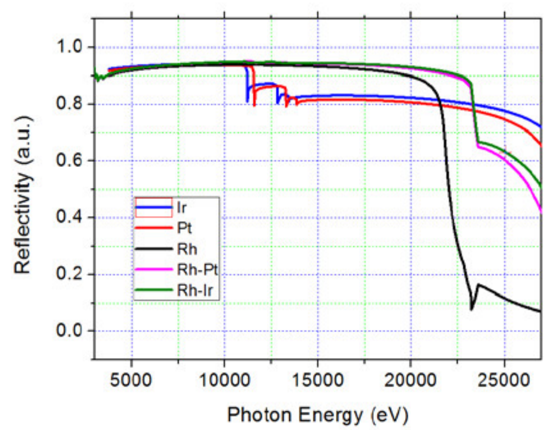



**FIGURE 3.** The material selection for coating on mirrors



Two sets slits are set as the second source. The spot size will be 1 µm when the vertical demagnification ratio is set at 77.2 and the horizontal is 179. The 0.5 µm beam will be achieved by diminishing the slits. The spot size will be 10 µm when the vertical demagnification ratio is set at 22.8 and the horizontal is 49.2. The beam could be adjusted from 0.5 µm to 20 µm by the control of slits based on the experimental requirement.

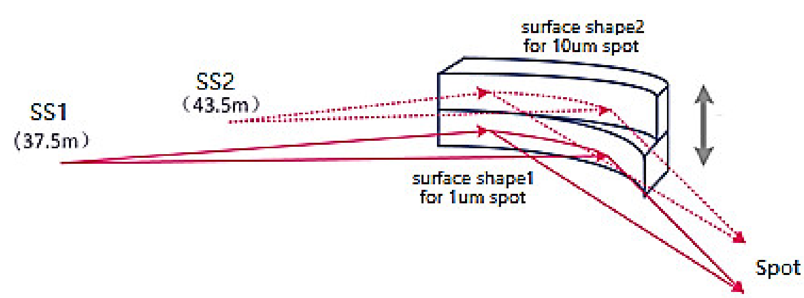



**FIGURE 4.** 'quick' switch method to change the beam size

To let the users achieve quick and reliable beam size changes, the second KB mirrors are fixed shaped with two surface shapes, shown in Figure 4. One surface shape will focus the beam from SS1 to 1 µm at the spot position, and another surface shape will generate 10 µm. Two surfaces could be switch by motors very fast. The slope error on the surface to realize 1 µm spot is less than 60 nrad, which is a very challengeable requirement.

## EXPERIMENTAL END STATION

The experimental end station of the MPX beamline will be installed to support these functions.

- High-resolution native data collection with experimental phasing method (SAD/MAD).
- High capacity auto-mounting robot, plus in-situ 96-well plates data collection
- Fast data collection, fine ϕ-slicing data collection.
- Rastering strategy for tiny crystals, and serial crystallography method for crystals on mesh/membrane mounted on gonio-head.

Currently the optical design of the beamline has been done and the manufacture or producing work is going on. The design of the end station, especially some part of the control system, is still open. More suggestions will be adopted from the end users to modify the design later.

## CONCLUSION

The design of the new sub-micron MX beamline, MPX, is described. It will be a high performance MX beamline with small spot size and high photon flux. CPMU and DMM are adopted to satisfy the flux-hungry experiments. An easy tuning of the beam-size will be made possible by adjusting the SS size and switching fixed shape mirrors. The commissioning of the whole beamline is planned at 2020.

## ACKNOWLEDGMENTS

The MPX beamline is part of the Phase-II project of SSRF, which will build 16 new beamlines. It is one of the two beamlines funded by ShanghaiTech University. The beamline design benefited from the assistance of colleagues from accelerator group, mechanics (or engineering) group, and optics group of SSRF. The scientists from the iHuman Institute of ShanghaiTech University also presented great suggestions.



## REFERENCES

1. J.H. He, X.Y. Gao. European Physical Journal Plus, **130**(2), Feb. 2015
2. Q.S. Wang, F. Yu, S. Huang, B. Sun, K.H. Zhang, K. Liu, Z.J. Wang, C.Y. Xu, S.S. Wang, L.F. Yang, Q.Y. Pan, L. Li, H. Zhou, Y. Cui, Q. Xu, T. Earnest, J.H. He. Nuclear Science and Techniques, **26**(1):12-17 (2015)
3. Q.S. Wang, F. Yu, Y. Cui, K.H. Zhang, Q.Y. Pan, C.Y. Zhong, K. Liu, H. Zhou, B. Sun, J.H. He. Appl. Phys. Review of Scientific Instruments, **88** (7) :073301 (2017)
4. J.L. Smith, R.F. Fischetti, M. Yamamoto. Current Opinion in Structural Biology, 22(5): 602-612, 2012
5. J. Jakoncic, M. Di Michiel, Z. Zhong, V. Honkimaki, Y. Jouanneau, V. Stojanoff. Journal of Applied Crystallography (2006). 39, 831-841
6. K. Takeda, H. Miyatake, S.Y. Park, M. Kawamoto, N. Kamiya, K. Miki. Journal of Applied Crystallography 37(6):925-933 (2004)