Improvement in stability of SPring-8 X-ray monochromators with cryogenic-cooled silicon crystals

Hiroshi Yamazaki¹,², Haruhiko Ohashi¹,², Yasunori Senba¹,², Tomoyuki Takeuchi¹,², Yasuhiro Shimizu¹, Masayuki Tanaka¹, Yasuhisa Matsuzaki¹, Hikaru Kishimoto¹, Takanori Miura¹, Yasuko Terada¹,², Motohiro Suzuki¹,², Hiroo Tajiri¹,², Shunji Goto¹,², Masaki Yamamoto¹,², Masaki Takata¹,², and Tetsuya Ishikawa¹,²

¹ Japan Synchrotron Radiation Research Institute (JASRI), 1-1-1, Kouto, Sayo-cho, Hyogo 679-5198, Japan
² RIKEN SPring-8 Center, 1-1-1, Kouto, Sayo-cho, Hyogo 679-5148, Japan

E-mail: yamazaki@spring8.or.jp

Abstract. SPring-8 standard double-crystal monochromators cooled with liquid nitrogen are being improved for providing a stable supply of intense nanometer-focused X-ray beams. The instability originates from the vibration and thermal deformations of the various stages of the monochromators: the former is caused by turbulent flow of the liquid nitrogen, and the latter is mainly due to unwanted cooling from the liquid nitrogen. A low-vibration flexible tube was devised to stabilize the coolant flow by covering the corrugations of the flexible tube with an alumina fiber textile. To achieve thermal insulation, we inserted a machinable ceramic block and a copper plate between the cooled crystal holder and the stages; the temperature of the copper plate was controlled to within ±0.01 °C using a sheet heater and a proportional-integral-derivative current controller. As a result, the vibration was reduced from 1″ to 0.15″ in terms of the misalignment angle between the two crystals, and a vertical focus size of 230 nm was achieved by demagnification projection of the real light source onto the focal plane. The angular instability due to the thermal deformation was suppressed to a rate of less than 0.2″/h. Furthermore, we discuss ongoing improvements for further stabilization.

1. Introduction

The nanometer-focused synchrotron X-ray is one of the most advanced probes for investigating materials for green nanotechnology. The standard undulator beamlines of SPring-8 have the potential to concentrate 15-keV X-rays with an intensity of $3 \times 10^{11}$ photons/s into an area of 100 nm × 100 nm with the use of Kirkpatrick–Baez mirrors [1]. The beam is generated by projecting the demagnified image of the real light source onto the focal plane in the vertical direction. The vibration of a beamline monochromator enlarges the vertical focal spot size because the effective light source is broadened by the fluctuation in the direction of the outgoing beam. We therefore require a high level of stability for the monochromators.

In our previous paper [2], we reported improvements in the stability of SPring-8 standard double-crystal monochromators (DCMs) [3] with water-cooled crystals. The instability of the
beam intensity occurs because of the loss of parallelism between the two crystals. Fast-varying instability originates from vibrations, and the slow-varying one arises from thermal problems. The improvements were mainly classified as addressing the following three issues: reduction in vibrations by smoothing water flow, temperature control of fine stepper stages, and reduction in radiation damage. We are presently focusing on improving DCMs with liquid nitrogen-cooled crystals in a similar manner. In this paper, we describe low-vibration flexible tubes, temperature control, and a radiation shield developed at SPring-8 (section 2); the present performance of the DCMs (section 3); and future plans (section 4).

2. Improvements
2.1. Low-vibration flexible tube
The liquid nitrogen paths of the DCM consist of pipes with a 0.5-in diameter and commercially available flexible tubes made out of stainless steel. Corrugations of the flexible tubes most probably disturb smooth flow and yield vibrations. Large vibrations remain when the flexible tube part is designed to be short. A simple solution is to insert smooth inner tubes in order to cover the corrugations of the flexible tubes. Polyurethane tubes are used for the water paths [2]. However, they harden at low temperatures.

Inner tubes must have flexibility, cold resistance, and radiation tolerance. We selected textile tubes made of alumina fiber. Figure 1 shows a cut model of the new flexible tube. A pipe end was welded to the flexible tube after the insertion of the inner tube. The flexibility (minimum bending radius) remained unchanged. The inner diameter of the inner tube was set to be the same as that of the pipe end (10.3 mm) in order to suppress turbulence. This tube is now available commercially [4].

Figure 2 shows the preliminary performance test results of the low-vibration flexible tube. The acceleration of vibration was measured using a pickup at the center of a 600-mm tube. The vibration was reduced to about one-fourth the previous amount for various flow rates of water at room temperature.

![Figure 1. Cut model of low-vibration flexible tube.](image)

![Figure 2. Vibration acceleration of conventional (black) and low-vibration (red) flexible tubes for various water flow rates.](image)

2.2. Temperature control
The DCM has two thermal problems. One is the thermal expansion of the first crystal caused by synchrotron radiation. The relaxation time is less than 2 h. The other and more serious problem is the thermal deformation of fine stepper stages; experimentally, this occurs mainly in
the tilt stages for crystal adjustment \[2\]. The gradient of temperatures in the DCM is over 220 K because liquid nitrogen is at 77 K. The reduction in stage temperatures continues during a three-month user operation and may not stop for a year. Heating from the synchrotron radiation hardly affects this problem because the heat transfer is blocked by the crystal holder with liquid nitrogen paths. Therefore, thermal insulation of the stages from liquid nitrogen is the most important issue.

To protect the tilt stage against cooling from the first or second crystal holder, we inserted an insulation block of machinable ceramics and a copper plate with a sheet heater; the copper plate was on the tilt stage. The heater current was calculated using a proportional-integral-derivative (PID) controller from the temperature of the copper plate monitored using a resistance temperature sensor. The temperature was kept at a preset value (25 or 27 °C) with fluctuations of less than ±0.01 °C.

In addition, the liquid nitrogen paths were hidden using a cryogenic insulator—ten-layer aluminum-deposition films spaced with nets—in order to reduce the absorption of heat radiation.

2.3. Radiation shield and improvement in tolerance
The synchrotron radiation impinging on the first crystal generates secondary electron and electromagnetic radiations, which cause radiation damage to the various components in the DCM. Figure 3 shows a schematic of a copper radiation shield used at some beamlines. The shield was rotated with the Bragg rotation. Most secondary radiations can be blocked using the shield, together with the second crystal, within the Bragg angles from 3° to 27°. The shield was indirectly cooled with water.

The materials of the cable jackets in the DCM were changed from cross-linked plastic to polyimide in consideration of the radiation tolerance.

![Figure 3. Positioning of the radiation shield for several Bragg angles θ.](image)

3. Present performance of DCMs
The vibration of the DCM caused a fluctuation in the intensity of the outgoing beam. Figures 4 (a) and (b) show the intensity plots of 1 Å X-rays with time obtained at the beamline (BL) 13XU \[5\] before and after the improvement, respectively. During the measurements, the first crystal stepped every 0.2′′ (arc second) over a time interval of 5 s. The integration time for each data point was shortened to 1 ms to increase sensitivity for the fluctuation; the average intensity over 100 ms appeared stable, as shown with the yellow line in Fig. 4(a). The misalignment angle due to the vibration was reduced from 1′′ to 0.15′′, and the fluctuation at peak intensity was reduced from 5.3% to 2.0%. At BL37XU \[6\], the misalignment angle was estimated to be 0.2′′ in the same manner.
After these improvements, we concentrated 15-keV X-rays with an intensity of $10^{12}$ photons/s into an area of 230 nm (vertical) $\times$ 270 nm (horizontal) by using a Kirkpatrick–Baez mirror at BL37XU. The vertical focusing was the demagnified projection of the real light source.

The slow-varying angular instability due to thermal deformation was suppressed to a rate of less than 0.2 $''$/h.

4. Remaining problems and future plans

The vertical focus size at BL37XU was theoretically estimated to be 100 nm at a position of 78 m from the light source by a magnification ratio of 1/170. The difference of the achieved vertical focus size originated primarily from the vibrations of the DCM placed at a position of 43 m. The angular vibration of the outgoing beam at the DCM caused a fluctuation in the virtual position of the light source, and the virtual source size was thus broadened. Supposing that both the intensity of the light source and the angular divergence were normal distributions, we roughly estimated the effect of the vibration to be $(230^2 - 100^2)^{1/2} = 207$ nm in terms of the dimensions of the focus size. The present target of the angular vibration is 0.05 $''$ to achieve 120-nm focusing. We are presently optimizing the performance of the flexible tube by varying its diameter and flexibility.

The reducing temperature of the tilt stages was not sufficiently suppressed. Currently, we are testing for further thermal insulation by using an offline test bench that includes DCMs and a liquid-nitrogen circulating system. After the above improvements, the temperature drop of the tilt stages was 4 $^\circ$C over three days from the start of cooling and showed no signals of stopping. This was attributed to the heat radiation from the stages to the liquid nitrogen paths. When an additional heater was inserted at a distance from each tilt stage in the opposite side from the copper plate, the temperature reached a constant value in a day. Other stages were also warmed effectively. This improvement will be tested on a beamline soon.

Acknowledgments

This work was partially supported by the “Low-Carbon Research Network” funded by the Ministry of Education, Culture, Sports, Science and Technology, Japan, and was conducted with the approval of JASRI (Proposal Number 2011B1059).

References

[1] RIKEN and JASRI 2012 SPring-8 Upgrade Plan Preliminary Report chapter 6 pp 96–97
[2] Yamazaki H et al 2010 AIP Conf. Proc. 1234 785
[3] Yabashi M et al 1999 Proc. SPIE 3773 2
[4] Osaka Rasenkan Kogyo Co., Ltd. “Clear Flow Flex” patent pending
[5] Sakata O et al 2003 Surf. Rev. Lett. 10 543
[6] Koyama T et al 2011 Proc. SPIE 8139 81390I