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X-ray beam stabilization at BL-17A, the protein microcrystallography beamline of the Photon Factory

Noriyuki Igarashi,[a]* Kazuyuki Ikuta,[a] Toshinobu Miyoshi,[a] Naohiro Matsugaki,[a] Yusuke Yamada,[a] Mohammad S. Yousef[a,b] and Soichi Wakatsuki[a]

[a]Photon Factory, Structural Biology Research Center, Institute of Materials Structure Science, High Energy Accelerator Research Organization, 1-1 Oho, Tsukuba, Ibaraki 305-0801, Japan, and [b]Biophysics Department, Faculty of Science, Cairo University, Giza 12613, Egypt. E-mail: noriyuki.igarashi@kek.jp

BL-17A is a new structural biology beamline at the Photon Factory, Japan. The high-brilliance beam, derived from the new short-gap undulator (SGU#17), allows for unique protein crystallographic experiments such as data collection from microcrystals and structural determination using softer X-rays. However, microcrystal experiments require robust beam stability during data collection and minor fluctuations could not be ignored. Initially, significant beam instability was observed at BL-17A. The causes of the beam instability were investigated and its various sources identified. Subsequently, several effective countermeasures have been implemented, and the fluctuation of the beam intensity successfully suppressed to within 1%. Here the instability reduction techniques used at BL-17A are presented.

Keywords: protein microcrystallography; Photon Factory; beamline development; X-ray beam stabilization.

1. Introduction

With advances in synchrotron radiation instrumentation, data collection using protein crystals of a few tens of micrometres in size is routinely achieved. However, in cases of biologically significant proteins like membrane proteins and supermolecular complexes, the production of such samples typically takes months or years and numerous iterations of crystal optimization trials followed by diffraction tests are necessary. Even when diffraction-quality crystals can be obtained, they may be very small, often less than 10 µm in one or more dimensions. In this regard, protein microcrystallography beamlines using microfocus X-ray beams have been recently demonstrated to be widely useful tools. These beamlines are now routinely used for data collection from protein microcrystals of a few micrometres in size, previously thought to be unusable. Such highly focused beams can no doubt significantly promote and facilitate structural biology research.

At highly focused beamlines, X-ray beam stability is one of the most important issues. In particular, the beam position is required to be stable on the order of micrometres around the sample. To achieve such high stability, it is necessary to detect, analyze and promptly eliminate any causes of instability in the beam. In this article we demonstrate the instability reduction techniques we have developed through our experiences at BL-17A, the protein microcrystallography beamline at the Photon Factory (PF).

2. Beamline configuration

BL-17A is designed for microcrystal structure analysis. In addition, an intense lower-energy beam at around 6 keV is used for structure determination by SAD phasing with light atoms. The source of the beamline is a newly developed short-gap undulator installed in one of four short straight sections of the PF 2.5 GeV ring. The main optical components of BL-17A are the vertically deflecting double-crystal monochromator (DCM) and the Kirkpatrick–Baez (KB) mirror system. The DCM is located at 17.5 m from the light source. Crystals are cooled indirectly by a liquid-nitrogen circulation system to reduce crystal deformation caused by high heat load (Mochizuki *et al.*, 2001). Following the DCM, the KB mirror system is used for fine focusing. Two sets of flat mirror benders are located at 24.0 m for vertical focusing with a focusing ratio of 2:1, and at 30.8 m for horizontal focusing with a ratio of 6:1. Both mirrors are Rh-coated Si crystals and perform well; their roughness and tangential slope error are within 2.0 Å (r.m.s.) and 1.0 µrad (r.m.s.), respectively. The second mirror has an elliptically bent surface which is achieved by the 'arm method' mirror bender, to reduce the aberration of the highly asymmetric 6:1 (H) focusing ratio (Kamachi *et al.*, 2002). Consequently, the beam cross section at the focal point (36 m) is measured to be about 0.03 mm (V) $\times$ 0.23 mm (H) which is about ten times smaller than that of the PF's two high-throughput beamlines, AR-NW12A and BL-5A. The measured flux through 100 µm $\times$ 100 µm and 20 µm $\times$ 20 µm collimation slits in both ranges is above 5×10^{10} and 5×10^{9} photons s^{-1}, respectively. The achieved intensities are high enough for both micrometre-size structure analysis and structure determination using softer X-rays (Igarashi *et al.*, 2007).

3. X-ray beam stabilization

BL-17A was opened for general users in June 2006 and very promising results were obtained. However, we initially observed about 10% instability of the beam intensity, even with slit sizes of 50 µm × 50 µm. We have investigated the relationships between beam intensity, beam position, vibration of the optical components, cooling device flow and ring orbit status. Consequently, we could identify five potential problems: (i) intensity fluctuations during the passage of heavy objects on the experimental floor close to the beamline; (ii) spike-like intensity changes every several minutes; (iii) high-frequency fluctuations of beam intensity; (iv) long-term drift of beam position; and (v) irregular step-like intensity changes. Further investigation allowed us to identify the causes of these problems. To overcome them, we considered the following countermeasures: (i) translocation of the mirror optics onto the stable floor frame; (ii) helium purge operation of the monochromator liquid-nitrogen flow; (iii) reduction of vibration around the optics; (iv) development of a feedback system for X-ray beam position; and (v) improvement of the ring operation. So far, most aspects of these countermeasures have been implemented and the stability of the X-ray beam at BL-17A has been successfully improved. Details of the problems and the results of the X-ray beam stabilization measures are described below.

3.1. PF floor structure

After the first operation of BL-17A in autumn 2005, we evaluated the beam performance of the beamline. At that time, intermittent beam intensity fluctuations were obvious. The fluctuations occurred most often when a heavy object is moved on the experimental floor near the beamline. We suspected that mechanical vibration somehow propagated through the experimental floor to one of the optical elements causing the beam instability. In order to analyze such a phenomenon, we used a rotary pump as a vibration source and placed it at various positions on the experimental floor. We then used a ground noise meter (AVT-104, Mitsutoyo) to measure the vibration propagating to each of the optical components. The measurements showed that placing the rotary pump in the shaded area shown in Fig. 1(*a*) strongly affected the vertical mirror. We also noticed that this vibration effect was minimal when the pump was placed on the concrete frame of the experimental floor. Thus, we decided to move the two mirrors upstream of the beamline so that they were anchored in the floor frame, as shown in Fig. 1(*b*). In summer 2006, the vertical and horizontal mirrors were displaced from 24.0 m to 22.2 m and from 30.8 m to 29.5 m distant from the source point, respectively. After the displacement work, the above-mentioned types of fluctuations were mostly eliminated and the beam stability was significantly improved. The displacement of the mirrors, on the other hand, has led to smaller asymmetric focusing rates and slightly compromised resultant focusing sizes. However, for a microcrystallography beamline, establishing reliable beam stability is much more critical because of the very narrow beam size.

3.2. Anti-bumping of the liquid-nitrogen cooling system

The most serious source of X-ray beam instability was related to the liquid-nitrogen cooling system, which protects against the large heat load on the DCM of the undulator beamline. We found out that the liquid-nitrogen flow rate sometimes changed quickly and sharply; whenever this occurred, a large vibration on the DCM was simultaneously observed. The problem was frequent, occurring every several minutes, and subsequent fluctuations in beam intensity and position were significant. To identify the cause of the problem, we monitored the liquid-nitrogen flow rate and the vibration on the DCM. We concluded that some unavoidable minor imperfections in the insulation of the liquid-nitrogen piping may have resulted in a sudden bumping of the liquid nitrogen which creates shock waves ultimately imping on the DCM crystals. Because the cooling system is operated at around 75 K, the liquid nitrogen can easily vaporize and expand when a minor heat leak takes place.

In our first attempt to solve the problem, we tried to substitute helium for nitrogen as a pressure control gas. Several iterations of helium injection into the system were sufficient to suppress the instability in the liquid-nitrogen flow as shown in Fig. 2. However, it is difficult to maintain helium gas inside the cooling system for long periods of time. Therefore we performed helium purge routinely, twice a day. Moreover, we found that it is effective to insert special

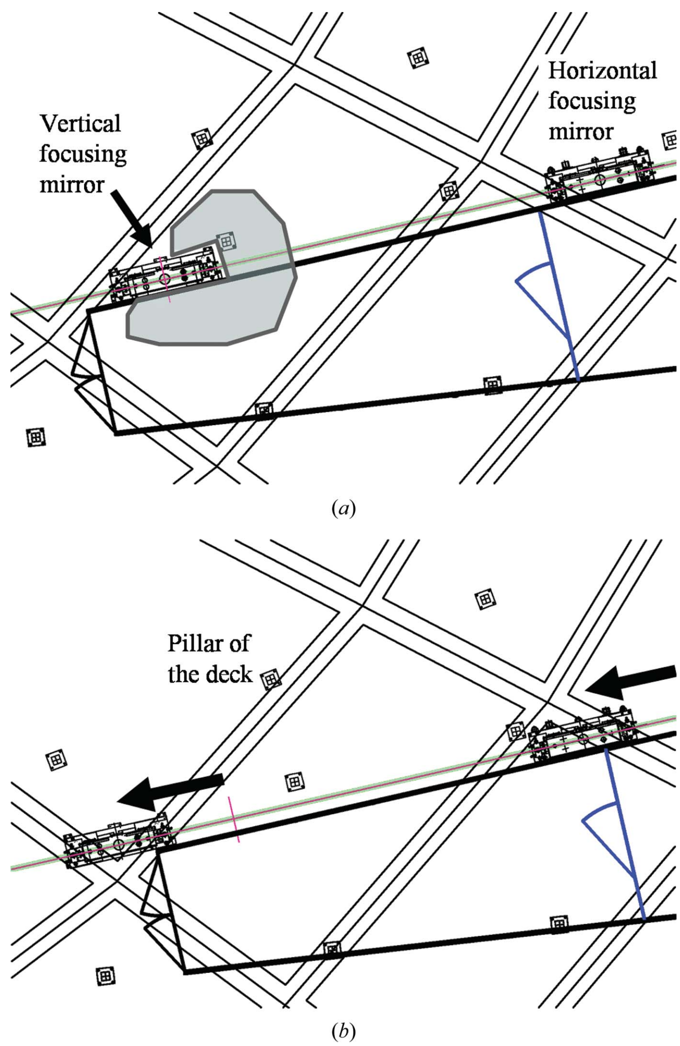

Figure 1
Top view of the KB mirror system superimposed on the frame architecture of the PF experimental floor. The left-hand side is towards the light source. (*a*) Before the displacement of the two mirror benders. The grey shadow shows the area where the vibration source had the greatest effect on the vertical mirror bender. (*b*) After the improvement. Two mirror benders were displaced to the floor frame as indicated by the two arrows.

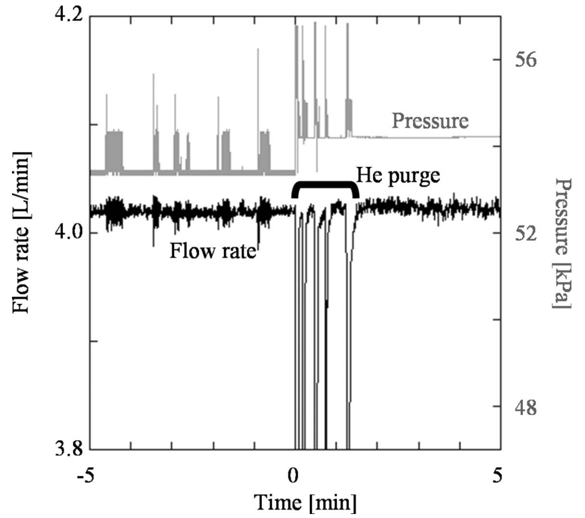

Figure 2
Effect of the helium purge on the liquid-nitrogen piping. Black and grey lines indicate the flow rate and the pressure of the liquid-nitrogen cooling system, respectively. Changes in the flow rate and pressure of the liquid nitrogen are not observed after several helium purges.

silicon adaptors into the joints of the insulated pipings. The combination of these two measures (helium purge and silicon adaptor) appeared to have eliminated the source of this problem.

3.3. Anti-vibration of optical components

In general, X-ray beam stability is limited by the vibration of the experimental floor. Fast ground motion has been studied extensively at the PF (Maruyama *et al.*, 2007). The authors reported that the vibration on the PF experimental floor has mainly faster frequencies than 25 Hz. In order to achieve greater beam stability, the effect of the fast vibration on the optical components must be eliminated. We tried to reduce the vibration by improving the beamline vacuum system and using anti-vibration materials (pads and sheets). Once an acceptable vacuum level is reached, we stopped the turbo molecular pumps (TMPs) and the scroll pumps around the optical components, and switched to the 'vibration-free' ion pumps. Furthermore, we inserted two types of anti-vibration products under the liquid-nitrogen flow pump and the refrigerator compressors, as shown in Fig. 3(*a*). The anti-vibration products used were a silicone gel sheet (BGEPM100, Misumi) and an anti-vibration pad (BPAS100-100, Misumi). The sheet and pad can absorb vibrations ranging from 60 Hz to 250 Hz and 25 Hz to 100 Hz, respectively. By using both products we hoped to cover a wider frequency range than any of the products can achieve solely. Ground motions slower than 25 Hz can be treated with the beam feedback system described in the following section. For effective absorption of vibration, we optimized the sizes of the anti-vibration products in order to support 50 kgf for every unit of each product. The result of the vibration reduction as a function of vibration source is shown in Fig. 3(*b*). Vibration was measured by the ground noise meter set on the DCM. The acceleration was reduced from 0.67 cm s^{-2} to 0.28 cm s^{-2} by the combination of anti-vibration products as well as stoppage of the TMPs and scroll pumps. At the same time, we investigated the beam intensity through a 50 μm × 50 μm collimation slit. As shown in Fig. 3(*c*), the fluctuation of the beam intensity was effectively suppressed and the beam stability was improved.

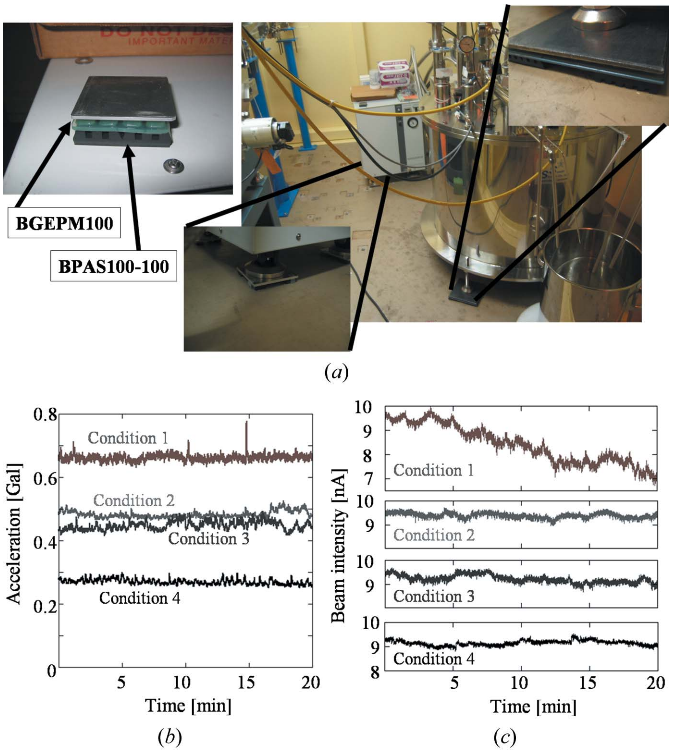

Figure 3
Effect of anti-vibration measures. (*a*) Left-hand photograph shows the combination of the two types of anti-vibration products. The anti-vibration products are set below the liquid-nitrogen vessel and the refrigerator compressor as shown on the right. (*b*, *c*) Vibration reduction and beam stabilization as a function of conditions: condition 1, no measures; condition 2, anti-vibration against the liquid-nitrogen vessel (flow pump); condition 3, with the addition of anti-vibration against the refrigerator compressor; condition 4, with the addition of stoppage of the TMPs and scroll pumps around the optical components.

3.4. Beam position feedback system

At a high-brilliance beamline, heat load is a critical problem. In particular, the PF ring has not yet been operated in top-up mode, and the heat load therefore varies depending on the ring current. After half a day, the beam position continuously shifts by 50 μm at the sample location, which is larger than the beam size of BL-17A. Even during a 20 min monitoring period, which is the typical data collection time at BL-17A, the beam position noticeably changes, as shown in Fig. 4(*a*). To overcome this problem we have developed a vertical beam position feedback system based on the piezo tuning of the first monochromator angle of the DCM. The heat load causes instability in the parallel arrangement of the double crystals, and it has been determined that the DCM control is effective for stabilizing the beam position (Kudo *et al.*, 2007).

The configuration of the system is shown in Fig. 5. The beam position is measured using a position-sensitive ionization chamber (Sato *et al.*, 1999), which is set on the diffractometer at about 700 mm upstream of the sample location. The signals from the beam position monitor (BPM) are fed to a high-speed current amplifier. The outputs from the amplifier are digitized by a fast ADC on an AD/DA PCI board with a TI ADS7809 processor. The PC is responsible for the energy function, which controls the gap of the undulator, the angles and positions of the DCM, and the gain of the amplifier. The BPM sum signal is used for the calculation of the feedback voltage, which is applied to a high-voltage (HV) controller (E-501.00, Physik Instruments) to drive a piezo transducer (PZT) through a DAC on the board. The ADC sampling rate is 10 μs and the BPM signals are

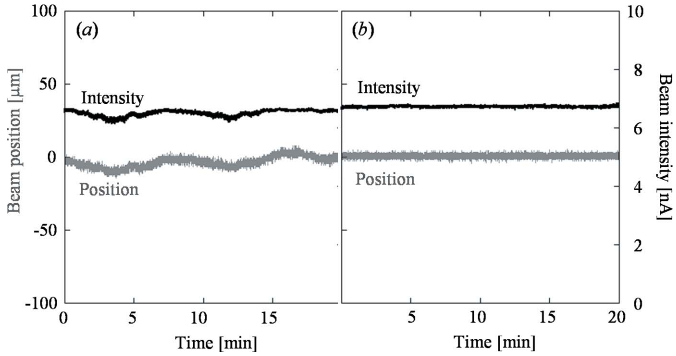

Figure 4
Beam stabilization at BL-17A (*a*) with and (*b*) without the feedback. Grey and black lines indicate the beam position and intensity, respectively. The beam energy was 12.4 keV. The beam position and intensity were measured using the position-sensitive ionization chamber and the standard ionization chamber, respectively. The standard deviation of the beam position and intensity during 20 min were effectively reduced from 3.6 μm to 1.3 μm and from 1.6×10^{-2} to 3.4×10^{-3}, respectively.

averaged for precise estimation of the beam position, and then an IIR filter is implemented for noise cancellation. The final feedback frequency is less than 20 Hz and the PZT periodically drives the first crystal of the DCM using the HV controller. The beam position and intensity, with and without the feedback, through a 50 μm × 50 μm collimation slit are shown in Fig. 4. With the feedback, the beam position could be kept at a fixed position, and the fluctuation of the beam intensity was improved to within 1% over a period of 20 min.

We have implemented this feedback system at BL-17A quite recently. The X-ray beam stability was effectively improved, but the fluctuation of the beam position is not yet negligible for a micro-crystallography beamline. Subsequently, we plan to optimize the feedback parameters and improve the noise cut function. Moreover, for more precise estimation of beam position, we will implement an in-line X-ray monitoring system before and/or after the DCM (Alkire *et al.*, 2000).

3.5. Improvement of ring operation

Fundamentally, stability of the light source is essential for utilizing these microbeams. As noted above, the PF ring is not operated in top-up mode, and we have learned that the current phase modulation technique for enlarging beam lifetime causes variation in the electron beam size. We are now engaged in discussion with the PF ring group to find solutions to this problem. At present, we have studied a top-up operation mode for the PF ring and developed a feedback system against longitudinal coupled-bunch instability by means of the RF phase-modulation technique (Obina *et al.*, 2007). The study of the top-up mode, together with implementation of a bunch-by-bunch feedback system, is scheduled for 2008 operation. At that time we expect to achieve more precise X-ray beam stabilization, of the order of sub-micrometres.

4. Summary

Here we have described our investigations of X-ray beam instability at BL-17A, as well as the countermeasures we took to suppress it. We identified five causes of the instability by monitoring beam intensity, beam position, liquid-nitrogen flow and vibration in the beamline components and experimental floor. We could significantly improve the beam stability by displacing the optical components on the stable floor frame, eliminating the bumping of the liquid-nitrogen flow, and reducing the vibration on the optical components. Finally, we have developed a beam position feedback system using a BPM and piezo drive for tuning the first crystal of the DCM. With this system we could control the beam position and suppress the fluctuation of the beam intensity to one-fifth of that originally observed. We are attempting to make improvements in the system, including implementation of more precise BPMs. Furthermore, modifications of the PF ring operation have been scheduled. Stabilization of both electron and photon beams will allow us to achieve efficient protein micro-crystallography at the Photon Factory. The reported stabilization techniques will not only be useful for currently operational synchrotron facilities, but will also be applicable to next-generation light sources such as those at the FEL and ERL facilities.

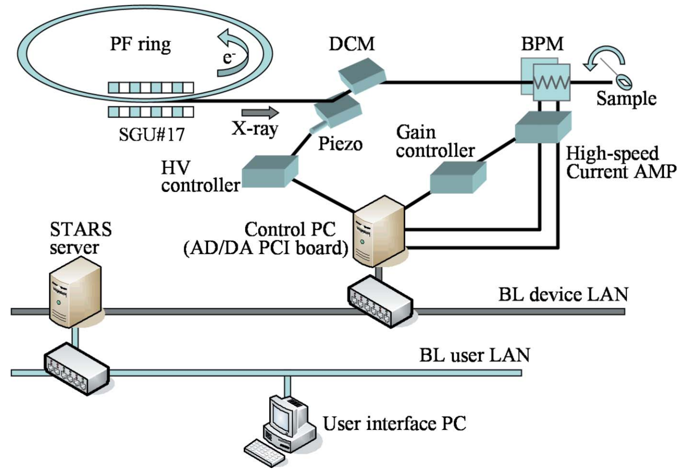

Figure 5
Configuration of the feedback system. The feedback is controlled by software on a control PC mounted on the beamline device LAN (local area network), which is separated from the beamline user LAN by a firewall. The STARS communication server runs on the firewall; users can send commands to beamline devices only through STARS.

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