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A fast mechanical shutter for submicrosecond time-resolved synchrotron experiments

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A new high-speed high-repetition-rate X-ray beam shutter for time-resolved photocrystallography at synchrotron sources has been developed and tested. The new design is based on a commercially existing DC servomotor and a frequency-lock control capable linear amplifier. Accurate speed control combined with an air bearing results in extremely low jitter in the motor rotation. Measured jitter at rotation speeds of 12000 to 30000 r min⁻¹ is less than 2 ns at a 6σ confidence level. The chopper disc is interchangeable, allowing maximum flexibility. The chopper disc currently installed has 45 radial slots which allows synchronization from the 12th to the 20th subfrequencies of the orbit frequency of the Advanced Photon Source storage ring, corresponding to X-ray pulse frequencies of 13.6 to 22.6 kHz. At 30000 r min⁻¹ the opening time window with a 350 µm slot size is 2.11 µs, and correspondingly less with smaller openings, which may be compared with the 3.68 µs orbit time of the Advanced Photon Source. The shutter provides high accuracy and efficient use of X-rays at a modest cost.

Keywords: fast shutter; phase locking; photocrystallography; air bearing.

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1. Introduction

Owing to the increased intensity and brightness of the now available third-generation synchrotron sources, time-resolved diffraction techniques have advanced rapidly during the last decade. The time resolution that can be achieved is only limited by the width of the synchrotron pulse, which at the Advanced Photon Source (APS) is currently 70–100 ps. In our experiments in the \sim 500–2 µs range the synchrotron was used as a continuous source pulsed by a mechanical chopper. Based on this experience we have designed a ring-frequency



Figure 1

Shutter with partially dismounted vacuum chamber.

synchronized mechanical shutter, which is described here. The shutter, shown in Fig. 1, is both economical (cost <\$25000 including the amplifier and one chopper disc) and highly accurate. It uses an air bearing and its speed is regulated by a frequency-controlled loop which can be phase-locked to the temporal structure of the storage ring. The current version has been optimized for use with a 25 keV monochromatic X-ray beam. The slotted wheel selected for the first applications allows repetition rates from 13.6 to 22 kHz, but is readily changeable. The chopper is capable of separating either the superbunch or the single bunch of special operating mode 1 (SOM1) at the APS.

1.1. Other approaches to isolation of short X-ray pulses at synchrotron sources

Several approaches have been described in the literature.

1.1.1. Choppers based on rotating crystals or rotating X-ray mirrors. These choppers are based on the small acceptance angle of the diffracting crystal or total reflection by the mirror. Because of the small acceptance angle of the diffracting crystal and the limited value of the total reflection angle of the mirrors, a crucial requirement for such choppers is extremely high rotational position control (Kosciesza & Bartunik, 1999; McPherson *et al.*, 2002; Norris *et al.*, 1992).

1.1.2. Choppers based on rotating apertures. (i) Rotation axis perpendicular to the direction of the X-ray beam. This

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Table 1

Comparison of currently operating choppers at APS (performance at maximum speed).

Chopper design	Opening size (mm)	Synchrotron ring frequency divider	Repetition rate (Hz)	Opening window (µs)	$ m r min^{-1}$	Reference
Rotating cylinder	0.5	102	2662	2.45	79867	McPherson et al. (2000)
Triangular rotor	1	300	905.2	2.1	54309	R. Pahl, C. Pradervand, S. Ruan & W. Schildkamp (unpublished)
Rotating crystal	15†	250	1086	0.023	32580	McPherson et al. (2002)
Rotating disc (current design)	0.35	12‡	22 629‡	2.1	30172	This paper

† Si crystal size. Note that crystal size is irrelevant for the effective timing window. ‡ For operation at other subfrequencies, see Table 2.

Table 2

Selected parameters as a function of the ring subfrequency for a 45-slot wheel with 350 μm slot opening.

Synchrotron ring frequency divider	Laser/X-ray frequency	r min ⁻¹	Transmission window full width (μs)
12	22 6 29	30172	2.11
16 20	16972 13577	22.629 18.103	2.81 3.52

design has several advantages. First, as the beam can pass through each channel every 180°, the shutter effectively opens and closes twice as fast as choppers with rotation axes parallel to the X-ray beam, which is the second option. Second, a larger amount of material can be used to block the beam compared with parallel design. A disadvantage of this solution is the reduced repetition rate and therefore reduced duty cycle of the shutters that have been described, which have been limited to the 1–2 kHz range. This geometry seems more suitable for use for highly intensive white or pink beam applications using Laue diffraction techniques (LeGrand *et al.*, 1989; Mills, 1989; Wulff *et al.*, 1997; McPherson *et al.*, 2000; R. Pahl, C. Pradervand, S. Ruan & W. Schildkamp, unpublished).

(ii) Rotation axis parallel to the X-ray beam. This design is preferred for high-repetition-rate choppers. As the chopper wheel can have multiple openings, the repetition rate will be a multiple of the rotation speed. In principle, repetition rates equal to the synchrotron ring frequency are achievable, the limiting factor being the machining of the slots and the rotation speed (LeGrand *et al.*, 1989; Mills, 1989; Coppens *et al.*, 2005).

The choppers that have been developed at APS are summarized in Table 1.

2. Shutter design

2.1. General characteristics

The new design is based on a commercially existing DC servomotor and a frequency-lock control capable linear amplifier (Motion Control Systems, VA, USA). Combination with the air bearing allows exceptional speed control owing to low error motion and consistent non-varying drag (or friction) characteristics. Drag torque 'ripple', owing to shear film losses,





is near zero, though, at high speed, air bearings are not friction-free as is sometimes assumed. On the other hand, bearings with rolling elements will always have some amount of torque ripple and lubrication is a problem at higher speeds.

To allow maximum flexibility a modular approach with an interchangeable chopper disc was selected. The current chopper disc is optimized for use with monochromatic radiation and is designed for operation with a rotation axis parallel to the X-ray beam. It has 45 radial slots which allows synchronization with the APS storage ring at 1/12 to 1/20 times the ring frequency, corresponding to X-ray pulse frequencies from 13.6 to 22.6 kHz (Table 2). Although the rotational speed is currently limited by the amplifier to $32\,000$ r min⁻¹, the motor and the chopper disc are capable of spinning at higher speeds. However, this option would require some design modifications.

2.2. Shutter components

The shutter system consists of five main components (Fig. 2):

A – brushless frameless DC servomotor. Stator, outer diameter 50.8 mm (2.000''); number of poles, 6. Rotor magnet, outer diameter 21.6 mm (0.850'').

B – shaft rotating in an air bearing. Since an aerostatic type of bearing was utilized, the bearing must be continuously pressurized at ~6 atmospheres. Air consumption is estimated to be below 25 l min⁻¹.

C – optical encoder with 360 lines, serving as a feedback device.

D – vacuum chamber. Vacuum is preferred for operation in order to minimize noise and pumping losses and to reduce the required motor torque and the spindle heating. The vacuum chamber serves as a protective shield for the rotating disc in the case of disastrous failure. Chopper containment will always be an important consideration, especially if the rotation speed increases further with a concomitant increase in stored energy. The vacuum chamber is currently equipped with one pair each of Kapton-foil X-ray windows and KB-7 glass windows for transmission of light signals for synchronization purposes. The chamber can be connected with a vacuum pump through an NW-25 adapter.

E-140 mm-radius interchangeable X-ray chopper disc. The steel disc currently installed has 45 radial slots of size 350 µm \times 5 mm. A timing diagram for the chopper rotating at 22629 rmin^{-1} and 0.100 mm effective aperture size is presented in Fig. 3. For a slot opening comparable in magnitude with the sample crystal, and auxiliary slit size larger than the size of the sample crystal, the crystal effectively acts as a slit, and its size affects the time profile of the diffracted beam. Under these conditions the transmission function has a trapezoidal form (Fig. 3), with a plateau from the time that the leading edge of the slot has passed the sample to the time that the trailing edge reaches the sample. The width of the plateau is such that it can accommodate the superbunch of SOM1 with a considerable margin. Note that the maximum allowed opening for superbunch separation is almost 3.68 µs, whereas it is less than 3.18 µs if the single bunch is to be selected

2.3. Attenuation coefficient

In our time-resolved experiments the 25 keV X-ray beam from an A-type undulator at the ID15B beamline is utilized. It has a flux of $\sim 2 \times 10^{12}$ photons s⁻¹ and a bandpass $\Delta E/E$ equal to 10^{-4} . In calculating the desired attenuation the ratio of the radiation allowed to pass through the shutter opening and the radiation that has to be stopped must be determined. We will consider the case in which the chopper is used with the asymmetric fill of the synchrotron ring (SOM1), which consists of a single bunch and a superbunch, the single bunch containing 5% of the ring current.¹ The most unfavorable situation occurs when the chopper is locked at the 24th synchrotron ring subfrequency and only the single bunch passes through the slot. In this case the ratio between radiation utilized in the experiment and the radiation to be blocked is $5/2395 \simeq 0.002$. Our requirement is that the radiation transmitted when the chopper is blocking the beam (the 'leakage') be less than 10^{-6} of the transmitted beam. At this level the secondary scattering from the air and the detector



Figure 3

Top: time structure of the X-ray beam in SOM1. Bottom: time profile of the beam passing through the 0.350 mm slots and diffracted by a 0.100 mm sample.

background are higher than the leakage. To obtain the 5/2395 ratio the desired attenuation must not be less than $I/I_0 = 2 \times 10^{-9}$, which is equivalent to $\mu t \simeq 20.0$. For 416 stainless steel, with a chemical composition equivalent to 13% Cr and 87% Fe and a density of 7.64 g cm⁻³, $\mu = 87.6$ cm⁻¹ (Chantler *et al.*, 2001), giving a minimum chopper thickness of 0.23 cm. The disc used has a thickness of 0.38 cm (0.15''), corresponding to an attenuation of 1.73×10^{-12} at 25 keV, but also adequate for photon energies up to 30 keV.

2.4. Selection of the chopper material and rotational stress calculation

We have chosen 30000 r min^{-1} as a practical limit for our design though higher speeds are achievable by simple modifications. We note that speeds well beyond $100000 \text{ r min}^{-1}$ are now routinely archived in ultracentrifuges or in other devices with high-speed spindles. Several metals, including copper, molybdenum, tantalum, tungsten or their alloys, may be considered as chopper materials. While brass, which has been used in a previous chopper, has several advantages, such as higher attenuation than steel or titanium, excellent machinability and low price compared with titanium alloys, its limiting factor in high-speed applications is its relatively low tensile strength.

Another important consideration in the spindle design is the thermal expansion coefficient of the materials used. The thermal expansion of the steel chopper matches that of the shaft and bearing assembly, which is optimal, as differing thermal expansion coefficients may cause the chopper disc to slip to an eccentric position on the bearing axis when the temperature increases. This will cause imbalance and can lead to bearing failure at higher speeds.

The 140 mm-diameter steel chopper can spin up to $78\,000 \text{ rmin}^{-1}$ before yielding and therefore strength is not a concern. Additional advantages of steel are its larger stiffness compared with other materials.

¹ The current in the single bunch has recently been increased from 5 mA to 8 mA, corresponding to almost 8% of the ring current.

Stainless steel 416 has a Poisson ratio $\nu = 0.133$ and a mass density $\rho = 7460$ kg m⁻³. The tangential stress σ of a uniform flat disc can be calculated by

$$\sigma = (1/8)(3+\nu)\rho\omega^2 R_0^2$$
 (1)

(Young & Budynas, 1989), in which R_o is the disc radius. With $R_o = 70 \times 10^{-3}$ m and using the numbers listed above we obtain $\sigma = 141.3 \times 10^6$ Pa, which is well below the maximum tensile strength of steel of $\sigma_{max} = 952 \times 10^6$ Pa (conservative estimate after heat treatment). The maximum allowable rotation frequency f_{max} is calculated by

$$f_{\rm max} = (1/2)\pi \left[8\sigma_{\rm max}/R_o^2 \rho (3+\nu) \right]^{1/2}$$
(2)

as $f_{\text{max}} = 1297 \text{ Hz} = 77820 \text{ r min}^{-1}$.

2.5. The synchronization (Fig. 4)

Both the motor controller and the laser are driven by a 43.99 MHz external reference frequency ($f_{\rm RF}/8$) derived from the APS storage-ring RF frequency. This signal is supplied by the accelerator control system. Programmable frequency dividers process this reference signal to lower subfrequencies appropriate for the motor controller and the laser. The phase shifts on both lines are corrected by a pair of digital delay generators. A schematic block diagram with divider ratios and corresponding frequencies is presented in Fig. 4. The synchronization between the X-ray pulses and the high-repetition-rate pump laser is established by a circuit consisting of an avalanche photodiode for the X-ray pulses and a fast photodiode for the laser beam. Photodiode II serves as a redundant optical encoder.

2.6. Performance test

The primary advantage of using an air bearing combined with the frequency-locked linear amplifier is the extremely high level of rotational speed control that allows precise timing. In the evaluation of the spindle, we measured the time



Figure 4

Block diagram for the experimental set-up, with division factor for the 16th subfrequency of the APS ring.

required for the rotor to make one revolution at different rotation speeds by recording the leading edge of the zeromarker pulses. A preliminary measurement with a LeCroy 7100 digital oscilloscope (1 GHz BW, 1 M points) showed the jitter to be of the order of nanoseconds, *i.e.* below the resolution limit of the instrument used.

For a more precise measurement, a HP 3325B Synthesizer/ Function-Generator to mimic the synchrotron reference signal and a HP 5335A Universal Frequency Counter were used. The reference 10 MHz signal for both devices was supplied by a PTS 1000 high-stability frequency reference. Three series of tests were performed with the chopper disc respectively rotating in air (up to 21000 rmin^{-1}), in a He atmosphere and in a 3 torr vacuum (up to 30000 rmin^{-1}). The air operation mode is not recommended above 15000 r min⁻¹ for permanent use because of excessive heating created by the motor and drag on the chopper disc. It may be noted that, in the vacuum mode, non-contact high-impedance-to-flow capillary sealing exists between the pressurized air bearing and the vacuum chamber. The jitter in the revolution time is presented in Fig. 5. No significant difference was observed between operating the chopper in a He atmosphere and in a vacuum. Therefore, only the jitter during the He atmosphere and air operation is shown in Fig. 5.

3. Error evaluation

For a chopper disc with multiple slots, several factors contribute to the overall accuracy. The first is the rotation time error, which has been measured to be below 2 ns in the 12000– $30000 \text{ r} \text{min}^{-1}$ range for a single rotation. For multiple rotations the error can accumulate to up to 20 ns. The second contribution, owing to eccentricity and inaccuracy of the encoder, may make the speed change slightly during a revolution. According to the manufacturer, comparison of arbitrary angular segments of the disc gives estimates for the error owing to the eccentricity and inaccuracy of the encoder of up to 30 arcsec. The third contribution is the radial and axial error motion of the spindle, which is specified by the manufacturer to be less than 0.05 µm at loads below 10 kg. The fourth and last contribution is the machining error for the radial slots. The



Figure 5

Jitter in the revolution time $(\pm 3\sigma \text{ level})$ as a function of rotation speed. RPT = room pressure and temperature. maximum deviation of the slot from its theoretical position has been measured as 22 arcsec, whereas the standard deviation of the positional deviations is found to be 7 arcsec. The relative error of the slot shape owing to the manufacturing process is found to be negligible, since the wire-EDM machine moved exactly along the same x and y coordinates for every slot. A precise rotary gear table was used for the angular positioning of the chopper disc before each cut was made. Note that this last type of error does not exist for a chopper wheel with a single radial or axial opening.

The overall error, which is mainly due to encoder and slotposition inaccuracies, is around 50 arcsec, which corresponds to $\sim 5\%$ of the 1070 arcsec opening size.

Generally, the above calculation simplifies and overestimates the error evaluation because the large inertia characteristic of this chopper design smooths and minimizes the speed variation given a low torque motor. As a result, the speed variation within each revolution owing to encoder inaccuracy vanishes at higher speeds. Since the commonly used samples have dimensions much smaller that the 350 μ m slot size (100 μ m less), the errors are negligible for all practical purposes.

4. Conclusions

A highly precise subsonic modular mechanical X-ray beam shutter has been designed and constructed. It can operate synchronously with the temporal structure of the APS storage ring. Currently its dimensions and performance are optimized for SOM1 operation at ChemMatCARS beamline 15. Accurate speed control combined with a precise air bearing results in extremely low jitter in the spindle rotation. Measured jitter at rotation speeds of 12000 to 30000 r min⁻¹ is less than 2 ns at a $\pm 3\sigma$ confidence level. The results demonstrate that low-cost components can be used to build an exceptionally precise instrument. The modular approach, which allows inter-

changeability of the chopper discs, makes the instrument suitable for a wide range of different time-resolved experiments. Because of the ability of the chopper to operate in a He atmosphere, the design can also serve for a synchronized millisecond or microsecond heat-load shutter.

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References

- Chantler, C. T., Olsen, K., Dragoset, R. A., Kishore, A. R., Kotochigova, S. A. & Zucker, D. S. (2001). X-ray Form Factor, Attenuation and Scattering Tables, http://physics.nist.gov/ffast.
- Coppens, P., Vorontsov, I., Graber, T., Gembicky, M. & Kovalevsky, A. Y. (2005). Acta Cryst. A61, 162–172.
- Kosciesza, D. & Bartunik, H. D. (1999). J. Synchrotron Rad. 6, 947–952.
- LeGrand, A. D., Schildkamp, W. & Blank, B. (1989). Nucl. Instrum. Methods Phys. Res. A, 275, 442–446.
- McPherson, A., Lee, W.-K. & Mills, D. M. (2002). *Rev. Sci. Instrum.* **73**, 2852–2855.
- McPherson, A., Wang, J., Lee, P. L. & Mills, D. M. (2000). J. Synchrotron Rad. 7, 1–4.
- Mills, D. M. (1989). Rev. Sci. Instrum. 60, 2338-2341.
- Norris, J. R., Bowman, M. K., Chen, L., Tang, J., Thurnauer, M. C., Knapp, G. S. & Montano, P. A. (1992). *Rev. Sci. Instrum.* 63, 1172– 1175.
- Wulff, M., Schotte, F., Naylor, G., Bourgeois, D., Moffat, K. & Mourou, G. (1997). Nucl. Instrum. Methods Phys. Res. A, 398, 69– 84.
- Young, W. C. & Budynas, R. (1989). *Roark's Formulas for Stress and Strain*, 6th ed. New York: McGraw-Hill.