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A compact superconducting ring as a radiation source for X-ray crystallography

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A compact superconducting storage ring installed at Ritsumeikan University is operated at an electron-beam energy of 0.575 GeV and an initial beam current of 300 mA. The radius of the circular electron orbit is as small as 0.5 m, suggesting that the radiation emitted contains short-wavelength components. With an imaging plate as a detector, X-ray precession diffraction patterns were recorded for organic single crystals within a reasonable period of time using radiation of wavelength 0.155 nm (8 keV) to 0.248 nm (5 keV). The use of the radiation in the structural study of organic crystals containing 3*d* metal atoms using the phenomena of anomalous scattering is described. If appropriately planned, X-ray diffraction and/or scattering experiments can be made at the compact ring without recourse to a large-scale ring.

Keywords: X-ray diffraction; compact superconducting ring; anomalous scattering.

1. Introduction

Wavelength tunability, characteristic polarization and other unique features of synchrotron radiation have offered more opportunities for X-ray crystallographic research than are attainable with the radiation from the ordinary laboratory sources and a number of advances have been made in the past two decades. Since the spectrum of the radiation emitted from the bending magnets in a storage ring depends on the electron-beam energy, X-ray diffraction and/or scattering experiments have been performed at facilities with a large-scale storage ring, such as the Photon Factory.

In view of the increasing demand for synchrotron X-radiation, it is desirable for many users to have sources with a short wavelength not only in large-scale facilities but also in facilities with a ring of a lower beam energy. One solution is to insert a wiggler (wavelength shifter) in the electron orbit in the ring.

There are storage rings in which superconducting magnets are used as the bending magnets. They were originally developed for X-ray lithography, a review of which is given by Wilson (1992). The high field strength generated by the superconducting magnets makes electrons move in an orbit with a small radius of curvature, resulting in a shift of the radiation spectrum towards the short-wavelength side. AURORA is one of the compact superconducting storage rings designed and manufactured by Sumitomo Heavy Industries (Takahashi, 1987; Yamada *et al.*, 1989). It consists of a single bending magnet and the electron orbit is exactly circular, the electron-beam energy being 0.575 GeV. The radius of the orbit is as small as 0.5 m and the critical wavelength λ_C of the radiation is 1.47 nm (844 eV in the critical energy). The spectrum of the radiation is shown in Fig. 1. λ_C is appreciably shorter than that of a ring consisting of normal bending magnets with approximately the same beam energy: for example, λ_C of the UVSOR ring (0.75 GeV) is 2.92 nm. After making a minor modification to the original design, AURORA was installed at Ritsumeikan University in April 1996 and has been operated successfully since then (Iwasaki, 1997; Iwasaki *et al.*, 1998). The small radius of the electron orbit allows the optical components of the beamline to be placed at a distance close to the source point so that it is possible to use a radiation beam with a relatively high photon flux. A feasibility study was carried out at the ring to investigate to what extent the X-ray component of the radiation can be used for diffraction experiments. The results are described below.

2. X-ray diffraction beamline at the superconducting storage ring

The spectrum shown in Fig. 1 indicates that, although the brilliance of the radiation from AURORA decreases rapidly with decreasing wavelength (or increasing photon energy), there still exists a non-negligible intensity at wavelengths in the range from 0.25 to 0.15 nm. A beamline was constructed at beamport No. 3 of the ring, where a double-crystal monochromator of the type originally designed by Golovchenko *et al.* (1981) was placed at a distance of 3 m from the source point. A pair of planar Si(111) crystals or Si(220) crystals were used as the monochromator crystal depending on the wavelength chosen. The monochromator chamber was kept in a vacuum of 2×10^{-6} Pa. The width of the entrance slit was set so that a radiation beam of 2 mrad in horizontal divergence was incident on the monochromator. The wavelength resolution $\Delta\lambda/\lambda$ was 2×10^{-4}. In order to remove the heat load on the monochromator crystal

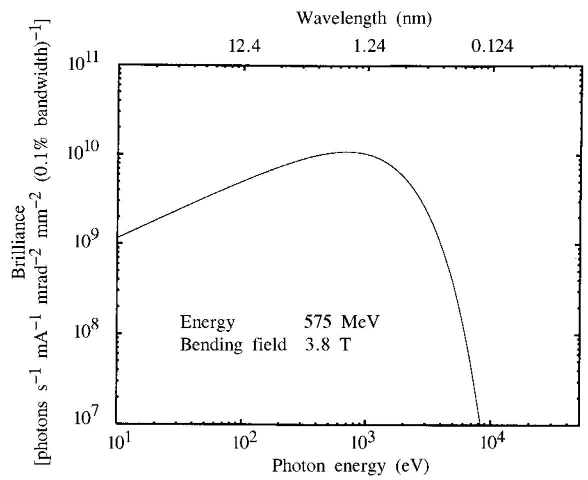


Figure 1
Spectrum of the radiation from the compact superconducting ring.

caused by irradiation by the primary beam, a graphite absorber, 0.3 mm in thickness, was inserted in the beam path. The ring was normally operated with an initial stored beam current of 300 mA.

The monochromated radiation beam exited the chamber through two kapton windows (12.5 and 50 μm in thickness) and was transported to the experimental station, where an X-ray precession camera with a single crystal on the sample holder was installed. The pinhole collimator attached to the camera shaped the incident beam to a cross-sectional diameter of 1–2 mm. Precession diffraction patterns were recorded with the imaging plate as a detector.

3. X-ray diffraction measurements using radiation from the superconducting storage ring

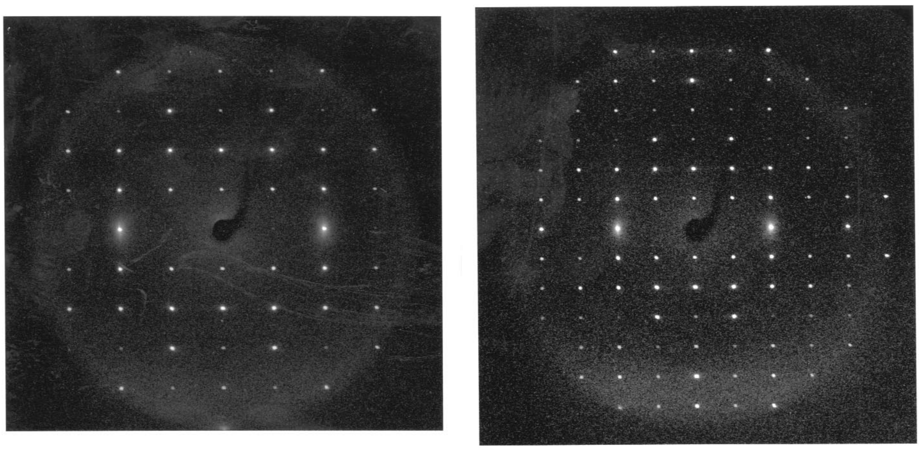
A series of diffraction measurements with changing wavelength were carried out using a single crystal of cholesteryl 2,2,3,3-tetrafluoropropionate ($C_{27}H_{45}OCOCF_2CF_2H$) (monoclinic $P2_1$ or $P2_1/m$, a = 1.244, b = 0.927, c = 1.330 nm and β = 106.0°) (Nakamura *et al.*, 1984) as a sample. Fig. 2 shows two of the precession diffraction patterns thus obtained, the wavelength of the radiation used being 0.207 nm (6 keV) (*a*), and 0.155 nm (8 keV) (*b*). The precession angle μ was 25°. The array of $hk0$-type reflections was recorded. The time required to take one pattern was 4.5 h, longer than that required at the X-ray beamline at the Photon Factory, as expected. The time with the 0.155 nm radiation is also longer than that for the measurements using radiation from a high-power X-ray generator with the rotating copper target. No comparable data are available for 0.207 nm radiation, but it is certain that the radiation from the superconducting ring is more intense for wavelengths longer than 0.155 nm, because there are no adequate target materials for a high-power X-ray generator.

The results of the diffraction measurements show that the radiation from the superconducting ring can be used in the wavelength range 0.155 nm (8 keV) to 0.248 nm (5 keV) for, *e.g.* intensity measurements of Bragg reflections from single crystals composed mainly of light atoms, that is, single crystals with low X-ray absorption. It is preferable to collect intensity data over an area of the reciprocal lattice in one pattern employing a two-dimensional detector such as the imaging plate, since this method is little affected by attenuation of the primary-beam intensity. Real-time measurements of structural changes employing counter-type detectors, on the other hand, are not appropriate, unless the diffraction intensity concerned is very strong.

Another example of the use of the radiation is a structural study applying the phenomena of anomalous scattering. One of the present authors made a structure analysis of a liquid-crystalline monosubstituted ferrocene derivative, {4-[ω-(cholesteryloxycarbonyl)decyloxycarbonyl]phenyl}ferrocene ($C_{55}H_{78}O_4Fe$) (Nakamura & Takayama, 1996). The structure is monoclinic, $C2$, with the lattice parameters a = 7.382, b = 0.588, c = 1.104 nm and β = 94.36°. The atomic arrangements were determined by applying direct methods. In order to lend support to the results obtained, the intensities of Bragg reflections were recorded using radiation of three wavelengths near and far from the K-absorption edge of the iron atoms on precession diffraction patterns, the absorption edge being at λ = 0.1743 nm (7.114 keV). Fig. 3 shows the wavelength dependence of the intensity of the $hk0$-type reflections. For example, the intensity of the 400 reflection decreases appreciably for radiation near the absorption edge, whereas that of the 800 reflection increases, the intensity of the 600 reflection remaining almost unchanged. This is in good agreement with what is expected from the atomic coordinates determined by Nakamura & Takayama (1996). It has been shown that the same is true for other reflections, indicating the correctness of the atomic arrangements determined. The phenomena can also be used in the course of a structure analysis to determine to which reflections the anomalously scattering atoms make a significant contribution and, in a favorable case, the information obtained leads to the arrangement of those atoms in a unit cell.

4. Discussion

As described in §1, there is an increasing demand for synchrotron X-radiation and users are always concerned about insufficient beam time for X-ray diffraction and/or scattering



(*a*) (*b*)

Figure 2
Precession diffraction patterns of a single crystal of cholesteryl 2,2,3,3-tetrafluoropropionate taken with radiation of wavelength 0.207 nm (*a*) and 0.155 nm (*b*). The exposure time was 4.5 h.

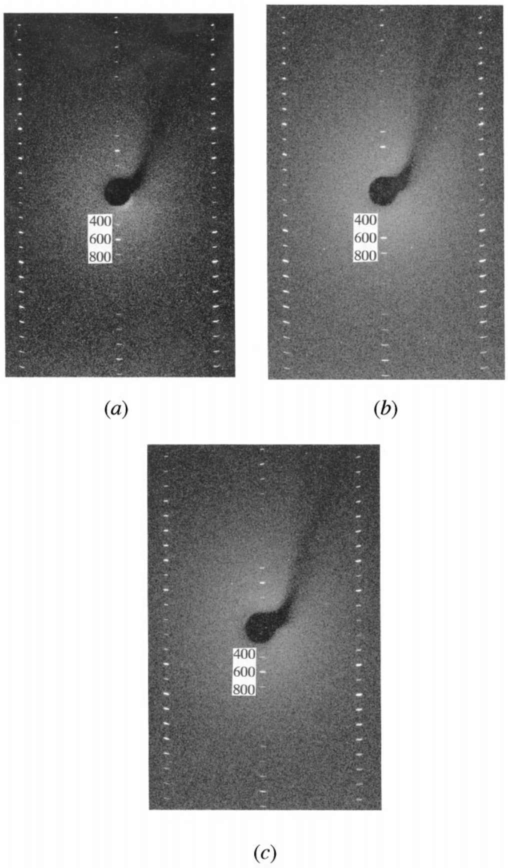


Figure 3
Wavelength dependence of the intensity of the *hk*0-type reflections of a single crystal of {4-[ω-(cholesteryloxycarbonyl)decyloxycarbonyl]phenyl}-ferrocene across the *K*-absorption edge of the iron atoms (λ_K = 0.1743 nm). λ/λ_K = 1.185 (*a*), 1.003 (*b*), 0.948 (*c*).

experiments. However, a large amount of investment is necessary for the construction of a large-scale storage ring, and it is not possible to have as many such rings as are demanded. On the other hand, the cost of construction of a compact superconducting ring is not prohibitively high and some X-ray diffraction and/or scattering experiments, which have previously been thought to be possible only at large-scale facilities, can, if appropriately planned, be made at a facility with a compact ring. Large-scale rings are mainly for those experiments which require radiation of shorter wavelength with a high brightness.

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