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Development of a compact six-circle goniometer

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A compact versatile six-axis goniometer has been built which can be used with the scattering plane vertical or horizontal for anomalous X-ray diffraction and diffraction X-ray absorption fine structure using a tunable X-ray undulator at SPring-8. The diffractometer consists of a four-circle goniometer for the standard ω, 2θ, φ and χ motions and a two-circle goniometer on the 2θ arm for mounting the detector and analyser crystal. The goniometer is controlled *via* Ethernet, and standard data-acquisition software with a graphical user interface has been developed. A closed-cycle helium cryostat is mounted on the ω axis for temperature-dependent experiments from 330 K to 14 K. Preliminary experiments on a $Bi_2Sr_2CaCu_2O_8$ single crystal demonstrate the performance. Superstructure peaks due to the one-dimensional spatial modulations in the *b* axis were recorded as a function of temperature above 20 K using an image plate.

Keywords: goniometers; XRD; DAFS; X-ray undulators; SPring-8.

1. Introduction

Recently, structural studies using synchrotron radiation have often required the combined use of X-ray scattering and X-ray absorption, or the simultaneous use of these two techniques, *i.e.* diffraction anomalous fine structure (DAFS) (Stragier *et al.*, 1992). For instance, in studies of high-T_c superconductors, where the CuO_2 plane shows a local deviation from the crystallographic structure (Toby *et al.*, 1990) or a spatial modulation (Yamamoto *et al.*, 1990), both local and superstructural information are necessary for understanding the lattice effects on high-T_c superconductivity. In a recently proposed model the two components, locally distorted (localized) and undistorted (itinerant) domains, form a spatially modulated two-dimensional superlattice, which is related to the enhancement of the critical temperature T_c (Bianconi, Saini, Lanzara *et al.*, 1996; Bianconi, Saini, Rosetti *et al.*, 1996). For single-crystal samples, X-ray absorption and X-ray anomalous scattering (Bianconi, Lusignoli *et al.*, 1996) provide the ratio of locally distorted to undistorted components and the modulation period, respectively. Combining these techniques, the width and frequency of the stripe structure are obtained.

It is desirable to measure both X-ray absorption fine structure (XAFS) in a fluorescence mode and anomalous scattering in the same experimental hutch. We have designed and built a compact Huber six-circle goniometer for joint experiments of XAFS and X-ray anomalous scattering or DAFS so that the two apparatuses are arranged in tandem at the undulator beamline XU-10 of SPring-8. In this report, we describe the fundamental specifications of the compact goniometer and evaluate its performance by observing the incommensurate modulation of $Bi_2Sr_2CaCu_2O_8$ (Bi2212) at various temperatures from 300 K down to 14 K.

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2. Diffractometer

2.1. *Goniometer*

We have built a small six-axis goniometer which can be used with the scattering plane vertical or horizontal for use with a tunable X-ray undulator. The diffractometer consists of a four-circle goniometer (Huber 424 with a 511.1 Eulerian cradle) for the standard ω, 2θ, φ and χ motions, and a two-circle goniometer (Huber 408 and 409) on the 2θ arm to mount the detector and analyser crystal. Since we plan to install the goniometer in the same hutch with an XAFS apparatus at the beamline XU-10 of SPring-8, efforts have been made to reduce the dimensions as much as possible yet to retain the precision required for a four-circle diffractometer. The inner and outer diameters of the χ circle are 250 mm and 340 mm, respectively, and the length of the 2θ arm is 550 mm. The angular resolution is 0.0001° for the ω, 2θ and ω_A axes, 0.001° for the χ axis, and 0.002° for the φ and $2\theta_A$ axes.

Fig. 1 shows a schematic illustration of the goniometer in a cradle for use in a vertical scattering mode. The left figure shows the front view of the goniometer looking upstream through the χ circle. The right-hand side shows the side view perpendicular to the incident X-ray beam. In this figure the X-rays enter from the left and pass through an entrance slit assembly and an ionization-chamber i_0 monitor before striking the sample. In a horizontal scattering-mode operation for either in-plane surface diffraction or use with a rotating-anode X-ray generator, the cradle is easily removed. The goniometer can be positioned using an air pad on a thin sheet of stainless steel in horizontal-mode operation.

For use at a synchrotron facility with vertical-mode operation, the six-axis system is mounted on a support stand with x, z and R_x (rotation) motions controlled by three stepping motors (the upper support stand in Fig. 1). The R_x motion rotates the goniometer and cradle in a horizontal plane about an axis which passes through the sample position at the centre of the χ circle. This motion is used for aligning the incident angle for grazing-incidence diffraction (GID) experiments (Feidenhans'l, 1989). The x-motion driving mechanism located underneath the R_x stage translates the goniometer in a direction perpendicular to the incident X-ray

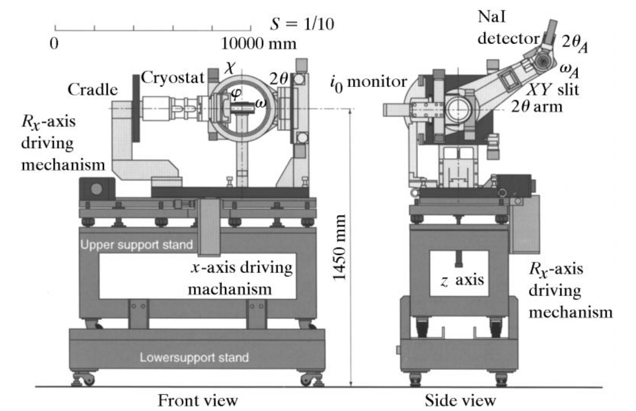

Figure 1
Front and side views of the six-circle goniometer with the scattering plane vertical. The left-hand figure shows the view looking upstream at the incident X-ray beam (front view) and the right-hand side shows the view perpendicular to the beam (side view).

beam. The z-motion mechanism lies beneath the x stage and controls the height of the entire assembly. The goniometer can be used at the Photon Factory (beam height 1225 mm) and SPring-8 (beam height 1445 mm). For use at the Photon Factory, only the upper support stand is used, while at SPring-8 both upper and lower support stands are required.

2.2. *Detector*

Fig. 2 shows a schematic diagram of the beamline optics and Huber control system. The incident X-ray beam intensity is monitored by an ionization chamber with a 140 mm-long electrode filled with dry N_2. A Bicron NaI(Tl) scintillation detector is routinely used on the 2θ arm. The output of a single-channel analyser (SCA) is counted by one of two programmable processor units (Labo Equipment, NT2400). The first programmable processor unit has two functions: as a stepping-motor controller for six channels and as a dual timer/scaler. A position-sensitive proportional counter (PSPC) with an effective length of 150 mm can also be used with two preamplifiers (EG&G ORTEC, Model 142A), two delay line amplifiers (EG&G ORTEC, Model 460) and a position analyser (EG&G ORTEC, Model 464). The second programmable processor unit is used as a multi-channel analyser. A Fuji BAS-III 20 × 25 cm image plate (IP) is used as a two-dimensional detector. Various combinations of adjustable slits and soller slits can be mounted on the 2θ arm. For high 2θ-resolution, a Si(111) analyser crystal is used, and for lower resolution but higher flux, an asymmetric (9° miscut) four-bounce Ge(220) analyser crystal (Crystal Scientific) is available. Ray tracing of the undulator optics (rotated inclined double-crystal monochromator and a double flat mirror) shows that a maximum flux of 10^{14} photons s^{-1} is achievable at the sample position.

2.3. *Cryostat*

For low-temperature diffraction experiments, a lightweight (3 kg) closed-cycle helium refrigerator (Air Products, Model 201M) can be attached to the Eulerian cradle. It has a cylindrical Be window, aluminized Mylar heat shield and a carbon-fibre vacuum shroud. Good thermal contact is achieved by both conduction and exchange with low-pressure helium gas in the internal shroud. With a Conductus LTC-10 temperature controller the cryostat provides a temperature range from 14 K to 330 K with ±0.1 K stability in most orientations. The temperature can be controlled either from the front panel of the controller or from the computer *via* an RS-232 connection.

2.4. *Data-acquisition system*

The goniometer is controlled by a custom-made workstation (Labo Equipment, UX-4100) with one Ethernet cable controlling all six axes and collecting data from the i_0 monitor, scintillation counter and PSPC (Fig. 2). For controlling six stepping motors, a programmable processor unit (Labo Equipment, NT2400) and six NIM-based stepping-motor drivers (Labo Equipment, SD-2020) are used. The former has the capability of independently controlling six axes and recording two outputs: the incident-beam intensity and the scintillation counter output. For the PSPC, the position is analysed and recorded by a programmable processor unit acting as a multi-channel analyser (Labo Equipment, NT-2400). The workstation runs either on a UNIX (Solaris 2.4) or Windows NT operating system. We have developed the data-acquisition (DAQ) software starting from a commercial four-circle goniometer control system, Gscan (Labo Equipment), using Microsoft Visual C/C++/Windows NT 3.5.1. The advantage of the present system is that all controllers are connected by Ethernet and a single Ethernet cable is introduced into the hutch. The DAQ software package features a user-friendly graphical user interface (GUI) making it easy to perform single-axis or $2\theta/\omega$ scans, two-dimensional reciprocal space maps or *hkl* scans using a slight modification of the standard formulation (Busing & Levy, 1967) for the different symmetry of this goniometer.

3. Performance

This system has recently been tested at the 27-pole wiggler beamline BL13B of the Photon Factory to measure X-ray

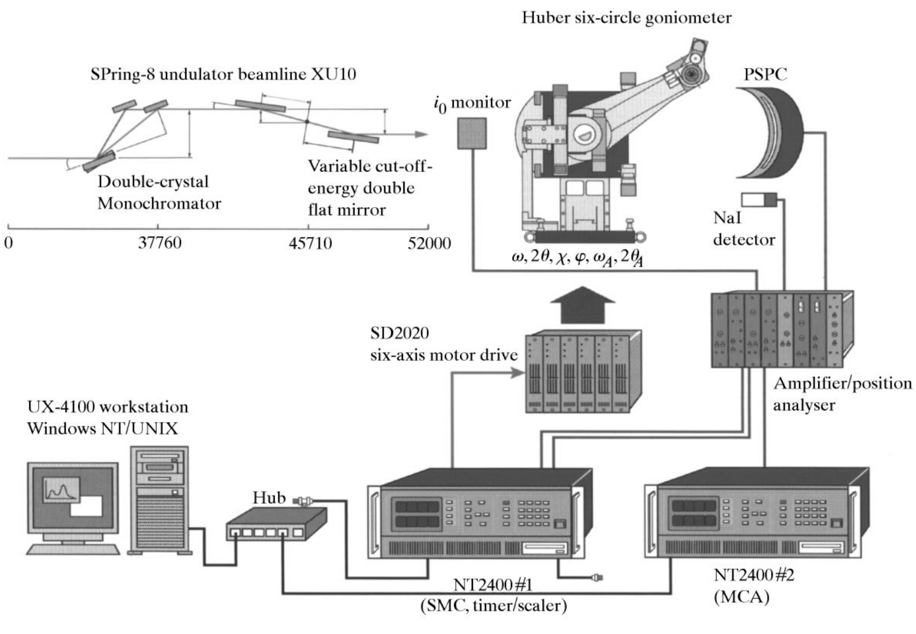

Figure 2
Schematic illustration of the optics and Huber system including a workstation, Ethernet, two intelligent processor units and drivers.

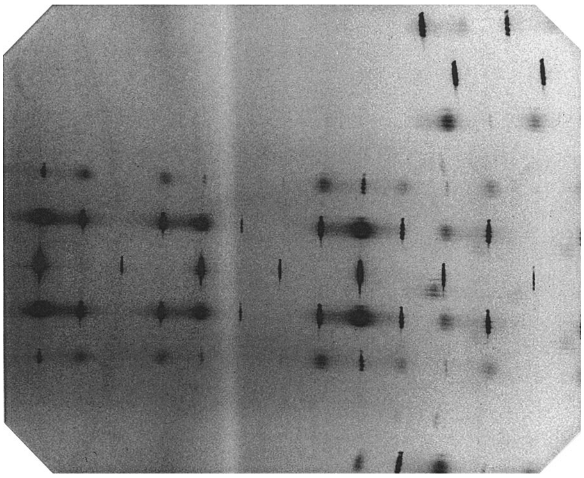

Figure 3
Image-plate data from single-crystal $Bi_2Sr_2CaCu_2O_8$ at 15 K obtained at the Photon Factory.

diffraction from a $Bi_2Sr_2CaCu_2O_8$ single crystal. This crystal has an incommensurate modulation with a superstructure vector of $\mathbf{q}_s = \beta\mathbf{b}^* + (1/\gamma)\mathbf{c}^*$ with $\beta \simeq 0.21$ and $\gamma = 1$. The sample was mounted on the cryostat without the cylindrical Be window, and image-plate data were taken at temperatures of 150, 100, 70, 30 and 15 K. Exposures were made at X-ray wavelengths of 1.4086 Å below the Cu *K*-shell absorption edge and 1.3788 Å above the edge. Fig. 3 shows image-plate data taken with the sample at 15 K and an X-ray wavelength of 1.4086 Å. Both superstructure spots and diffuse spots can be clearly seen. The indices range from $l = 12$ to 24 and $k = -5$ to 4, with $h = 0$. The data were obtained using the swing-scan mode, in which the φ axis (long axis of the cryostat) was continuously oscillated over a 25° range with a period of 12.5 s for a total exposure time of 500 s.

4. Summary

We describe a compact six-circle diffractometer designed for anomalous X-ray diffraction and DAFS using an X-ray undulator. Despite the small dimensions, the Eulerian cradle can support a lightweight closed-cycle helium cryostat with a high angular resolution. Stepping motors, counters and MCA are controlled by a workstation *via* Ethernet. This replaces intricate wiring into the experimental hutch by a single Ethernet cable. As a preliminary test, the modulated structure of the CuO_2 plane in $Bi_2Sr_2CaCu_2O_8$ single crystal has been investigated at various temperatures in the range 300–15 K. The installation of the goniometer requires a small area (1500 × 1000 mm), which is advantageous when sharing the hutch with other apparatus.

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