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Focusing monochromator and imaging-plate camera for grazing-incidence diffraction studies of thin films

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A multiple-imaging-plate detector system and focusing monochromator have been developed and successfully applied to the time-resolved study of phase transitions in Langmuir–Blodgett films by grazing-incidence X-ray diffraction (GIXD). The monochromator described here combines fixed-exit-beam height with sagittal focusing of the second crystal. The design is similar to that of Matsushita *et al.* [Matsushita, Ishikawa & Oyanagi (1986). *Nucl. Instrum. Methods*, A**246**, 377–379], with the exception that the motion of the first crystal is achieved *via* a computer-controlled *X*-*Y* translation table rather than a set of cams. The second crystal is a ribbed Si(111) wafer mounted in a four-point bending mechanism. The first reported application of imaging plates to a GIXD study was carried out by our group and proved to be very successful in the determination of thin-film structure [Foran, Peng, Steitz, Barnes & Gentle (1996). *Langmuir*, **12**, 774–777]. To extend the capabilities of this system, an imaging-plate camera was designed and built which can accommodate up to 13 imaging plates (40 × 20 cm) inside the vacuum chamber of the main diffractometer at the Australian Beamline at the Photon Factory.

Keywords: imaging plates; grazing-incidence X-ray diffraction; Langmuir–Blodgett films; phase transitions; focusing monochromators.

1. Introduction

The Australian National Beamline Facility (ANBF) has been in operation at the Photon Factory since 1993 (Foran *et al.*, 1994). The primary instrument, a large multi-configuration diffractometer, was originally designed as a high-resolution high-speed powder camera (Barnea *et al.*, 1992; Garrett *et al.*, 1995). However, its unique combination of imaging-plate (IP) detection and vacuum operation has proven particularly suited to several other techniques, especially grazing-incidence X-ray diffraction (GIXD). The combination of this instrument and a newly developed focusing monochromator has given the ANBF a unique capability in solid-surface GIXD.

In this paper we report an outline of the system used for GIXD experiments, especially the focusing monochromator which has made time-resolved experiments feasible.

2. Instrumentation

2.1. *Monochromator*

The key to being able to perform time-resolved GIXD is having sufficient X-ray flux at the sample position. This is achieved with a monochromator combining a fixed-exit-beam height with a sagittally bent second crystal.

There have been several designs published wherein fixed-exit-height capability is achieved with a single wavelength drive by translating one or the other crystal *via* a mechanical linkage (Golovchenko *et al.*, 1981; Cowan *et al.*, 1983; Garrett *et al.*, 1992; Matsushita *et al.*, 1986). The monochromator described here is conceptually similar to the Matsushita *et al.* (1986) design but with the important difference that the motion of the first (upstream) crystal is achieved *via* a computer-controlled *X*-*Y* table which replaces the mechanical cams.

A photograph of the internal workings of the monochromator is shown in Fig. 1 and a schematic representation of the principal elements is shown in Fig. 2. The monochromator operates in-vacuum ($\sim 10^{-7}$ torr). The wavelength rotation is produced by a Huber 420 rotary stage mounted outside the vacuum chamber, coupled to the monochromator mechanism *via* a double O-ring sealed shaft. Also, outside the vacuum on the wavelength rotation is a Heidenhain ROD 800 optical encoder. The first crystal [Si(111)] is mounted on a pair of cross-roller-bearing linear tables giving 250 mm travel parallel and 100 mm perpendicular to the crystal surface. The monochromator offset is fixed at 25 mm, necessitating 133 mm and 1.84 mm of motion, respectively, to achieve a fixed-exit-beam height over the energy range 4–20 keV. The parallel (X) and perpendicular (Y) positions for the first crystal are given by

$$X = \text{offset}/2\cos\theta\tan\theta,$$

$$Y = \text{offset}/2\cos\theta,$$

where θ is the Bragg angle.

The first-crystal mount incorporates a pair of cross-roller-bearing arcs for Bragg- and tilt-angle adjustments, and a three-

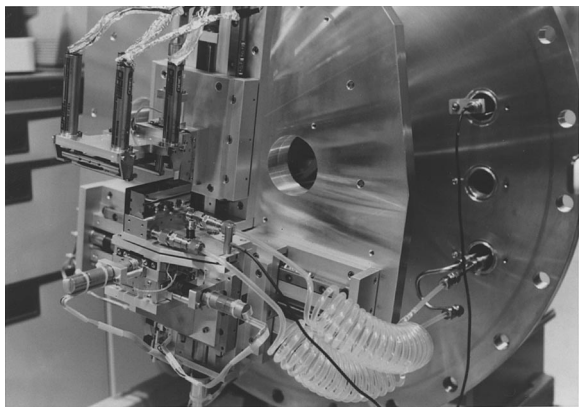

Figure 1
Photograph of the interior of the focusing monochromator looking from the upstream side. The water lines leading to the monochromator crystal are in the foreground. The DC encoder micrometers of the four-point bender are visible on top.

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point kinematic mount with a piezo actuator at one vertex for fine tuning of the Bragg angle. The crystal was manufactured by M. Hart and incorporates his water-jet-cooling scheme (Berman & Hart, 1991). All motions inside the vacuum are *via* DC encoder motors.

The second crystal is a ribbed Si(111) wafer mounted in a four-point bending mechanism adapted from the design of Stephens *et al.* (1992), in contrast to the triangle bender employed by Matsushita *et al.* (1996). The four-point bending mechanism gives superior control over such crystal distortions as twist and spiral compared with triangle bending mechanisms, and a cone figure can be introduced if required. Another advantage over triangle benders is that the horizontal position of the focus does not change as the crystal is bent. Prior to polishing and etching, the crystal dimensions were 90 × 40 mm, × 0.5 mm thick, with 5 mm-high ribs. It is held between four centreless ground stainless-steel rods. The inner two rods are fixed, and the outer pair are moved by four Oriel DC encoder micrometers, which have a resolution of 0.025 μm. The fixed rods were set parallel to within 5 μm, and any distortions of the wafer (*e.g.* twist) can be removed with the four actuators. The crystal ribs were cut with a diamond saw, after which the diffracting surface was polished to 400 mesh, and the whole crystal was lightly etched. This preserved the flatness of both sides of the wafer. The second crystal and bender assembly is mounted on a linear table allowing the crystal to be moved perpendicular to its surface to position precisely the crystal on the main monochromator axis of rotation and to allow the position to be fine tuned as the bend radius is changed.

The monochromator is positioned 10.9 m from the source. The usual focal position is inside the diffractometer at about 14 m, giving approximately the ideal 3:1 focus condition for a sagittally bent crystal (Sparks *et al.*, 1982).

The monochromator has proven to be very stable in operation, especially considering the number of motion stages in use. This is certainly in part due to the very effective first-crystal cooling provided by the water-jet system. As mentioned above, a piezo linear actuator is available for fine tuning/detuning of the Bragg angle, and a feedback system is available to control it. However, experience has shown that it is not necessary to make use of this system to achieve stability of the monochromator even over a period of days and the cross-roller-bearing arc is more than sufficient to tune the Bragg angle.

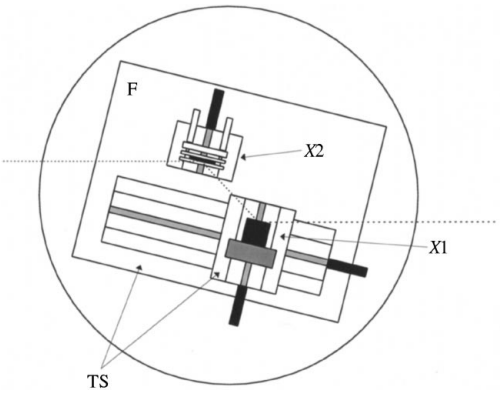

Figure 2
Schematic representation of the important elements of the fixed-exit-beam-height focusing monochromator at the ANBF. *X*1 = first (upstream) water-jet-cooled crystal, *X*2 = ribbed wafer second crystal mounted in a four-point bending mechanism, TS = translation stages for positioning the first crystal, F = flange on which the crystal mounts sit and which rotates about a horizontal axis through the surface of the second crystal.

The monochromator is primarily used for GIXD at fixed wavelengths, with a sagittally focused beam. The bent crystal accepts 1.8 mrad horizontally and focuses to an almost Gaussian profile spot with 0.8 mm FWHM at 14 m. This focus is approximately source-size-limited. At 1 Å the intensity gain was approximately 20, measured as flux into a 100 × 100 μm aperture with and without focusing. The vertical beam size increases slightly as a result of sagittal focusing. The gain into a horizontal defining slit (*i.e.* integrating vertically) was about 32. The monochromator rocking curve (rocking the first crystal) increased from 8 arcsec FWHM with an unbent second crystal to 11 arcsec with the focused beam.

2.2. *Diffractometer*

The multipurpose diffractometer in use at the ANBF has been described in detail previously (Barnea *et al.*, 1992; Garrett *et al.*, 1995). This diffractometer is particularly well suited to the type of experiment described herein as it incorporates in one instrument the features of precision, remote-controlled goniometers, a two-dimensional detector system (IPs) and the ability to operate in a vacuum environment.

2.3. *IP camera*

The primary design parameter for the IP camera was to allow multiple IPs [200 × 400 mm (Fuji)] to be loaded, exposed and stored inside the vacuum chamber of the diffractometer without the need to vent the chamber and re-evacuate between plate exposures. A detailed description and photograph of the IP camera may be found in the report of Foran *et al.* (1998).

3. Experimental technique and conditions

3.1. *X-ray beam*

The beam was focused in the plane of the IP detector to a horizontal FWHM of 0.89 mm. Slits were used to define a beam height of 50 μm in the vertical direction. These settings gave a beam with dimensions 1.6 × 0.05 mm (H × V) at the sample position. The vertical beam size was chosen so as to just cover the full length of the sample surface under grazing-incidence conditions. The angle of incidence used in these experiments was 0.18°.

3.2. *Exposure conditions*

Following sample alignment, IP/backing-plate assemblies were loaded into the camera and the diffractometer was sealed and evacuated to a pressure of <1 torr. Typical exposure times for a single IP were of the order of 2 min although this could be reduced to about 20 s without significant degradation of the signal-to-noise ratio when the low-Q and/or specular profile were the main area(s) of interest. The ability to scan such a wide area of Q space in such a short time has until now been impossible for traditional GIXD instrumentation. After the completion of a series of exposures, the diffractometer was vented and the IPs removed from the camera and loaded into magazines for scanning. A full description of the experimental procedure and data processing has been given by Foran *et al.* (1998).

4. Conclusions

The monochromator and detector system described allows the diffraction signal from a monolayer or multilayer sample on a solid substrate to be recorded over a large range of Q space in

much shorter times than previously possible with scanning detectors without compromising angular resolution or signal-to-noise. Utilization of the focusing monochromator and IP camera makes time-resolved GIXD experiments feasible. The usefulness of the technique has been demonstrated through the observation and measurement of a temperature-induced phase transition and intermediates in Langmuir–Blodgett multilayer samples of cadmium fatty acids (Peng *et al.*, 1997, 1998).

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