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The commissioning of a flexible low-cost multipurpose X-ray beamline at SRRC

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A low-cost multipurpose X-ray beamline has been commissioned at the bending magnet B15 of the Synchrotron Radiation Research Center (SRRC). The beamline is constructed in such a way to accommodate the various needs of small research consortia, from universities or research institutes, whose requirements of a beamline facility are quite diverse while under limited funding support. Flexibility is the special feature of this beamline. It is capable of performing quick test measurements without a prolonged reviewing process. Switching between different techniques, such as white-beam irradiation, EXAFS and X-ray scattering, can be achieved within 1 h. Novel experiments, such as energy-dispersive small-angle scattering experiments and energy-dispersive reflectivity measurements, can also be performed.

Keywords: X-ray beamlines.

1. Introduction

Recently, a flexible low-cost multipurpose X-ray beamline has been commissioned at the bending magnet B15B of Taiwan Light Source (TLS) in the Synchrotron Radiation Research Center (SRRC). In the following, we introduce its design philosophy and give two examples of novel experiments performed in this beamline.

In a modern synchrotron facility, the user demand for X-ray beamlines can be separated into two categories. The first is the dedicated X-ray beamline optimized for a single type of experiment. The best experimental results can be obtained without altering the beamline set-up, so that the beam time operation efficiency can be high. This is currently the most popular type in major synchrotron radiation centres. However, there is also another trend in synchrotron beamline use – the low-cost multipurpose beamline. Unlike most beamlines, which are optimized for single experimental methods, this beamline can be built at a low cost, without the complex set-up necessary for obtaining extremely high-quality experimental data, to provide quick access for materials or chemical scientists. The major design principle of this beamline is to accommodate the various needs of small research consortia, from universities or research institutes, whose requirements of a beamline facility are quite diverse while under limited funding support. Multipurpose beamlines are preferred because, for a small research team, it is difficult to form a group of researchers interested in using the same measurement technique and equipment. Such researchers are also unwilling to wait for a long reviewing time in order to obtain a few hours of beam time on a single-purpose beamline just for a quick check of their sample. Usually, routine measurements or quick tests of samples in the course of their research projects are needed occasionally and unpredictably. Therefore, flexibility is the most important feature of this beamline. To increase the flexibility, part of the beamline can be modified by users in a short period of time by the insertion of a section of vacuum pipe for any new novel experiment. At present, the beamline consists of three sections in series: the irradiation or lithography section, the X-ray absorption spectroscopy section and the X-ray scattering section. Switching between the three sections can be achieved within 1 h.

This beamline will serve the following purposes: (i) quick routine measurements, especially for industrial applications, (ii) quick beamline access, without a prolonged reviewing process, for the quick testing of new equipment and newly prepared samples, (iii) preliminary studies before a formal synchrotron run, and (iv) as a training ground for new students and first-time users.

The beamline configuration was laid out according to a survey of beam time demand. Routine powder diffraction and EXAFS are the two most time-demanding measurements. We put these two experiments in cascade, so that scattering experiments are performed downstream of the beamline to take advantage of the small beam divergence. Some of the time-demanding novel experiments, such as *in situ* or in-vacuum scattering experiments, are also performed downstream. Preparation work for these experiments can be taking place while spectroscopy experiments are being performed upstream.

2. Beamline layout

Fig. 1 shows a schematic drawing of the B15B beamline. The software-controlled double-crystal monochromator (DCM) (Hwang *et al.*, 1998) is the only major component in the beamline. On the upper stream of the beamline, a branch shutter consisting of a 15 cm tungsten block acts as a *bremsstrahlung* shielding for safety in the operation of downstream equipment. Two cascaded beryllium windows, each 125 μm thick, are placed after the branch shutter to isolate the beamline vacuum from the ultra-high-vacuum environment of the electron storage ring. Three experimental stations at different locations are operated in time-sharing mode. The first part of the beamline is a white-beam test-station for irradiation tests or deep lithography work. There are 150 CF six-way crosses located between the Be window and the monochromator. A filter ladder and sample-holder rod can be inserted into the six-way crosses through two 35 CF flanges for irradiation experiments. The second part is an X-ray absorption spectroscopy station which consists of two ion-chambers enclosed inside a 15 cm vacuum pipe. The white beam can be delivered downstream of the beamline if the first crystal of the DCM is moved out and the Ar gas inside the ion chambers is evacuated. The third part is a scattering experimental station. The energy range available is from 3.5 to 20 keV. Simple and routine Laue experiments, white-beam topography experiments and powder diffraction or other detector-testing experiments can also be performed with a flexible time schedule inside a small shielding box at the end of the beamline.

The final cost of this beamline is estimated to be a third of the cost of a dedicated beamline at NSLS or SRRC. The cost can be significantly reduced because (i) the beamline is only operated at 1 mtorr, (ii) there is no mirror on the beamline, (iii) there is no cooling water for the crystals in the DCM, (iv) there is no hardware linkage between the two crystals for the fixed exit configuration of DCM, (v) the DCM arm is small, (vi) a small shielding box (0.8 × 0.9 × 0.9 m) was used instead of a large shielding hutch, and (vii) no extra exclusive zone barrier is required because of a small 20 × 10 mm opening in the branch shutter which defines a small radiation-exclusive zone within the vacuum pipe along the beamline. The drawback of the beamline being operated at 1 mtorr is the loss of the capability to operate the beamline at photon energies below 3.5 keV. This drawback is partly compensated by the flexibility of changing the beamline configuration due to the low-vacuum environment. The drawback of having no mirror on the beamline is the loss in the intensity of the photon flux (by about two orders of magnitude) if a small spot (1 × 1 mm) on the sample is needed. Without the high-energy cut-off of the mirror, the highest energy range for EXAFS experiments is achievable. Without the need to consider the mirror contamination, operation of the whole beamline in a low-vacuum environment becomes possible. Furthermore, the beam divergence can be well defined by slits without complication. The beamline configuration becomes flexible, as there is no focal point defined by the pre-figured mirror, and different kinds of experiments can be performed at any location along the beamline. The DCM with no cooling water on the first crystal is stable if the vacuum chamber is back-filled with He gas. Since the critical energy of the TLS is only 2.14 keV, the first two Be windows actually remove almost all the deposited heat. Even without the He cooling, the temperature rise on the first crystal is less than 10 K. The software-driven DCM, without precision machining for the linkage mechanism between the crystals, further reduces the cost and enhances the flexibility of adjusting the offset between the incident and exit beams. This adjustability allows us to reduce the offset and hence shorten the length of the DCM arm when EXAFS experiments are performed at high energy ranges. Overall, the low vacuum, lack of cooling requirements, short rotating arm and software-driven DCM make the machine much more easy to fabricate and save a great deal of expense. Without the focusing mirror, the flux at 20 m is only 10^9 photons mm^2 s^{-1} (0.1% bandwidth)$^{-1}$ at 8 keV, which is about 100 times smaller than that of a dedicated beamline at SRRC at the focal point, but several times larger than the characteristic X-rays from a high-power rotating-anode X-ray machine. Typical EXAFS experiments can be completed within 1 h. The best way to utilize this beamline is to take advantage of the continuous spectrum and the flexibility of the experimental set-up. In fact, in our energy-dispersive experiment, which will be described later, the maximum counting rate is limited by the throughput of the energy-dispersive detector instead of the intensity of incoming photons of the beamline.

3. Examples

Routine experiments can be performed, including white-beam irradiation, X-ray spectroscopy, white-beam scattering experiments and mono-energetic beam scattering experiments. At the white-beam irradiation station, routine exposures of interesting materials were carried out. For example, the exposure experiment on PMMA (polymethylmethacrylate) is an essential step in the fabrication of micromachining parts. An LIGA (X-ray deep lithography *galvanik abformung*) pattern with 10 μm linewidth and an aspect ratio of more than 20 has been fabricated in this beamline (Cheng *et al.*, 1995). Although in this irradiation station the energy deposited on the sample by changing the filters is not well defined, the time flexibility provides a good opportunity for a quick and preliminary run. With narrow slits and a Ge(111) DCM in place to define the energy resolution, excellent quality traditional X-ray absorption spectroscopy experiments such as EXAFS and XANES can be performed. A solid-state detector can be set up on the side-flange of the sample chamber for X-ray fluorescence experiments. With mono-energetic photons delivered to the downstream shielding box, routine X-ray scattering experiments can be performed. In case high resolution is not required in a powder diffraction experiment, the DCM crystals can be changed from Ge to graphite within 1 h to enhance the intensity of the beamline.

Two novel experiments, energy-dispersive small-angle X-ray scattering (EDSAXS) and energy-dispersive reflectivity measurements, were performed during the first year of beamline commissioning. The energy-dispersive measurement is a unique method taking advantage of the continuous spectrum of the synchrotron radiation source. In an energy-dispersive measurement, all energies are registered in the detector simultaneously and the maximum counting rate is typically limited only by the maximum throughput of the energy-dispersive detector. Intensity is sufficiently high even without a focusing mirror. The EDSAXS

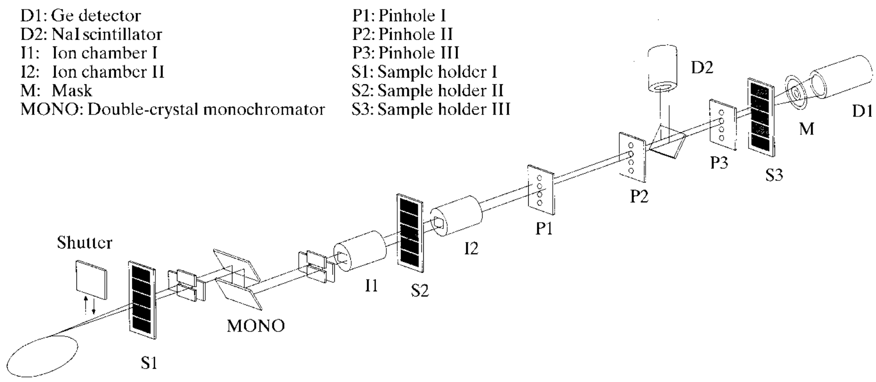

Figure 1
Schematic drawing of the multipurpose B15B beamline at SRRC.

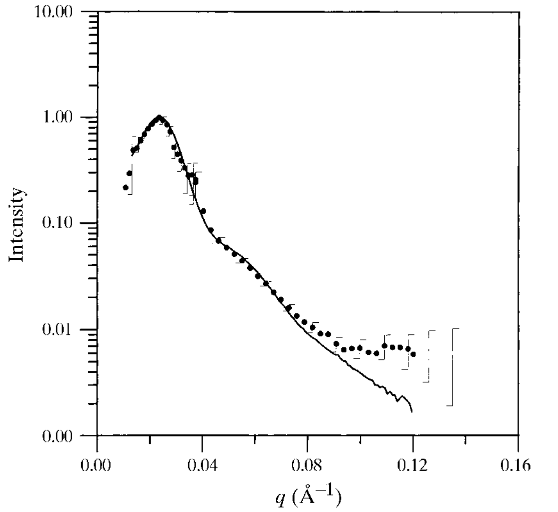

Figure 2
Experimental results of energy-dispersive small-angle X-ray scattering. The sample is cross-linked polyethylene. The experimental data (points) after correction are in good agreement with the measured results using Cu $K\alpha$ (solid line) photons.

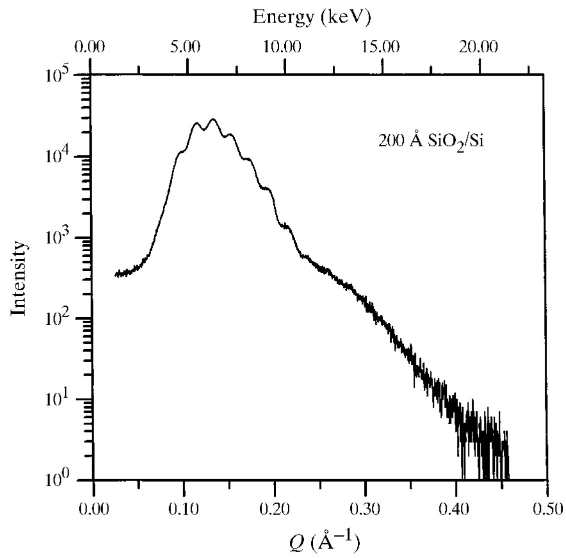

Figure 3
An example of the energy-dispersive reflectivity measurement (rough data before correction). The sample is SiO_2 on an Si wafer. The oscillation intensity of measurement indicates that the thickness of SiO_2 is 200 Å. The incident X-ray angle is fixed at 1.2° and the detector fixed at 2.4°.

is a suitable way to study the phase kinetics and/or morphology changes of solutions, sol–gels and other advanced material systems as a function of time.

In an EDSAXS measurement, a three-pinhole system was used to collimate the incident X-ray and to eliminate the parasitic scattering from the edges of previous pinholes. Downstream of the sample chamber, a pure Ge detector was placed inside the shielding box to collect the scattering photons of different energies. The scattering angle was defined by a mask placed before the detector. To align the whole EDSAXS system, all the pinholes and masks were coated with P-43 phosphor powder for easier visible alignment through the viewports. The resulting scattering vector range is from q = 0.005 Å^{-1} to q = 0.5 Å^{-1} with a resolution of about 8%. A standard sample, cross-linked polyethylene, was used to test the performance of this experimental method (Yu *et al.*, 1998). The measured result (shown in Fig. 2) is compared with those from the same sample measured with a conventional 18 kW rotating-anode machine with a 10 m SAXS set-up. We can see that the EDSAXS spectrum after correction looks very similar to the results of mono-energetic measurement and the counting time is actually 50 times smaller to obtain the same data quality.

We also used a similar set-up to perform the energy-dispersive reflectivity measurement. The sample was mounted on a rotation feedthrough for improved alignment. During the measurement, both the sample and detector were kept at fixed positions. The data were obtained within 1 min. In Fig. 3, we show an example of silicon oxide on a silicon wafer. The oscillating fringes reflect the thickness of the thin film correctly.

4. Conclusions

In conclusion, we have built a low-cost, flexible, multipurpose apparatus which is capable of performing X-ray scattering and absorption spectroscopy experiments *etc.* The flexible arrangement of this beamline allows a small research team with a limited budget to access synchrotron facilities to perform routine or novel experiments and to check samples quickly using the synchrotron light source.

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