High Resolution Inelastic Scattering (BL35XU)

1. Introduction

A great deal of progress has been made at BL35 since the 1998 report. On the one hand, there has been extensive work on the beamline itself, including installation of the undulator and front end, construction of the hutches, installation of the transport channel and optics tables, and first light in February of 2000. On the other, beamline personnel have been involved in a large R&D effort on beamline components (backscattering monochromator, analyzer crystals, temperature control system, NRS optics, detectors, timing electronics) using other beamlines, X-ray generators and BL35 itself. Here we describe some of this work. A sketch of the beamline is shown below (Fig.1). For details of the beamline design and background, please see [1].

2. Installation/Fabrication of Main Components

SPring-8 standard items, including the undulator [2] (Sumitomo) and front-end components [3] (Anelva), were installed in the storage ring tunnel in the summer of 1999. Hutch construction [4] (Kawasaki Heavy Industries) was completed late in the fall of 1999. Shortly there-after the monochromator mechanics [5] (Kohzu), transport channel components [6] (Ayumi) and optics table [7] (Kohzu) were installed, as well as the safety control system [8] (Hitachi Zosen) and the control system for the undulator, front-end and optics hutch [9].

3. First Light and Safety Tests

The shutter for the beamline was opened for the first time (under high power load) on 4 February 2000. Safety tests were completed for the optics hutch and the first two small experimental hutches (for nuclear resonant scattering). Safety approval of the two large hutches for inelastic scattering was received in June of 2000 and they will be tested in the Fall.

4. LN_2 Cooled Monochromator

The monochromator uses a new type of cooling system based on recirculating liquid nitrogen [10]. It was the first of this type of system installed at SPring-8. It performed sufficiently well for some commissioning work, but improvement is needed. In particular, the cooling capacity (~300 W) limits the maximum flux at small undulator gaps. This will be improved to >500W by increasing the capacity of the present refrigerators (summer of 2000) and adding one more refrigerator (by January of 2001). A more serious problem is the vibrations introduced by the cooling system. There are relative motions of the two crystals of the monochromator by ~ 5 μrad, sufficient to significantly disrupt the output X-ray divergence and to reduce the flux at higher energies. Presently work is under-way to both modify the LN_2 pump and to change the crystal support structure to reduce the vibration.

5. Measured Fluxes

Flux measurements were carried out using ionization chambers (and absorbers, as needed) with the results shown in Table 1. Note that these are preliminary *results only*, the flux is expected to improve as the monochromator cooling is improved.

Energy (keV)	Gap (mm)	Front-End Slit Size (V×H mm^2)	Flux in Si(111) (at 100 mA)
14.4	20.05	0.8×1.7	4.0×10^{13}/2.5eV
17.8	28.90	0.8×1.2	1.1×10^{13}/3.2eV
21.7	11.77	0.8×1.2	2.9×10^{13}/4.1eV
25.7	13.22	0.8×1.2	2.4×10^{13}/5.3eV

Table 1. Preliminary measurements of the X-ray flux after the Si(111) monochromator.

6. IXS Backscattering Monochromator

We investigated the use of a single reflection backscattering crystal as a monochromator for inelastic X-ray scattering (IXS). Proper functioning of

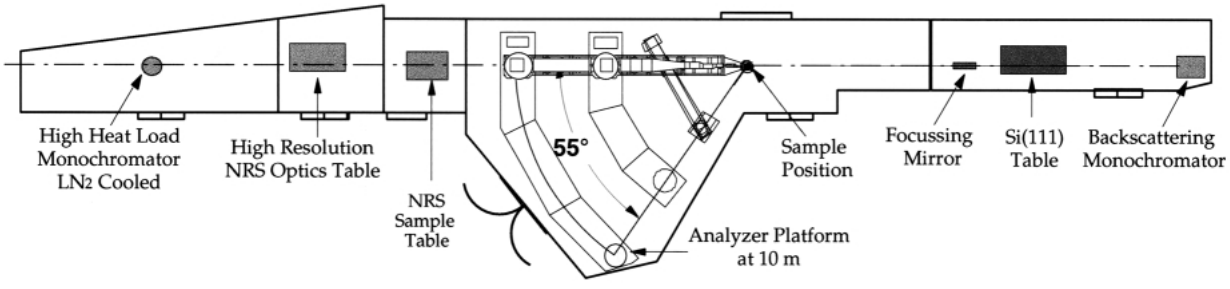

Fig. 1. Layout of BL35XU

the monochromator and, especially, the ~ mK temperature control are crucial to the beamline performance. After appropriate setup at BL47XU we were able to measure rocking curve as shown in fig. 2[11]. This is an important and necessary confirmation of the quality of our present system.

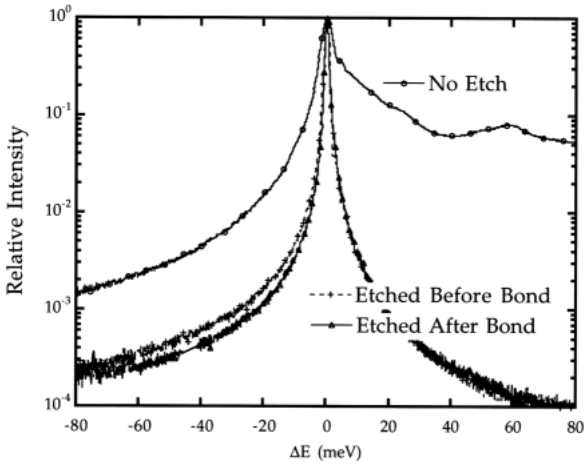

Fig. 2. Measured monochromator response functions - temperature (energy) scans in a two-crystal backscattering geometry.

7. IXS Analyzer Crystals

A crucial element of the inelastic scattering spectrometer is the analyzer crystal. This must be a large perfect crystal, but also must be curved into a focussing geometry. The required level of perfection means that it is not possible to simply bend a silicon wafer – the strain introduced by bending is too large. One solution (see [12]) is to glue many (>10,000) small (~0.6 × 0.6 × 2.9 mm^3) crystallites onto a substrate of appropriate curvature. However, this is a complicated technical problem requiring first careful cutting of silicon, then careful gluing and etching.

Beamline personnel have been collaborating with NEC Fundamental Research Laboratories to produce good analyzer crystals. While the use of several glues has been investigated, the desired goal is to use a high temperature, metal, diffusion bond. This bond is relatively insensitive to the etch and should allow one to do the etch only as the last step in the process – possibly improving the registration of the crystallites on the curved substrate. Figure 3 shows the response of a crystallite etched before bonding and one etched only after bonding [13]. The responses are essentially the same, showing that first bonding and then etching is a viable option.

In addition to being strain free, the crystals must be glued to the surface of the substrate in a way that keeps the appropriate orientation. This orientation has been mapped using an X-ray generator. Two early examples are shown in Fig. 4, demonstrating the

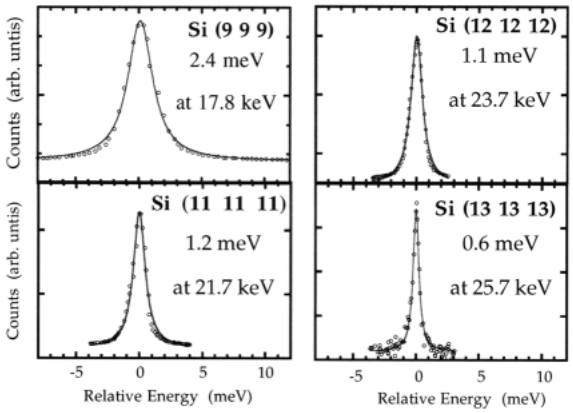

Fig 3. Strain in bonded crystallites. Measured using the Si (11 11 11) reflection.

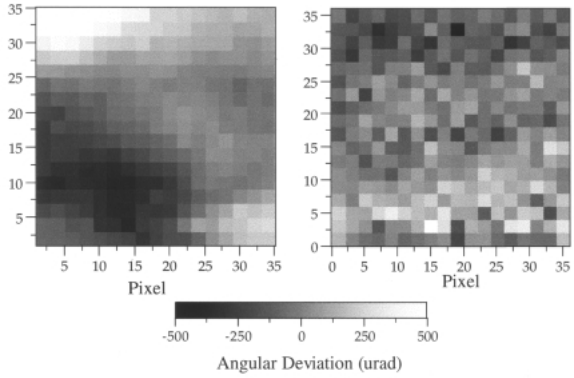

Fig. 4. Angular deviation of crystallites expoxied (left) and diffusion bonded (right) to a flat substrate.

difference in two types of bonding. Recently one sample (flat, ϕ10 cm) has shown an rms. deviation from ideal as small as 22 μrad [14] . This is cause for some optimism.

8. IXS Spectrometer

The main component for the inelastic scattering experiments is a large spectrometer with a 10 m horizontal arm and a 3m vertical arm (Huber Diffractiontechnique). This was delayed and installation should be completed during November of 2000.

9. Nuclear Resonant Scattering

The last two cycles of the 2000A were used to begin to commission the nuclear resonance scattering (NRS) station. New detectors were used, including a thick, large area, device from Perkin-Elmer (formerly EG&G) and a thinner one from Hamamatsu (see [15]). The former (10×10 mm^2 × 185 μm active thickness) provides high efficiency and a good signal to noise ratio, while the latter (ϕ3mm × 20 μm active

thickness) showed a good time response (< 200 ps FWHM, with small tail) and excellent signal to noise characteristics.

We commissioned a new type of high resolution monochromator [16] optimized for the 25.6 keV resonance of Dy. This had a bandwidth of 0.5 meV and a throughput of ~3.5×10^7 photons /sec. The bandwidth was exactly as expected from theory, but the throughput was about a factor of four less than hoped, due, primarily, to the vibrations of the LN_2 cooled monochromator (mentioned above).

10. Conclusion

Commissioning of BL35XU has begun in earnest, and most components are reasonably on track. With the safety approval of the last two hutches and installation of the spectrometer, improvement of the monochromator setup and analyzer crystals will become significantly easier.

References

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Source
Standard SPring-8 In Vacuum Undulator
139 × 32 mm periods, 8 mm min. magnet gap.
Fundamental: 6-18 keV; 3rd Harmonic: 18-50keV.
Electron Beam Size (FWHM):
35 μm Vertical × 950 μm Horizontal

X-Ray Beam Properties
X-Ray beam Divergence (FWHM):
~12 μrad Vertical × ~40 μrad Horizontal
Flux after Si (111) monochromator at 100 mA:
~ 10^{10} photons/sec/meV below 26 keV
Flux onto sample: Optics Dependent.

Experimental Facilities
IXS Spectrometer with Vertical and Horizontal Arms.
Optics Table for High Resolution (in line) Optics.
4-Circle Diffractometer (H. Scatt. Plane)
Closed cycle He cryostat (~10-300K).
LN_2 cryostat (~80-300K).
Furnace (~300-1200K).
APD, Si Diode and NaI Detectors.