BL35XU
High Resolution Inelastic X-Ray Scattering

1. Introduction

During FY 2001, BL35XU made excellent progress, as we completed in months work that has required years at other facilities. This very rapid progress can be attributed to good design [1], excellent preparation [2] and the hard work of the staff and good quality of the facilities at SPring-8. Specific milestones, after completion of the spectrometer mechanics in April, include first beam from an analyzer in May, commissioning of a full 4 analyzer high-resolution setup after the summer shut-down, first user experiments in October, and then commissioning of a phonon (medium-resolution) setup in February, as well as continued work on optics and detectors. One notes that while this rapid progress has been good for users, it has come at the expense of severely limited time for beamline staff to carry out their own research.

2. A Single Analyzer

After completion of the spectrometer mechanics, the beam was guided through our optics onto the sample position. Notably, our torroidal mirror performed well, giving a best focus of ~70x90μm² FWHM (and ~160x180μm² FWTM). First beam off the analyzer crystal gave a resolution of 1.7 meV for a reduced detector size (~1.4kHz maximum count rate from PMMA). Confirmation of the performance was done by examining phonons in silicon at the zone boundary, $\overline{Q}$ = (0.5, 0.5, 0.5), and the results of an over-night run are shown in figure 1. Note that the phonons appear very clearly even in this rather un-favorable (low-q) geometry. Note also that the elastic background from this perfect crystal is rather smaller than the phonon signal.

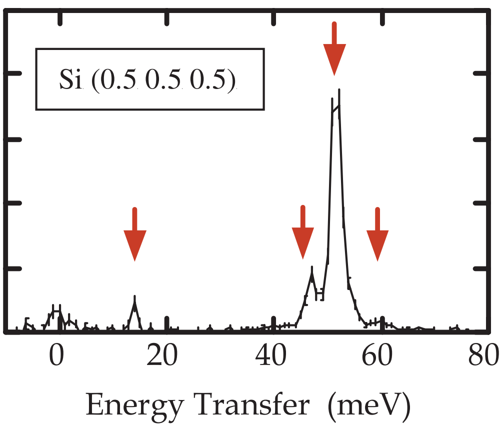

Figure 1. Phonons in silicon measured by IXS. Arrows show the expected energies from neutron scattering work as given by Kulda et al, PRB (50) 13347

3. Heat Load

One early problem that appeared was that the heat load from the ~100 mW of monochromatic beam from the Si (111) monochromator strained the backscattering crystal, leading to broadening of the energy resolution by a few tenths of a meV at 22 keV. This was fixed by replacing the normal incidence backscattering crystal with a grazing incidence one, reducing the heat-load per unit area by more than one order of magnitude, with minimal impact on beamline operation.

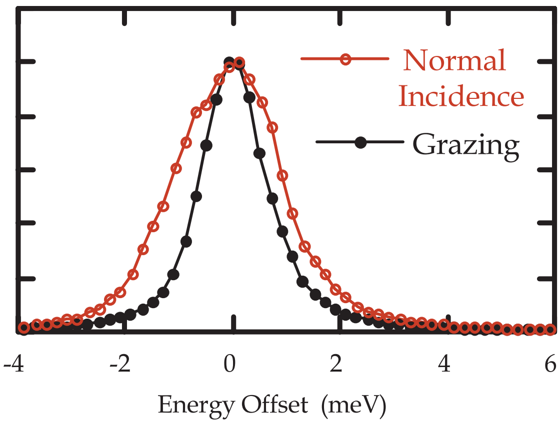

Figure 2. Normal vs. Grazing incidence. Going to grazing incidence removes the heat-load induced strain in the backscattering crystals.

4. Multi Analyzer Setup

Following the first tests of the single analyzer, we installed a 4 analyzer setup over summer shutdown. Aside from the obvious work involved in multiplexing from one channel to four (additional stages, additional temperature monitoring equipment, more complicated controls, etc), the main task here was replacing the single element detector with a multi-channel one. In principle the detector is straight-forward, requiring only that one have several elements located on a few mm spacing, with each channel needing high efficiency and low noise. However, this turns out to be somewhat difficult in practice. After extremely unpleasant experience with a 5 channel cooled silicon device from

Figure 3. Four analyzer crystal setup.

Eurisys Measures (it was delivered broken several times), we worked with Hamamatsu to make a 4-channel CdZnTe room temperature detector. This detector performs excellently in the area of interest, with background rates of about 2 cts/channel/1000s at 16 keV and 1 ct/channel/1000s at 22 keV.

5. Water

First performance tests of the beamline (after the silicon phonon measurement) were conducted using a water sample. This sample has been extensively studied at ESRF [3], while at SPring-8, the improved count rate (about a factor of 40 relative to that early work at ESRF), makes this a convenient test of the beamline performance, given the rather limited beam time before user proposals. Figure 4 shows some typical spectra and the fits. The measured dispersion of the mode agrees with earlier work [3].

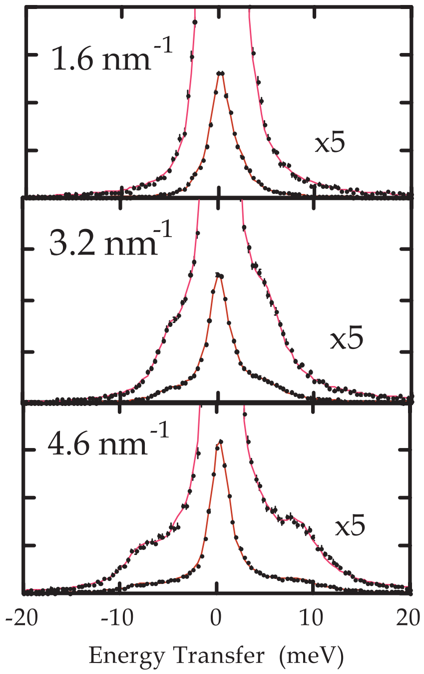

Figure 4. IXS from Water. Solid lines are fits.

6. Analyzer Improvements

In parallel with beamline commissioning we have been carrying out analyzer R&D with NEC Fundamental Research Laboratories. Beginning some 4 years ago, this has now led to reasonably consistent, high quality analyzer crystals, with improved count rates and resolution. The FWHM of the response has been improved, as well as the tails and the count rate. At present our best crystal gives ~1.5 meV resolution (FWHM) at 22 keV and about a factor of 3 higher count-rate than our first tests. At the end of FY2001, our 4-analyzer setup has crystals that vary from 1.5 to 1.9 meV resolution. One crystal is shown in figure 5 while resolution functions are shown in figure 6, and numerical values are given in Table 1.

Figure 5. Analyzer Crystal.

7. Phonon Setup

The high resolution (1.5 to 2 meV) setup is well suited to some classes of experiments : liquid excitations and other relatively low energy or narrow excitations. However, for other experiments, one would happily accept poorer resolution to get increased count rate. Thus a high-flux, low resolution setup is interesting. In particular, in response to user requests, such a setup was commissioned using the (888) back-reflection at 15.8 keV. This provided a resolution of 6 meV and a flux on the sample about an order of magnitude more than 1.5-2 meV setup at the (11 11 11). This was then applied by users to measure phonons in several materials. The various resolutions measured are shown in the figure and the table.

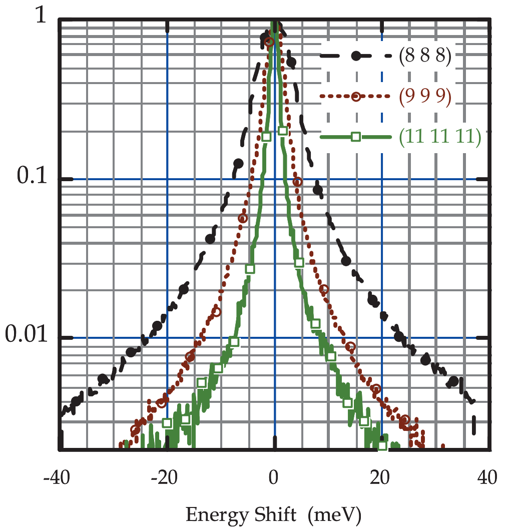

Figure 6. Measured resolution functions.

Si (n n n)	FWHM meV	FWM/10 (meV)	FWM/100 (meV)	Rel. Flux
(8 8 8)	6.3	15.3	47	8
(9 9 9)	3.1	8.3	29	2 *
(11111)	1.5	4.2	16	1

Table 1: Measured widths and relative flux at various silicon orders.

8. User Experiments

Beginning after the short test with water, the beamline was opened for user experiments. While detailed descriptions are available via the user reports, we note that successful measurements have been carried out with several sample, including liquid mercury, liquid magnesium, liquid silicon, molten salts, superconducting materials and a binary quasi-crystal.

9. Nuclear Resonant Scattering

While most of the time during FY2001 was devoted to IXS, some small part of the time was used for NRS measurements. Several user experiments employed the ^{161}Dy setup, which is now providing ~3e8 photons/s into a 0.5 meV bandwidth at 25.65 keV, with well optimized detectors for incoherent and forward scattering. Also, first commissioning of a setup for ^{119}Sn was carried out with a sub-meV bandwidth monochromator providing ~1e8/s. While this will be improved, it is sufficient to investigate nuclear inelastic scattering (NIS) and nuclear forward scattering (NFS) in various materials. In particular, first studies of diffusion using nuclear scattering in a non-iron systems were done on Cu_3Sn.

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