

## BL35XU High Resolution Inelastic X-Ray Scattering

### 1 . Introduction

During FY 2002, BL35XU<sup>[1]</sup> shifted to a user operation mode from a mixed commissioning / operation mode of the year before. As such, the emphasis on beamline activities shifted from that of basic operation to improving reliability, more understanding of detailed beamline performance, and, in some cases, improving convenience.

### 2 . High Heat Load Monochromator

The BL35XU high heat load monochromator was the first re-circulating LN<sub>2</sub> system installed at SPring-8, and has required some improvements. Notably, the cooling capacity was increased from 2 refrigerators to 3. With a cooling capacity of ~ 150 W/refrigerator, this is a significant improvement for smaller gap operations - allowing us to increase the front-end slit size at smaller gap settings without overloading the cooling. The path of the LN<sub>2</sub> through the crystals was changed from a parallel arrangement (with a "T" joint, and therefore some turbulence) to a series arrangement. Improved insulating ceramics were also put in, and heaters were added to stabilize the temperature of the stages after the insulators. Taking all of these together, increased fluxes onto the sample of ~ 30 to 40% were achieved, in the higher heat load setups (small undulator gaps).

A MOSTAB<sup>[2]</sup> was added to use feedback to stabilize the relative angle of the two silicon crystals. Note that we use feedback on the rather narrow 3<sup>rd</sup> harmonic radiation (Si (333) rocking curve) which means that even with a low set-point, the change in relative angle of the crystals is < ~ 1 urad over the course of the fill. At smaller gap settings, there remain some slow drifts in energy of the mono associated with current decay in the storage ring, but one hopes these will be removed when top-up operation is established.

### 3 . Backscattering Monochromator

The backscattering monochromator was improved by going from a 10 degree grazing angle (see last years report) to a 2.5 degree grazing angle. This has minimal effect on the diffraction properties of the crystals, but helps to spread the resultant power load of the incident (monochromatic) beam over a larger area - thereby reducing further the heat-load effects. This initially appeared to be sufficient to remove all heat load effects, and even to allow some margin for increased power load. However,

coincident with the improvement of the storage ring emittance we noticed some small worsening (~ 0.1 to 0.2 meV) in resolution that seemed to be, again, due to power load effects. This is currently being investigated.

### 4 . Analyzer Crystals

There has been general improvement in the analyzer crystals from NEC, but also some setbacks. It seems that resolutions of about 2 meV are relatively consistent, but it is not always possible to directly get to 1.5 meV - sometimes some additional processing is needed. At present we have three crystals that give 1.5 to 1.6 meV resolution, while the fourth gives ~ 1.8 meV resolution (at the (11 11 11) reflection, 21.747 keV). Nominally, the conditions of fabrication were the same for all crystals, so it seems there remain some subtleties in the process. Work is ongoing.

We investigated the effect of multi-beam contributions on the resolution function. Backscattering in silicon is typically not a unique reflection condition: for example, at exact backscattering at the (11 11 11) reflection, one also excites some 20 other Bragg reflections, the (2 14 8), the (5 5 15), etc. These reflections can lead to both reduction of the backscattered intensity (as x-rays are reflected in other directions) and to contamination of the reflected radiation by unwanted energy components. Thus, when operating just off of exact backscattering (as we do at BL35), one of the goals is to choose the azimuthal angle about the Bragg vector to minimize the effect of these other reflections. For example, figure 1 shows the resolution function measured from one analyzer as it is

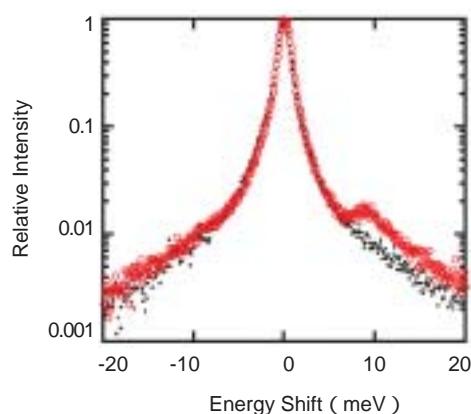


Fig 1 Measured resolution of one analyzer at two different rotations (a 15 deg. rotation about the (11 11 11) reflection.)

rotated about the (11 11 11) scattering vector (Y rotation). The appearance of the small bump in the tail in the resolution function is a multi-beam interference effect.

## 5 . Vertical Arm

We performed very first tests of the vertical arm of the spectrometer. This arm is interesting to go to higher momentum transfers (it goes to nearly 170 degrees in two-theta - as opposed to the horizontal arm which is limited to 55 degrees) and for the possibility of accepting a large solid angle (the smaller radius - 2.8 m- and larger analyzer crystals - 15 cm diameter lead to a factor of 20 increase in solid angle as compared to a single analyzer on the horizontal arm). However, the vertical motion of this arm and the short radius add some complications. In particular, the short radius both makes the space constraints at the sample severe and forces operation of the analyzer crystal further from backscattering. Thus the best observed resolution with this arm was about 3 meV. The vertical motion means the direction of the gravitational force on the analyzer crystal and its positioning mechanics is dependent on two-theta angle, leading to shifts in the analyzer alignment that must be corrected as two-theta is changed. Finally the smaller (2.8 instead of 10m) analyzer crystals do not yet seem to be of as good a quality as the 10m crystals used in the horizontal. All of these problems were noted during the first tests and are now being considered and worked on. More tests will be made.

## 6 . Software & Controls

Experimental control undergoes continual improvement (evolution) at BL35. Most control is done through computers running Linux (RedHat) and SPEC - using a mixture of VME (rcp-base) interfaces, Ethernet, and GPIB. The present IXS control system has evolved into a set of 6 interlocking spec versions. One control system (spectci) operates the transport channel components (monochromator, front end slits, optics hutch slits) via socket i/o with the beamline workstation. As these motors are not often moved, and as the i/o is slow, they are separated from the other control programs. Meanwhile most of the motors for beamline control are in another program (specixs) and the control for the temperature (energy scans) scans in yet another program (spect). In the background, there are three temperature readout programs that monitor the hutch temperature (specrtm) with ~ 30 mK precision, the backscattering crystal temperature (specbtm) with 0.3 mK precision, and the analyzer crystal temperature (specatm), also 0.3 mK precision. The room temperature measurement is primarily for reference and is not used on line, while the others (backscattering and analyzer crystal temperatures) interface with

the other spec via arrays in shared memory.

The use of multiple specs allows increased flexibility and speed. In particular, the separation of the temperature scan control and the motor control means that, if needed, positions of motors can be peaked up during a scan. Meanwhile the temperature readout, involving ~ 15 sensors, is relatively slow (due primarily to the speed on the interfaces and to the settling time as one switches between different sensors) and thus it is very useful to have it going on in parallel.

## 7 . User Experiments :

In general, user time is about equally divided between disordered (glass and liquid) systems and crystalline systems, with the emphasis for the crystalline system being strongly on superconductors. Thus over the past year we have investigated 5 different superconductors (mostly high Tc materials), 3 liquid systems (mostly at high temperature and/or high pressure), two glassy systems, as well as one quasi-crystal (and related materials). We also did our first work with samples in diamond anvil cells (pressures up to ~ 80 GPa). One notes that there were no nuclear resonance scattering proposals accepted in this period.

## References

- [ 1 ] A. Q. R. Baron, Y. Tanaka, S. Goto, K. Takeshita, T. Matsushita and T. Ishikawa : J. Phys. Chem. Solids **61** ( 2000 ) 461,  
A. Q. R. Baron, Y. Tanaka, D. Miwa, D. Ishikawa, T. Mochizuki, K. Takeshita, S. Goto, T. Matsushita and T. Ishikawa : Nuclear Inst. Meth. **A 467-8** ( 2001 ) 627.
- [ 2 ] Originally from DESY, this was imported to SPring-8 thanks to the efforts of Y. Nishino with help from T. Kudo.

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