

# BL35XU

## High-Resolution Inelastic X-Ray Scattering

### 1. Introduction & Overview

During FY 2010 (April of 2010 - March of 2011) the BL35XU scientific staff included A. Baron, S. Tsutsui and H. Uchiyama, with additional support on specific issues from D. Ishikawa, H. Fukui and D. Ellis. Technical support, from SES, was T. Oguchi with help from M. Hanada.

Scientific work at the beamline continued along similar directions as previously, including both crystalline and disordered materials. Work on high pressure systems expanded, while the iron arsenide materials dropped nearly to none.

### 2. Continued Benefit of the ID Upgrade

In the spring of 2010 the ID at BL35XU was upgraded, and while this was mentioned in last years report, it is worth mentioning again. In particular the x2 to x3 increased flux allowed some proposals to be shortened, and, in some cases, where single spectra were measured for two or three days, allowed very beautiful data to be collected, for example, confirming the presence of transverse acoustic modes in a liquid.

### 3. Sensitive flat panel detector

An important improvement at BL35XU this year was from the installation and commissioning of a flat-panel detector. Several experiments benefit from this, most notably those at high pressure. The detector installed is a Hamamatsu Photonics C9732DK which has 2400 x 2400 Pixels (50  $\mu\text{m}$  pixel size) over a 120x120 mm<sup>2</sup> area. It is mounted on a motorized stage allowing it to be moved into place reproducibly: to use the detector, the large two-theta arm is moved to high angle, and the detector moved in from low-angle side, about 120 mm behind the sample. It sees, mostly, negative two-theta angles. Figure 1 shows a typical measured pattern from a polycrystalline sample under pressure.

The detector is used to confirm the sample has been properly placed into the beam (the absorption contrast of the very thin samples in high pressure experiments is frequently very small), to check the orientation of single crystals, and to confirm the quality of the sample – texture, uniformity, and, in some cases, lattice constants (using the IPAnalyzer software developed by Yusuke Seto) – see figure 2.



Figure 1. Powder pattern from an iron foil in a diamond anvil cell (DAC) at 53 GPa (work by Ohtani, *et al.*). Rings are from the Re gasket and the Fe sample. Purple sections were not included in the analysis, and the grey patch at the center of the rings is the beam stop. Some care is required because the direct beam, with > 10 photons/sec is enough to damage the detector.

### 4. Backscattering Crystal Quality Issues

There were significant problems during the year with the resolution of the backscattering monochromators. BL35XU operates with two independent backscattering crystals, allowing a second crystal to be (*relatively* quickly) swapped in if the resolution of the one crystal degrades. This typically is needed on a few-month time scale (depending on detailed operating conditions), as the footprint of the monochromatic beam builds up and leads to local strain,  $\Delta d/d$ , at the level of a few parts in 10<sup>8</sup>. However, during this year it was found that the newly polished crystals performed poorly, with typical resolution of 1.7 to 2 meV, or worse, and sensitive position dependence, where 1.4 to 1.5 meV had been previously observed. This appears to be the result of a bad polishing run, with the polishing introducing deep strain into the crystals that was not removed with the usual application of a “strain-free” polishing step at the end of fabrication. Significant in house time was then used to try to understand this, and, as a stopgap measure, also to find positions on the re-repolished crystals with good resolution for user experiments.

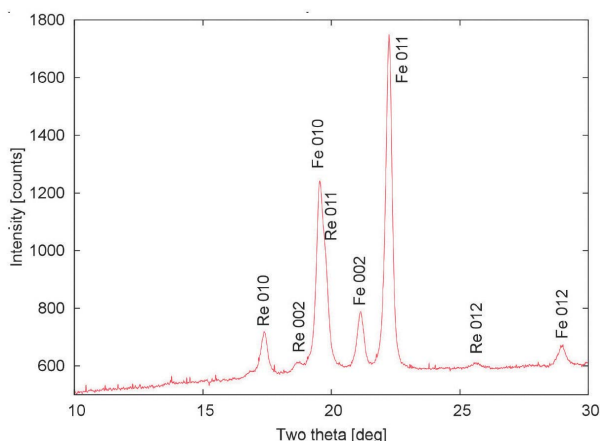


Fig.2 Projected results from figure 1 with the various rings identified. There are contributions from both the Fe sample and the Re gasket.

Figure 3 shows one of the polished crystals after applying a light etch. The presence of scratches on the polished side is what led us to wonder about the polishing run. Meanwhile the bottom, which showed no scratches at that level, had better resolution. Unfortunately, polishing is required to keep the beam size small – using an un-polished surface increase the beam size at the sample by nearly a factor of two.

At present (summer of 2011), we are trying alternative crystal fabrication processes to get rid of this problem – comparing polishing from two different companies, and enlarging the crystal size.

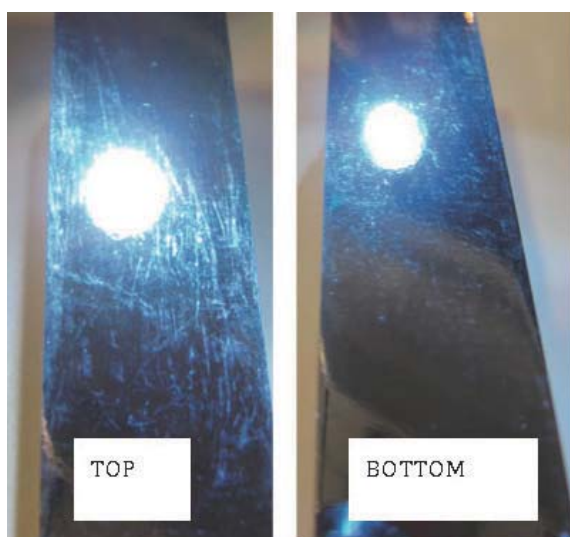


Fig.3 Backscattering monochromator crystal after a light etch. The top side side was polished, and before etching, looked pristine, without any scratches. The bottom side was not polished, and no scratches appeared after etching.

## 5. Next Generation Beamline

Effort continued to focus on BL43LXU, especially A. Baron, D. Ishikawa and H. Uchiyama. Work in this period largely focused on installation and interfacing of various components, details of implementation, getting control systems running, testing of delivered components, etc. This had great help from the SPring-8 Optics group, the XFEL alignment group and technical support from Yamamoto-san's group in RIKEN, with occasional instruction from T. Oguchi.

## 6. Other

Other improvements, reports, and changes include:

- The granite base for the spectrometer was re-aligned in the winter of 2011 but did *not* require re-alignment in the summer of 2011.
- Off-line tests of a new temperature control system were favorable, with sub-mK stability achieved (D. Ellis, D. Ishikawa).
- The computers at the beamline were upgraded (H. Uchiyama) to more modern machines with a recent operating system (Fedora 10). Some difficulties with serial port configurations with the older Heidenhain readouts have been noted.
- A remote controlled system of attenuators was installed.
- Temperature control electronics for the analyzer crystals were moved inside the hutch and onto the large two-theta arm to avoid moving the cables when the arm moved. This was an effort to improve stability and avoid occasional jumps of ~10 mK in the control point.
- A cooled Compton shield was installed on the mono, but then removed due to vibration issues.
- The control and readout electronics for the backscattering crystals were moved inside the hutch, near to the backscattering chamber to improve the stability of the AC bridge readout (successful).

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