BL35XU
High-Resolution Inelastic X-Ray Scattering

1. Introduction & Overview

During FY 2011 (April of 2011 - March of 2012) the BL35XU scientific staff included A. Baron, S. Tsutsui and H. Uchiyama, with additional support on specific issues from D. Ishikawa and H. Fukui. Technical support, from SES, was mostly 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 systems under high pressure in diamond anvil cells (DACs) continues to expand, including both solids and liquids.

2. More Extreme Conditions

BL35 XU has gradually been putting together a reliable setup for measuring samples in extreme conditions in diamond anvil cells. This began with the installation of a KB setup providing ~17 micron (FWHM) beam sizes and continued last year with the introduction of an area detector and the associated software to allow in-situ lattice constant determination (which is especially important when high temperatures are needed).

FY 2011, a high-resolution (~0.1 mm) long-focal length camera was installed to provide a precise reference for the sample position (see figure 1). When used with the area-detector, it is expected this will allow reproducibility of the sample-to-area-detector distance to better than 0.2 mm, as can be needed for exact determination of material lattice constants. One should note that, at the 0.1 µm level, it appears as if the sensitive surface of the detector may not be a perfect plane, so at large scattering angles the powder "rings" are not simple.

In collaboration with user groups, additional sample containers have also been designed to allow both internal and laser heating of samples in DACs. This is slightly tricky due to the small scattering angles desired in such measurements and the rather restricted space around the sample position, especially if the KB setup is installed. Good performance was achieved, however, with up to 1200 K via internal heating and up to 1800 K with external laser heating (though only from one side).

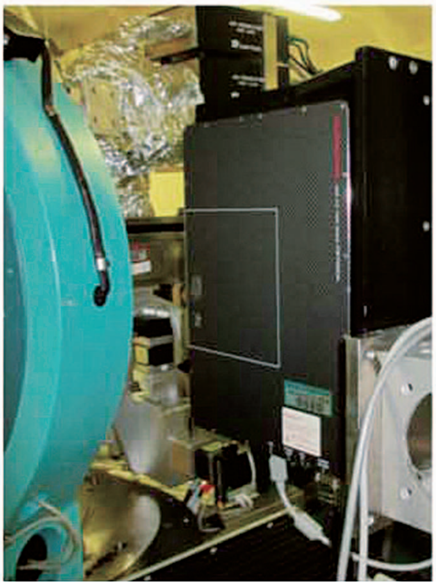

Figure 1. The upper panel shows the long focal length camera (right side) looking at the sample positions from a distance of about 1.5 m. It has a resolution of about 0.1 mm, The lower panel shows a photograph (from the opposite side of the spectrometer) of the area detector located behind the sample.

3. Severe losses at 7 GeV

Tests during 7 GeV running showed that the losses were severe, as expected from calculations of the ID performance. In the 1.5 meV resolution setup at 21.7 keV, the intensity on the sample was about 40% of that at 8 GeV. For poorer resolution at lower energy, 3 meV at 17.8 keV, the reduction was more manageable, with only ~20% losses. However, the bulk of BL35XU experiments require the higher, 1.5 meV, resolution. Further, it is expected that the highest (1 meV) resolution setup at 25.7 keV (which we will return to in 2012B) will be impossible at 7 GeV. For BL35XU, 8 GeV running is greatly to be desired. This sensitivity comes from having carefully tailored the ID period, 20 mm, to match precisely the desired operating conditions at 8 GeV energy.

4. Backscattering Crystal Quality Issues

After serious problems last year with the backscattering monochromator quality, a new polishing method has been employed and gives good resolution without sensitive position dependence.

5. Modified Monochromator Mount

During FY 2011 the backscattering monochromator mount, for one of the crystals, was upgraded. This, slightly bulkier design (see figure 2) included a relatively robust was of attaching sensor wires with a large teflon interface plate. It standardizes the electrical connections and seems to behave reliably.

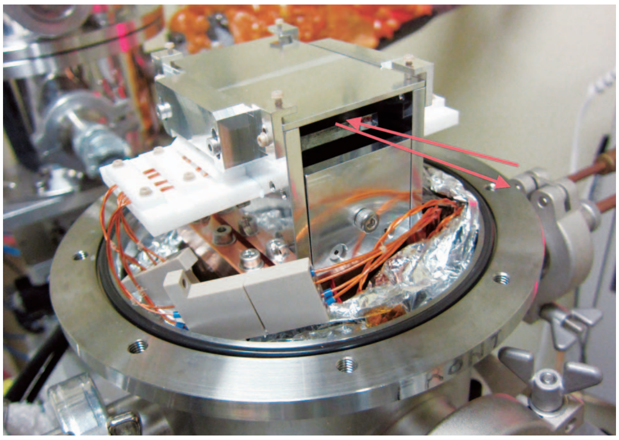

Figure 2. Backscattering crystal and its vacuum chamber. The crystal sits in a temperature-controlled cage inside of vacuum, surrounded by thermal insulation (largely removed in the present picture) and with a teflon interface for the several temperature sensors. In this picture the silicon is barely visible just inside the cage, and the beam directions are as indicated.

6. Low Temperature Refrigerator

Several years ago a low-temperature (He-flow) cryostat was purchased from Taiyo-Nissan in order to allow lower temperatures to be reached. A pulse tube setup was also considered but available designs were too heavy for the Eulerian cradle. It was expected that the He-flow cryostat would provide a minimum temperature of <3.5 K (as opposed to the ~10K typically available with the previous closed cycle system). However, this setup has been problematic – with acceptable stability at the lowest temperature but a lack of stability at higher temperatures, and a rather large He consumption (about 100 l/day). Many fixes were tried, and the problems have finally been traced to poor design of the He transfer tube. Borrowing a different transfer tube greatly improved the stability and reduced the He consumption (about a factor of two improvement at the lowest temperature). A new transfer tube is now being purchased.

7. Next Generation Beamline

Effort continued to focus on BL43LXU, especially A. Baron, D. Ishikawa and H. Uchiyama spent large amounts of time at BL43LXU. Work in this period largely focused on installation and interfacing of various components, details of implementation, testing of delivered components, etc. Commissioning of the BL43LXU started in this period.

8. Other

Other improvements, reports, and changes include:

- The granite base for the spectrometer largely remained stable and was not re-aligned during this period. This is a first, and may indicate, finally, stabilization of the floor under the hutches.
- Laser interlock systems were installed in both the preparation room and the large analyzer hutch to allow laser heating setups to be used with DACs.
- Ethernet cabling is now being upgraded from ~10 Base T to GBit with new hubs and cables installed where needed.
- It has been necessary to slightly close the FE slit size when operating at the Si (999) setup at 3 meV. This may indicate a loss of cooling power of the liquid nitrogen cooling system.
- After the upgrade of the computer system last year, there were intermittent problems with the ethernet interface turning off every few weeks on the main experimental station control computer, requiring a reboot. This, with lots of fiddling, now seems to be fixed.

Alfred Q.R.BARON
Satoshi TSUTSUI
Hiroshi UCHIYAMA
Daisuke ISHIKAWA
Hiroshi FUKUI