BL35XU
High-Resolution Inelastic X-Ray Scattering

1. Introduction

During FY 2007 (April of 2007 - March of 2008) the BL35XU scientific staff included A. Baron, S. Tsutsui and H. Uchiyama, with additional support on specific projects from members of the Materials Dynamics Laboratory, D. Ishikawa and H. Fukui. Technical support, from SES, was primarily T. Oguchi and H. Yahata. Aside from the usual tasks involved in keeping the beamline running, work focused on developing a long-scan-range monochromator for electronic excitations, optimization of a setup for high pressure diamond anvil cell (DAC) work, and a setup for liquid surface scattering. Effort, especially in collaboration with the Beamline Group, was also devoted to design of a next-generation beamline for IXS using a long-undulator. Some other issues are also discussed at the end of this report.

Scientifically, work continued in both crystalline and disordered materials, with publications in Nature Materials, Journal of the Physical Society of Japan, Physical Review Letters, Physical Review B, etc. Specific topics included phonons in quasi-crystals, superconductors, skutterudites, relaxors, as well as excitations in several high temperature liquids (Se, Fe, etc) and glasses Here we focus primarily on the instrumentation development allowing future innovative work and new directions.

2. Long Scan Range Monochromator

In recent years there has been increasing interest in using IXS to investigate electronic excitations, and, if possible, to do measurements with high resolution. Such experiments need to scan over larger energy transfers, with a desired range of～eV to make contact with other work. A backscattering monochromator while being easy and efficient for small scan ranges (<0.5eV), is difficult to scan over a large range－the same temperature scans that are comfortably matched to small energy changes, are awkward for larger transfers, with, as seen in fig. 1, an～80K scan range is needed to reach～3eV energy transfers. This was implemented by using a special low-temperature chiller and bath fluid, and required some effort to get the shielding and the sensor attachment appropriate to the large temperature differences needed.

This monochromator was successfully used for a first high resolution measurement of an electronic excitation at BL35XU, with an order of magnitude improvement in energy resolution compared to other previous work－in particular a d-d excitation in NiO[1]. The main issue in the experiment, after making the long-scan-range mono work, was signal rate, and signal to background

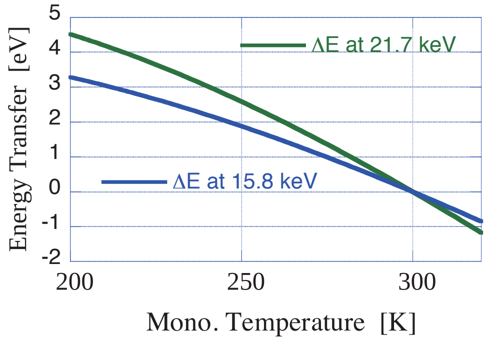

Figure 1　Energy offset between the backscattering monochromator and analyzer as a function of the monochromator temperature the (888) and (11 11 11) reflections (15.8 and 21.7keV, respectively).

ratio. Here the very low noise of the CdZnTe (CZT) detectors at BL35, with most channels having dark rates of about 1/hour, was crucial for observing signals of a few tens of counts per hour.

3. High Pressure Cells and Elastic Constants

High pressure experiments, e.g. in DACs, in principle, are not different than usual IXS experiments. In practice, however, they require experience and specialized knowledge to handle small samples in a cell that creates large background and limits angular access. We began a more concerted effort toward this type of work. The newly delivered Kirkpatrick-Baez (KB) mirrors were used to focus simultaneously in the vertical and horizontal, giving a spot of less than 20 microns in diameter (see fig. 2) and a throughput of about 50%. Proficiency was also improved in the setup, which requires refocusing the main beamline mirror to accommodate the KB mirrors. Work is now in progress to improve the flexibility of handling DAC－including easier access to more scattering geometries, an on-line

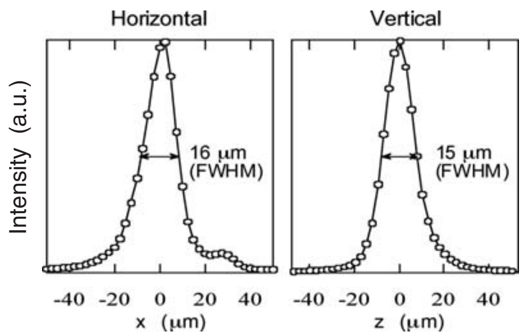

Figure 2　Focus measured with the KB setup.

alignment system. Separately, the requirements for precision measurement of elastic constants (of great interest for geology), were carefully investigated, with a demonstrated improvement of nearly an order of magnitude improvement over previously published work[2]. Finally, the use of diamond flight path for a cell with a thick epoxy gasket allowed experiments to be carried out at low momentum transfers without huge backgrounds from the small angle scattering from the epoxy[3].

4. Liquid surfaces

First test were made of a grazing incidence setup for measuring liquid surfaces. This, in principle, allows one to study the dynamics of the top few nm of a liquid, which can sometimes show layering, and can be expected to have a rather different dynamics than the bulk. The setup included the addition of a deflection mirror in the KB setup, to allow control of the angle of the x-rays onto the liquid surface, an active vibration suppression stage. First results with a Hg surface in air showed a strong effect. We are now in the process of commissioning a chamber for ultra-high vacuum (UHV) measurements, to allow a cleaner and more stable surface (fig. 3)[4].

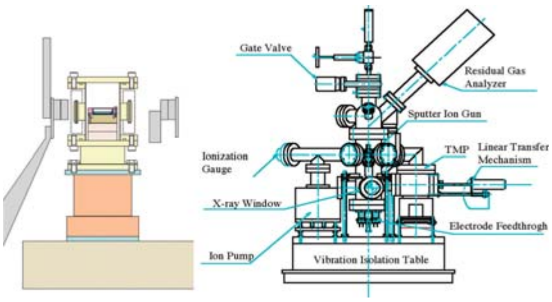

Figure 3 Setup for measuring a liquid surface. The left is a test chamber for air measurements while the right panel shows a chamber now being commissioned for UHV.

5. Next Generation Beamline

Work continues toward a next-generation beamline for IXS using a long undulator. More detailed design considerations were discussed with the beamline group and with the project appearing feasible despite the unprecedented power densities. The improvement, including an order of magnitude more flux (see fig. 4) onto the sample than presently available, should allow new experiments and new science, and is especially interesting for high-resolution investigation of electronic exciations.

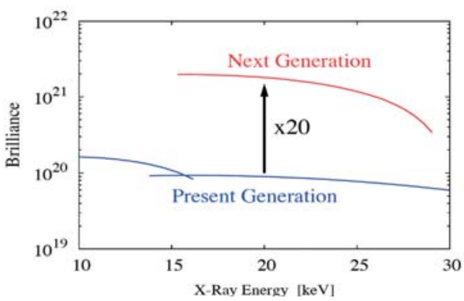

Figure 4 Improved Brilliance with a 20m ID.

6. Other

Two notable problems during FY2007 included monochromator vibrations and instability of the experimental floor. The former caused difficulties during the fall of 2007 and the problem turned out to be due to a mixture of an intermittent fault from a vibrating fan, and a general worsening of the monochromator behavior. The fan had been installed several years previously, but degraded and started to vibrate during 2007, leading to an intermittent intensity loss. As for the floor, during the first pat of 2008, drifts in the angle of the granite base for the two-theta arm, led to one occasion where the two-theta arm stopped moving. The immediate problem was corrected by beamline staff, but later those responsible for the original installation came back to re-align the granite. However, by summer of 2008 there were again indications of misalignment, possible due to continues settling of the granite, but, more probably, due to the nearby construction of new beamline, which may have affected the BL35XU experimental hutch floor. This will be corrected during the summer shutdown of 2008.

Other work included the purchase of the magnets for the eventual upgrade of the BL35XU undulator (expected in the summer of 2009), and during the summer of 2007, the removal of a Pt coating from the main BL mirror and replacement with a Ru, to improve the reflectivity by some about 7% − one notes that the focusing properties of the mirror were not adversely affected. A l-He refrigerator capable of reaching temperatures <3K was purchased at the end of the fiscal year. The closed cycle system, while occasionally reaching 7K, especially after a recent cleaning, is really only reliable to about 10K. Occasional user experiments require lower temperatures. A pulse tube design was also considered but was too heavy and large for the BL35XU Eulerian cradle. Finally a special collimator (similar to a Soller slit) was fabricated and tested - it should allow effective removal of background in some experiments.

[1] A. Baron et al.: work in progress.
[2] H. Fukui et al.: J. Synch. Rad., accepted.
[3] Y. Yomogida, T. Hattori and Y. Katayama : work in Progress.
[4] D. Ishikawa et al.: work in progress.

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