

10.3.2 User Newsletter

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This is the seventh user newsletter for Beamline 10.3.2, and the first which will be distributed only over the beamline website.

Website

Bob Sublett has done an excellent job of creating and maintaining the beamline website. It's your one-stop source for all beamline documentation, software, and links to other X-ray related sites. If you have a problem with the software, make sure you've downloaded the current version, as I make frequent small tweaks and bug fixes. Each executable is flagged with its modification date. If you have LabVIEW 6.1, you can use the sources and get the full functionality of the LabVIEW controls, which is not available in the executables. For instance, in a LabVIEW program, you can change the defaults and ranges on controls. This facility isn't available in the executable.

Where is the website? Since you're reading this, you've already found it, but may not have known it exists or where it was until getting an email notification. No, we didn't go out of our way to hide it; LBL has stringent policies about getting links to pages put into official web pages. They're quite picky about it. For instance, no page may be 'Under Construction' and no button may be dead. The page then must be approved by various people. Until all that happens, there won't be a reference to the site in any obvious place. Sorry!

Schedule

I've just seen the December-May schedule. It's really tight this time. The entire month of April is a shutdown for realignment of the ring. They've decided to do this every year instead of waiting three years. Also, half of December is already spoken for. I've also been asked to rein in the schedule to get rid of the spillover. Therefore, there are a lot of disappointed users. I apologize for this, but that's what I have to work with.

Jeremy Coyne and Gary Krebs are starting to take over user scheduling. Please CC them on any time requests.

Optics and Vacuum

We put in the old spare M2 and the beam looks much better. There's still some vertical scatter, but it's not nearly as bad as the most recent users have seen it. Still, I can't seem to get the vertical spot below about $7\mu\text{m}$, for reasons which elude me. The horizontal is good, bottoming out at around 3-4 μm . Thus, if you need really small spots, you might want to make the horizontal the small direction. The M2 we had in there got coated with more hydrocarbons, showing that even with the Huber slits out, there's still something smelly in there. We've since fixed some leaks in the mirror cooling lines and the vacuum in the box is in the high 7's. An RGA scan was just done and showed some hydrocarbon peaks at 53,55 and 57amu, the biggest at 57. A likely culprit is the 3" segment of vinyl roughing hose connecting the optics box with the tube entering the hutch. We've just replaced this tube with a bellows section. When the money turns on in October, I'd like to install an ion pump on the cross just downstream of the roll slits, where the PIN diode monitor is. Another thing to do when money reappears is to buy a new M2 so that we have a spare.

We've added a new type of current monitor to the above-mentioned cross, one which doesn't require that beam be interrupted. This consists of a 25 μ m Be window mounted on insulators, on the same stick as the PIN diode. It's biased through some battery boxes and we read the drain current from it. It's not as sensitive as the PIN diode is, but you can use it while scanning. We've ordered a thinner window so that it won't interfere with low-energy running. Eventually, we will have software which will automatically tune M1 roll when requested. The request can be issued directly by the user or automatically every N scans by the EXAFS program, N being user-settable. As vaporous as this all sounds, there is a real plan to do this, with a commitment of time on the part of Ed Domning, who's done similar things for 7.3.3.

We have had some trouble with stray beams which don't get monochromated and which hit the I_0 chamber. Even when such beams don't make it out of the I_0 chamber, they cause failures of normalization. These beams have been traced to small-angle vertical scatter from the roll slits and M2. We've dealt with these by adding a beam scraper between M2 and the mono, modifying the collector plate over M3 so that it leaves a smaller clear space above the mirror, and adding Pb-tape slits to the Be window at the exit of the box, just inside the I_0 chamber.

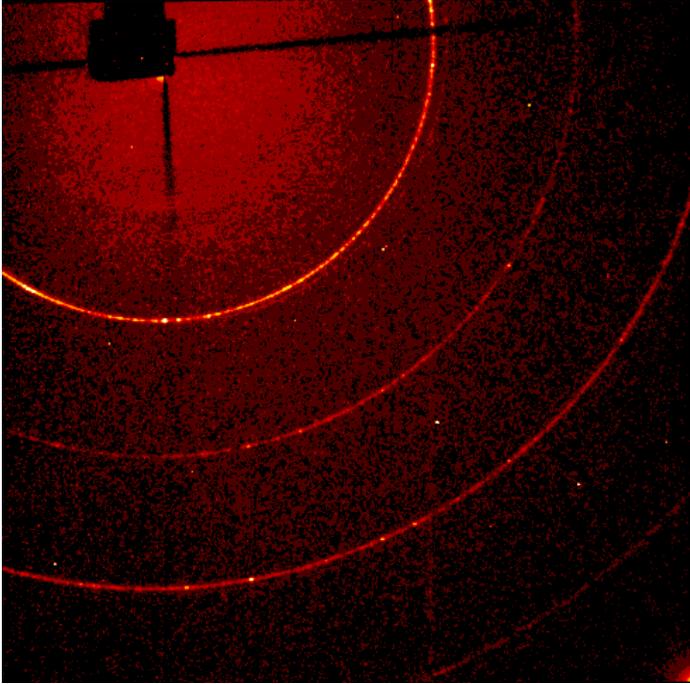
We have had a user who did Cl-edge EXAFS. He got his data, but at the cost of many hours spent building a beam-bag around the sample area. This area is very crowded with all sorts of things poking into it, so beam-bagging is a non-trivial exercise. Anyone thinking of low-energy work should be aware that it will take more time than you might think. Also, although M2 is better than it was, there's still a large amount of

vertical scattering which is worse at low energies (fractal roughness?) and shows up as a loss of contrast in maps and a loss of energy resolution.

Diffraction

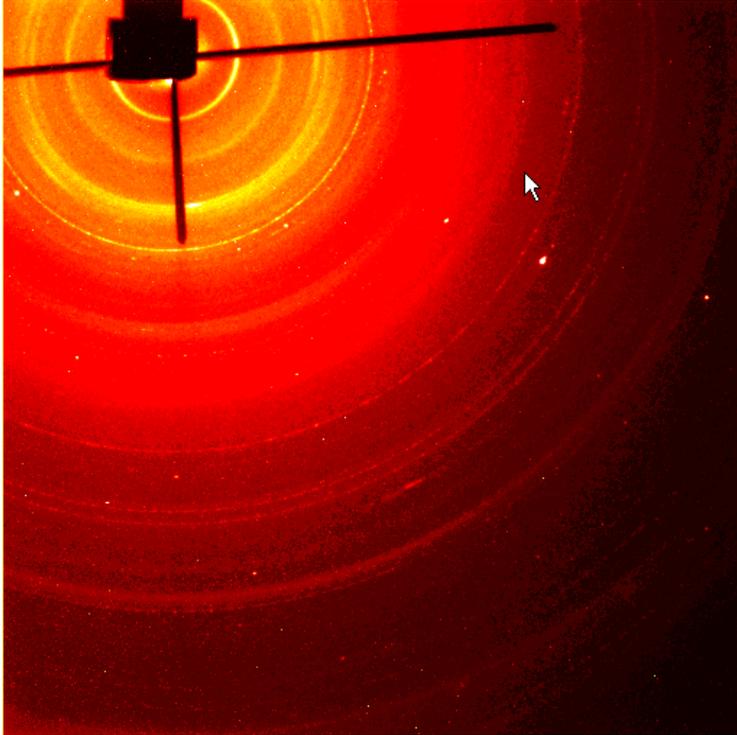
We have use the two-bunch time to get the diffraction camera working. We will post pictures of the setup on the website. The camera is a Bruker SMART6000. The software presently consists of the SMART and GADDS packages from Bruker. I'll have to work with Nobumichi Tamura on better analysis software. The detector can be moved along the beam, up and down, and rotated to catch the appropriate piece of k-space. In order to use the detector, one must dismount the regular EXAFS sample holder arm and install one we made from Newport parts and which has a single gripper to hold the sample instead of the kinematic mount. Thus, you can't put in a sample, take it out, and put it in again at the same place. However, you can do EXAFS and diffraction (transmission mode) at the same sample spot. This special arm might make beam-bagging a little easier in that a thin rod, rather than a big slab, holds the sample.

Here is an example of a diffraction picture for a 0.5 μm film of Fe, taken at 15keV, during 2-bunch mode, with a 10 second exposure time:



The fourth ring is at a d-spacing of 1.01 \AA . This was taken with the detector as close as possible to the sample. The dark cross visible at pattern center is a beamstop, consisting of a cross of W wire ($200 \mu\text{m}$ diameter). As you can see, it's slightly misaligned. Since some scattered light gets by the cross, we have a secondary beamstop consisting of a bolt mounted close to the detector; its shadow is visible coming down from the upper left. The bolt alone is insufficient because the primary beam causes intense air scattering. One ring is visible at 17 keV with a 1 second exposure time.

The Fe sample is useful for calibration, but isn't much like a real sample. In order to see if we can detect clays, we tried a sample containing a known clay species. This sample is a dried smear of an anti-diarrhea preparation (store-brand version of Kaopectate). The label claims the active ingredient to be attapulgite, which is a clay also known as palygorskite. A diffraction pattern, taken at 12 keV , 2-bunch time (40 mA), for 60 sec , is shown here:



The inner ring is at a d-spacing of 10.5 \AA . The sharp, spotty, intense ring is probably due to TiO_2 , which is claimed on the label. There is also evidence of texture, which is presumably due to flow alignment. We see that even with the detector close in (the geometry is the same as for the Fe picture) a 10 \AA ring is easily resolved. With the detector pulled back, we could probably get a 50 \AA spacing.

Our first experiment after two-bunch involved a liquid crystal made of CdSe nanoparticles. We used the diffraction camera to judge the amount and direction of the nematic ordering, based on the (002) ring, whose wavevector is along the long axis of the particle.

Detector

As mentioned in the last newsletter, the 7-element detector was getting gassy and some channels weren't working well. We've used the 2-bunch break to warm it up, pump it out for a couple of days, then cool it down again. The results are quite

gratifying. All elements now work properly, with the resolution they once had, and the low-level noise is much reduced. The total dark count, including the lowest channels, is down to 100/sec., whereas it used to be a couple of thousand. The background induced by a peak at energies lower than that peak is also down considerably; this will make it much easier to look at Mn in the presence of lots of Fe, for instance. There must be a leak in the dewar somewhere, though we were unable to locate it using the acetone squirt test. We didn't try squirting the Be window, out of fear for what might happen if we did.

Software

Almost all the programs now have manuals. We also have some new programs. For XRF maps, we have a program called 'Mix and match' which lets you take a channel from one map and transplant it into another. Thus, if you do a difference map, you can put the difference result for the element of interest into the map containing the other elements, making a composite map with all the information in one place. Another XRF program is called 'Decontaminate'. This is used when you have signal from one element which leaks into the bin from another, e.g. Fe getting into the Mn channel. The program lets you subtract some proportion of the contaminating channel from the counts in the contaminated channel. Use this with caution, as it's hard to know how much to subtract.

On the EXAFS side, I've fixed a bug in the non-linear least-squares (multishell) fit program. This bug disabled the 'Slave E0' mode. I've also written a new program, modeled on one from France, which allows you to test all possible linear combinations of one, two, ..., components to see what works best. It reads a reference database file which points to a set of reference spectra, tries all the combinations up to a specified number of

components, sorts them by goodness-of-fit, then reports the results. This program is as-yet undocumented.

I've just written a troubleshooting document which groups problems by the symptoms they cause. Bob plans to turn this into part of the web site, using frames to group related problems.