10.3.2 User Newsletter

January 2, 2005

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This is the eleventh issue of the user newsletter for 10.3.2. It's been more than a year since the tenth, so here's another issue.

Publications

We have 12 pubs with 2006 dates on them. Thanks and congratulations to the users. None of them are in *Science, Nature* or *PRL* yet, but not for lack of trying.

Shutdown and next cycle

As you're probably aware, we're coming off a long shutdown, which is why I had the time to write this. The intent of the shutdown was mostly to prepare for top–up operations, which will result in a large increase in average beam current as well as a decrease in the vertical source size. It remains to be seen whether we can take advantage of the smaller source. If nothing else, we should be able to put more flux into a 5μm beam than we do now. Watch out for that radiation damage! Here's what the ALS internal newsletter had to say about the shutdown:

"The ALS Fall 2006 shutdown is now underway. The facility is shut down for two and a half months, starting October 16, to accommodate some extensive work required in preparation for the transition to top-off injection. The main power supplies for the booster bend and quadrupole magnets are being replaced as well as the power supplies for the booster-to-storage ring transfer-line bend magnets. The booster radio frequency (rf) system is also undergoing major upgrades, with the replacement of the transmitter, the associated power supply, and a new transmission line into the booster. Significant changes and upgrades to the control and timing systems are also being done to ensure that all the new equipment works together.

Storage-ring work includes a major upgrade to the the Beamline 5.0 front-end optics, installation of a high-order-mode damper on rf cavity #1, outfitting all sectors with new beam-position monitors, installation of an experimental camshaft bunch kicker, and our annual swap out of the superbend magnet cold heads. User shifts are scheduled to return during the first week of January 2007. The plan is to operate the ALS with full-energy injection shortly after the installation shutdown. We anticipate slowly migrating to full top-off operation during the second half of the 2007 calendar year."

What you may or may not have heard is that an important power supply, which was delivered three months late, failed with series of loud reports and lots of flying sparks. That's why we won't get beam until January 17. Even then, it will take longer to inject than it used to because we'll be running in a kludgy mode which will require ramping from 1.2GeV instead of 1.5GeV. Two users have already been informed that their time has disappeared. There are discussions going on now as to what official ALS policy should be concerning loss of beamtime due to events like this power-supply failure.

Unfortunately, 2–bunch will not benefit from top–up, since the upgrade needed to perform bunch cleaning under top–up would cost too much. There is a move in the works to implement what's called 'quasi single–bunch mode' in which the camshaft bunch gets kicked into a different plane than the others, so a beamline looking at that plane would get only light that flashes on once per revolution. No word yet as to when or if this will happen, or how much 2–bunch, if any, it will displace.

What you may not know is that there's going to be another shutdown in May. This is to install the MERLIN undulator and also as contingency time in case top–up doesn't get done during the winter shutdown. That's why beamtime is extremely tight this coming cycle. If your proposal didn't get time, it may be just bad luck. In allocating, we had to make some tough and essentially arbitrary choices among proposals whose rating distribution was very tightly peaked.

Branching out – update

Last issue, I talked about nanoparticle and space–related projects. One of the nanoparticle projects, on looking at dichroism in CdSe nanorods [Deborah Aruguete, et. al.], is grinding slowly through the publication mill. Another, on using microfluidics to measure fast reactions [Emory Chan, UC Berkeley] is being written up. One on doped ZnO nanowires [Ben Yuhas, Sirine Fakra, et. al., UC Berkeley, ALS] has been submitted.

With Andrew Westphal, we have been looking at comet dust collected by the Stardust probe, not Genesis, as reported in the previous issue. Stardust is the one which *didn't* crash into the desert. Westphal gave one of the plenary talks at the ALS 2006 User meeting about this, highlighting 10.3.2 results as well as others. I don't want to be specific about results because there's a *Science* paper on the way regarding bulk composition, in which our work is merged with that from 5 other light sources, and there should be other papers with our XANES/EXAFS/XRD data.

Another user has started a project to look at Mesopotamian cuneiform clay tablets, with the idea of being able to correlate provenance with chemical and phase composition.

Changes

As usual, there have been lots of incremental changes to the beamline and analysis software. A prominent user pushed me to add certain features to the k–space&background removal and FT programs. He gave me source code for a tensioned–spline routine which I translated to LabVIEW and used to make the requested changes and add a smoothing function to the 2–column Editor program. The EXAFS Editor has a Noise Analysis routine which works something like the Measure panel in the EXAFS running program. It allows you to see how many effective counts you have, in terms of equivalence with ideal Poisson statistics.

The XY display program has a new feature: you can read in the header of an EXAFS file and have the cursor point to where that file was taken. This is handy for documenting which spectrum goes with which spot.

There's a new version of the MCA program which can automatically move from point to point and energy to energy, taking MCA spectra as it goes. I just wrote it during shutdown, so it hasn't been tested with actual beam yet. There's a possibility that we could integrate the Bruker detector into the system, which would allow an automatic multipoint XRD capability. It isn't quite diffraction mapping, but it's getting there. Another untested program is a new version of the XY mapping program which will allow linescans of the sort the STXM does, in which the sample scans in X and instead of Y incrementing at each line, the energy changes. Thus, reading vertically gives you an XAS scan of a point and reading horizontally gives you a linescan. This new mode could be useful for spectroscopy on rad–sensitive materials. The utility of this new capability remains to be seen; we'll just have to try it and see. Also tested, but not with beam, is the long-awaited version of the XY map program which lets you program in multiple maps, varying the positions or energy from map to map. This program will make difference mapping much easier and also has the linescan feature described above. Setting up scans is less convenient than with the single-scan program, so I anticipate using the old program for most things and the new one for difference maps and unattended operations, once it has proven itself reliable.

There's a program for generating k–mode scan definitions, in which the energy spacing and the time per point increase as you go out. This avoids the vast tedium of designing these by hand. Using k–mode seems to save time because you take fewer points, hence incur fewer instances of the 1.9sec overhead per point.

We have found that the monochromator glitches provide a nice internal energy calibrant. We still run foils at the beginning of a XANES run, but we find that the results are consistent with our glitch calibrations. We now use as calibration points the elemental edge energies from a paper (Kraft, et. al., *Rev. Sci. Instrum.* 67,681 (1997)) which tabulates newer, more accurate values for edge energies than those in the commonly–used tables. Thus, if you're used to the Fe K–edge being at 7113eV, don't be surprised to see it quoted and calibrated to as 7110.75eV. We keep a directory of scan definitions in which the region around a glitch is finely tabulated in order to enable internal calibration There is also a new button in the Scan Editor of the EXAFS code which enables one to add a region, even if the new region overlaps existing ones.

Spurred on by the Stardust project, we have a large set of Fe XANES standards, all calibrated to FeK=7110.75eV. This has proven very useful for various projects. Thanks to all the people who have contributed spectra to our database.

Another user wanted to run the fluorescence detector with a shorter shaping time than normal in order to get high count rates so as to deal with a severe background problem. We tried it and it works. Someone who has Zn in the presence of lots of Ni, for instance, might want to try this new mode. This "fast" mode does result in some loss of energy resolution, so it's not for every project.

We have an improved system for mapping an analog quantity such as the output of a transmission chamber. We feed pulses from the V/F into a converter box which makes them into step–functions that look like what comes out of a detector preamp, and then feed this into the unused 8^{th} channel of the XIA signal–processing modules. Our previous system was a horrible kludge in which pulses were jammed into the test inputs, which caused them to splatter over many channels. The new system makes a nice, narrow peak whose position may be adjusted as needed. It's also more linear than the old system. We've used it for transmission radiography as well as X–ray beam induced current [Istratov, Buonassisi, et. al.]. In principle, we could do this with any analog quantity or even pulse stream of appropriate frequency range. Thus, if you have a phosphor you want to map, you could set up a PMT or other light detector with a suitable collecting optic and stick the output of this into the appropriate input of our system.

We're still using the Bruker 6000, so I think this detector may be said to be permanently ours. We had a $6000 failure which turned out to be due to water condensing inside the box where the circuit boards are. I knew there was trouble when water dripped out of the detector after it was turned off. The Bruker tech put together a nitrogen purge line system which should prevent this from happening in the future. There's now a hard–stop which defines a standard detector–sample distance, thus easing calibration. One improvement I hope to make is to replace the secondary beam stop with a short He–purged flight path, sort of a cross between a SAXS flight path and the detector condom. A mechanical engineer has designed it and built a prototype out of cardboard, with a sheet-metal unit to come if the cardboard one works and doesn't bump into things too much. If it works, it will make XRD on weakly–scattering samples like phyllomanganates or ferrihydrite much easier.

Valeriy Yaschuk and Greg Morrison adjusted the KB set so that the M3 and M4 mirrors are properly orthogonal and that one of the picomotors isn't hitting the inside of the optics box as it used to. This enabled Sirine Fakra to tune the mirrors using knife–edge scans to a 3μm spot size at 10keV. This is about the best it's ever been. Also, I came up with the idea for, and Greg and Val built, a device for tuning the M2 (vertical collimator) mirror. It's a small, fixed–energy channel–cut mono using one of the crystals from the old 4–bounce mono that used to be on the beamline. The idea is that by running this in dispersive mode with respect to the main monochromator, angle gets encoded as energy, so an energy scan with the main mono yields information about the vertical divergence of the beam from M2. In order to do this test, M3 has to be dropped out of the way, for which there's (fortunately) enough range on the M3 translator. This device allows me to tune M2 without having to open the box, and is more quantitative than running Cu foil or organobromine–in–lexan scans.

We have a new computer to replace UXAS_DATA. Its name is UXASDATA. The FTP site is therefore ftp://uxasdata.als.lbl.gov/ . This new machine has a 250GB disk so it can retain all your data without the need for moving old stuff off to another drive. In addition, it has a 300GB drive with automatic backup which will back everything up each week. The new machine is faster and cleaner than the old one. We also got a bigger monitor, which makes it easier to find your way through the maze of desktop icons or to keep multiple windows open. The old machine is still up and running, just in case.

We've bought a new roughing pump to replace the loaner we've been using for the past two years and have had it installed on the roof so it doesn't heat up the hutch. Without the pump, the temperature equilibrates at 26C with no additional air conditioning. Moving the dry-air generator out also contributes to keeping our cool.

Low-energy upgrade

As mentioned last year, the low–energy upgrade is still on hold. However, the prospects for capital money are a little better than they were, so it's possible that we will get it. The upgrade would be more expensive than first estimated, since calculations show that the mono must be cryo–cooled in order to avoid having a big thermal bump on an InSb crystal. At this point, don't hold your breath waiting for it, but don't give up completely.

I'm trying to ride microdiffraction's coattails in order to get a new set of KB mirrors in hopes that they will solve the long–standing problem of the spot–size blowing up at low energies. This will be a major project if it happens at all, and would occupy some shutdown or 2–bunch time.

SAXS/WAXS

Beamline 7.3.3 is being changed from a microdiffraction line to a SAXS/WAXS line, while microdiffraction is moving to 12.3.2. How's that relevant to 10.3.2? I'm going to be in charge of 7.3.3 when it gets built, and will therefore split my time between 7.3.3 and 10.3.2. Thus, users will see less of me than they're used to. I've been promised that somebody will be hired to backfill me on 10.3.2. Anyway, I hope to figure out some experiment which uses both beamlines.

Thanks again!

I got an Outstanding Performance award, mostly on the strength of the scientific program at 10.3.2. This could not have happened without a steady stream of users bringing in good science, nor could it have happened without a lot of hard work from Sirine Fakra. She deserves a lot of credit for her work here, both in user support and in her own projects.