

Annual Meeting

— of the —

Advanced Light Source

Users' Association

October 20-21, 1994

Lawrence Berkeley Laboratory

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Organized by the
ALS Users' Executive Committee

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MASTER

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Preface

News of the exceptional science emerging from the first year of the ALS user program attracted more than 250 attendees to the Advanced Light Source Users' Association Annual Meeting, held at Lawrence Berkeley Laboratory (LBL) on October 20 and 21, 1994. Five new user beamlines had joined those available a year before, and it was clear that promises of "unique research opportunities" and "experiments not possible anywhere else" made at the inception of the ALS were indeed coming true.

The first day's program began with recent highlights from the ALS, chaired by the ALS User Executive Committee Chair, Michael White (Brookhaven National Laboratory). ALS Director Brian Kincaid welcomed attendees, and Bill Oosterhuis of the U.S. Department of Energy, Office of Basic Energy Sciences (BES), congratulated the ALS for having exceeded user expectations. Oosterhuis pointed out that like other BES-supported facilities, the ALS is budget-limited in its ability to provide beamtime to users, and he described current efforts to increase ALS funding in FY96. Kincaid picked up the budget theme by detailing how the ALS would use the proposed additional funds; for instance, a 10% increase above President Clinton's FY95 budget would allow the ALS to reach a 7-day operations schedule, increasing user shifts from 9 to 16 per week (a 78% increase) while improving user support services.

Ben Feinberg, Head of Operations, began a "nuts and bolts" segment of the program by describing recent work at the ALS designed to improve operations by tracking down and eliminating causes of down time. These efforts produced a phenomenal 93% efficiency rating (actual/scheduled user beamtime) for April–September 1994. Accelerator Group Leader Alan Jackson described how the ALS has responded to user needs by operating the storage ring over its full energy range of 1.0–1.9 GeV, improving single-bunch purity, and using feedback systems to correct multibunch instabilities. Accelerator group priorities for the coming year include improving beam position stability during undulator gap scans, commissioning new photon beam position monitors, and using information from the diagnostic beamline to improve short- and long-term beam stability. Howard Padmore, Experimental Systems Group Leader, extended the nuts-and-bolts talk to beamlines, highlighting the successful commissioning of undulator beamlines 7.0 and 9.0.1 and bend-magnet beamline 9.3.2. He also reported on upcoming developments, including two nearly-finished beamlines: undulator beamline 9.0.2 (chemical dynamics) and bend-magnet beamline 9.3.1 (atomic and materials sciences), both due to begin operation in 1995.

ALS Scientific Program Head Neville Smith concluded the first session by giving highlights from some of the first operating beamlines and describing industry involvement at the ALS. He issued a call for proposals from independent investigators, due December 1, 1994, for research periods beginning in April 1995 (the next round of proposals will be due June 1, 1995, for research beginning in October 1995). Later, User Liaison Group Manager Fred Schlachter offered details on the proposal process.

Lunch time brought an opportunity for participants to explore the ALS experiment floor and 28 vendor exhibits. Afterward, Nora Berrah (Western Michigan U.) chaired a session in which

current users showed the possibilities offered by the bright beams of the ALS by describing their own results. Brian Tonner (U. of Wisconsin, Milwaukee) began by summarizing developments at Beamline 7.0's SpectroMicroscopy Facility, where photoemission, photoelectron diffraction, soft x-ray fluorescence, and soft x-ray microscopy are being used to study detailed electronic structures of a variety of materials. In the ultraESCA (electron spectroscopy for chemical analysis) project, for example, Tonner's group is using photoemission to study actinide particles on the microgram scale; these particles have such low levels of radioactivity that they can be safely handled without the elaborate precautions necessary for larger samples.

Another group at Beamline 7.0, led by Joseph Nordgren (Uppsala U.), has been performing high-resolution soft x-ray fluorescence studies of organic and inorganic molecules. Nordgren discussed the group's work with buckyballs, benzenes, and substituted benzenes, with a particular focus on distinguishing the symmetries of molecular orbitals in these molecules.

Undulator Beamline 9.0.1 has been host to several research groups in its short history. Denise Caldwell (U. of Central Florida) reported on her group's initial results from high-resolution photoemission studies of simple atoms and molecules. Their photoelectron spectra from gas-phase experiments, with resolutions rivaling those of absorption spectra from other facilities, promise new opportunities in atomic physics.

Anders Nilsson (Uppsala U.) described a collaboration between Uppsala researchers and IBM to commission a new endstation at Beamline 8.0 and to study resonant photoemission processes in metal systems, with the goal of gaining a better understanding of multilayer interfaces. Adding to the good news from Beamline 8.0, Tom Callcott (U. of Tennessee) reported on studies which have taken advantage of the ALS's brightness to map band structures, characterize chemical bonding in buried monolayers, and generally expand the previous limits of fluorescence studies.

Keith Jackson (Center for X-Ray Optics, LBL) outlined progress at LBL in micromachining using deep-etch x-ray lithography. ALS Beamline 10.3.2 had just been commissioned as a dedicated beamline for this work, after demonstration experiments at neighboring 10.3.1. Jackson described the micromachining process from fabrication of x-ray absorbing masks to molding of final products for use in computer, biomedical, aerospace, and other industries.

The first day's activities culminated in a banquet, where speaker Jay C. Davis (Lawrence Livermore National Laboratory) provided a first-hand account of his experiences as a member of the United Nations inspection team in Iraq in his talk, "Nuclear Non-Proliferation and the UN Nuclear Inspections of Iraq."

Steve Cramer (U. of California, Davis) chaired the morning session on the second day of the meeting, which featured speakers describing opportunities at the ALS in several fields of research. François Wuilleumier (U. of Paris) began by speaking about experiments in atomic physics that would make use of the high brightness, flux, and polarization of ALS undulator beams. He urged researchers to focus on developing detectors with resolutions to match those of ALS photon beams; progress in this direction has already begun with innovative detector designs for a protein crystallography beamline, scheduled for completion in 1996. A talk by Robert Stroud (U. of California, San Francisco) described applications of synchrotron-based

protein crystallography to rational drug design. He pointed out several advantages of the ALS as a source for x-ray diffraction studies including the increase in speed of data collection compared to conventional laboratory x-ray sources, and the advantage of a tunable source in the technique of multiple-wavelength anomalous diffraction (MAD) to determine phases directly from diffraction amplitudes.

Specific industrial applications for ALS light were the focus of talks by Richard Brundle (an independent consultant, formerly of IBM/Almaden) and Harald Ade (North Carolina State U.). Brundle enumerated several advanced materials applications for synchrotron radiation, including those in the semiconductor and magnetic recording industries, and described what changes will be necessary for the national synchrotron facilities to become effective partners with industry in these fields. Ade then spoke on the potential of x-ray spectromicroscopy to solve industrial problems involving polymers; compositional and orientational sensitivity give x-ray spectromicroscopy unique capabilities in analyzing materials such as Kevlar® fibers and multilayer polymer films.

Gwyn Williams (NSLS) focused on infrared microscopy, describing an ALS bend-magnet infrared beamline scheduled for availability in 1995. Infrared techniques are known to be valuable research tools in materials science, and can be even more effective using a synchrotron source. Williams also chaired an afternoon workshop on Infrared Microspectroscopy with Synchrotron Radiation.

UEC Chair Michael White closed the session with election information for the ALS Users' Executive Committee. Participants went next to the ALS for lunch, tours, and a poster session. Attendees, crowding around poster displays and asking questions, showed their intense interest in the work already being done at the ALS, and many looked forward to proposing their own research to take advantage of its bright beams.

Report from the DOE

William T. Oosterhuis

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I would like to welcome you to the ALS Annual Users' Meeting on behalf of the Department of Energy (DOE). It is quite clear that a substantial amount of progress has been made at the ALS in the past year: the accelerator and storage ring have worked extremely well; the quality of the beam has met or exceeded the expectations of many of the experimenters, and the ALS has proved itself a reliable performer. I know all of you would like to see the operations extended from the current 9 shifts per week to possibly as much as double that; unfortunately, I don't think we have a way of making the week longer than 7 days!

Many more beamlines have appeared on the experiment floor in the last year, and they are producing qualitatively new results at a remarkable pace. I admit we still have a long way to go before the ALS is fully instrumented, but we are definitely making steady progress toward this goal. I especially look forward to the science that is going to be done with the elliptical wiggler beamline as it will provide some very unusual and unique opportunities. All in all, I think that Director Kincaid and his staff at the ALS are to be congratulated on a job extremely well done.

I have a few comments on the fiscal 1995 budget. The operating budget for the ALS will be some 2% to 3% less than its 1994 budget. All facilities share the same fate, as does Basic Energy Sciences (BES) support for research. This is only going to make it more difficult to exploit these marvelous new tools. Each of the BES-supported facilities is budget-limited in its ability to provide users with more beamtime, and also with more efficient use of that beamtime because of beamline upgrades that need to be made.

This problem is being addressed, though, and all user facilities have documented their needs. The budget shortfalls at each facility were identified for FY95. The facility directors, led by Arthur Bienenstock, have made a case for a significant budget enhancement in order to operate the facilities more fully in FY96. I can say that this effort, which I'll call the Bienenstock Bump, has been very well-received at the Department of Energy, Office of Management and Budget (OMB), and Office of Science and Technology Policy. As many of you know, user organizations at each of these facilities have been asked to respond to OMB questions about the needs at their facilities. I take this as evidence that the issue is being taken very seriously. If this effort is successful, we could see a substantial increase in operating time here at ALS and at other facilities, perhaps by a factor of almost two at ALS. At other facilities it might be a 50% enhancement which might take the form of beamline upgrades.

Finally, I would say that DOE will continue to support the development of new beamlines. The development schedule will probably be much slower than we'd all like to see, but realistically, I think that development will continue. Let me close by wishing you all a good meeting and success in the coming year.

ALS Director's Report

Brian Kincaid

Director, Advanced Light Source
Lawrence Berkeley Laboratory
Berkeley, CA 94720

My talk will concentrate on a few of the most important areas that I have responsibility for as director of the ALS: our organizational structure, budget strategy, staff, and commitment to quality. I will also give an overview of current and future beamlines as an introduction to the ALS presentations which will feature more specifics on accelerator operations and experimental systems projects.

We made a few changes to the ALS organization during the past year and the current management structure seems to work extremely well. We now have two program heads: Neville Smith, who joined the ALS in March as head of the scientific program, and Ben Feinberg, who has been our head of operations for over a year. Basically, Neville handles the scientific program and the users, while Ben's responsibilities center around operations. The group leaders communicate with each other over a sort of "data bus," horizontally, and these connections have proved most effective (see Figure 1). Our management structure is more or less copied from Denis McWhan at NSLS and it works. Rather than inventing a new organization, we used what had already proved itself.

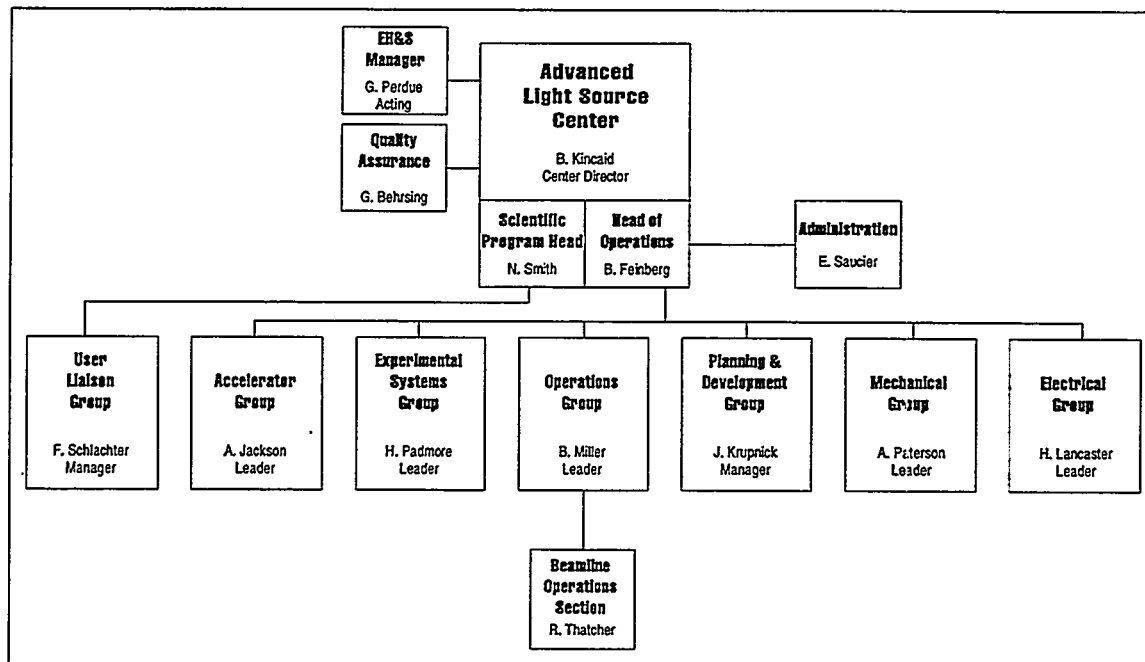


Figure 1. The organizational structure of the ALS.

The story on our budget figures is a simple one. The projected operating budget for this year, in the original construction project, was supposed to be \$24M. What we ended up with was \$22.5M, and it has gone down since then. Inflation further reduces the value of the funds we receive, and some involuntary layoffs have been necessary for us to stay within budget. If you started with the original number and did a trivial escalation, you would get \$27.7M as a natural equilibrium value for the budget of this facility, so that figure is the one we will use in our FY96 budget request.

Let me show you the positive side of what we could do with this increase in funding. Figure 2 shows the result of an exercise we did to prioritize the needs of the facility and the user community according to what would happen if we got an incremented budget of \$2M over our present budget, and what would happen if we got two other \$2M increments, bringing us up to nearly \$28M. One of the most dramatic changes the additional funding would make would be to increase our available user time. We have reached a plateau of 3,000 hours of user time per year, but that is only about 60% of the time available. We could run full time, 5,370 user hours per year, with only \$2M (a 10% increase) added to our budget. That \$2M would also buy a more appropriate level of user support for the increase in users we expect with each year of operation. If we received an increase of \$4M, we could also do significant industrial outreach and expand our research and development activities to update equipment designed ten years ago. Some of our users are developing the next generation of instrumentation in some areas, but we should be leading that effort and we don't have the money. For \$6M, along with all of the above, we could add technical staff and facility infrastructure to improve our throughput by keeping all operations timely. My prediction is that we will only see the first \$2M increase, and perhaps less, but that the increase will come because of direct pressure from our users. The message that U.S. light sources need increased funding must go to Washington from all the facilities, not just one. Arthur Bienenstock, Denis McWhan, Dave Moncton, and I are working on such a unified approach, and we should find out about the results of that in the next year or so.

Meanwhile, we are focusing on quality work to save money and to attract industrial customers. One of the tenets of quality management is that the quality is free. That may sound strange, but we have seen first-hand the benefits of quality in the engineering program that went into building the ALS, where beamlines have been commissioned in one hour. The storage ring was commissioned in a matter of weeks. This is a direct reflection of the quality process that went into building this ring; some beamlines around the world have taken years to commission.

There is now an international standard for quality called ISO 9000, which essentially gives a prescription for checking to see whether an organization has the checks and balances, the feedback loops, and the responsible people necessary to deliver what it says it can deliver. This standard is mandatory for all companies doing business with the European Community, so our industrial customers in the Silicon Valley have requested that we use ISO 9000. If they want to do any work here, they need to be able to say that work done at the ALS is consistent with ISO 9000 standards for their own customers. The benefit to us is that by improving the quality of the operation, we can have our people work more efficiently and effectively without having to work harder. We avoid making the mistakes that cost so much money to fix.

ALS FY95 Operating Budget Scenarios

Priority	Hires	Cost	(FY95k\$)	Totals
1 Nominal Operations				
◆ Reach Steady-State 21 Shift Operation				
Accelerator Operators	2	250		
Electronic Maintenance Techs	2	220		
User Coordinators	2	250		
Supplies & Expenses		100		
Power		<u>250</u>		
	6	1,070		
◆ Provide Appropriate User Support				
Scientific Liaison	1	125		
User Administrator	1	125		
Clerical Support	1	125		
Beamline Techs	2	220		
Supplies & Expenses		100		
Misc. User Support		<u>350</u>		
	5	1,045		
Total			2,115	
2 Industrial Outreach & R&D Programs				
◆ Initiate Industrial Outreach Program				
Industrial Liaison Scientists	2	250		
Special Outreach Materials		75		
Supplies & Expenses		<u>35</u>		
	2	360		
◆ Support Basic R&D Activities				
Vac. Eng., Controls,	3	525		
R&D Support		550		
Visiting Scientists		360		
Students, Post-docs		360		
Supplies & Expenses		<u>110</u>		
	3	1,905		
Total			2,265	
3 Infrastructure Support				
◆ Ensure Timely Beamline Installation				
Mechanical Technicians	2	220		
Mechanical Designers	2	250		
Electronic Installation Techs	1	135		
Electronic Coordinators	1	125		
Supplies & Expenses		100		
Beamline Maintenance & Spares		<u>290</u>		
	6	1,120		
◆ Proper Facility Infrastructure				
R&D Support		450		
Quality Assurance	0.5	95		
Budget, Misc.	1	150		
EH&S Staff	2	230		
Supplies & Expenses		50		
Mgmt. Reserve		<u>125</u>		
	3.5	1,100		
Total			2,220	
Increase Totals			6,600	
President's FY95 Budget			21,900	
ALS FY95 FTP Budget			28,500	

Allows full utilization of the ALS by moving to 7 day/week operation, providing 16 user shifts/week instead of only 9. Hire operations staff and pay for increased power.

Provides for proper level of support for scientific users. Expect rapid growth in scientific utilization of facility.

Begin necessary outreach activities with full-time liaison scientists in order to educate industrial and other users about ALS capabilities.

Support advances in key instrumentation needed to develop industrial and other scientific use of the facility.

Provide adequate staff to ensure timely assembly and installation of beamlines and experimental equipment to support user research.

Maintain leadership position in key areas of research. Ensure proper facility infrastructure to support both accelerator and experimental facility operations.

Figure 2. Priorities for use of proposed additional ALS funding.

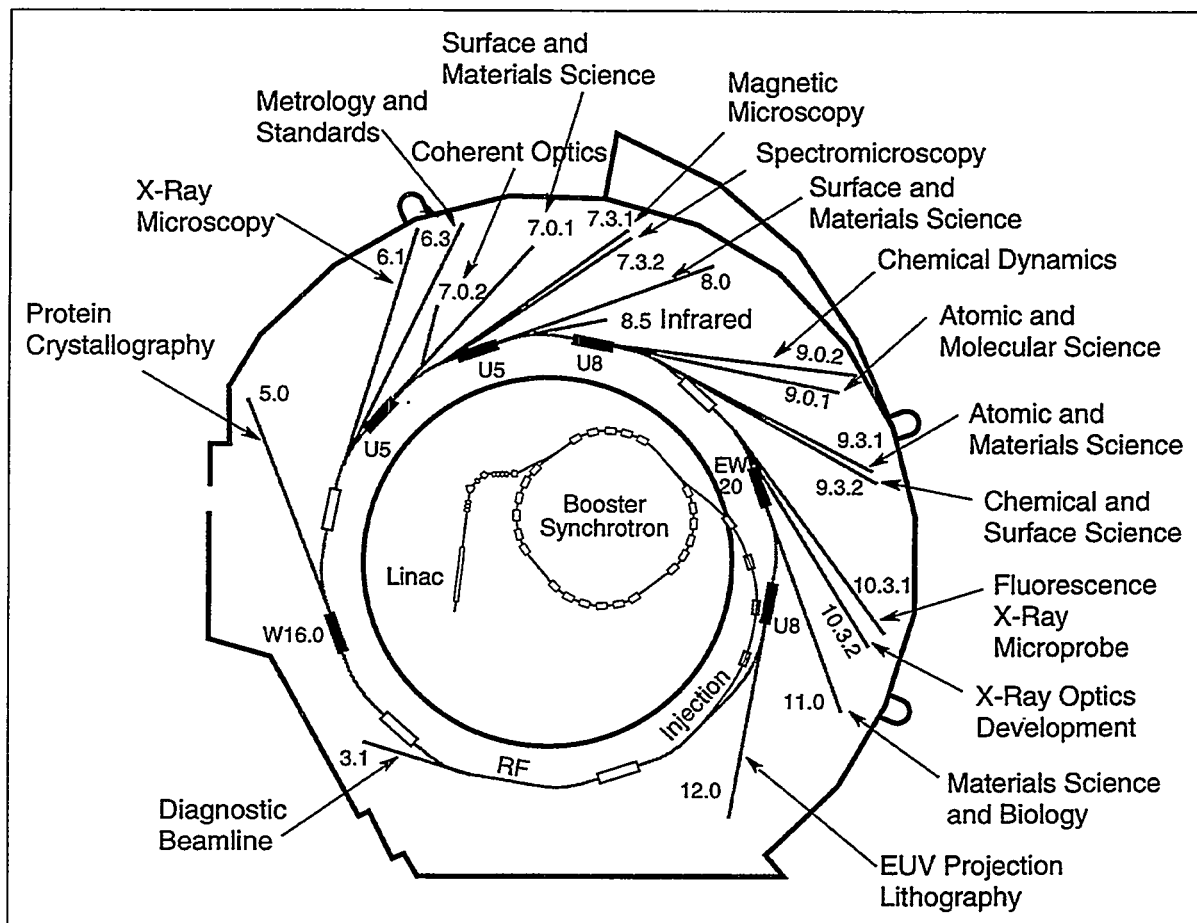


Figure 3. Plan view of ALS beamlines for 1994-1996.

Now let me give you an update on how far we have come in the beamline world. The policy of ALS management in the past year has been to build as many beamlines as possible, and that is happening. At this time last year, we had two undulator beamlines (7.0 and 8.0) and one white-light bend-magnet beamline (10.3.1) operating, and we had first light from the diagnostic beamline (3.1). The initial group of user beamlines has grown from three to seven, with eleven more beamlines scheduled to begin operation by the end of 1996, for a total beamline population of 18 beamlines (Figure 3 and 4). The recently commissioned undulator beamline (9.0.1) and the three newly operational bend-magnet beamlines (6.1, 6.3, and 9.3.2) are already producing some amazing results.

I will finish by summarizing the progress of the ALS as of today: first, the original vision of the ALS conceived of in 1982 has been realized. Second, the commissioning and early operation of the ALS have been successful. We have the high brightness, the high flux, and the tunability of undulators. The challenges solved and instant fixes produced by the accelerator group, even as their staff has been reduced, have been amazing. Third and finally, new scientific frontiers have been opened at the ALS. Some of the techniques people proposed for the ALS in soft x-ray fluorescence and scanning soft x-ray fluorescence microscopy were deemed completely inconceivable before the ALS turned on, but today you can go and see the experimental results from those techniques.

ALS Beamlines for 1994–1996

Beamline	Source	Research	Energy Range	Avail.
3.1	Bend magnet	Diagnostic beamline	200–280 eV	1994
5.0	W16 wiggler	Protein crystallography	4–13 keV	1996
6.1	Bend magnet	High-resolution zone-plate microscopy	250–600 eV	Now
6.3	Bend magnet	Metrology and standards	50–4000 eV	Now
7.0.1	U5 undulator	Surfaces and materials, spectromicroscopy	70–1200 eV	Now
7.0.2	U5 undulator	Coherent optics experiments	70–650 eV	1995
7.3.1	Bend magnet	Magnetic microscopy	600–900 eV	1995
7.3.2	Bend magnet	Spectromicroscopy	100–1500 eV	1996
8.0	U5 undulator	Surfaces and materials	70–1200 eV	Now
8.5	Bend magnet	Infrared spectromicroscopy	0.1–2 eV	1995
9.0.1	U8 undulator → U10	Atomic physics and chemistry	20–300 eV	Now
9.0.2	U8 undulator → U10	Chemical dynamics	5–30 eV	1995
9.3.1	Bend magnet	Atomic and materials science	700 eV–6 keV	1995
9.3.2	Bend magnet	Chemical and materials science	50–1500 eV	Now
10.3.1	Bend magnet	Materials science and advanced microprobe instrumentation	3–12 keV	Now
10.3.2	Bend magnet	LIGA, total reflection x-ray fluorescence	3–12 keV	Now
11.0	EW20 elliptical wiggler	Materials science and biology, magnetic materials	50 eV–10 keV	1996
12.0	U8 undulator	EUV projection lithography, optics development	60–320 eV	1996

Figure 4. ALS beamline “scoreboard” with specific information on beamlines for 1994–1996.

ALS Operations Update

Ben Feinberg

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I will begin by showing a graph of ALS operations for April to September, 1994 (Figure 1). The bottom line is that we averaged 93% for actual vs. scheduled beamtime: an excellent record of performance. The gaps between operations are weekends when we do not operate because of funding limitations, or when we had a shutdown for equipment installation.

Of course it is our intention to continue to improve our performance and quality of operations. To do this, we have instigated a rigorous analysis of what causes our beamtime "out-ages" (i.e., the missing 7%) so that we can devise procedures or improvements which will eliminate or minimize them. Part of this analysis is making sure all our feedback loops are in

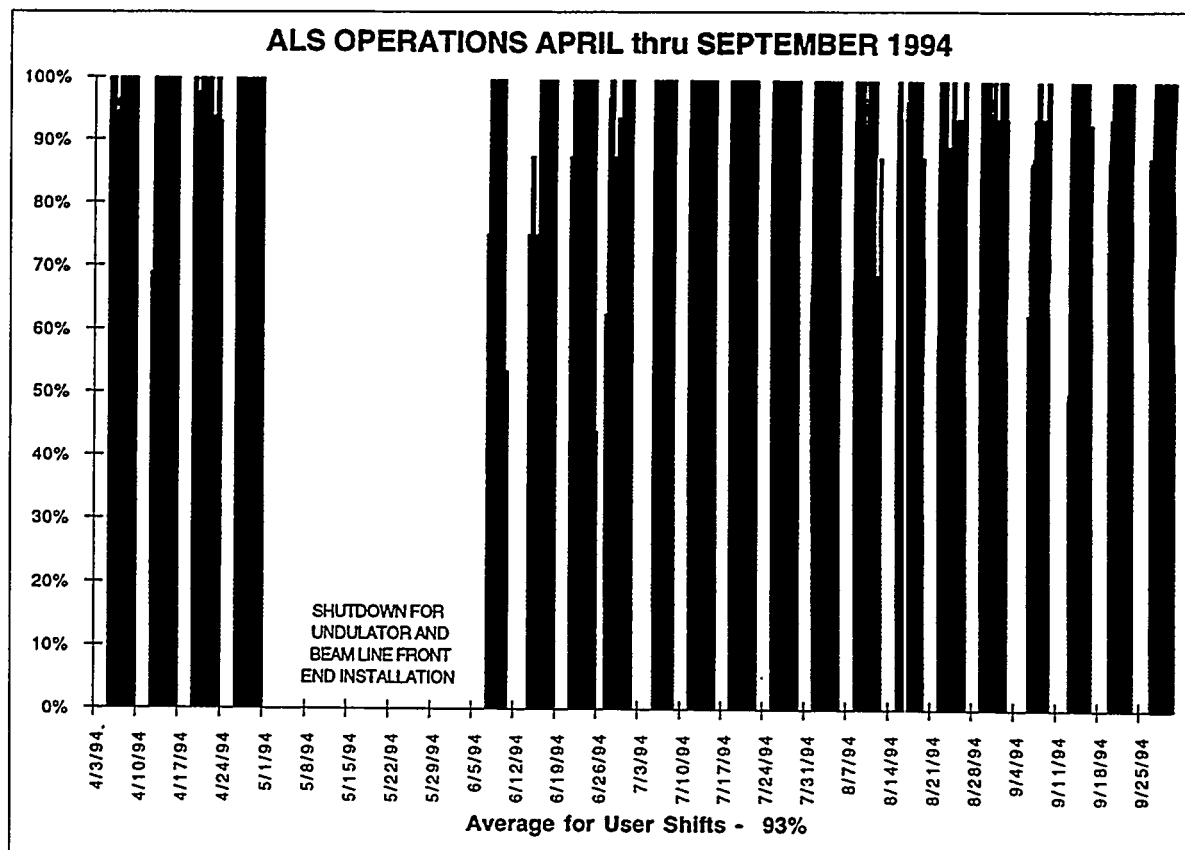


Figure 1. The ALS achieved a 93% performance level for actual vs. scheduled beamtime for April-September 1994.

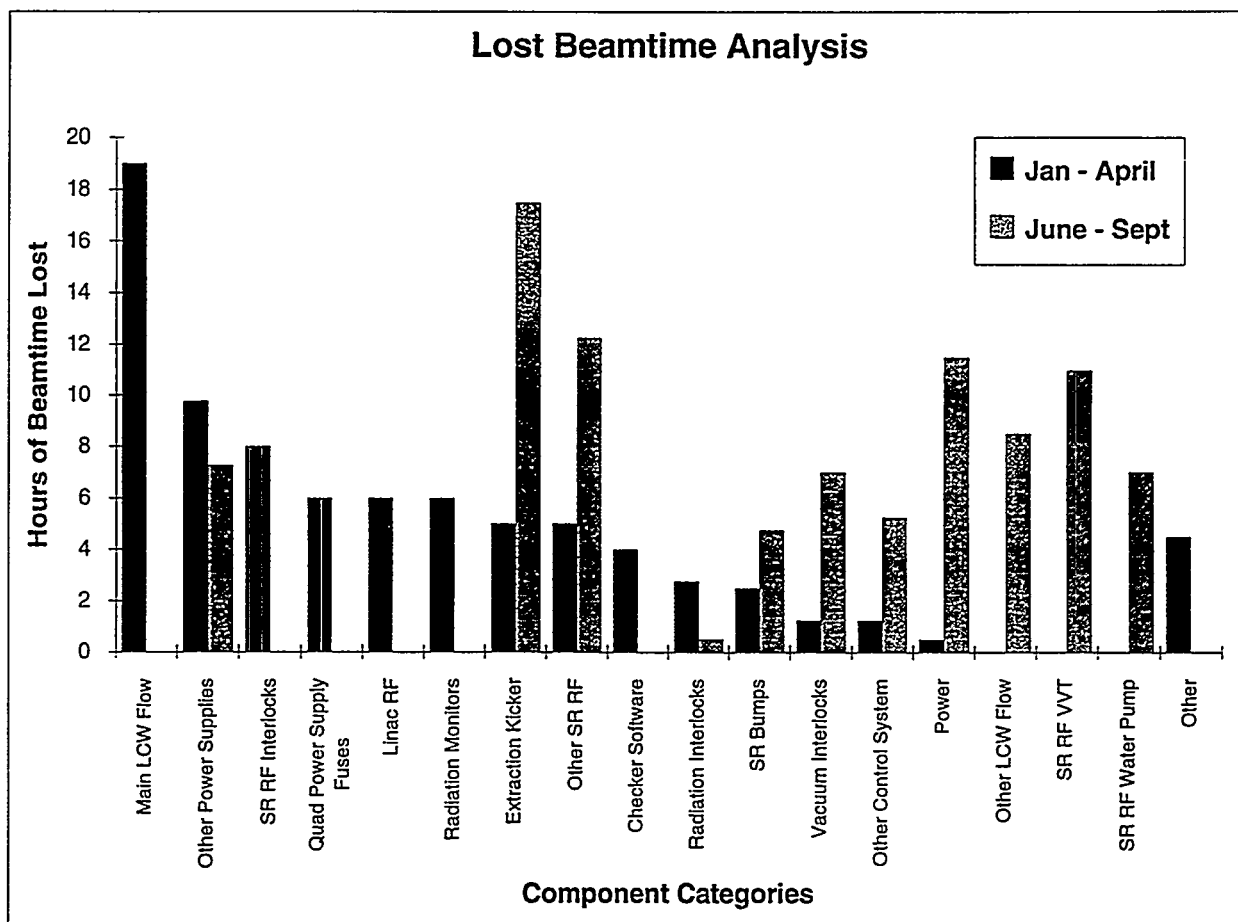


Figure 2. An analysis of beamtime outages for January-April and June-September 1994.

place as I believe that effective feedback is one of the keys to our success. Figure 2, a graph of the different component categories contributing to lost beamtime vs. hours lost, shows the positive results that came from an analysis of operations we conducted this year. For example, problems with our main low-conductivity water (LCW) flow, which caused 19 lost hours from January to April, were analyzed and corrected so that we reduced this number to 0 hours for June to September, and the situation was the same for most of the other major problems during this time frame. Now other problems are cropping up, and those will be what we focus on next in order to reach our goal of 100% delivery of scheduled beamtime to users.

Now I will present some long-range scheduling information. During the spring 1994 shut-down, we installed two beamline front ends and an 8-cm-period undulator for Beamline 9.0. Our next shutdown will be January-February 1995, when we will install a small-gap chamber in the undulator for Beamline 7.0. One might think that this would be relatively easy, just swapping out a vacuum chamber, but actually we have to remove the undulator from the machine, take out its present vacuum chamber, install the new vacuum chamber, and re-install the undulator. Some of the other activities planned for the seven-week shutdown include the installation of the front end for bend-magnet Beamline 7.3, and a monochromator for Beamline 9.0.2.

We hope we will see some increases in operations funding during the next few years so that we can expand the number of shifts for user operations. At this point, we are operating only 9 user shifts per week, with 5 shifts for maintenance, startup, and accelerator physics; leaving 7 shifts when we cannot operate for lack of funds. We are doing some shuffling of our user shifts so that users can get more beamtime in less calendar time; but the only way we can really get more beamtime is to get more funding. With the \$2M funding increase that Brian Kincaid mentioned earlier, we could run full-time at 16 user shifts per week. That represents a 78% increase in operating time for a 10% increase in funding, and is the pitch that we have made to our funding agencies. We aim to get that funding increase in 1996, but that is still uncertain. We seem to have the support of the current administration, but we will also need Congress on our side. They have made huge capital investments in user facilities of all kinds, and many of those facilities cannot operate full-time because of lack of funds. A modest increase in operating funds will buy immense returns at all these facilities, and particularly at the ALS.

Recent Results in Machine Physics

Alan Jackson

Accelerator Group Leader, Advanced Light Source
Lawrence Berkeley Laboratory
Berkeley, CA 94720

INTRODUCTION

The ALS Accelerator Group and our collaborators made great advances in understanding the operational characteristics of the storage ring, and in establishing optimized parameters for routine user operations during 1994. A major accomplishment of direct benefit to user operations was the development of procedures to rapidly set the correct rf frequency (which determines the circumference of the electron orbit), establish minimum closed-orbit distortions, adjust quadrupole settings for correct dispersion and optimum betatron tune, and set the sextupole magnets to give zero chromaticity. The importance of this capability was aptly demonstrated "in the heat of battle" during the startup period after the May shutdown, and again after the Northridge earthquake in June, when user beam was re-established within two days.

Measurements of the electron beam made at low beam current, where collective effects (due to large charge density) and beam instabilities are negligible, show that the parameters of the beam match the design values. However at larger currents, such as the 400 mA routinely used as the starting current for user operations, the quality of the electron beam is degraded, mainly due to coupled-bunch motion that increases the energy spread of the beam. We are developing a feedback system to correct this problem, and tests with a prototype indicate that it will be able to restore high quality beam up to the full operating current. We expect this system to be put into operation in 1995.

Beam stability has been another major area of study this past year. We analyzed movements in the beam caused by the small residual dipole fields in the undulators and developed feed-forward algorithms to minimize the effects of these fields. Other investigations led to the correlation of periodic beam motion, with periods of around 10 minutes and 1 hour, to changes in the low conductivity water (LCW) temperature and the local air temperature respectively. This work is still underway and will continue in the coming year.

Much of the work described in this report was accomplished in collaboration with the ALS operators, instrumentation engineers and controls engineers, the ALS Experimental Systems Group, our users, the Beam Electrodynamics Group (led by John Corlett) from LBL's Center for Beam Physics (CBP), and the Stanford Linear Accelerator Center (SLAC) Feedback Group (led by John Fox).

BEAM CHARACTERISTICS AT LOW CURRENT

Prior to the full installation of Beamline 7.0, a transmission grating spectrometer (TGS) was used to characterize the radiation from the U5.0 undulator. The TGS can only operate at low

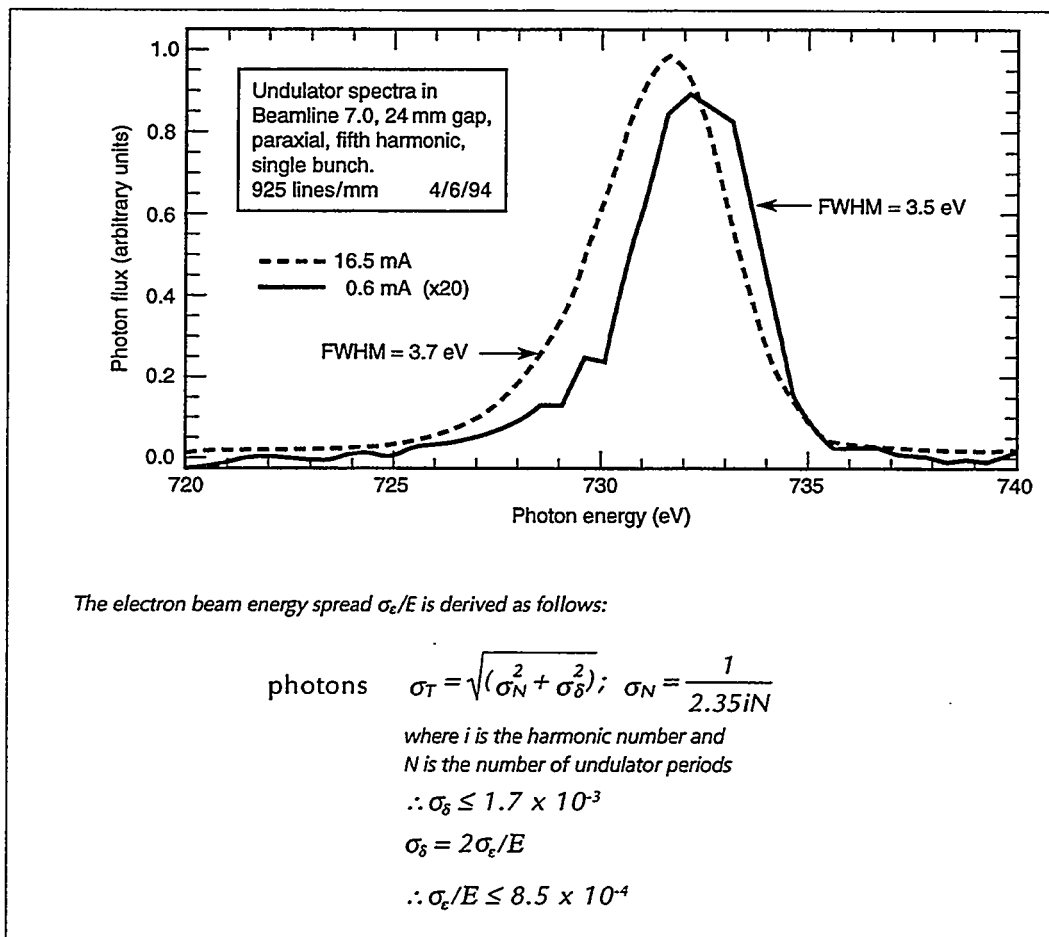


Figure 1. (Above) Spectra of the undulator fifth harmonic in single bunch mode on Beamline 7.0. (Below) Derivation of the electron beam energy spread.

beam intensity because the grating is set normal to the photon beam, so the measurements were taken using < 0.5 mA of current. By observing the angular widths of the red-shifted radiation produced away from the undulator axis, it is possible to calculate the beam emittance. This work, undertaken primarily by the ALS Experimental Systems Group, gave an upper limit on the horizontal emittance of 5×10^{-9} mrad, in good agreement with the design value of 3.6×10^{-9} mrad.

When completed, Beamline 7.0 was used to measure the line-widths of the first, third, and fifth harmonics of the undulator spectrum with a single bunch in the storage ring. Analysis of this data (shown in Figure 1) indicates an electron beam energy (or momentum) spread of $\sigma_e/E \leq 8.5 \times 10^{-4}$, close to the design value of 8×10^{-4} . Also it shows a negligible increase in energy spread as the current is increased from 0.5 to 16.5 mA per bunch.

When the single bunch current was increased to around 30 mA, another phenomenon set in. The beam lifetime was observed to increase by a factor of six or more. Since the single bunch

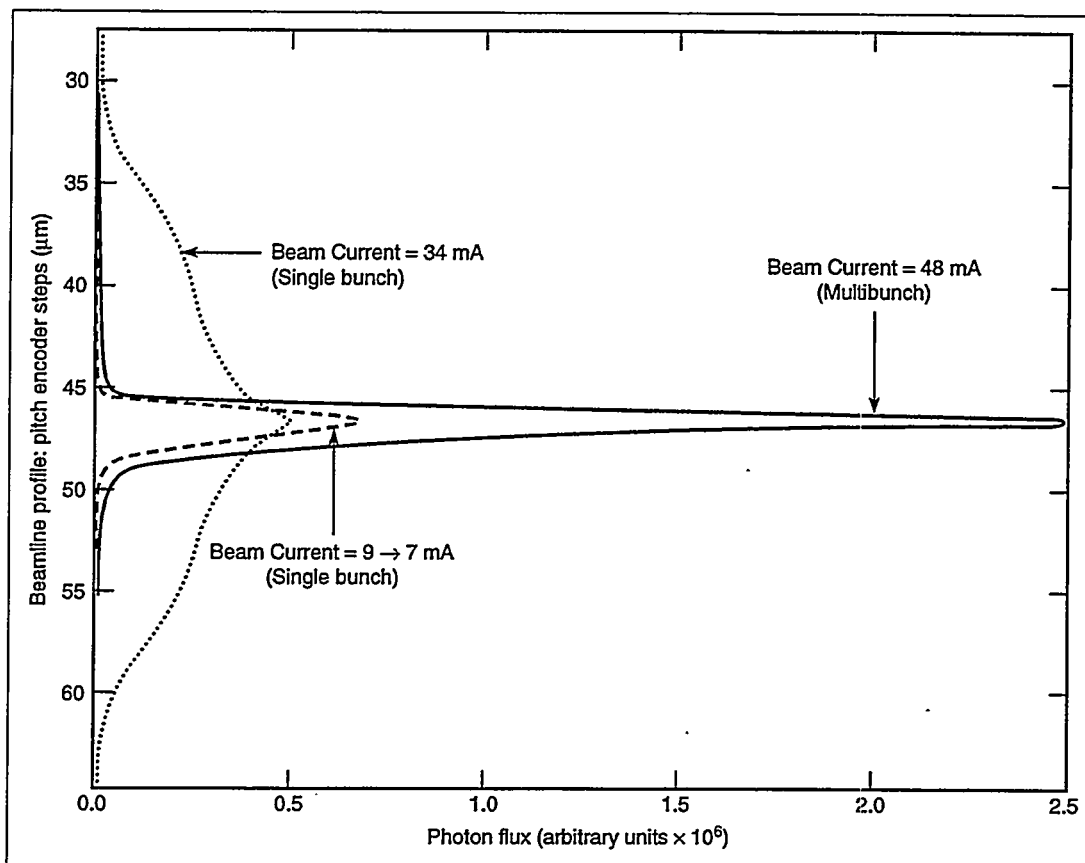


Figure 2. Vertical beam blow-up due to transverse mode-coupling instability; measurements taken on Beamline 9.3.2.

lifetime is dominated by the Touschek effect, this indicates that the beam is blowing up transversely, longitudinally, or both. Evidence from changes in the beam synchrotron-oscillation frequency suggested that we were engaging the vertical transverse mode-coupling instability (TMCI), as predicted by theory. Experiments conducted in collaboration with Beamlines 7.0, 9.0 and 9.3.2 confirmed that the beam blowup was indeed in the vertical plane only, consistent with the TMCI. Figure 2 shows the vertical beam profile measured on Beamline 9.3.2.

STORAGE RING PARAMETERIZATION—MEASUREMENT-BASED MODEL

Naturally, one of the key functions of the Accelerator Group is to improve the control of the electron beam to match the ever more demanding requirements of our users. As such, we were forced to abandon our simulation-based model of the storage ring and develop a better measurement-based model incorporating algorithms based on how the accelerator actually behaves. To derive the model, we make changes in a single device, a corrector magnet for example, and measure the effects the changes have on different parameters (such as the closed-orbit, chromaticity, betatron tune, etc.). The resulting response- or sensitivity-matrices are then used in control applications: for example, to minimize the closed-orbit changes caused by undulator gap changes.

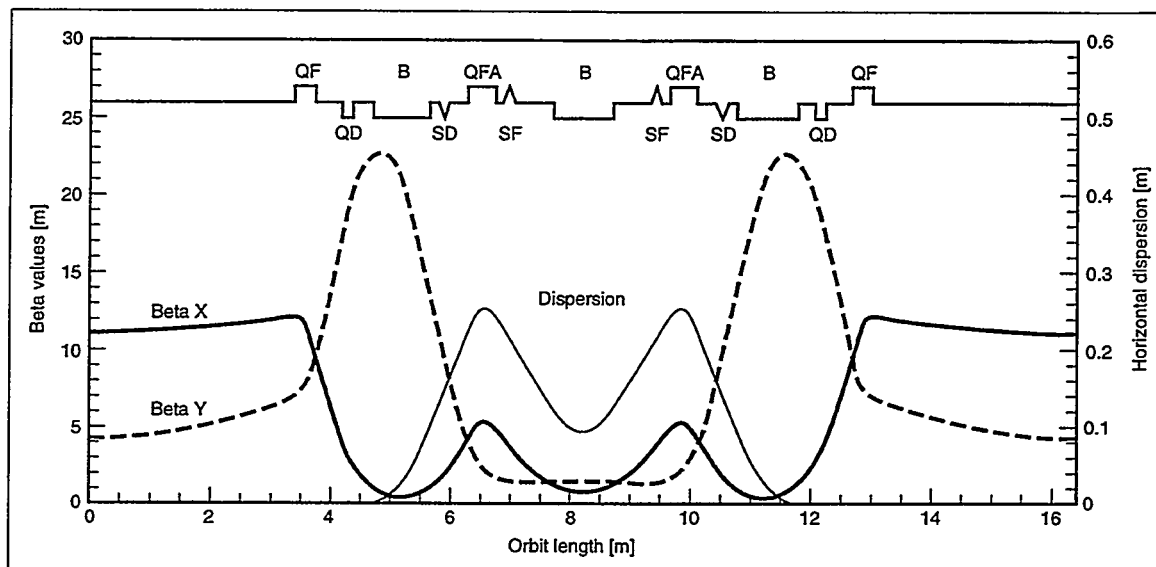


Figure 3. Lattice parameters in one superperiod of the ALS triple-bend achromat lattice.

These measurements also show how well the storage ring conforms to the perfect twelve-fold symmetry with which it was designed. Figure 3 shows the optical functions of a perfect lattice through one-twelfth of the storage ring. A compilation of three measurement methods giving the vertical β -function at twelve homologous positions around the circumference appears in Figure 4. The results confirm that the ALS magnet fields are within the 10^{-3} tolerance band, and that the magnets have been installed with an accuracy of 0.1 mm.

In establishing the closed-orbit, we rely heavily on the accuracy of the beam position monitors (BPMs). Their positions around the ring are determined by the rigid one-piece vacuum vessel to which they are attached, and the position of the vacuum vessel which is determined by survey. The electrical centers of the BPMs are electronically calibrated to an accuracy of about 30 μm . However, our beam-based measurements revealed that some monitors had internal resonances at frequencies not covered in the calibration process, and gave readings with errors as high as 1.5 mm. Therefore we developed an automated calibration procedure that uses the beam to measure the difference in position of a BPM compared with an adjacent quadrupole magnet, whose centers are well established by survey. This allows the relative offsets of the BPMs to be automatically incorporated into the orbit correction algorithm.

BEAM STABILITY

Photon beam drift and jitter was a major issue in 1994, and will continue to be into 1995. Part of the problem is the difficulty in separating the contribution of the source (the electron beam) to photon beam motion from the motion of the photon beam caused by movement of the optical elements in the beamlines. Unfortunately, the BPM system is not sufficiently sensitive to detect electron beam motion at the necessary level to establish how the electron beam is affecting photon beam motion. However, a prototype electron beam monitor that is being developed for the undulator radiation "errant photon beam" protection system proves to have a sensitivity of better than 1 μm , and is perfect for observing electron beam jitter. Also, be-

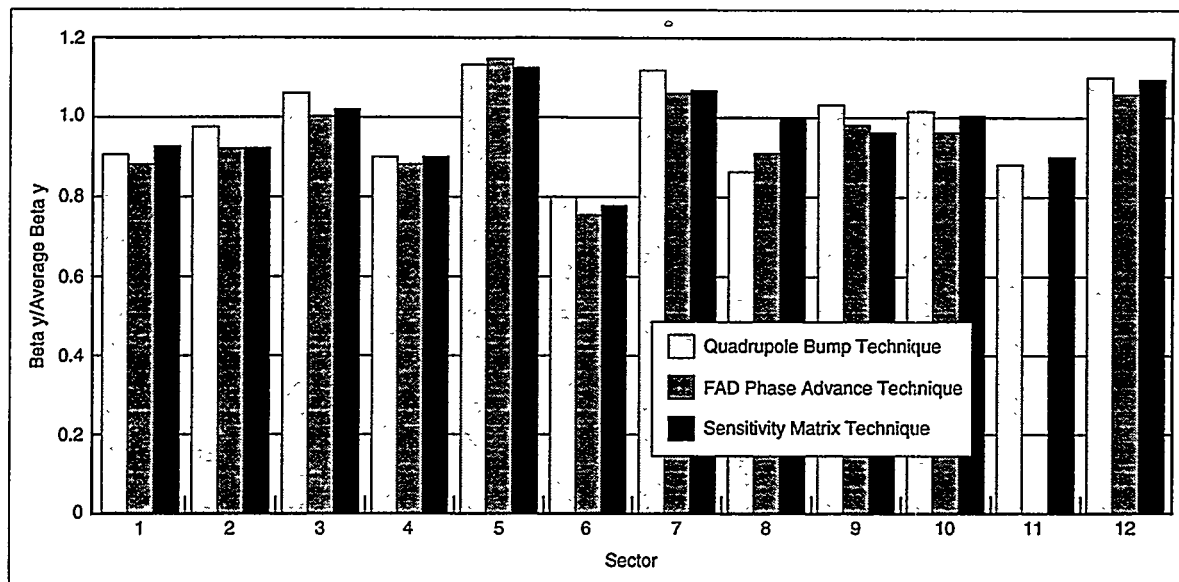


Figure 4. The data show good agreement between the three techniques used to measure β -function, and confirm that the ALS magnet fields are within the 10^{-3} tolerance band.

cause the head of the electron beam monitor is solidly anchored to the floor, the device is ideal for investigating longer term drifts.

Using this new diagnostic, we measure a beam jitter of $20\text{ }\mu\text{m}$ peak-to-peak in the horizontal plane, and $4\text{ }\mu\text{m}$ in the vertical plane, well within the specified tolerance of 10% of the RMS beam sizes (see Figure 5). The jitter has a strong frequency component at 12 Hz which corresponds to a torsional oscillation mode of the girder on which the magnets are mounted. Such a source of motion would also account for the difference in amplitude observed between the horizontal and vertical planes. Further, we were able to correlate cyclic vertical motion of $5\text{ }\mu\text{m}$ peak-to-peak with a period of about 10 minutes with cyclic changes in the temperature of the low conductivity water (LCW) cooling system (also shown in Figure 5). In a later experiment, some evidence was found to correlate the large horizontal motion of $50\text{ }\mu\text{m}$ with air temperature. In 1995 the prototype monitor will be supplemented with six more such devices situated at each end of the three undulators. Also, the diagnostic beamline will be brought into service in 1995, adding to the armory of diagnostics that can be focused on the problem of beam stability.

LONGITUDINAL AND TRANSVERSE COUPLED-BUNCH INSTABILITIES

A much faster beam "jitter," occurring at frequencies above 10 kHz, is produced when the electron bunches interact with each other through self-induced electromagnetic fields in the rf cavities and/or in the vacuum chamber. These fields drive motion that can become unstable in the so-called longitudinal and transverse "coupled-bunch instabilities (CBI)." In the ALS, the longitudinal CBI, caused by excitation of high frequency resonant modes in the rf cavities, are responsible for an increase in the energy spread in the electron beam by up to a factor of 4. This, in turn, increases the contribution of energy spread to the line-width of the undulator spectrum, causing it to become the dominant factor in determining the line-width of the third,

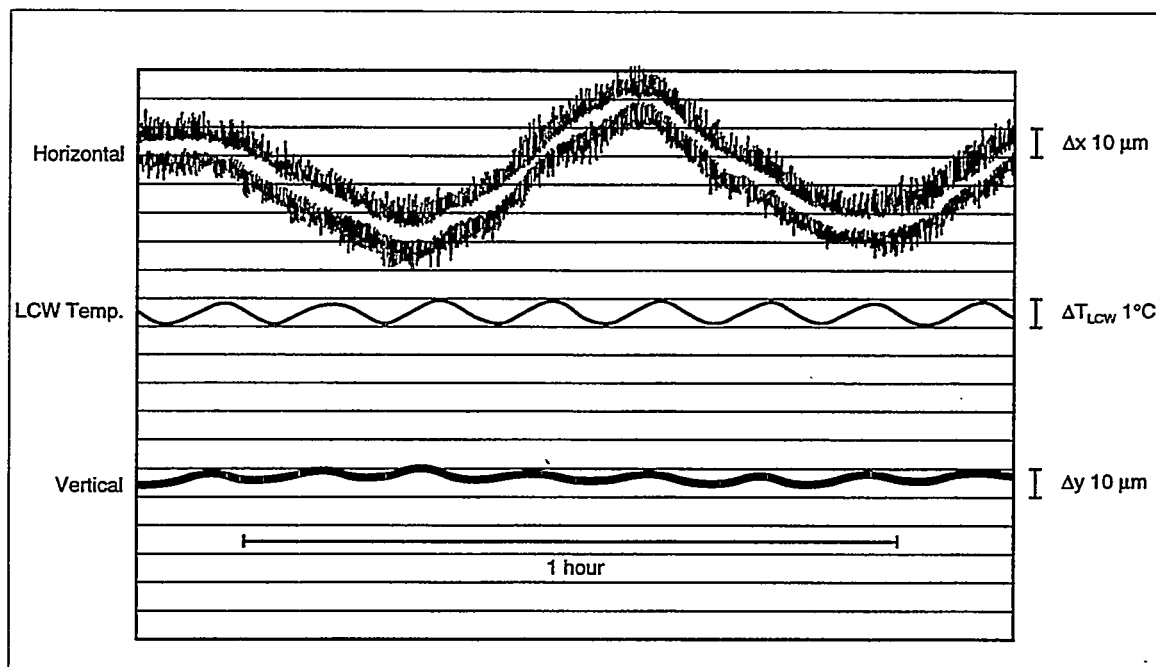


Figure 5. Beam jitter in the horizontal and vertical planes measured by the prototype electron beam monitor. We were able to correlate the cyclic vertical motion of the beam to the changes in the LCW system temperature (center line).

fifth, and higher harmonics for the undulators. That this is indeed the case has been confirmed in collaborative experiments with Tony Warwick using Beamline 7.0.

To dampen the longitudinal oscillations in multibunch operation, and thus narrow the energy spread in the storage ring, a novel feedback system utilizing state-of-the-art digital processing techniques is being developed jointly with the SLAC Beam Feedback Group and the CBP Beam Electrodynamics Group (Figure 6). A prototype system has been tested with 84 bunches in the storage ring and shows the expected improvement in spectral performance (see Figure 7). The production version is expected to be installed early in 1995.

When the longitudinal instability is brought under control, we observe a transverse instability driven by the interaction of the electron bunches with the walls of the vacuum vessel that increases the vertical emittance of the beam. The feedback system for this instability has been developed and successfully commissioned by the CBP Beam Electrodynamics Group.

EXPANDING THE RANGE OF OPERATING CONDITIONS

Routine operation at the ALS has come to mean: an energy of 1.5 GeV; a starting current of 400 mA; 320 consecutively filled bunches out of a maximum 328, with a gap for "ion clearing;" and an anomalously long 12 hour lifetime, as a direct consequence of the large energy

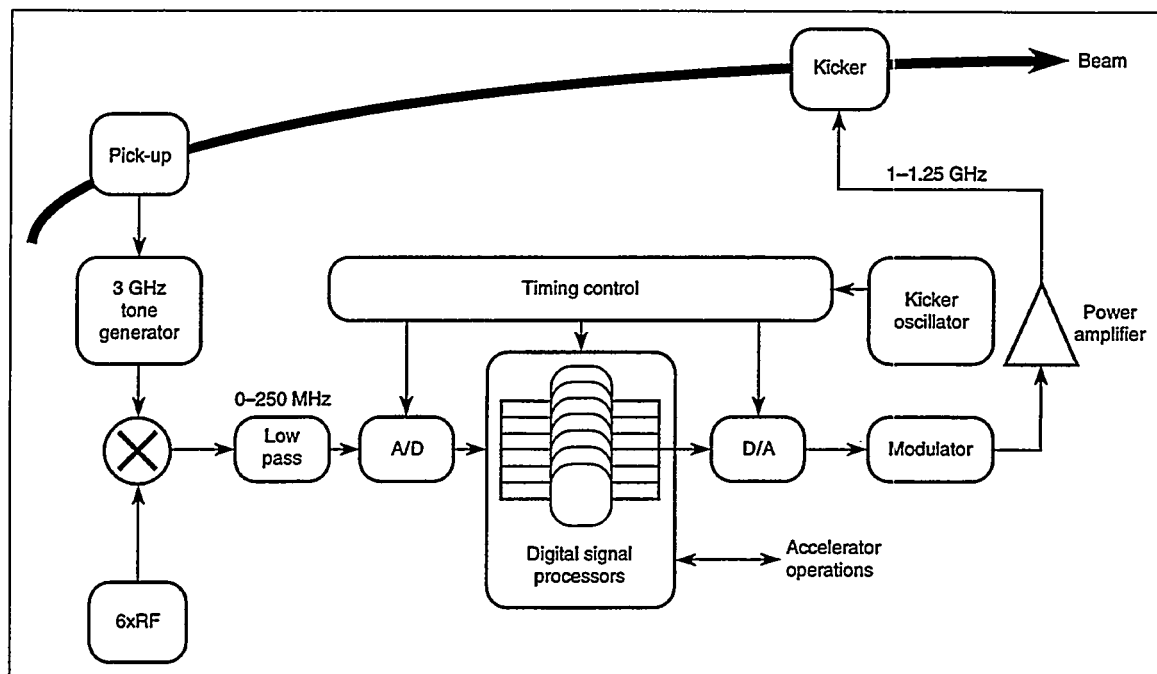


Figure 6. Schematic diagram of the longitudinal bunch-by-bunch feedback system.

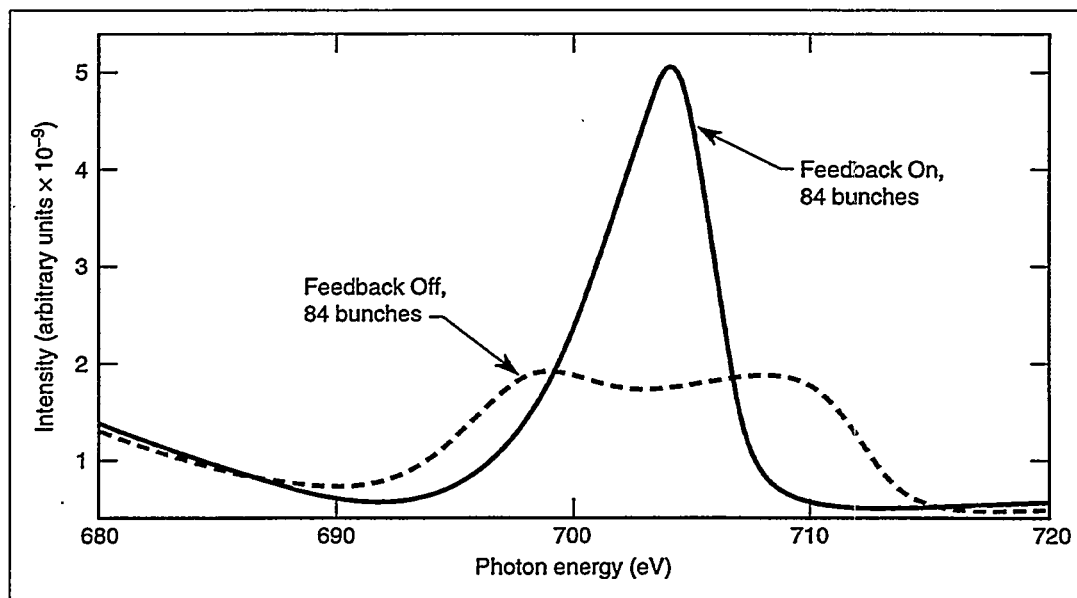


Figure 7. Measurement of the fifth harmonic on Beamline 7.0 with and without longitudinal feedback.

spread caused by coupled-bunch motion. Other parameter sets have been requested and supplied:

Operation at different energies. The storage ring has been fully characterized at 1.3 GeV and the injection system has also been set up for this energy. In preparation for 1.9 GeV operation in 1995, the storage ring has been ramped from its maximum injection energy of 1.5 GeV to 1.9 GeV. This was accomplished using only minor corrections to magnet settings predicted from magnetic measurements made four years ago. After these corrections were applied, a 50 mA beam was ramped, *without any loss of beam*, in 20 minutes. Future work will concentrate on improving the rf system, so that higher currents can be accelerated (the immediate goal is 250 mA), and on improving the ramping speed. As an exercise, it was also shown that the beam could be ramped down from 1.9 GeV to 0.8 GeV, again without any loss of beam.

Single- and two-bunch operation. Single- and two-bunch modes of operation at 1.5 GeV were implemented for time-resolved studies on Beamline 9.0. Here the current was limited to 20 mA per bunch due to a hardware limitation on the storage ring transverse kicker electrodes. In any case, an upper limit of 25 mA per bunch will be necessary to avoid the TMCI described above. The beam lifetime under these conditions is relatively short, about 30 minutes, and was increased to 2 hours (by sitting close to a betatron coupling resonance) at the expense of an increased vertical emittance.

The bunch purity, defined as the amount of beam in what should be empty buckets, was 1%; much larger than the requested value of 0.01%. Investigations found that this beam is being generated right back at the electron gun. Some dynamic "tricks" involving changing gun characteristics, and injecting on the side of the booster injection kicker pulse will be tried, but it is likely that we will have to change the cathode itself to get to the required conditions. This is planned for the January shutdown.

PLANS FOR 1995

Let me conclude by presenting a list of our top priorities for 1995. The order in which they will be pursued will depend to a large extent on user requests.

- Energy Ramping: Goal is 250 mA at 1.9 GeV in a ramp time < 5 minutes.
- Beam stabilization: Improve drift and jitter.
- Magnet "feed-forward" through a server: Required particularly for undulator steering and tune compensation, but will also facilitate faster energy ramping.
- Multi-bunch feedback: Required for higher quality undulator spectra.
- Single bunch (or few bunch) purity: Goal is 0.01%.
- Improve the storage ring injection efficiency: This will improve filling times and facilitate trials of top-off operation.

To aid in these activities our existing diagnostics will have to be augmented. Here our priorities are:

- Diagnostic beamline (Beamline 3.1): This will enable us to study such parameters as beam size, beam stability, bunch length, and bunch purity.
- Undulator beam-position monitors: The primary purpose of these new monitors is detecting beam motion that could lead to catastrophic failure of the vacuum system. However, they can also be used as very sensitive detectors for jitter and drift, and possibly in a feedback loop for undulator photon beam steering.

Progress in Beamline Commissioning and Overview of New Projects

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A year ago my report presented the results from our initial work to characterize the U5 undulator, the first measurements from the diagnostic port, and a description of our plans for completing and commissioning several new beamlines. Since that time, a huge amount of work has been done to bring these systems on line, and this has been accomplished in record time and with remarkable results.

This year my report will again feature an update of recent beamline construction, commissioning, and operation activity; and an overview of several of the new projects currently underway. The work described represents the efforts of the Experimental Systems Group, with excellent support from the Mechanical and Electrical Engineering Groups at the ALS. The fact that our most complex systems worked first time with little or no adjustment is a tribute to the superb work of these groups over a number of years.

ANALYZING UNDULATOR PERFORMANCE

We extended the work we began last year on measurements of the angular and spectral characteristics of the 5-cm-period undulator (U5) for Beamline 7.0 to include a full and detailed measurement and analysis of the undulator's performance. As before, this work was primarily carried out by Phil Heimann and Dmitri Mossessian.

The measurements were taken using an ALS-designed transmission grating spectrometer (TGS) that allows characterization of the angular as well as the spectral behavior of the light. The spectral distributions were measured on-axis for a range of K values, and the angular distributions were measured for both on-harmonic and red-shifted energies. As expected, the measurements in the low-energy or red-shifted tail of an odd harmonic indicated that, as the observation energy was tuned away from the harmonic, the emission was localized in an off-axis hollow cone that became larger as the difference between the observation and harmonic energies increased. Also, the angular width of the cone section decreased rapidly as the difference between the observation and harmonic energies increased.

For observation at an energy detuned from an odd harmonic by several times the harmonic energy width, the effective width of the cone section is given by the finite emittance of the electron beam. This technique was used to measure the electron beam emittance, giving values of 4×10^{-9} mrad and $< 2 \times 10^{-10}$ mrad in the horizontal and vertical directions respectively. (In the vertical direction, it is only possible to set an upper limit due to uncertainty caused by near field effects. The effect of observing in the near field is currently being assessed.) These values of emittance are considerably better than the "book" values previously

published for the ALS, although it should be noted that our measurements were made at currents of < 0.5 mA because of restrictions imposed by the maximum power load that the TGS can handle.

Since the optical components of the spectrometer had been precisely calibrated, we could accurately compare its data to theoretical determinations for the brightness and line shape of individual harmonics. Taking into account the measured emittance and the electron energy spread which had been determined from the widths of the harmonics, the calculations based on data collected with the TGS typically showed agreement in peak brightness within 5% of the measured magnetic field values for up to the 5th harmonic. The calculations used the measured magnetic field of the undulator, hence including errors, but the effect of the errors even in the 5th harmonic was less than 20%. The conclusion from this work is that the U5 undulators are essentially perfect—even in the higher harmonics—and the quality of light they deliver is dictated by the properties of the electron beam in terms of emittance and energy spread.

In August, the TGS was re-enlisted in order to characterize the light from the newly installed 8-cm-period (U8) undulator for Beamline 9.0. Again, measurements confirmed the near-perfect performance of the device. As this is a higher-field device than the U5, it was expected that many more higher harmonics would be observed at high K values. Figure 1 shows that about 100 harmonics can be observed at $K = 5.2$, eventually merging into a continuous bend magnet spectrum.

Another important characterization of the undulators was performed jointly by Tony Warwick of the Experimental Systems Group and by the Accelerator Group. This involved measurements of the motion in position and angle of the electron beam induced by uncompensated dipole errors in the undulators as the undulator gap was closed. The motion was measured with both photon and electron beam position monitors, and the measurements showed that the uncompensated dipole error fields are extremely small. In addition, the Accelerator Group developed a new algorithm for feeding forward error correction signals into adjacent steering magnets which holds beam position distortions under 30 μm for all adjustments.

COMMISSIONING OF UNDULATOR BEAMLINE 7.0

Beamline 7.0 is a state-of-the-art spherical grating monochromator (SGM) system, covering the photon energy range from 50 to 1200 eV. It consists of a spherical vertically focusing and deflecting premirror to focus light onto the entrance slit of the monochromator, three interchangeable spherical gratings, a translating exit slit, and post-exit-slit refocusing optics. The refocusing optics include a plane mirror, a vertically reflecting mirror that reflects light from the exit slit to the sample, and a horizontally reflecting mirror that focuses the light from the source onto the sample. The system was designed by Tony Warwick, and the testing reported here was carried out by Tony with assistance from Dmitri Mossessian.

The commissioning of the beamline proceeded in the following steps:

Focus size on entrance slit. The focus size at the entrance slit was measured by rotating the premirror and monitoring the signal immediately after the slit and after the monochromator. The beam size measured by monitoring the signal after the monochromator revealed a full

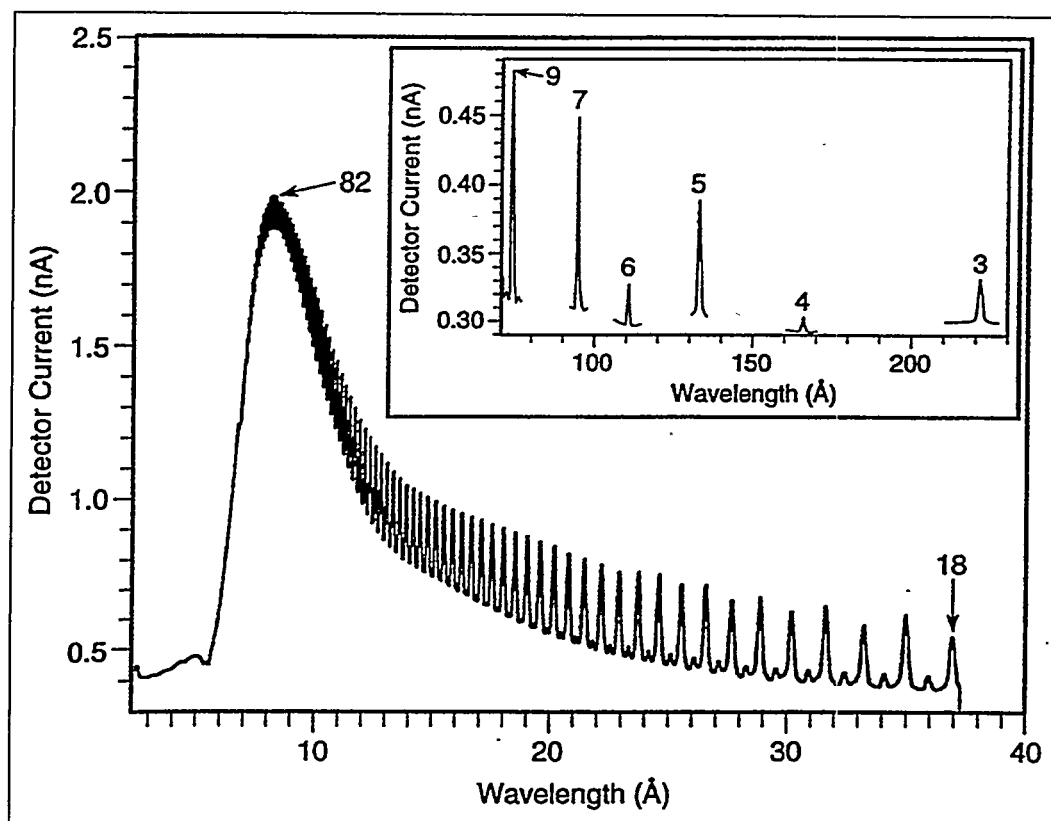


Figure 1. Spectrum of the 8-cm-period undulator taken by the TGS, showing harmonics from the 3rd to the 82nd (and beyond) at a value of $K = 5.24$ (25 mm gap).

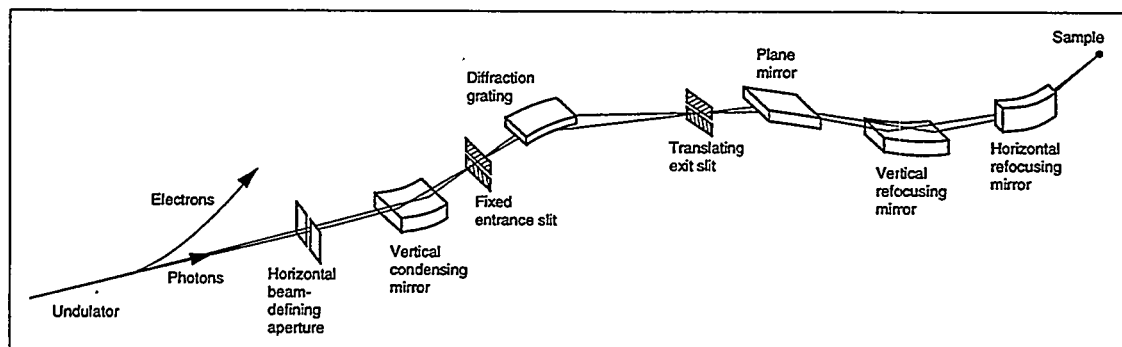


Figure 2. Path of the synchrotron light through the optical components of Beamline 7.0.

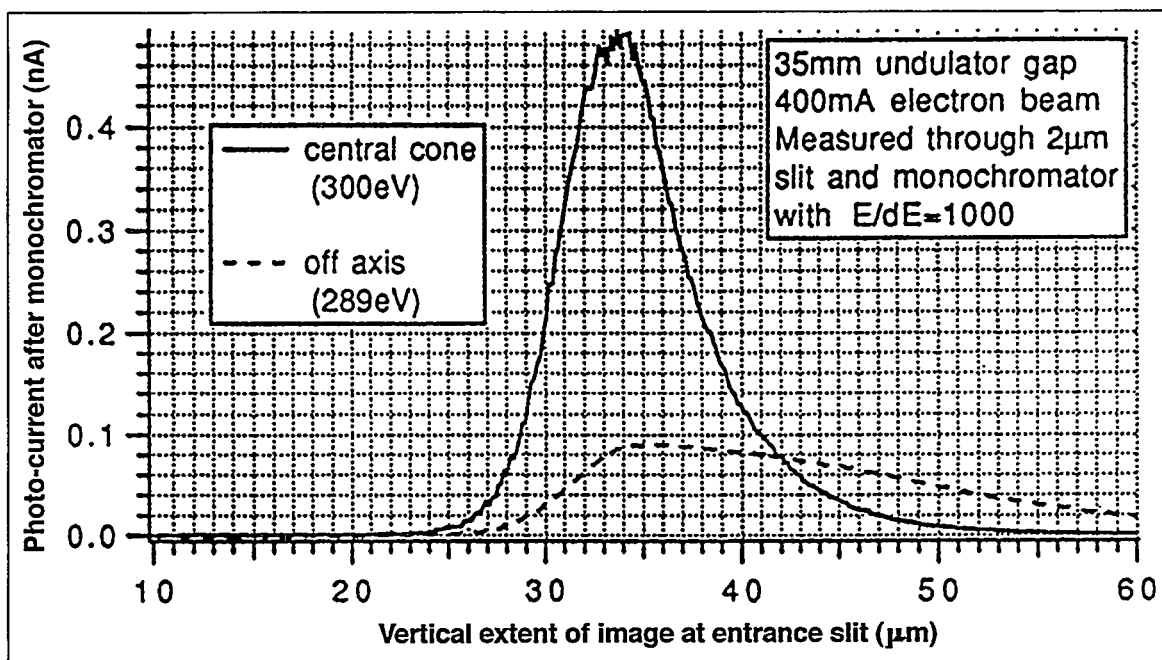


Figure 3. The focused monochromatic light image measured on the Beamline 7.0 monochromator entrance slit has a FWHM of 8 μm .

width at half maximum (FWHM) of 8 μm (see Figure 3). This was for the condition where the monochromator was tuned to the harmonic energy of the undulator. If the monochromator were detuned to the low-energy side of the harmonic, then the emission would be off axis and hence fill the pre-mirror more than in the on-axis case. This leads to coma aberration, and to a significantly enlarged image size. The 8 μm FWHM focused image size is in agreement with expectations, and demonstrates that the premirror and entrance slit are working perfectly.

Directly downstream from the gratings are the zero order light baffles. These are phosphor coated, and the dispersed light from the various undulator harmonics can be observed at this location (as shown in Figure 4). The light is observed to be distributed in parabolic arcs, with structure within each arc dependent on the harmonic number. For example, the fundamental has a single on-axis node, the third harmonic has an on-axis node and two symmetric off-axis nodes, and the even harmonics have an on-axis antinode. The general parabolic shape results from the correlation between emission energy and observation angle: the emission energy decreases as the observation angle increases, and the diffracting power of the grating increases as the photon energy decreases.

Resolution. To test the resolution of Beamline 7.0, we used the standard resolution test of measuring the nitrogen K edge $1s \rightarrow \pi^*$ absorption spectrum in N_2 . As expected, the vibrationally split series was observed, and, after a few hours in which the exit slit motion was calibrated, the expected resolving power of around 10^4 was obtained (see Figure 5). No fine alignment of the monochromator, slit heights, rotations, etc., was required to reach this result. The rapidity with which this very high value of resolving power was measured is a tribute to the mechanical engineering of the beamline, and to the accuracy with which all the components were surveyed and aligned.

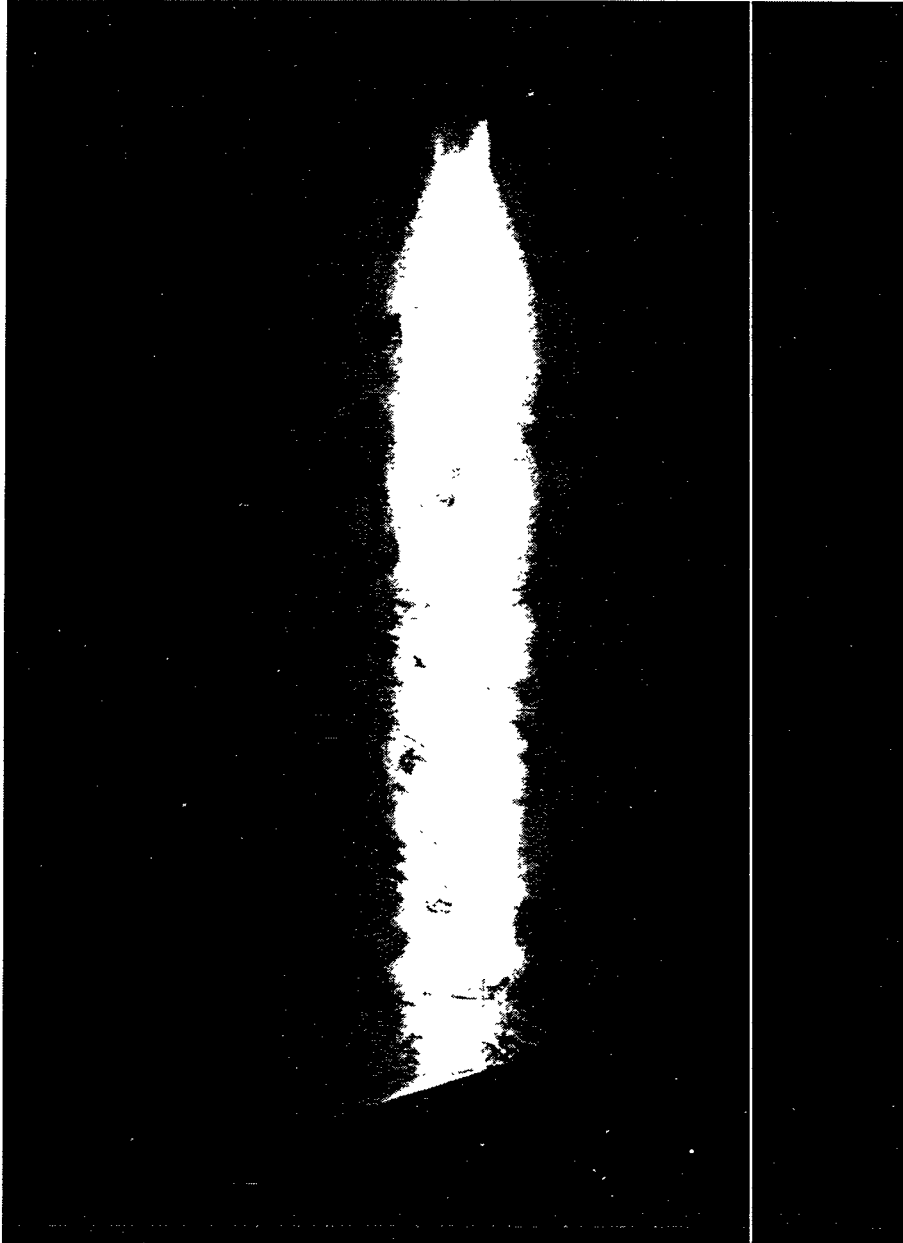


Figure 4. Undulator radiation incident on the phosphor-coated paddle located downstream from the monochromator on Beamline 8.0. A similar pattern was observed on Beamline 7.0.

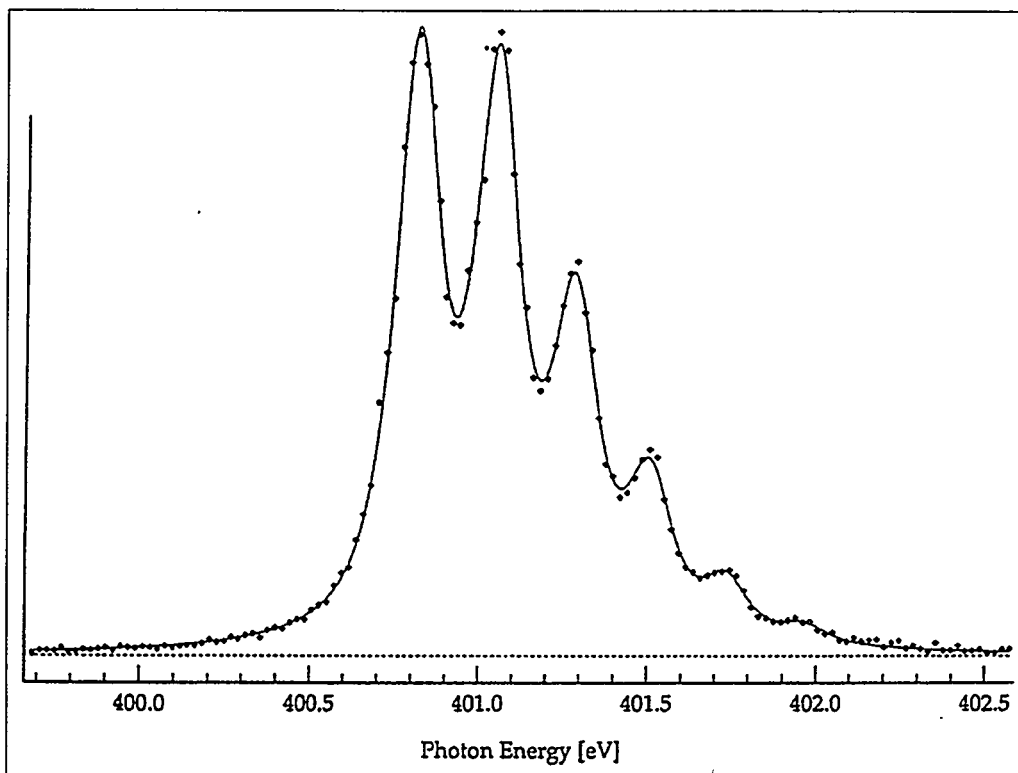


Figure 5. The resolution of Beamline 7.0 was tested by measuring the fine structure in the nitrogen K edge region of N_2 . Results showed a resolving power of around 8300 at 400 eV; equal to the best ever recorded.

Beam size at sample. One of the problems with a standard SGM beamline is that as the photon energy is changed, the exit slit position changes in order to keep the beam in focus. Since Beamline 7.0 is designed to supply light to microscopes, and since these microscopes require a fixed object position, the beamline's vertically focusing mirror was designed to be bendable so that it could image the variable position object (the exit slit) to a fixed image position. The mirror, shown in Figure 6, is based on a design of Malcolm Howells in which the thickness of the mirror varies as the cube root of distance along the mirror. This structure is machined into a single Glidcop™ block using the technique of wire electric discharge machining (wire EDM), and, with the addition of EDMed flexural hinges, is a single monolithic bending block. The device also accommodates a piezo drive system which lets us obtain a required radius of bend by applying a certain voltage to the piezo.

The horizontal focusing mirror refocuses the source in the horizontal direction at 14:1 demagnification onto the sample; the nominal vertical magnification is 1:1. The focused beam size at the sample was measured using a knife edge technique, and yielded sizes of 45 μm (h) by 20 μm (v), in agreement with expectation. The horizontal refocus mirror system has two mirrors that can be interchanged by lateral translation. These mirrors deflect light into two endstation branchlines and allow easy switching of light into either station with minimal realignment, giving optimum use of the undulator radiation.

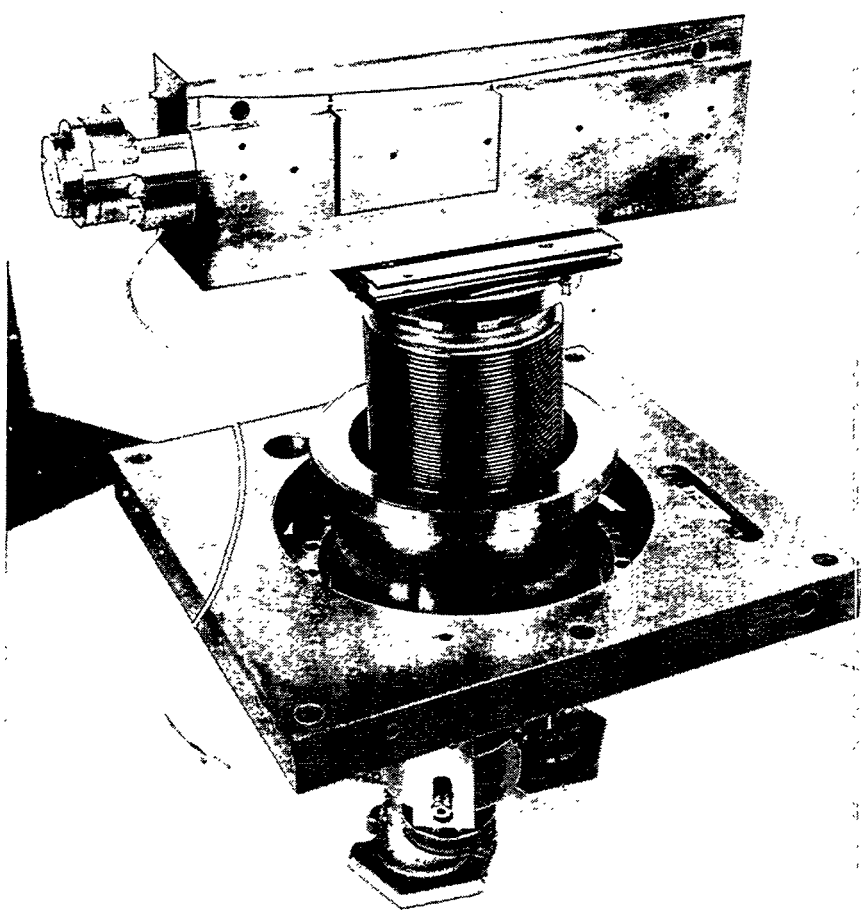


Figure 6. The Beamline 7.0 integral-bendable x-ray mirror.

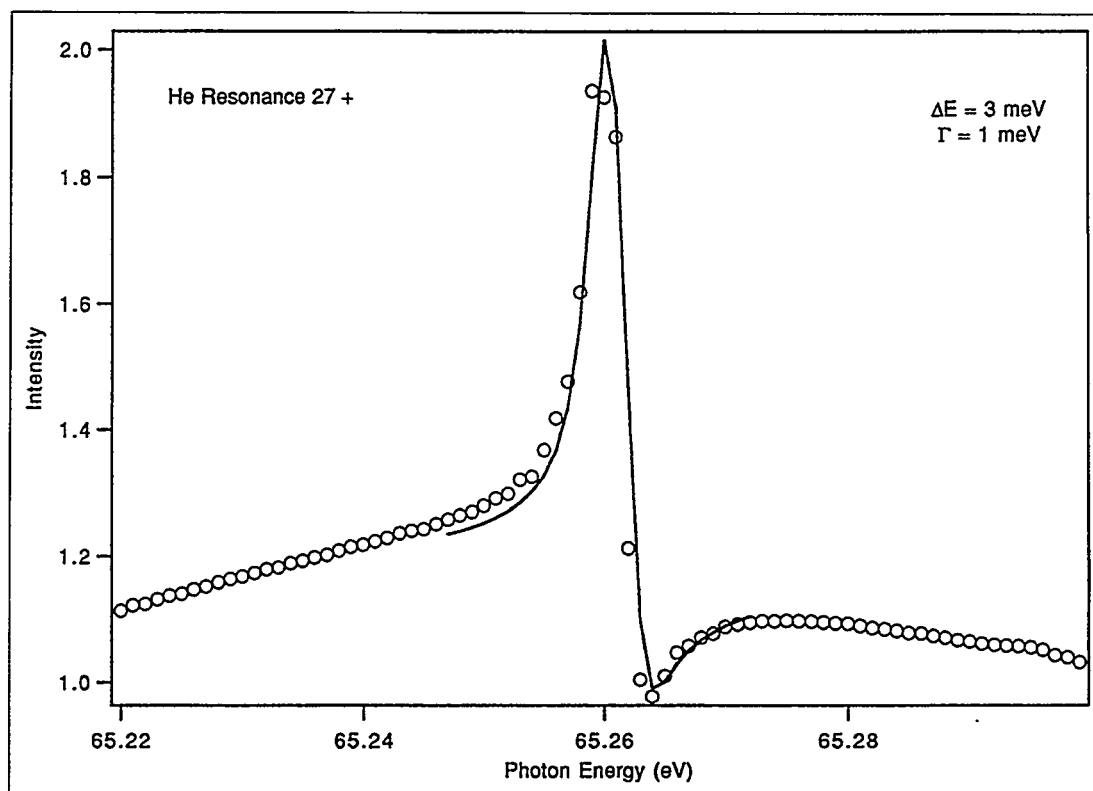


Figure 7. The absorption spectrum of helium double ionization series used as a resolution test for Beamline 9.0.1.

Undulator beamline 7.0 was commissioned in record time and its design parameters have been met or exceeded. This beamline has been in constant use since commissioning and has proved to be extremely reliable and robust. This has enabled a tremendous range of science to be performed in a relatively short time and gives a very good indication for the future.

COMMISSIONING OF BEAMLINE 9.0.1

This undulator beamline is similar to Beamline 7.0, except for the addition of a horizontally deflecting mirror as the first element in the beamline. The monochromator covers the energy range from 15 to 300 eV and will be used for a range of atomic and molecular physics. As in the case of 7.0, after the performance of the undulator was measured using TGS, the performance of the beamline was measured using a range of techniques. This report gives only the resolution and flux.

Resolution. The standard resolution test in this photon energy regime is the absorption spectrum of helium in the region of the double ionization series. The lifetime broadening of the higher term members of the series is extremely small and gives us an excellent resolution test. Measurements taken at approximately 65 eV indicate that the resolving power is close to 20,000, giving a line width of about 3 meV (see Figure 7). This indicates that the system is extremely well aligned (as with Beamline 7.0, this is thanks to the accurate initial survey), and that the grating used for this measurement has a very low slope error.

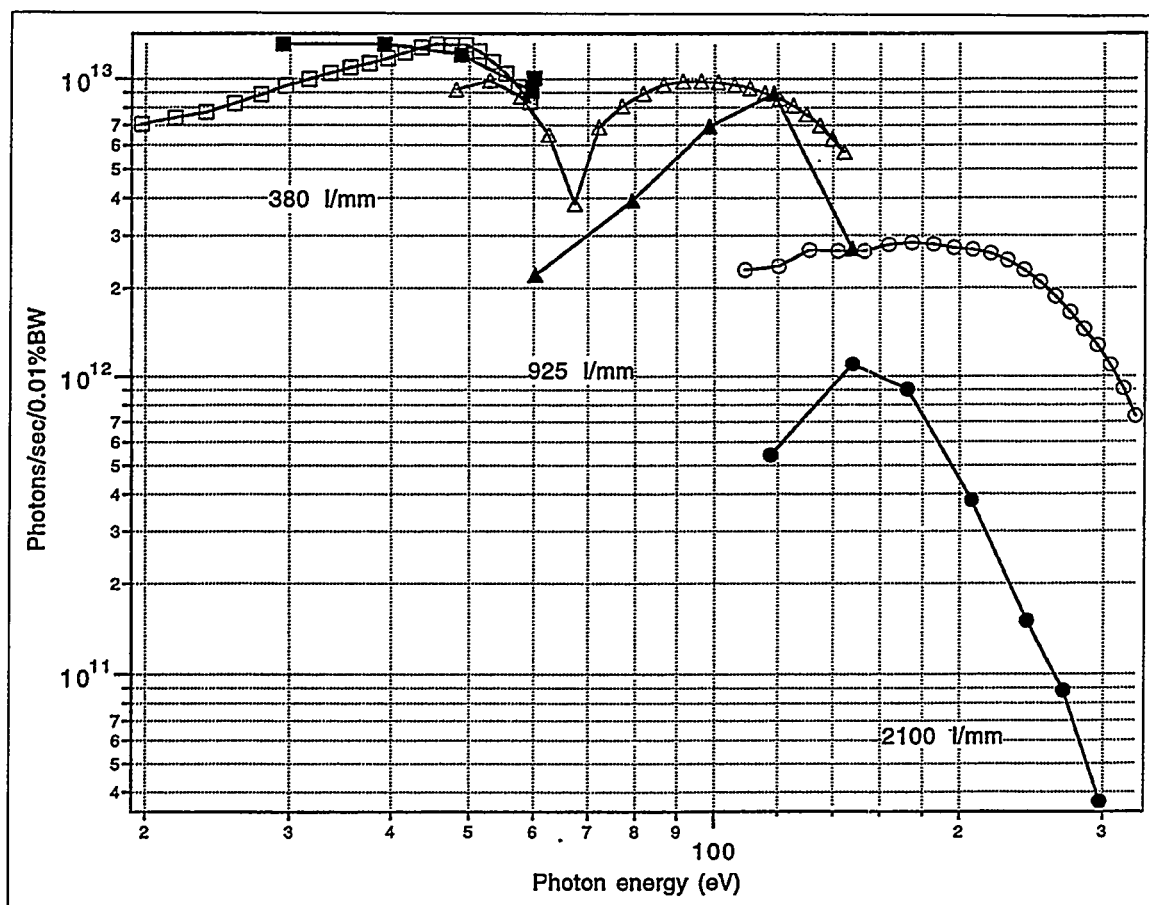


Figure 8. Flux measured at the 9.0.1 endstation focus for the 380, 925, and 2100 lines/mm gratings (solid symbols) compared to theory (open symbols) at a resolving power of 10^4 .

Flux. The flux at the sample has been measured over the full energy range up to 300 eV. The flux is as expected for the 380 lines/mm and 925 lines/mm gratings. However, in the energy range above 100 eV using the 2100 lines/mm grating and the third harmonic, there is a significant discrepancy between theory and experiment. Some of this is due to the reduction in peak brightness caused by excessive electron beam energy spread, but some is due to as-yet-unresolved differences between the two models which we have used to calculate the grating efficiency: the diffraction efficiency model and the full vector scattering model. Nonetheless, over much of the photon energy range, the resolved flux at a resolving power of 10,000 is 10^{12} – 10^{13} photons/sec, dropping to around 10^{11} at a photon energy near 300 eV.

Beamline 9.0.1 was commissioned extremely rapidly and has been in routine operation for about one month. Phil Heimann, Dmitri Mossessian, and John Bozek did a remarkable job in commissioning the beamline and bringing it to successful operation in record time.

9.3.2 BEAMLINE

This bend-magnet beamline is a standard SGM design covering the photon energy range from 30 to 1500 eV. The beamline's SGM, developed as the prototype ALS monochromator, was

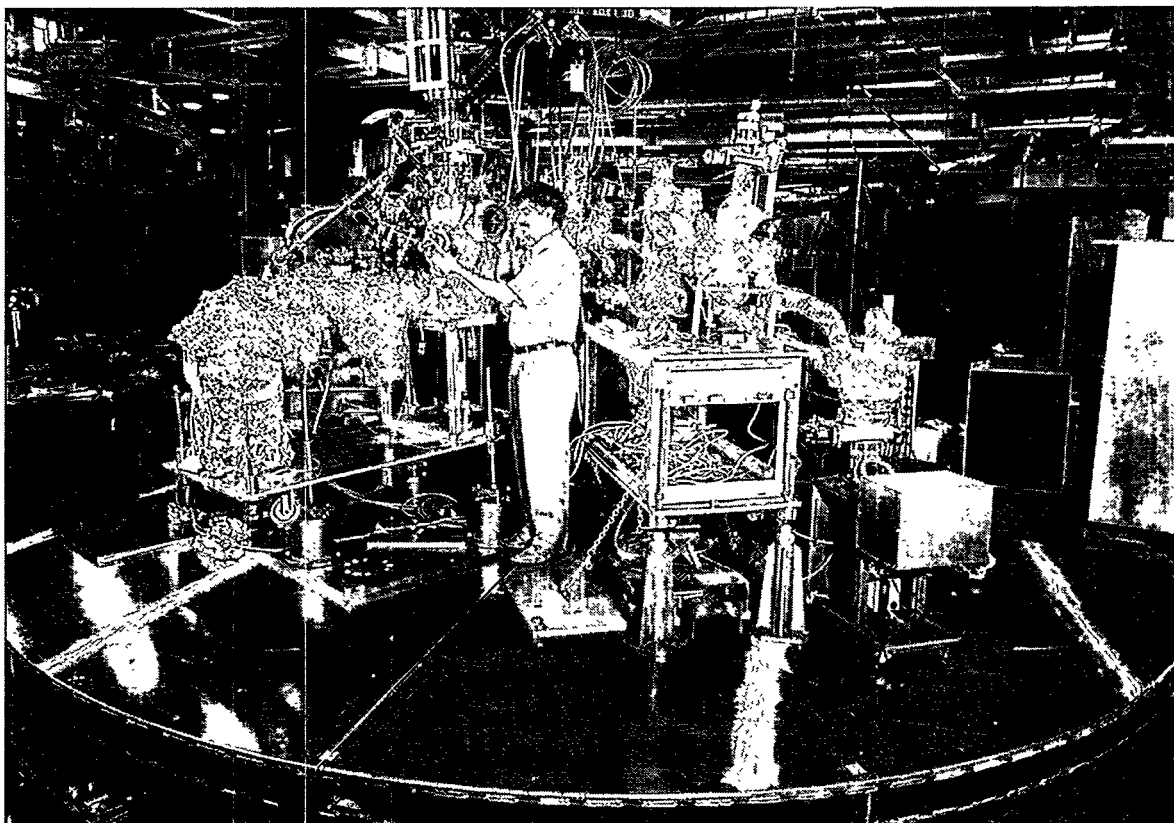


Figure 9. Zahid Hussain makes an adjustment to the angle-resolved photoemission spectrometer installed on Beamline 9.3.2's rotatable platform.

initially installed and tested on Beamline 6.0 at SSRL. It has now returned to the ALS and is undergoing its first tests of operation at Beamline 9.3.2, after being redesigned by a group led by Zahid Hussain (see Figure 9). The beamline underwent many modifications to make it compatible with the high brightness of the ALS, including a new cooled horizontally deflecting premirror, and a new vertically focusing mirror.

Another significant upgrade to the beamline was the design and construction of a rotating platform for the endstations. This allows two systems to be mounted on the platform at once, so that light can be switched from one to the other without breaking vacuum. The beamline received its first light in June, and, using a simple photoabsorption cell, the standard resolution tests indicated a resolving power of around 10^4 . The commissioning of the main system, a Scienta-based photoelectron spectrometer, is now underway, and on the evening before the Users' Meeting, the first data on the 4f states from tungsten were recorded. Although work is proceeding, beam instabilities, probably caused by the vibration of the vertical focusing mirror, are causing some problems. These are currently being investigated, and a new mirror mechanism will be fitted by December 1994.

CHEMICAL DYNAMICS BEAMLINE 9.0.2

The installation of the chemical dynamics branchline on Beamline 9.0.2 is making steady process. The "white light" branch (9.0.2.1) should be operational just after the shutdown, in late February. By insertion of a mirror into the 9.0.2.1 branch, light can be deflected into the 9.0.2.2 branch and into a 6.65-m off-plane Eagle monochromator. This monochromator is designed for extremely high resolution in the scanning mode and is currently on order for April delivery, with first beam for commissioning work a couple of months later. This effort is being led by Phil Heimann, working with the chemical dynamics PRT, and with Masato Koike who is assisting with optical design.

NEW PROJECTS

Infrared. There has been considerable interest in the construction of an infrared spectromicroscopy facility at the ALS. Design work has now started with initial efforts aimed at coming up with a mirror design and retraction mechanism. The mirror design is difficult due to the extreme power density caused by the proximity to the tangent point (0.75 m), and due to the need to integrate the system within the machine vacuum chamber. Funds have been allocated for the construction of these key components, and it is hoped that with further funding the beamline can be installed in the fall 1995 shutdown.

Magnetic- and Spectro-microscopy. In response to the need to establish additional facilities to perform characterization spectromicroscopy, to provide a location for a CRADA-funded beamline for magnetic microscopy, and to provide another bend-magnet beamline, we have designed and constructed a new front end for bend-magnet Beamline 7.3 which will be installed in the January-February 1995 shutdown. There will be two side branches, each with a premirror inside the shield wall at a glancing angle of 2° and a central 6 mrad fan. Design for one of the branchlines is well advanced and will accommodate an "entrance-slitless," single-grating beamline designed to cover from 600 to 900 eV. It is designed to achieve the maximum photon-flux density on a sample, and it will be used with a full-field photoelectron microscope. The second branchline is a more versatile and general-purpose beamline and is being designed for a range of industrial spectromicroscopy applications such as microparticle identification.

Protein Crystallography. Our project to build a protein crystallography beamline at the ALS has passed through various phases since last year from the publishing of a white paper, through the submission of a formal proposal, to the review of the funding by the Department of Energy and the start of preliminary construction. The beamline itself will consist of 3 branchlines, two identical side stations for "fixed wavelength" experiments within the wavelength range 1.5 to 0.95 Å, and one central station for rapidly tunable multiple wavelength anomalous dispersion (MAD) measurements over the range 3 to 0.95 Å. The engineering of the side stations has been proceeding for some time, funded by a monochromator research and development project from the Office of High Energy Research (OHER). The key component in the side stations is the high heat load curved crystal monochromator, and detailed design and prototyping is underway. This station will be the first to be constructed and will be operational in May 1996. The wiggler itself is a 38-pole, 2-T device, now under construction. This was funded from an initial \$0.5M from University of California through LBL Director Charles Shank, as well as \$200K from OHER, and will be completed from new FY95 funding. The wiggler should be ready for installation in December 1995.

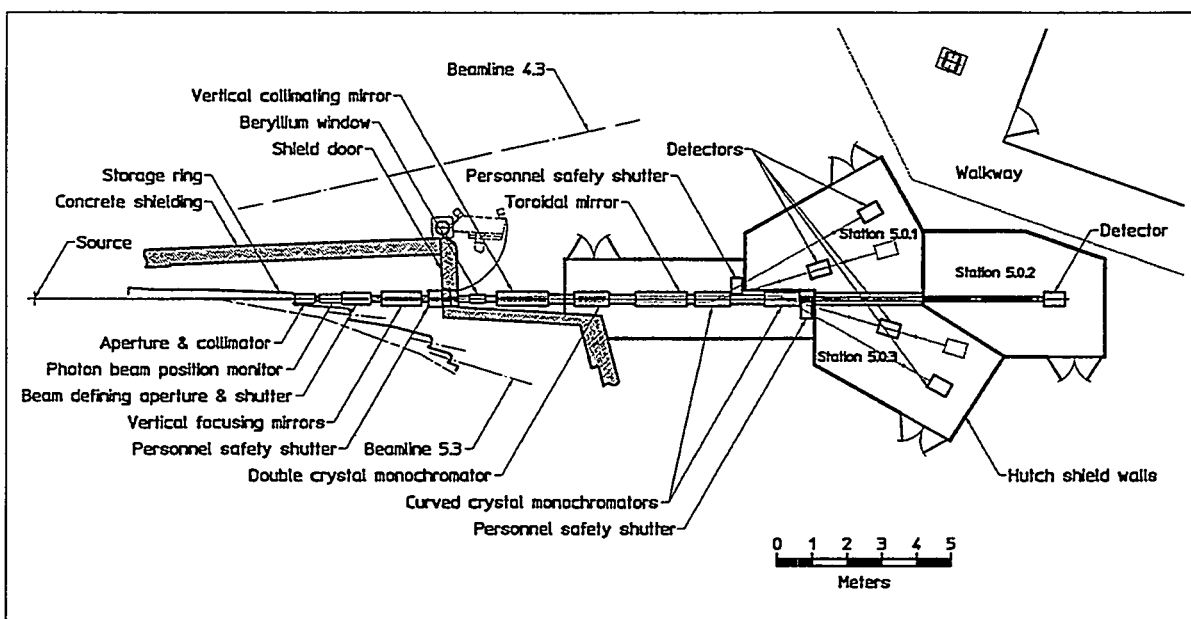


Figure 10. Layout of the Protein Crystallography Beamline.

Detailed comparisons have been made between this facility and other crystallography beamlines, for example bend-magnet sources at NSLS and at the APS, as well as multipole wiggler sources at CHESS, APS and SSRL. The conclusion of this work is that this facility will be fully competitive with these sources (excepting the APS multipole wigglers and undulators), and that for a typical crystal, the recording time will be around 5 secs/deg of crystal rotation. At these fluxes, absolute flux is not important, but detector readout time becomes critical. This problem has been addressed in the ALS facility by the use of state-of-the-art matrix CCD detectors. These have high quantum efficiency, high dynamic range, a small point spread function, as well as a readout time of about 1.7 seconds.

The key design feature of this beamline will be ease of use, brought about by a large degree of automation in sample and beamline alignment, camera calibration, and automatic data reduction and transfer. It is expected that the first branchline side station will be finished in May 1996. In support of the beamline, a separately funded \$7.9M Structural Biology Support Facility is now under construction. This project will be completed in late 1996 and will provide comprehensive support facilities in biochemistry, computing, and off-line analysis, as well as providing the necessary office and laboratory space for support of all activities in structural biology at the ALS.

Elliptical wiggler. The elliptical wiggler is in its final stages of design, with construction of some of the key components about to start. One of the major activities of the year has been a study of the radiation properties of the device, and in particular to study the magnitude of noise caused in a magnetic circular dichroism (MCD) spectrum by perturbations of the source. For example, these perturbations could be changes in the horizontal field, changes in aperture, or changes in position. We have also assessed the combination of horizontal and vertical fields that gives the highest figure of merit for single-event-counting MCD experiments.

This work has recently led to a comparison of elliptical wiggler and bend-magnet sources for this type of experiment using the 9.3.2 SGM optical system. We find that for typical conditions at the Fe L_3 edge, the gain factor of the elliptical wiggler is around 13, compared to the number of poles (28). We have identified a way to improve this by demagnifying onto the entrance slit, and aberration-correcting the monochromator, and it appears possible to get a gain factor of at least 30. This will be a major advantage in many types of MCD experiments, but particularly for experiments in which the magnetic system is dilute. The collinear nature of the left and right circularly polarized radiation will lead to a dramatic reduction in beam-position-induced noise caused by the above- and below-plane angular chopping of conventional bend-magnet systems. The beamline design is being intensively studied, and a complete optical design will be finished in spring 1995. Currently the beamline construction is being funded from Accelerator and Reactor Improvement and Modification (ARIM), and would take several years, but we have a proposal being considered by OHER for funding of construction.

The ALS Scientific Program

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My appointment as the first Scientific Program Head of the ALS began in April, with responsibility for the overall scientific program of the facility. Today I would like to talk about some of my reactions to the first months on the job, and about recent developments in the scientific and user program at the ALS. I will also be talking about our industry involvement, the proposal process for independent investigators that is underway, and some of the strategies we have in mind for "filling the ring."

The development of the ALS scientific program in the last year can be summarized in one line:
 $P = R + iI.$

That is, the scientific program (P) now has a large real part, not just an imaginary part—and I would like to begin my talk by describing some of the new science that has come out of the ALS. I had contemplated focusing on the more recent highlights, but there are so many of those that I thought if I selected just one or two, I would end up alienating almost the entire user community. So instead, let's look at some of the ones that I saw when I first arrived. At that time there was buzzing and excitement about the new results in soft x-ray fluorescence spectroscopy just coming out on the first undulator beamline, Beamline 8.0, done by Dave Ederer, Tom Callcott, and their collaborators. They did these experiments at a point when the beamline's monochromator did not have a grating in it, but they were not deterred by an obstacle like that; they used the undulator harmonic itself for monochromatization. They looked at a thin film of boron nitride, sweeping across the boron K edge. As they sat at the K edge, they saw a huge resonance at the $1s \rightarrow \pi^*$ transition; and off resonance they saw another, broader peak, which is actually the density of states (Figure 1). The thing that got us all excited about this is that the boron nitride layer was buried under a relatively thick layer of carbon. That's exciting—a spectroscopy which can be used to study buried interfaces.

Interestingly enough, when the second undulator beamline came into operation, the lead-off experiment there was also soft x-ray fluorescence spectroscopy. The research group on Beamline 7.0 was Joseph Nordgren and his co-workers and this time the sample of interest was buckyballs. The history of his first experiment is a perfect example of what you heard this morning:

- Day 1 – Beamline is opened to the ring for the first time, beam delivered to sample
- Day 2 – First spectra obtained
- Day 3 – Experiment completed with publishable data

and that pattern of quick turnaround has been repeated over and over again at the ALS. When I saw Nordgren's results (Figure 2), it gave me a tingle of excitement because it reminded me

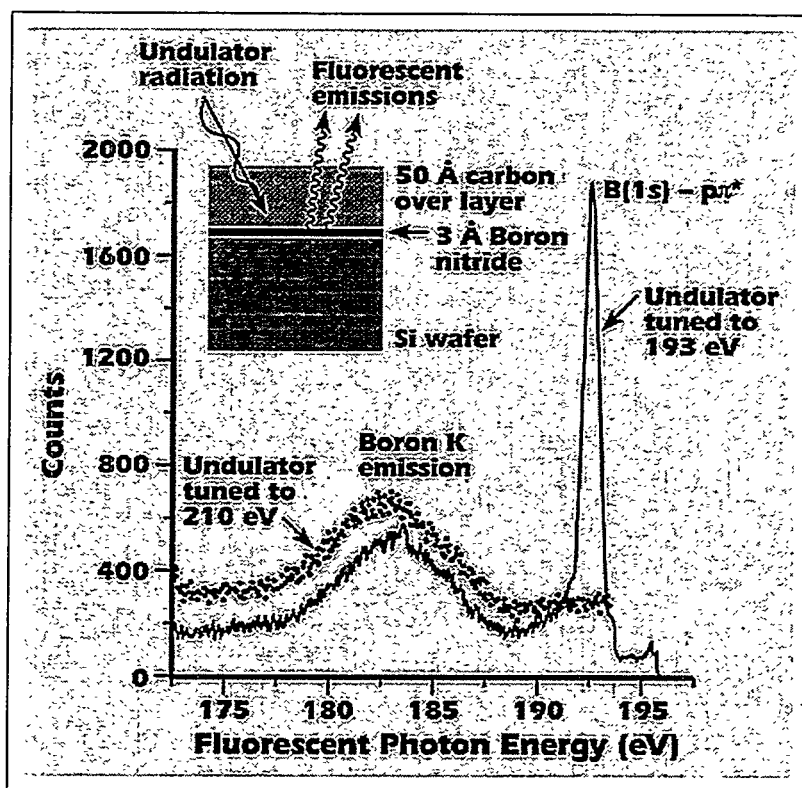


Figure 1. Soft x-ray fluorescence spectra taken at Beamline 8.0 before its monochromator grating was installed showing the boron density of states and the $B(1s) \rightarrow \pi^*$ transition from a thin layer of boron nitride buried beneath a thick layer of amorphous carbon. The photon-in, photon-out nature of this technique is what makes the buried layer accessible; the brightness of the ALS is required to create sufficient fluorescent yield from light elements to make this a viable research technique.

of what photoemission spectroscopy was like 25 years ago when I took my first photoemission spectra. What you see are very, very rich spectra of emitted x rays as a function of incoming x-ray energy. In the spectra you can see the valence band density of states, and some interesting dynamics close to threshold. Beyond threshold, the spectrum settles down to look something like the valence band density of states. So these fluorescence spectra look very much as photoemission spectra looked 25 years ago, with valence band density of states, rich structure, many peaks, good quality data, and good resolution.

The technique used in Nordgren's experiment, which is made possible by the brightness of the ALS, has the power of photoemission but can be used to study systems not accessible to ordinary photoemission. Since the ALS beam allows you to put the light into a very small spot on the sample, you can get a lot of photons through the entrance slit of the second spectrometer. I find this very exciting, and I think this technique will really take off—but it can't become too widespread because the ALS is the only place in the United States where you can actually practice this technique!

Now let us move on to where I think the mainstream of our scientific program is going to go—microscopy—which benefits greatly from the brightness of the ALS. One way you can do microscopy is to put undulator radiation through a Fresnel zone plate into a small spot on the

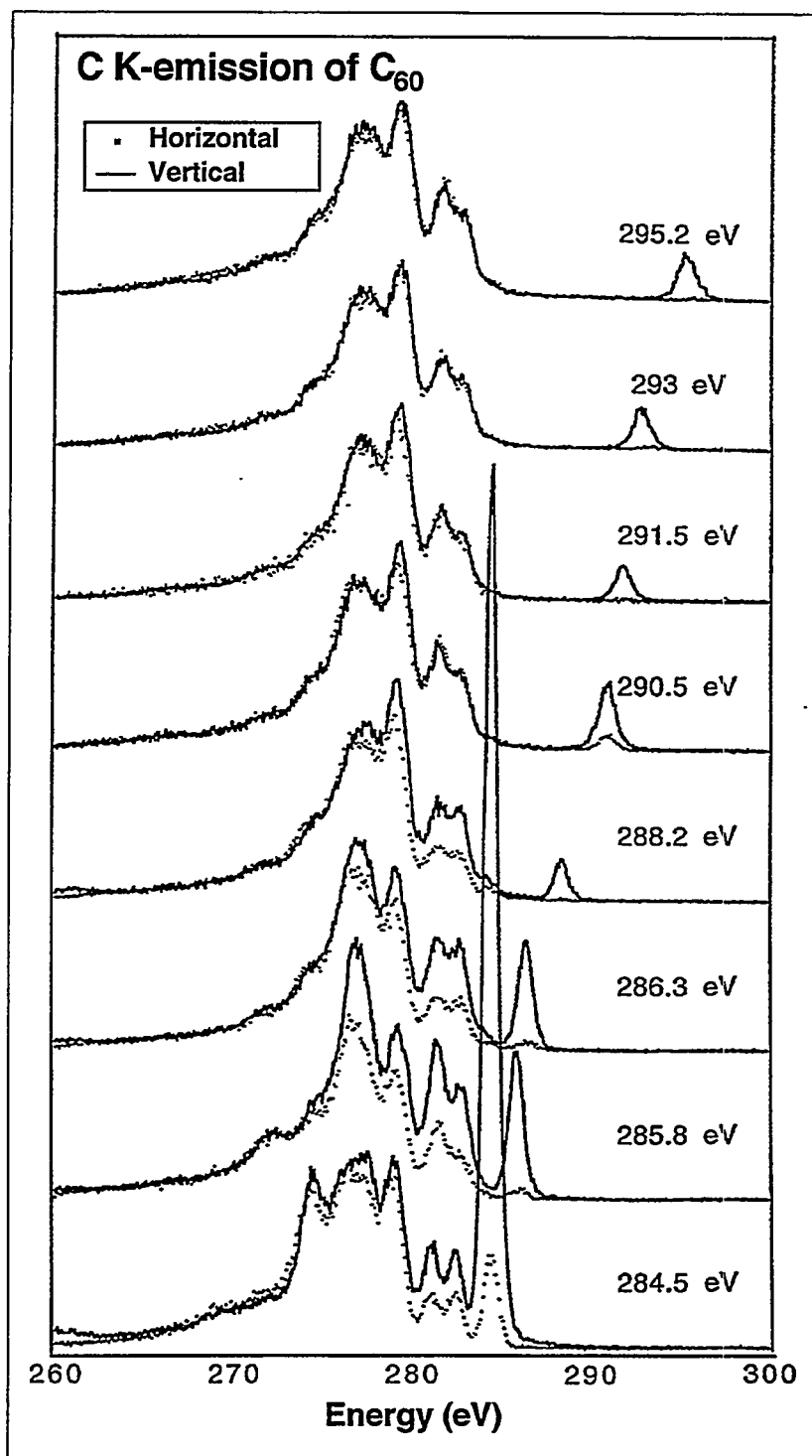


Figure 2. Spectra taken at Beamline 7.0 from buckminsterfullerene (buckyballs) using soft x-ray fluorescence spectroscopy. The uppermost spectrum, taken at 295.2 eV photon energy, approximates the density of states for the fullerene, while the spectra taken closer to the threshold energy show markedly different effects. Because of the high brightness of ALS undulator light, Beamline 7.0 researchers are able to produce fluorescence spectra which rival photoemission spectra in their richness, resolution, and short data-collection times.

sample and do photoemission. You can scan the sample through the spot and get elemental sensitivity and chemical sensitivity. Why do you have chemical sensitivity? In photoemission, chemical sensitivity comes out as a shifting of the core-level peaks. Another way to get chemical sensitivity is to do XANES (x-ray absorption near-edge structure), and a third way is using left and right circularly polarized light, with magnetic circular dichroism (MCD).

The fact that much of ALS science activity is likely to be in microscopy means the problems we can address are problems of concern to industry. This is very fortunate—the ALS has in a sense lucked out. We are under political pressure now to become responsive to societal needs for industrial competitiveness, environmental remediation, and so on, and here we are sitting on an instrument that is capable of being responsive! So we can move in this direction, and rather than being artificial or forced it is the natural evolution of the program. I admit this comes as a certain relief to those of us who have steered the program in this direction. I have for a number of years been the chair of the Program Review Panel, so I shared that responsibility. One of our nightmares was that after we built these microscopes, there would be nothing worth seeing with them, but now we know that there are things of interest on the “nanoscopic” scale that these microscopes can address.

In the vein of increased industrial collaborations, we have initiated a vigorous outreach effort to try to attract industrial users to the ALS, plus we should never forget that IBM, a major player at the ALS, has been involved for ten years now. They own and operate their own beamline, and, if that weren't enough, they have now entered into a cooperative research and development agreement (CRADA) with us to further develop magnetic microscopy.

New industrial contacts we are developing include Intel Corporation and Charles Evans and Associates in the semiconductor industry; Chevron in the petrochemical industry; and Amgen, Syntex, and several other biotechnology companies who are interested in our new protein crystallography beamline. I will use the semiconductor area to give an example of how these relationships evolve. These companies are interested in doing microcontamination analysis using techniques like total x-ray reflection (TXRF) and micro-XANES. The one thing that they have impressed upon us is that if these are to be useful analytical tools for their industry, a company has to have very rapid turnaround on an around-the-year basis. We've responded to this by forming an alliance with SSRL so that at any given time, the ALS, or SSRL, or both, will be in operation. We also participated in a meeting of Sematech on August 29th along with SSRL, and it looks very likely that some of the microcontamination analysis techniques to be available at the ALS are going to be included in the semiconductor industry association roadmap. This will open up avenues to new funding sources for this kind of work by giving us the seal of approval of the semiconductor industry.

Let me switch gears again and talk about the program for independent investigators. We are putting out a call for proposals from independent investigators; in fact, I'm calling for proposals right now. We have tailored our proposal process (outlined in Figure 3) to incorporate the best features of the procedures now being used at Brookhaven, Stanford, and Wisconsin. The ALS has two scheduling cycles per year: April–September and October–March; and proposals for beamtime from October 1995 to March 1996 are due June 1, 1995.

One of my other responsibilities is to chair the Program Advisory Committee (PAC), formerly the Program Review Panel (PRP). The role of this committee was recast in June from review-

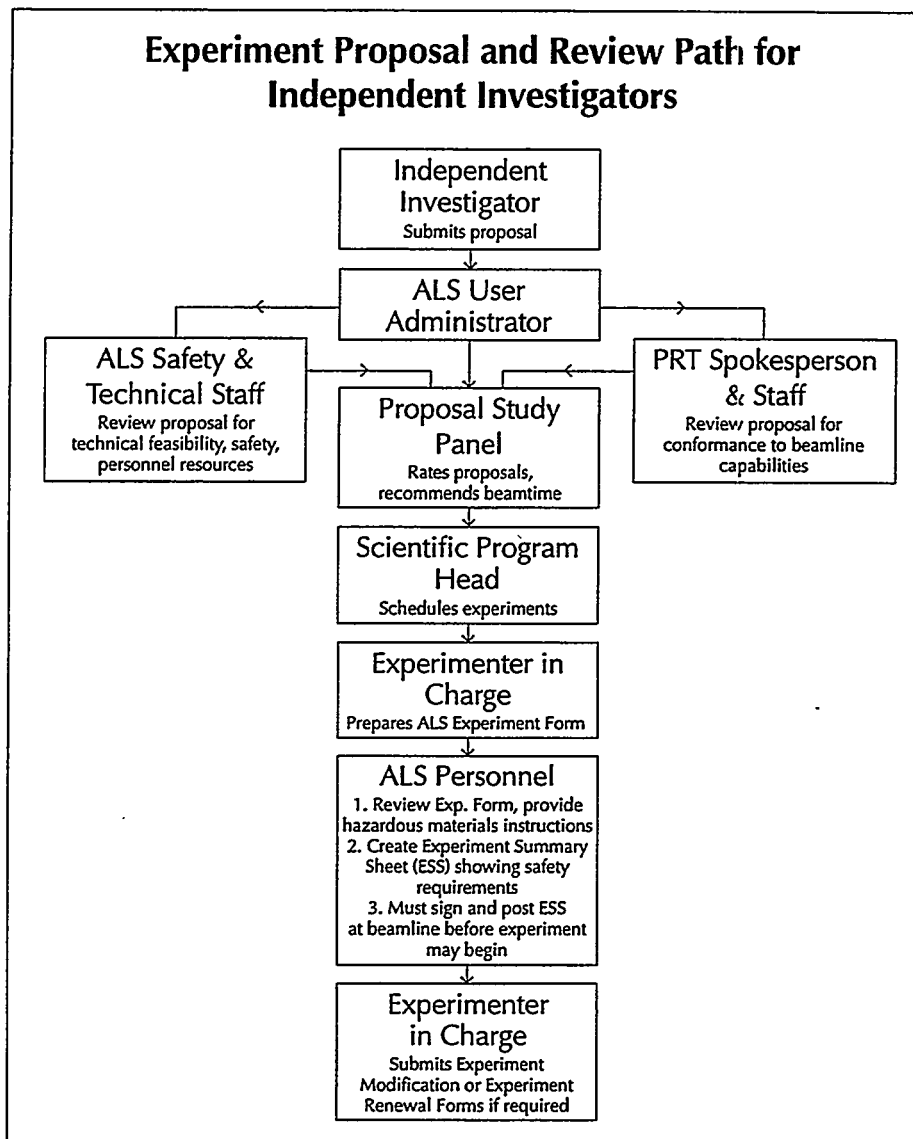


Figure 3. The process by which Independent Investigator proposals are evaluated and scheduled for ALS beamtime. After a proposal is accepted, the experimenter-in-charge prepares an ALS Experiment Form for the experiment which goes through a review process (last three steps on flowchart) to make sure all safety issues have been identified and resolved before the experiment begins.

ing and approving proposals for new PRTs to serving as a "board of directors" working through ALS Director Kincaid to advise the LBL Director on current ALS operations, allocation of facility resources, strategic planning, budget development, and other major issues. The members of the PAC for the next year are Franco Cerrina, Charles Fadley, Keith Hodgson (new), Christof Kunz, Jerry Lapeyre, Bob McDonald (new), Dave Shirley (new), and François Wuilleumier. Let me say a few words about the new members and why I asked these particular people to serve on this committee, because that in itself will give you the flavor of where we see our scientific program going. Keith Hodgson is the deputy director at SSRL and a life scientist. He will be wearing retiring member Bill Orne-Johnson's former hat, representing life science interests, and will help us cement cooperative relations with SSRL. Bob McDonald is a manager in the research organization at Intel Corporation. Finally, the ALS is an outcome of

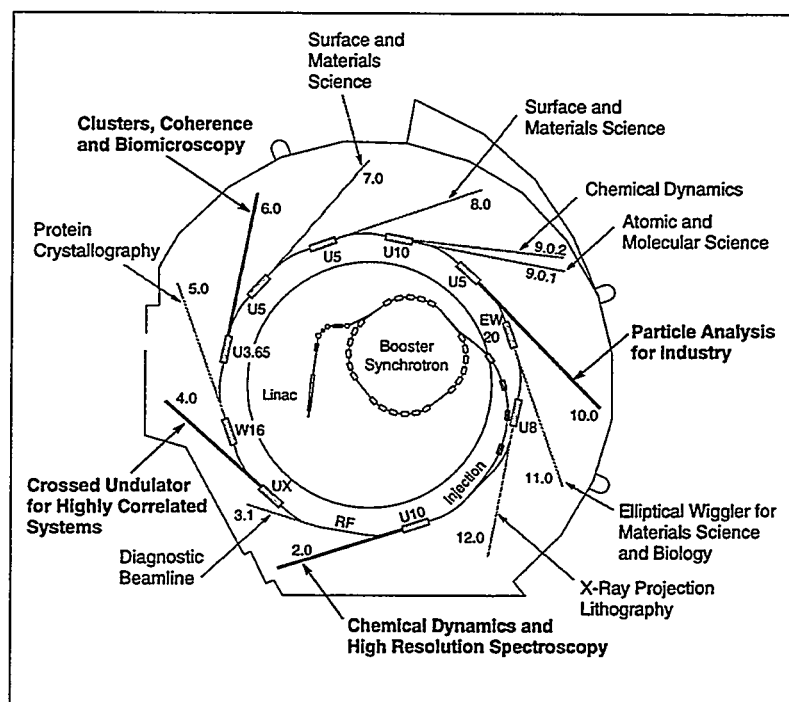


Figure 4. ALS floor plan showing the location of insertion-device beamlines for the scientific program, as well as the accelerator diagnostic beamline 3.1. Beamlines presently operating or under construction are shown in background, whereas proposed beamlines are highlighted.

Dave Shirley's vision, and we wanted him to come back and give us another infusion of vision for the next few years.

Let me finish by summarizing where we would like to go from here. Figure 4 shows one strategy for filling the ALS ring. It includes existing insertion devices, those under construction or in the design stages such as the protein crystallography wiggler and the elliptical wiggler, and suggestions for the four straight sections presently not spoken for. It shows our vision of the way we see the facility evolving: our highest priority is particle analysis for industry, because we think this ability to study small particles on wafers and systems of interest to industry is extremely important. Chemical dynamics will move from its present location on Beamline 9.0 to its own beamline. Another elliptically polarizing beamline, working in the low photon energy region, will complement the elliptical wiggler beamline. And we would like to see another beamline to incorporate some of the biomicroscopy you heard about this morning.

First Results from the SpectroMicroscopy Beamline

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The SpectroMicroscopy project is really a collection of experiments designed to maximize the benefits of the undulator and monochromator on Beamline 7. The experiments have a common theme of combining high spatial resolution and high energy resolution, for either mapping or structure determination with chemical-state selectivity. There are a number of ongoing projects. These include the ultraESCA program, x-ray photoelectron diffraction (XPD), the PRISM photoelectron microscope, and two zone-plate microscopes, one for high pressure studies and the other for ultra-high vacuum surface studies. First results have been obtained from ultraESCA, XPD, and PRISM, portions of which are summarized here. The zone-plate microscopes are under construction, with the first images expected to be taken shortly after the winter '95 shutdown.

THE ULTRAESCA PROJECT

We have constructed a photoemission system using a multi-channel detector photoemission spectrometer and automated 5-axis sample manipulator, to get the best performance from the combination of small-area, high intensity, and high energy resolution that the undulator beamline can provide. This project is called "ultraESCA", which stands for *UnduLaToR* Assisted Electron Spectroscopy for Chemical Analysis.

The idea is to try to simultaneously make major improvements in x-ray photoelectron spectroscopy (XPS or ESCA) in terms of small analysis area, overall spectral resolution, and counting rate. This results in a figure of merit, $G=I/(dA*dE)$, which characterizes the sensitivity of the tool. Compared to sophisticated laboratory ESCA instruments ($G=1$), ultraESCA has an enhancement in merit of $G=100,000$. Some examples of surface-sensitive, high energy resolution, high statistical quality core-level spectra from reconstructed silicon surfaces have been taken with total spectrum acquisition times of 15 seconds or less. Surface sensitive core-level spectra from Si(100) and Si(111) give count rates in excess of 1 MHz from areas of less than 100 micron diameter, with total system energy resolution of 0.08 eV. This type of performance has had a tremendous impact on the field of XPD (see below).

The ultraESCA instrumentation allows us to create maps, or images, of photoelectron spectra as a function of position on the sample, with a spatial resolution set by the monochromator spot size. Using the adaptive refocusing optics of BL7, this spot size can be adjusted to about 30 microns, with no penalty in count rate, at high energy resolution ($R \sim 8000$). There are numerous applications of this technique of scanning ESCA, but one in particular has had interesting initial results.

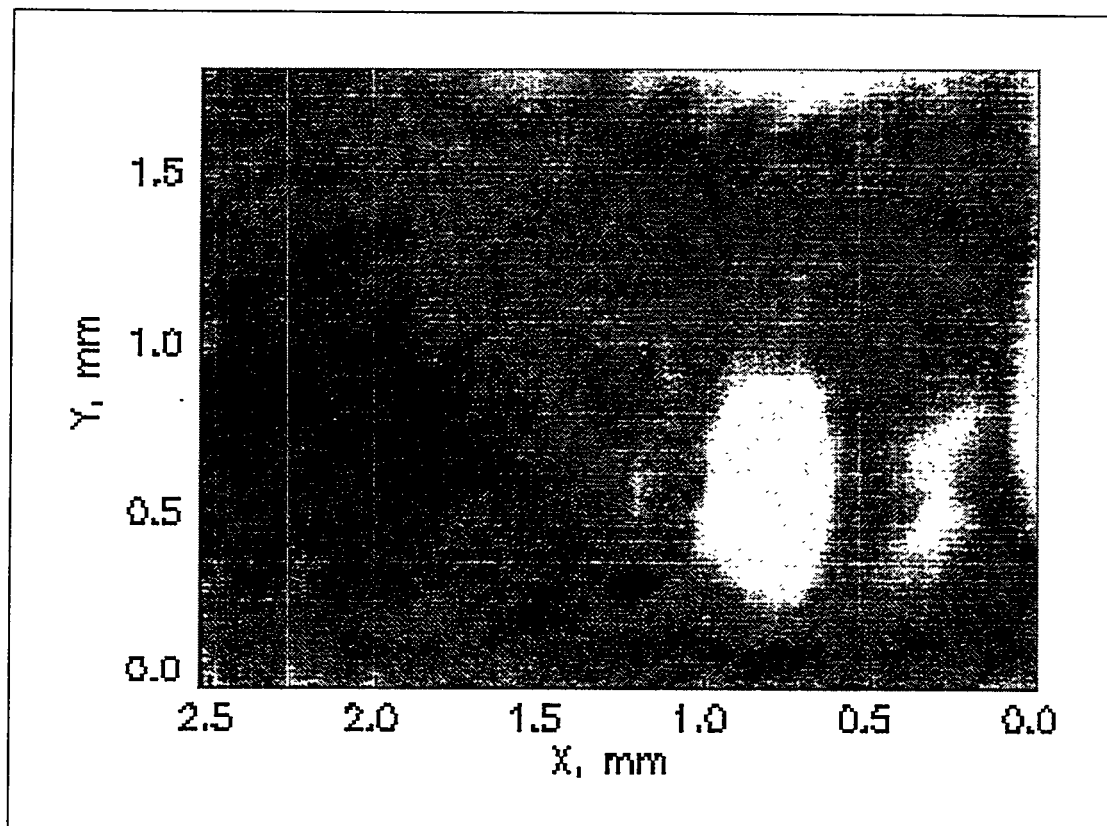


Figure 1. Scanning photoelectron spectroscopy image of a small particle of curium compound, where the image intensity is proportional to the Cm 4f core-level count rate. The bright spot in lower right is the Cm oxide particle.

We have used ultraESCA to study small particles of actinide compounds, primarily curium compounds. Ordinarily, the problems associated with radioactivity and toxicity of actinides makes it extremely difficult to perform spectroscopic measurements at facilities, without extraordinary sample handling precautions. By reducing the sample dimensions and volume, and immobilizing the samples on substrates, these problems can be greatly simplified. We have shown that photoemission cross-sections, as a function of incident photon energy, can be readily measured from samples of less than 3 nanograms mass and 3 picoCuries of activity. Some of the first soft x-ray absorption spectroscopy on actinides has been done this way, as well.

In an ultraESCA experiment on a small particle, the mapping capability is first used to locate the particles on the substrate. An example image is shown in Figure 1, where the intensity of emission from Cm 4f core-level is plotted as a function of position on the substrate. The substrate appears dark, with a high-contrast bright white dot showing the location of the actinide particle. This specific particle is large, about 250 micron by 500 micron, so individual points on the particle surface can be chemically analyzed. A typical photoemission binding energy spectrum from a spot on the particle is shown in Figure 2. The core-level spectrum shows features in addition to the 4f-7/2 and 4f-5/2 main lines, which may be due to additional chemical states. The area of analysis is about 75 microns across.

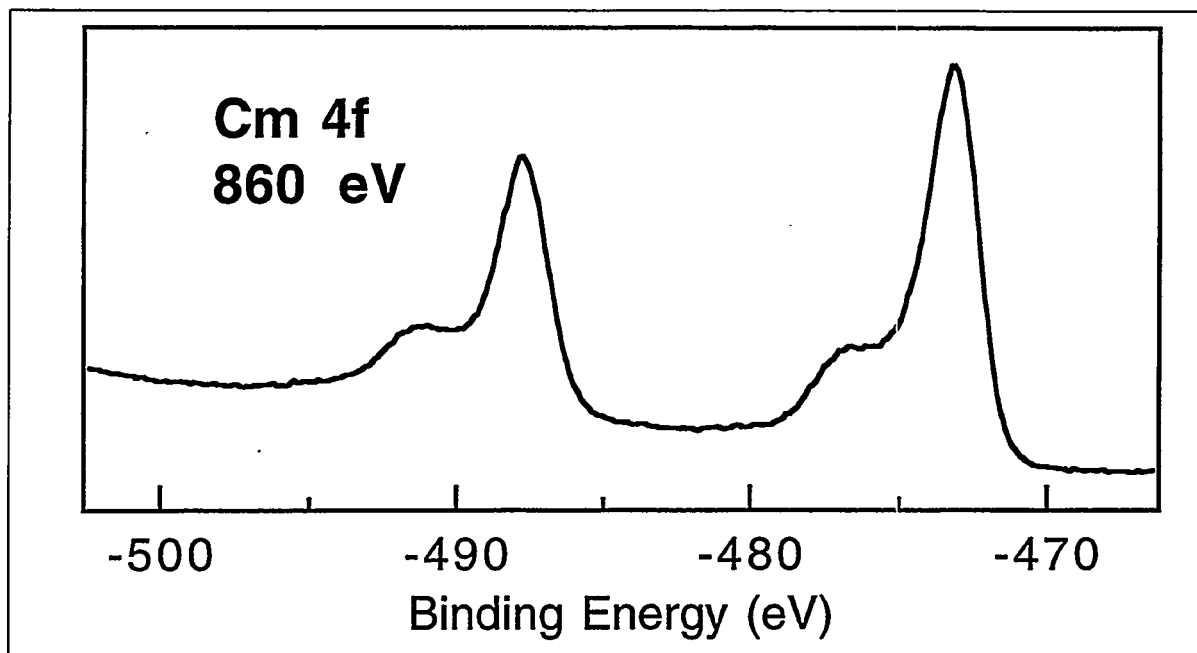


Figure 2. Photoelectron spectrum of a small Cm containing particle, located by scanning photoemission microscopy using ultraESCA.

We have scheduled several important extensions of this method for the future. The actinide work will continue, to look at a variety of compounds and measure the x-ray absorption spectra in correlation with XPS. In addition, a number of small particle analysis problems can be elegantly solved using this instrument. These range from soil contamination issues to chemical composition of airborne particles.

THE PRISM PHOTOELECTRON MICROSCOPE

A series of important benchmarks were reached in the PRISM photoemission microscopy project. PRISM, which stands for Paraxial Ray Imaging SpectroMicroscope, is the result of a collaboration between UW-Milwaukee and IBM-Almaden. It is a new design of a direct-view imaging photoelectron microscope, which uses electron optics to make a magnified image of a sample surface using the photoelectrons that are emitted. The electron optics consist of a high-voltage electrostatic magnification stage, bridged to a hemispherical electron spectrometer for selection of the electron kinetic energy. By selecting low energy electrons, which are copious, high spatial resolution images of the surface can be viewed at standard video rates. To map the surface chemical composition, the analyzer is set to pass electrons from a specific core-level, and the image is integrated to build up statistics.

An example of the first photoelectron core-level test pattern images taken with this instrument is shown in Figure 3. The sample was a mesh of Au (a standard electron microscopy mesh), which was sputtered to remove the contamination layer. After sputtering, the strong Au 4f core-levels are nicely resolved, as shown in Figure 4. The photoelectron microscope image was acquired by setting the analyzer to pass the Au 4f core-level. The image displayed took

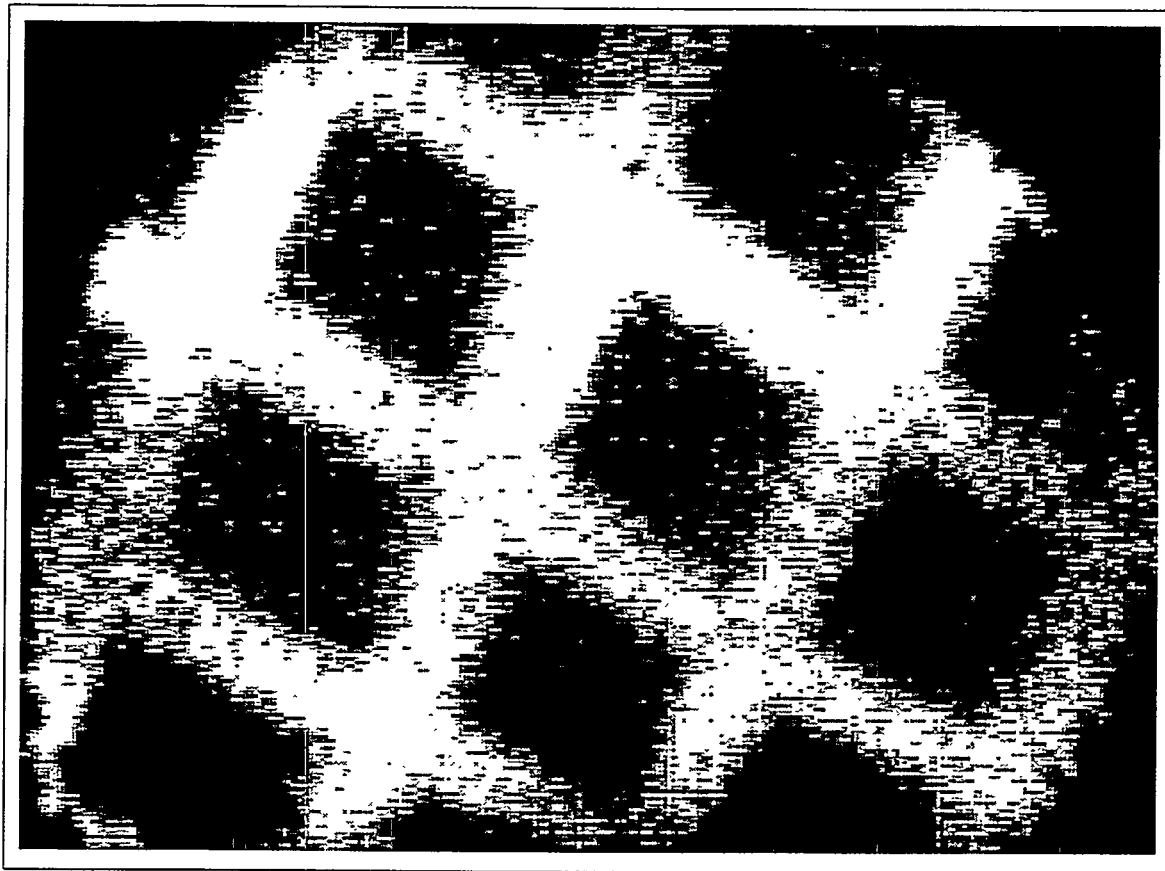


Figure 3. Photoemission microscopy of a Au grid, using the Au 4f core levels, acquired in 34 seconds. The grid spacing is 25 μm .

34 seconds to build up the statistics shown, which are sufficient to see the main structures of the grid.

Earlier tests of the spatial resolution of PRISM were made using lithographically printed photoresists, and video-rate microscopy with secondary electrons. Linewidths as small as 0.2 micron were easy to see at these speeds, and 0.1 micron is discernible in frame-grabbed pictures. The secondary-electron mode of PRISM also is used for chemical analysis, by sweeping the incident photon energy to generate an x-ray absorption near-edge spectrum (XANES). Micro-XANES mapping is being applied to a variety of problems, from magnetic domain imaging to the identification of the chemistry of contamination particles.

PHOTOELECTRON DIFFRACTION

Photoelectron diffraction is a method for getting precise structural information about atoms at surfaces from the energy and angle dependent variations in emission intensity of photoelectron peaks. Since photoelectron binding energies are sensitive to the local chemical environment, photoelectron diffraction techniques are one of the few structural probes available that can map out bond lengths and angles for individual chemical species in a material.

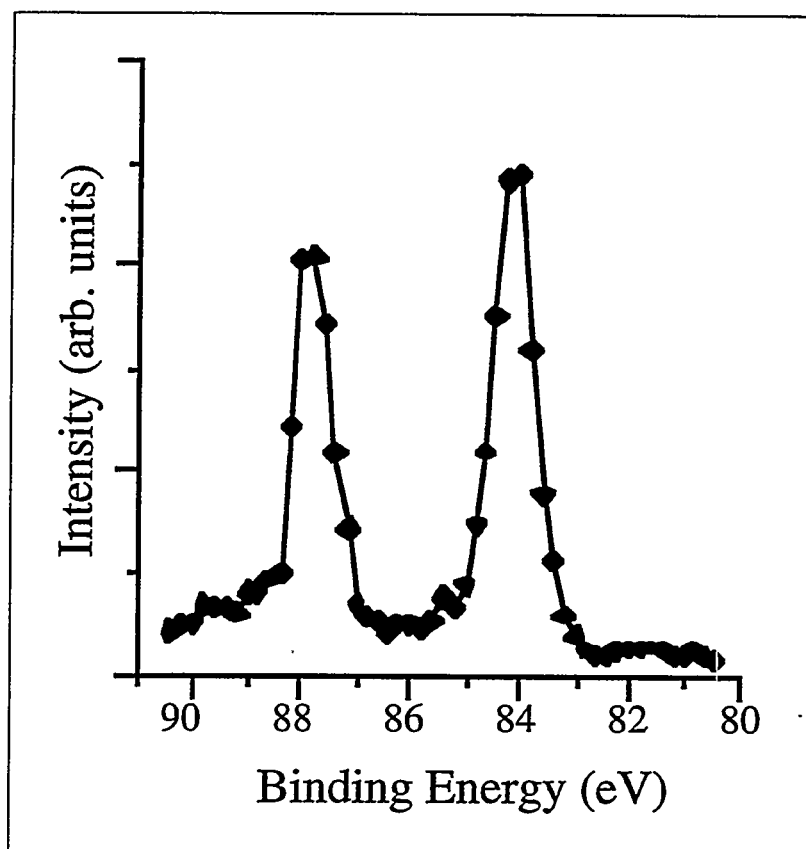


Figure 4. Photoemission binding energy spectrum of Au 4f core-levels from a small section of one of the grid bars (about 2 microns in diameter). This high quality spectrum was acquired with 3.3 seconds of time per point.

A photoelectron diffraction pattern for a particular electron kinetic energy is a map of the emission intensity into the hemispherical space above the sample substrate. An energy dependent photoelectron diffraction curve is the intensity oscillation for a specific emission direction, but with the kinetic energy varying. Both of these types of diffraction experiment, angle and energy dependent, contain structural information which results from the complicated scattering of the outgoing photoelectron wave from the neighboring atoms in the solid. With modern multiple scattering calculations, it is possible to model experimental data and achieve excellent agreement with theoretical models. This can result in bond-length measurements with an accuracy of 0.05 Angstroms or better.

One of the main project goals for the SpectroMicroscopy Facility was to optimize the experimental acquisition of complete photoelectron diffraction data sets, covering both the angle- and energy- dependence. Since the angular dependence can be expressed in terms of electron momentum parallel to the surface, and the energy dependence is in terms of perpendicular momentum, a data set comprehensively showing the diffraction pattern in K_x , K_y , and K_z space can be thought of as a photoelectron diffraction "volume." In order to see the value of such a large data set, we have constructed a diffraction volume for a Cu(100) surface. In Figure 5 we show the full angle-dependent patterns for a set of discrete photon energies. The patterns show the high sensitivity to the electron kinetic energy, since each has its own unique

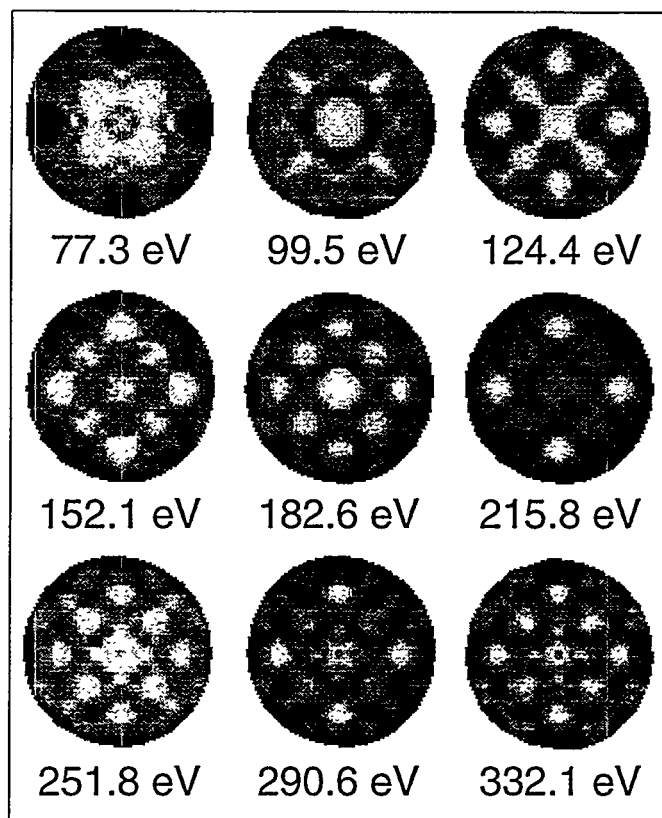


Figure 5. Set of complete photoelectron diffraction patterns from Cu(100) crystal at several kinetic energies.

signature. Common features include bright spots at directions corresponding to atomic rows, which arise from forward scattering along the atom chain.

These individual patterns are combined with others to create a diffraction "volume", shown in Figure 6. This image, which is the first of its kind in the field of photoelectron diffraction, displays in a rich way some of the fundamental physics of the diffraction process. The bright vertical columns of intensity are energy-dependent diffraction oscillations along a specific emission direction. The bright column on the left of Figure 6 is the (110) direction in Cu(100). The intensity oscillations are due to backscattering events in the atom chain. The horizontal planes are the angle-dependent patterns, which contain information about the forward scattering events.

Photoelectron diffraction is a powerful structural tool, but its full potential places high demands on the sensitivity of a photoemission experiment. In order to fully exploit the technique, high energy resolution is needed to extract small chemical shifts, and high intensity is needed to accumulate the large required data sets in a reasonable time. An example of a challenging problem is to use photoelectron diffraction with very high energy resolution to study the diffraction effects of surface atoms in the Si(111)-7x7 reconstruction.

The high resolution spectrum of the Si 2p atoms from Si(111)-[7x7] is shown in Figure 7. The overall envelope of the 2p core-level surrounds emission from as many as five different atomic

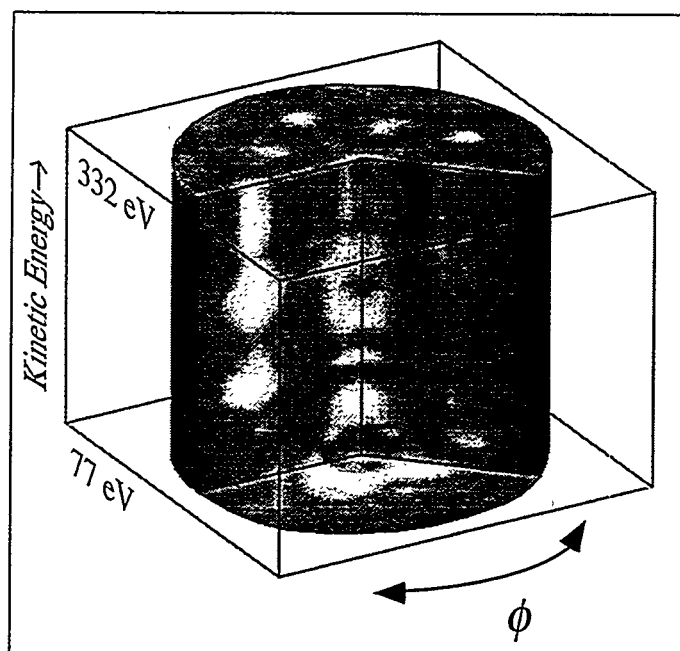


Figure 6. Photoelectron diffraction "volume" in momentum space constructed from a set of individual patterns. The energy dependent XPD appears as vertical columns, the horizontal planes are patterns for a specific energy.

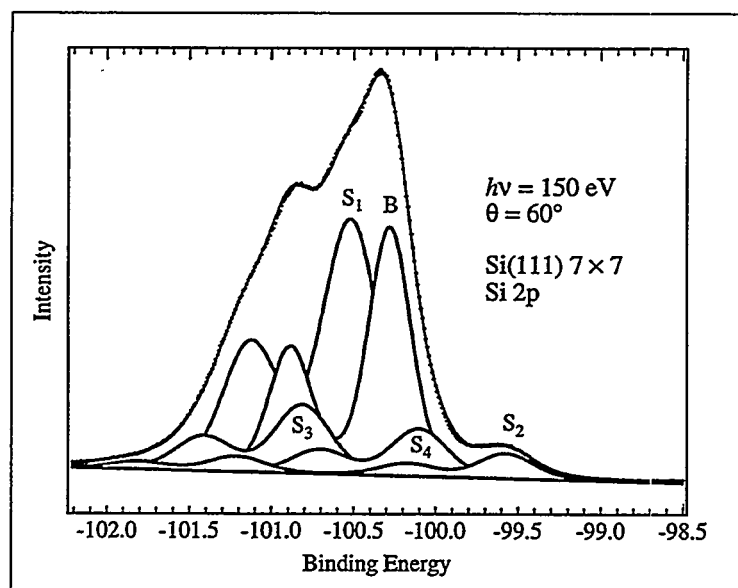


Figure 7. High resolution spectrum of Si 2p core-levels from a Si(111)-[7x7] surface, showing four surface atom peaks in addition to the bulk peak.

species, "bulk" atoms plus four types of surface atoms in the [7x7] reconstruction. Very clean, well ordered surfaces, and high energy resolution, are needed to see the small distortions (shoulders) in the 2p envelope that uniquely pin down the parameters of the multi-peak curve-required for this complex surface.

There remains a considerable debate over the origin of the various spectral components in core-level spectroscopy from this surface. We have used the high intensity and energy resolution of the SpectroMicroscopy Facility, along with a photoemission "robot" to scan the angles and energies, to collect a photoelectron diffraction "volume" for the [7x7] surface reconstruction. The results have superb statistics, and reveal the diffraction patterns of each of the components in the 2p line¹. Since each atom which has a distinct local structure will produce a unique diffraction pattern, we can identify the origin of the spectral features with certainty. The data analysis employs both the conventional approach of model calculations, and direct inversion using photoelectron holography techniques.

Related experiments which have been completed are a surface core-level shift photoelectron holography study of Si(100)-[2x1], and a surface-shift diffraction study of W(110). Ongoing experiments include studies of magnetic ultrathin film structures and molecular adsorbate geometry.

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Soft X-Ray Fluorescence Spectroscopy of Solids

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Emission spectroscopy in the soft x-ray range, defined here as energies between about 100 and 1000 eV, has long played a role in studies of the electronic properties of solids. In contrast to other techniques, such as photoemission spectroscopy, it produces density of states information that is selective for chemical species (local) and angular momentum state (partial), so that it provides a local-partial-density-of-states (LPDOS) that is particularly useful for the analysis of complex materials.

The major factor limiting the use of soft x-ray emission (SXE) has been the very low quantum yields that produce spectra of very low intensity, which have limited both the development of conventional x-ray sources and their utilization for electronic states spectroscopy. This limitation has been largely overcome by the development, in the last decade, of new emission spectrometers utilizing large, photon-counting area detectors, and by the availability of intense synchrotron sources that permit photon, rather than electron, excitation of spectra. With the advent of third generation synchrotron sources utilizing undulators and high-resolution, high-throughput monochromators, core hole generation with photons can be achieved at rates comparable to those available with electron excitation. These photon-generated emission spectra are commonly referred to as soft x-ray fluorescence (SXF) spectra.

Photon excitation has tremendous advantages compared with traditional e-beam excitation. It permits selective excitation of core levels, which is useful for sorting out overlapping spectra of complex solids. It eliminates Bremsstrahlung and dramatically reduces background radiation, permitting the study of weak spectra from low-concentration species, and from M and N spectra of heavier elements. It is less damaging to fragile samples. The long x-ray absorption lengths make it a bulk probe useful for the study of bulk impurities and buried structures. And perhaps most significantly, it permits excitation into selected states near an x-ray threshold, under conditions that couple the excitation and emission processes.

This talk features spectra that illustrate many of the points made above. $L_{2,3}$ spectra of CdS and ZnS are presented that illustrate the conventional use of SXE spectroscopy for the study of LPDOSs and bonding (see Figure 1). The spectra show features characteristic of both ionic and covalent bonding, as well as overlap features from the d-bands of Cd or Zn.

Two studies of threshold effects are presented.

- The K-spectra of graphite illustrate an inelastic scattering process in which the photoelectron and valence hole resulting from the scattering are correlated in k-space (see Figure 2). These are the first spectra clearly showing dispersion of spectral features in SXF spectra.

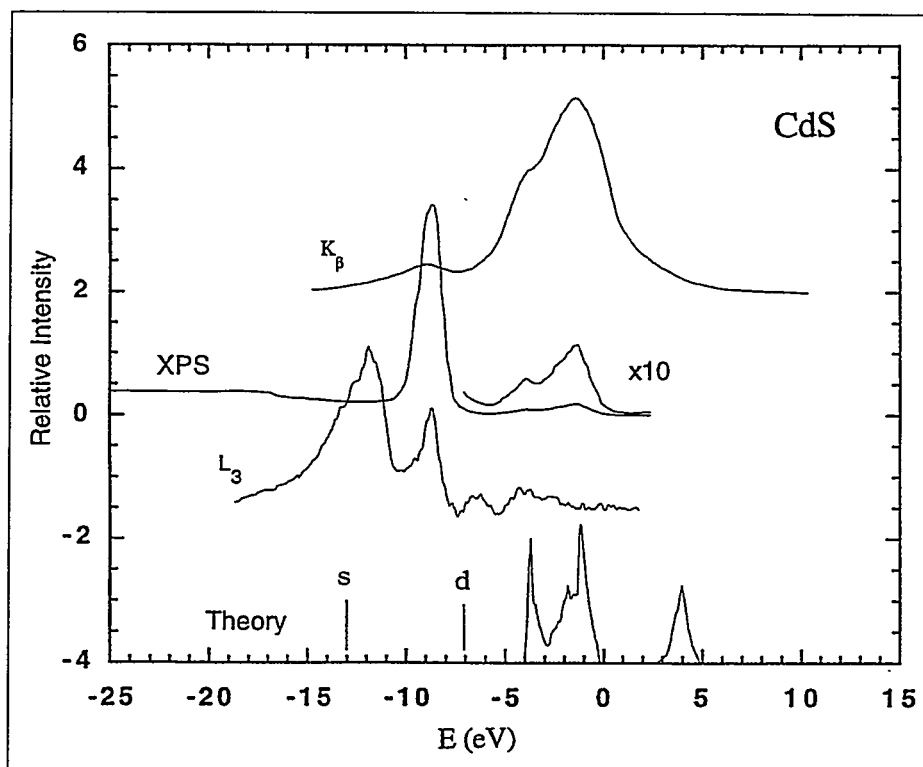


Figure 1. The L_3 spectrum of CdS shows structure derived from sulphur s -electrons (-10 to -15 eV), sp^3 bonding region (0 to -4 eV) and overlap peak from Cd d -states (-9 eV). Comparisons are made with the Cd K spectrum, photoemission results and theory.

- The boron K -spectra of hexagonal boron nitride (h-BN) exhibit a very strong resonant excitation level bound by about 1.8 eV below the conduction band edge. At energies well below (5 eV) the resonance, elastic scattering, and an inelastic spectrum produced by an electronic Raman process is visible (see Figure 3). On resonance, a resonant Raman process produces the same final state as a resonant "spectator" fluorescence process. The interference between these two processes greatly enhances the intensity of the SXF spectrum and produces modifications of the spectrum associated with the opening of the second channel. With still higher energy excitation into the delocalized conduction band, the spectrum resembles that excited by energetic electrons.

Finally, a study of the boron K -spectrum of a monolayer of BN deposited on either Si or amorphous carbon, and covered by a thick overlayer of carbon, demonstrates the ability of SXF studies to provide electronic states information for low concentrations of elements in buried structures (see Figure 4). These studies, like the threshold studies described above, were impractical or impossible prior to the development of sources such as the ALS.

These studies were carried out as a collaboration of researchers from many institutions, including the University of Tennessee, Tulane University, Lawrence Berkeley Laboratory, Lawrence Livermore National Laboratory, the National Institute of Standards and Technology, Oak Ridge National Laboratory, and the IBM Laboratories, Yorktown. The research was supported by the Division of Materials Research of the National Science Foundation, by Basic Energy Sciences of the U.S. Department of Energy, and by the participating institutions. The experiments described were carried out at the ALS.

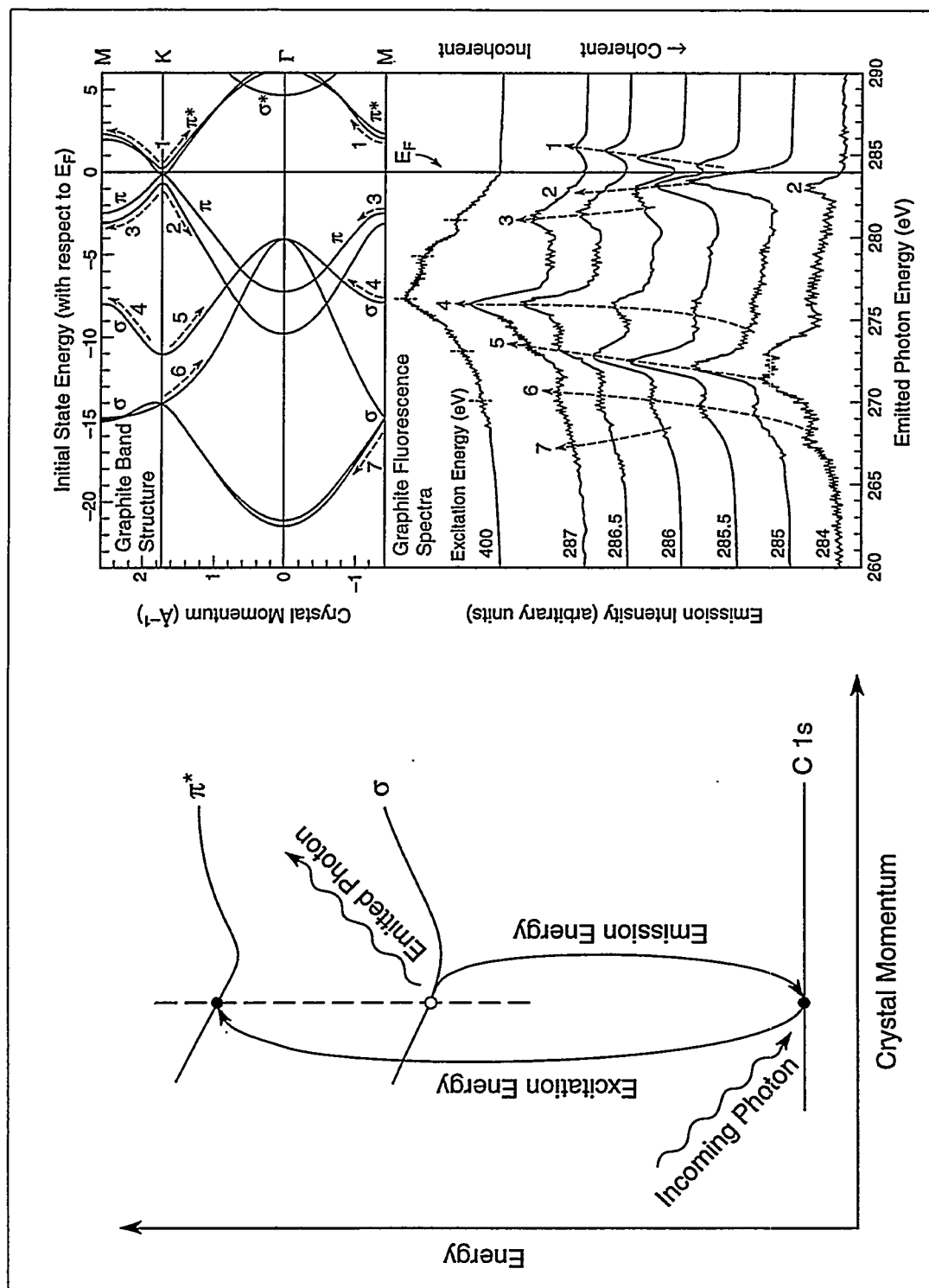


Figure 2. In graphite near the K threshold, an inelastic scattering process is observed in which the photoelectron and valence hole resulting from soft x-ray fluorescence are correlated in k -space. a) A diagram of the process. b) SXF C-K spectra excited near threshold showing dispersion and relation to the band structure of graphite.

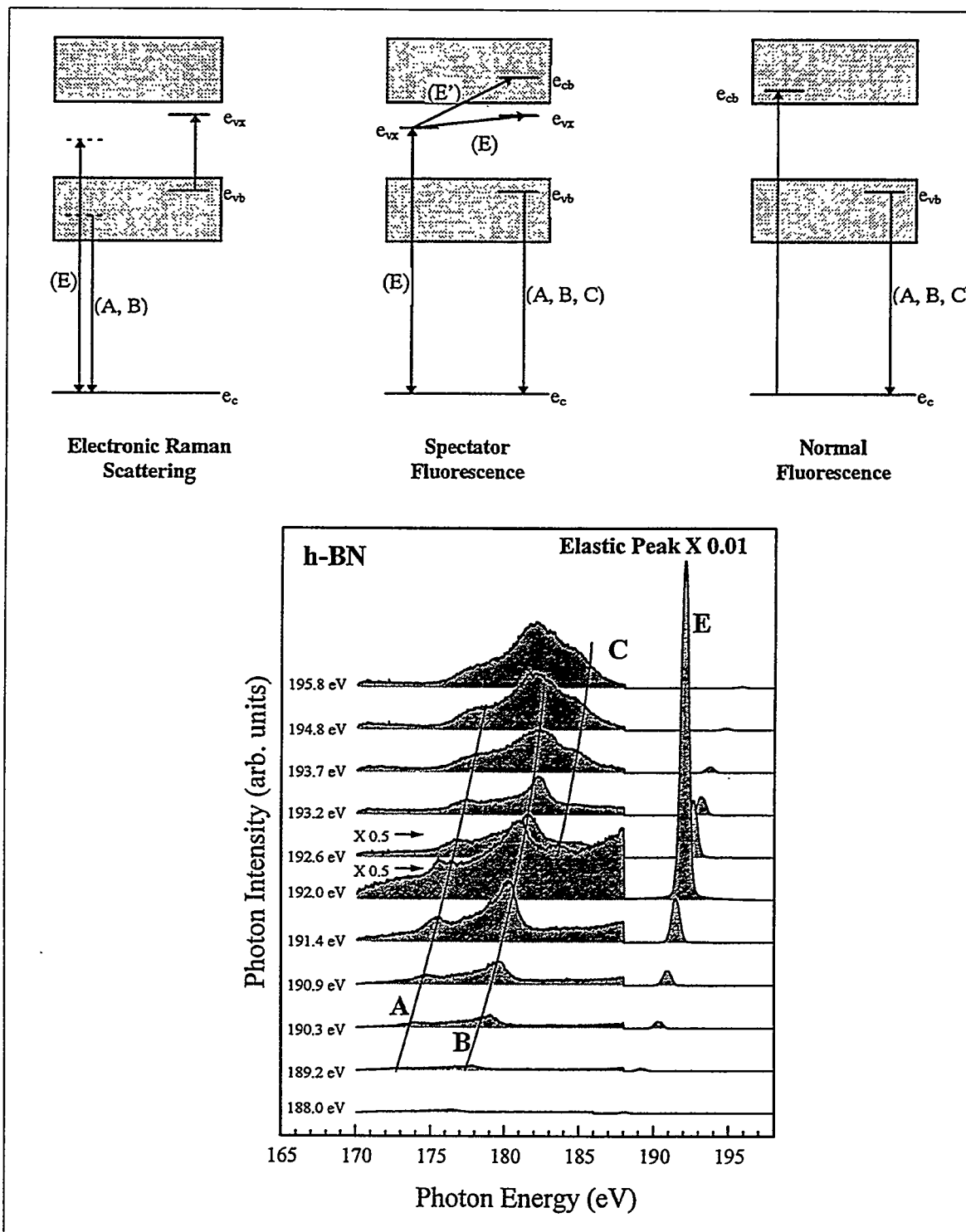


Figure 3. In h-BN, an inelastic electronic Raman process is observed well below the threshold resonance, and resonance scattering and normal fluorescence are observed with higher energy excitation. Top) The various processes contributing to the spectra are illustrated. Bottom) SXF B-K spectra observed excited near threshold showing structure due to Raman, spectator and normal fluorescence processes.

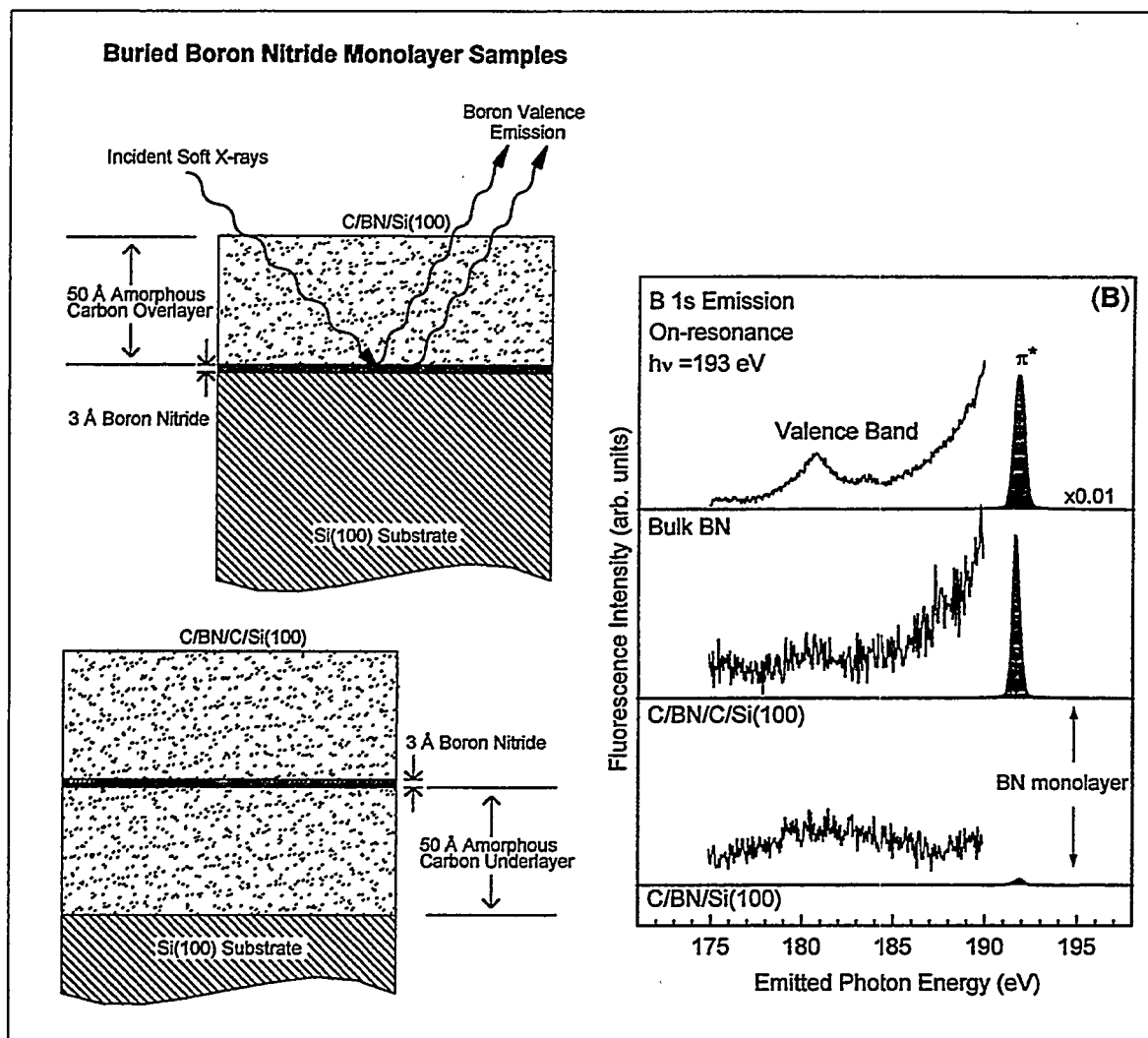


Figure 4. The electronic structure of buried monolayers of BN may be characterized from their B-K spectra. Left) Geometries of buried monolayer of BN. Right) SXF B-K spectra showing detectability of B and its retention of the prominent resonance peak associated with hexagonal symmetry when buried between layers of carbon.

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Soft X-Ray Fluorescence Spectroscopy of Molecules

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The potential of soft x-ray fluorescence spectroscopy (SXRF), previously presenting considerable obstacles for its exploitation due to low yields and low instrument efficiencies, has become accessible due to the high brightness performances of the third generation synchrotron radiation (SR) sources. The inherent selective properties of x-ray emission, together with the additional selectivity offered by the use of monochromatized synchrotron radiation are important elements of this progress. The polarization properties of SR and novel angular resolution capabilities of the recording instrument are other assets which present many opportunities for new research.

A number of SXRF experiments leading to new information about the electronic structure of matter have already been carried out at the Advanced Light Source. One such class of experiments is narrow bandpass excited soft x-ray emission spectra of molecular systems. The finding that resonant inelastic x-ray scattering (RIXS) is a significant process in threshold excited soft x-ray fluorescence spectroscopy of periodic solids¹ suggests a new method of studying also molecular symmetry and to address questions about dynamics of core state excitation².

In recent studies at beamline 7.0 at ALS, we have investigated resonantly excited soft x-ray emission spectra of a number of molecules in solid and in gas phase. One example is benzene in condensed form, shown in Figure 1. Soft x-ray fluorescence was excited at photon energies starting several eV below the x-ray absorption edge and going up to energies above the ionization threshold. Two of the spectra recorded are shown in the figure, namely at an excitation energy corresponding to the first x-ray absorption state (π_u) and at an energy above the ionization threshold, respectively. In the figure is also indicated the various molecular states of *gerade* or *ungerade* symmetry. One observes that the emission spectrum recorded at excitation to the π_u state lacks the lines corresponding to the *gerade* molecular orbitals, whereas they are present at higher energy excitation. The figure also shows inserted spectra calculated *ab initio* in a model assuming a one-step scattering process where there is no intermediate state of broken electronic symmetry. One observes a very good agreement between experimental and theoretical results, indicating that indeed there is no breaking of the symmetry in this core excitation process³.

The small disagreement that is present is ascribed to a vibronic coupling effect. This effect causes symmetry breaking upon core excitation if the lifetime of the process is long enough, as observed e.g. in the high energy excited x-ray emission spectrum of gaseous CO_2 ⁴. In further experiments at ALS we also studied gaseous O_2 where vibronic coupling is not present and we observe indeed that the "one-step" symmetry selection rule is strict. For gaseous CO_2

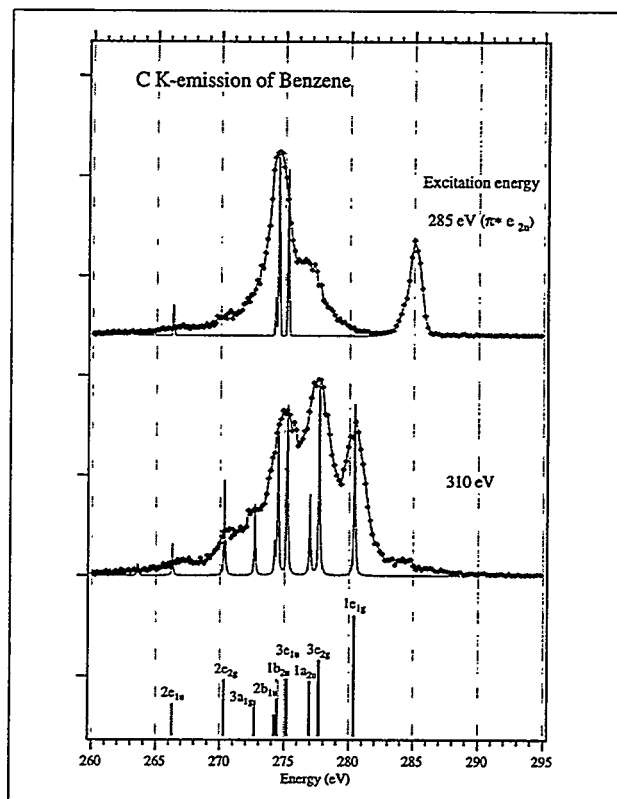


Figure 1

excited resonantly we observe a modulation of the intensity of forbidden lines, which suggests that we are dealing with a case where symmetry breaking is partially present (due to vibronic coupling), and this offers an opportunity to study the dynamics of the core excitation process⁵.

One way to view the symmetry selection properties of resonant inelastic scattering is that for symmetric molecules degenerate molecular orbitals give rise to interference of scattered x-rays such that symmetry selection rules come in operation. This leads to strong intensity modulations when the excitation energy is tuned to the various absorption states of defined symmetry. For the C_{60} molecule, which has I_h symmetry one observes that excitation to states of for instance *gerade* symmetry enhances the intensity of *gerade* states in the emission spectrum, i.e. there is a selection rule stating that parity must be unchanged in the process. Figure 2 shows a series of C K emission spectra of solid C_{60} recorded at various excitation energies chosen at the different peaks in the x-ray absorption spectrum. There is also some contribution of "forbidden" transitions appearing in the spectra. This is due to both tail excitation, i.e. the tail of the excitation excites nearby absorption states of different symmetry, and also to an incoherent contribution caused by vibronic coupling.

Previous studies of polarization properties of molecular x-ray emission showed strong polarization effects for low symmetry molecules, and an interpretation was made in terms of molecular alignment selection and molecular orbital symmetry properties⁶. This is also observed as an angular dependence in the RIXS spectra of symmetric molecules originating in

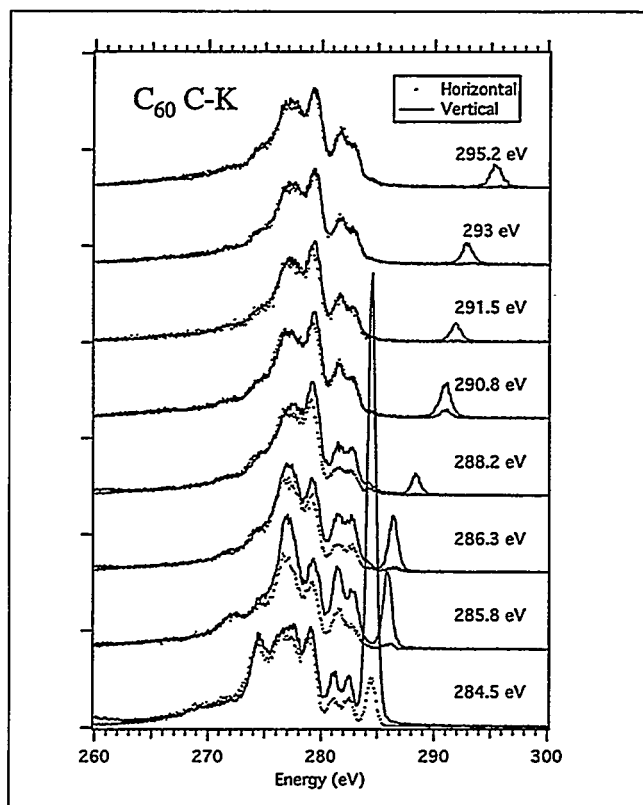


Figure 2

the defined polarization of the exciting radiation and the symmetry properties of the molecular orbitals involved. Figure 2 shows x-ray emission spectra recorded in the plane of polarization of the incoming synchrotron radiation (horizontally) and perpendicularly to this plane (vertically), respectively. Significant differences are observed reflecting the symmetry properties of the molecular states involved in the process. *Ab initio* HF calculations of the angular resolved RIXS spectra have been made showing good agreement with experiment⁷.

Excitation below threshold gives rise to a Raman type scattering, which was observed in the x-ray range by Sparks⁸, and discussed theoretically by Åberg and Tulkki⁹. In the sub-threshold excited emission of C₆₀ this kind of process is observed, and the molecular symmetry is reflected in the shape of the spectra, see Figure 3. Also broadenings are observed which can be described in terms of so-called Stokes doubling, offering a method to measure the lifetime width of the core state¹⁰.

In summary, the study of resonant and sub-threshold inelastic soft x-ray scattering of molecules provides a new tool to study molecular electronic structure, owing to the strong symmetry selection properties. Also, for low symmetry molecules, where core hole localization is defined, selective excitation allows one to obtain site-selective probing of electronic structure. For smaller systems, where *ab initio* calculations are feasible, detailed comparisons of advanced theoretical descriptions of molecular orbitals and chemical bonding can be made with experiment, free from irrelevant overlapping spectral features from multiple excitation

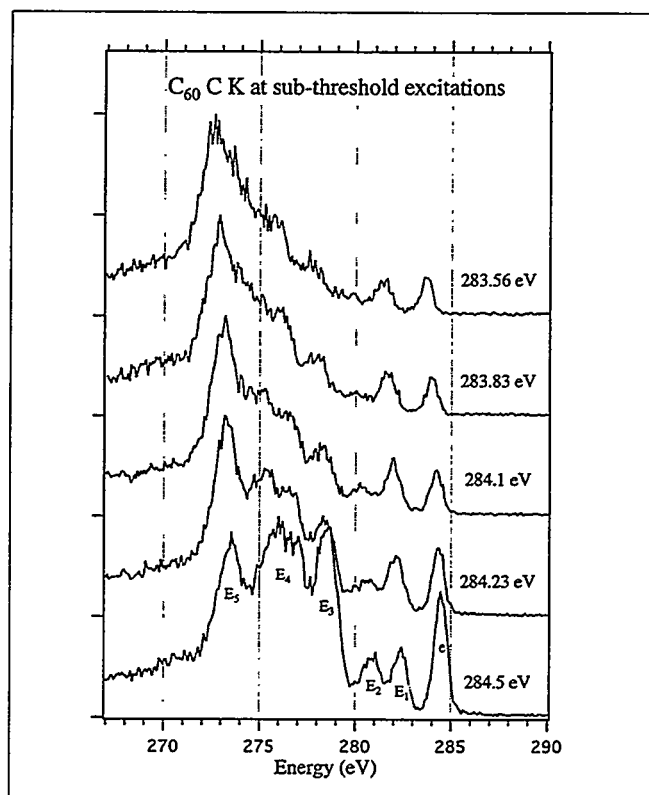


Figure 3

processes and near-lying states. It is a foreseen possibility that in the future these inherent local and symmetry selective properties of x-ray emission can be applied in the study of systems with high complexity where only approximative and simpler theoretical methods are applicable, such as for organic molecules.

ACKNOWLEDGMENT

This presentation at the ALS User's Meeting was based on significant contributions from a number of co-workers, in particular J.-H. Guo, P. Skytt, N. Wassdahl, H. Ågren, Y. Luo, P. Glans, K. Gunnelin, M. Magnusson, C.-J. Englund, T. Warwick, P. Heimann, E. Rotenberg, and J. Denlinger.

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Microstructures and Micromachining at the ALS

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LIGA¹ is a combination of deep x-ray lithography, electroplating, and injection molding processes which allow the fabrication of microstructures with lateral dimensions in the micron range, vertical dimensions several hundred microns high, and submicron tolerances. The techniques of deep x-ray lithography are used in the construction of micromotors, acceleration sensors, microgears, and linear comb drives. This type of lithography depends on the existence of synchrotron radiation sources which can provide a sufficient flux of highly parallel x rays in the 3-10 keV range, and the Advanced Light Source is an excellent source of radiation for this application.

A Participating Research Team (PRT) at the ALS to explore the science and technology of high aspect ratio microfabrication in a variety of materials using the LIGA process was formed on October 1, 1994. Initial participants in the PRT are the Center for X-Ray Optics at Lawrence Berkeley Laboratory, the Jet Propulsion Laboratory, Lawrence Livermore National Laboratory, and Sandia National Laboratory. The PRT will address extensions and research questions of the LIGA technique.

Micromachining covers a range of fabrication technologies. Micromachining processes are based to a large extent on standard semiconductor processes and most importantly on lithographic pattern transfer. The penetrating power of x rays in this region of the spectrum allows the fabrication of structures which have vertical dimensions on the order of millimeters and horizontal dimensions which can be as small as 1 micron. These are 3-D microstructures defined by 2-D lithographic patterns. The height-to-width aspect ratios in structures made using this technique can easily reach a factor of 100. This type of high-aspect-ratio design is relevant to the manufacturing of miniature components that can withstand high pressure and temperature, and can transfer useful forces or torques. The possibilities for inventive designs that save energy, reduce cost, add functionality, and improve reliability are limited only by the lack of basic research.

The LIGA technique originated at the Karlsruhe Nuclear Research Center in Germany. As originally implemented, highly parallel x rays in the 3-5 keV range from a bend magnet or wiggler are incident on a mask patterned with high Z absorbers (see Figure 1). The absorbers on the mask are thick enough to prevent the penetration of x rays. In the open areas of the mask, the radiation passes through and exposes a layer of PMMA (polymethylmethacrylate) resist. The radiation has a 1/e absorption length of over 100 μm at 3-5 keV. The resist is then developed and the resulting PMMA structure is replicated using electroplating techniques. The electroplating is either the final step in the processes or the electroplated part is used as a mold. The resulting mold can then be used to mass produce other parts out of a variety of

¹ LIGA is an acronym taken from the German words that describe the process: Lithographie (lithography) Galvanoplastie (electroforming) Abformung (plastic molding).

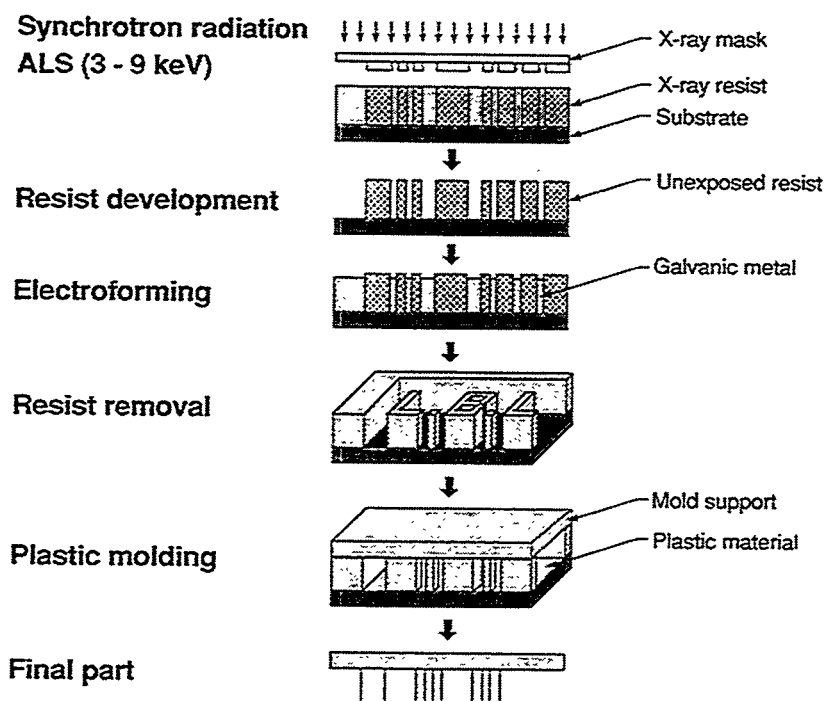


Figure 1. The LIGA process.

different materials: this is one of the great advantages of LIGA. We plan to build microstructures out of metal, plastics, and ceramics.

Initially the PRT will limit its ALS activities to branchlines 10.3.1 and 10.3.2 of the Center for X-Ray Optics (CXRO) Microprobe Beamline. There will also be some activities at Stanford Synchrotron Radiation Laboratory. These branchlines are currently part of the microprobe PRT, who has agreed to let our LIGA team use the 10.3.2 branchline. This branchline has recently been completed and will be used at least 70% of the time for LIGA related activities. This approach allows maximum leverage of existing facilities and minimum buy-in for the initial LIGA PRT members. The LIGA PRT expects to use 10.3.2 for a duration of two years. This period may be extended if it is mutually agreeable to the LIGA and microprobe PRTs.

The proven applications of micromachining technology have excited private industry and the scientific community. Figure 2 shows a critical part used in a thermal sensor for the automobile industry produced using deep-etch x-ray lithography at the ALS. In this process, highly parallel x rays in the 3-5 keV range from a bend magnet are incident on a mask patterned with absorbers. The absorbers on the mask are thick enough to prevent the penetration of x rays. In the open areas of the mask, the radiation passes through and exposes a layer of PMMA resist. The resist is then developed and the resulting 3-D PMMA structure shown in the picture remains. This demonstration project was performed jointly with the Jet Propulsion Laboratory (JPL), and the electroplating was done by Sandia National laboratories. JPL expects to use this technique to fabricate a precision collimator grid for x-ray astronomy.

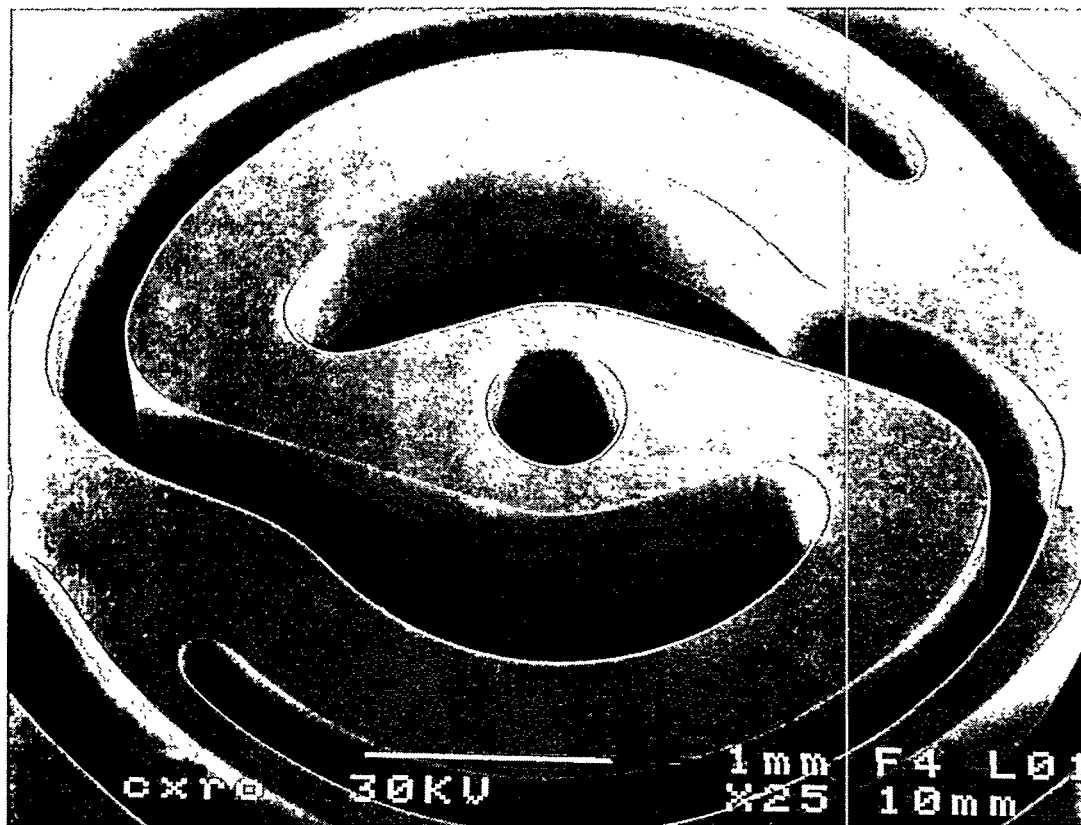


Figure 2. This microstructure, fabricated at the ALS by deep-etch x-ray lithography, could be used in the manufacture of thermal sensing units for automobile engines. The high precision of the lithography process enables complex structures like this to be manufactured with submicron accuracy.

High-Resolution Photoemission from Simple Atoms and Molecules

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Until recently, the potential of newly developed monochromators for synchrotron radiation sources has only been exploited for measurements in absorption. Because of the lower signal levels, the more demanding and more highly differentiating emission studies have rarely been attempted. Using beamline 9.0.1.2, we have been able to demonstrate that it is possible to perform energy- and angle-resolved electron emission studies at a photon resolution comparable to that previously exhibited only in absorption measurements.

The electron spectrometer which we used consists of three mutually perpendicular spherical sector plate electron energy analyzers mounted on a platform which is free to rotate about the direction of the incoming photon beam. A source cell surrounding the scattering region permits the detection and energy analysis of electrons having both low and high kinetic energies, in our experiments ranging from 0.4 eV for helium to 250 eV for methane. Measurements are made in the constant ionic state mode of analysis, in which the instrumental resolution is determined solely by the photon bandwidth.

The greater part of our analysis focused on the determination of the differential cross section for the decay of the two-electron excitations in helium. This simplest multi-electron atom continues to serve as the paradigm for electron correlation studies and the influence of the electron-electron interaction on the structure and dynamics of electron motion within atoms. We observed the "positive" ($2n^+$), ($3n^+$), ($4n^+$) and ($5n^+$) series through decays into the $\text{He}^+(N=1,2,3, \text{ and } 4)$ final ionic states. Working at a photon bandpass of 6.5 meV, we identified resonances up to $n=15$ in the ($2n^+$) series and the three lowest negative resonances. The linear polarization of the photon beam was measured to be 0.97, and we determined angular distribution parameters for all the decays which we observed into $\text{He}^+(N=2,3, \text{ and } 4)$.

On beamline 9.0.1.3 we made a series of measurements of the resonance Auger decay spectra of the carbon K-shell excitations in methane. In spite of its relative simplicity, this molecule continues to pose a challenge to theoretical analysis of electron excitation and decay involving the inner shells of molecules. Prominent among our observations was the role which the "participator" process plays in the decay. Using the constant ionic state technique, we observed angular distributions of the participator electrons throughout the resonance region.

This work was performed at the ALS by Sean Frigo, Alexander Menzel, Denise Caldwell, and Manfred Krause. It was supported in part by the National Science Foundation under grant PHY-9207634 and in part by the Division of Chemical Sciences, Office of Basic Energy Sciences, Department of Energy under contract DE-AC05-84OR21400 with Martin Marietta Energy Systems, Inc.

X-Ray Diffraction at the ALS

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The ALS is poised to begin x-ray diffraction. X-ray diffraction uncovers structure at the resolution adequate to resolve atom positions. The dynamic range of structure analysis is from about 1 Å (strictly approximately $\lambda/2$) to about 500 Å. This encompasses the range useful for initiating drug design strategies based on the structure of protein molecules as targets. Thus applications to contemporary industry abound. Several corporations including Agouron Pharmaceuticals, Vertex Pharmaceuticals, and Arris Pharmaceuticals have strong programs which utilize x-ray diffraction in this mode. High throughput is a premium in such evolution. Other corporations, including Genentech and Amgen, utilize x-ray diffraction in the design of new protein molecules as therapeutics. The ALS will provide a source sufficiently intense to address the issues of high throughput structure analysis.

In addition, high intensity monochromatic x-ray sources offer several other unique advantages for structure analysis by these essentially optical methods. These include the diminished radiation decay of protein crystals, and the enhanced resolution attainable at synchrotron light sources which can be absolutely critical to structure analysis in individual cases. Access to a tunable source of x rays also offers special advantages in the use of anomalous dispersion from elements in biological samples, especially sulfur. Metallo-proteins which include transition state elements, or heavy metal derivatives based on lanthanides then provide an alternative basis for solving the "phase problem" of protein crystallography. Phases can then be determined directly by observations made by tuning the x-ray beam in the region immediately adjacent to absorption edges. The change of phase of the scattered rays from these elements permits direct phase determination from the entire biological protein structure.

The use of diffraction for drug design has been validated with organic molecules developed by iterative structure determination based upon the structure of a protein molecule target. Thymidylate synthase is one such excellent drug target with importance for cancer therapy. Compounds designed by this route have now reached the clinical stage through the research at Agouron Pharmaceuticals and at UCSF, where we determined the initial structure of the protein target (Perry et al., *Science*).

A second area where structure determination depends upon synchrotron radiation concerns membrane proteins. Usually membrane proteins are crystallized by solubilization in detergent, followed by crystallization of the soluble detergent complexes. Such crystals often give limited resolution in conventional laboratory sources and so become tractable only in the intense, highly monochromatized beam attainable from a synchrotron source. Membrane proteins can often be crystallized; indeed, we have succeeded in our laboratory with acetylcholine receptors, bacteriorhodopsin, the CHIP-28 water conducting osmoregulatory chan-

nels, and colicin Ia. In this case, a large biological molecule was determined using mutagenesis, heavy metal labeling, and entirely by the use of synchrotron radiation without which structure analysis could not have been carried out. The resolution attainable in a laboratory source was 7 Å. At a synchrotron radiation source, the resolution was 3.3 Å. After cooling to liquid nitrogen temperature with an attendant increase in the lifetime and resolution of crystal in the beam, a resolution of 2.3 Å was attained. The structure was solved initially at 3 Å resolution and extended to 2.3Å (Ghosh et al., *Nature Structural Biology*).

Very few membrane protein structures have been determined at atomic resolution, though often crystals can be grown. Limiting resolution and radiation damage can be catastrophic limitations and while it is not clear that synchrotron radiation can always alleviate the problem, synchrotron radiation always provides the best resolution and lifetime available. Thus the use of the ALS can make intractable problems tractable and can lead to the highest resolution analysis from difficult structural problems. As such it will become the standard for diffraction analysis of proteins and membrane proteins.

The level of university and corporate interest in a reliable, high intensity, tunable x-ray source is potentially very high. The characteristics most valuable to the community will be monochromatic radiation with wavelengths between about 1 Å and 1.8 Å. High intensity radiation emitted using a wiggler magnet will be critical. Tunability within the accessible wavelength range will access a wider area of structure determination dependent upon anomalous dispersion from sulfur and other transition state and lanthanide elements.

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Utilizing Synchrotron Radiation in Advanced Materials Industries

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Two factors make it now feasible and even likely that synchrotron radiation will be used in the near future for "real world" materials characterization (surfaces, interfaces, thin films) in the high-technology industries (semiconductor manufacturing, including packaging, magnetic and optical storage; and the industries feeding off these, e.g. process equipment manufacturers). These factors are (i) the availability of high brightness sources, and (ii) pressure to make DOE facilities impact U.S. technological competitiveness. Without both of these, the outlook would be bleak for significant involvement in the real world issues in advanced materials industries, which often involve processing, reliability, or failure analysis problems during development and manufacturing cycles. These are not of a "research program" nature, as understood by academia, even though they are often at the cutting edge of materials characterization and analysis.

The materials problems facing the process engineer, the materials specialist, and the analyst in the above-mentioned industries often revolve around the apparently simple questions of what is there, how much is there, and how is it distributed—but on ever finer scales of sensitivity, accuracy, spatial and depth resolution, etc. A wide range of material types, from organic molecules through to metals, are involved, and in material forms ranging from particles through to multi-layer thin film structures and finely patterned parts.

In-house characterization capabilities may sometimes be inadequate, even though these industries are usually well-equipped with multi-million dollar commercial techniques. Industries then resort to external analytical houses on the basis of cost, availability, turn-around time, long-term reliability, and confidentiality. A synchrotron facility will be viewed in exactly the same manner:

"What added-value do I get, for what cost, compared to attempting in-house solutions?"

Considering only one technique, XPS (x-ray photoelectron spectroscopy), for the sake of brevity, the technical advantages of using a high-brightness synchrotron source compared to a laboratory-based source, are, by now, reasonably well appreciated by materials laboratories in industry. They are:

- (1) Vastly increased count-rate for a given set of spatial/spectral resolution conditions. This translates into speed and cost effectiveness and allows the exploitation of (2) - (5).
- (2) Spectromicroscopy possibilities down to well below 1 μm . Lab-based instruments have been stuck at around a value of 100 μm for many years, though very recently this has been dropping.
- (3) Improved spectral (energy) resolution and therefore chemical speciation information.

(4) Variable photon energy leading both to the possibility of non-destructive near-surface depth profiling and to exploitation of resonance effects.

(5) Magnetic information utilizing the polarization aspects of synchrotron radiation.

The practical aspects of making use of these technical advantages are all in the "value-added/cost" bottom line. The hopelessness of the situation, until very recently, can be best exemplified by the not too uncommon case where to actually have a chance of utilizing the synchrotron radiation, during an all-too-infrequent "run," required bringing along every conceivable item and spare part (or risking physical violence over "sharing" sparse and usually inadequate "user" equipment). Also, only a novice would be so naive as to expect that a staff scientist, whose title sounds as though he were responsible for beamlines, endstations, or whatever, would actually be available to assist while light was available (and therefore available also for his experiments!). In short, synchrotron facilities are not operated (so far) like commercial analytical laboratories. The internal infra-structure necessary to make efficient use of the multi 100-million-dollar facilities has been woefully inadequate for anything other than basic research groups committed to learning and doing everything themselves. Their access, however, is essentially free. Real-world industries expect to pay (handsomely), but can only come if the infra-structure is right, which means addressing questions of continuous running, dedicated endstations (or even beamlines), dedicated staff, and the complementary availability of the normal materials laboratory equipment (such as optical microscopes, SEMs, sample preparation facilities) one would expect to find "at home."

It is easy to list a number of past crises and on-going (i.e., almost quality-control like) materials issues where synchrotron-based XPS would have been the choice if it had been feasible from a practical standpoint. These include numerous aspects concerning the chemical nature, thickness, spatial distribution, and surface degradation of magnetic disk lubrication material—a whole industry in itself for the commercial analytical houses—circuit board and magnetic recording head issues involving adhesion, corrosion, and contamination; and RIE and other etching or deposition processes involved in Si/SiO₂ and magnetic recording head lithographies.

Currently the lab-based techniques routinely used for characterizing small particles on Si wafers and for detecting trace amounts of metals at wafer surfaces (two of the most critical yield detractors) are becoming inadequate as Si wafer design rules shrink and, therefore, specifications tighten. There are opportunities for synchrotron radiation-based methods to fill the gaps (e.g., micro-XANES, micro-XPS, TXRF).

A survey I undertook in industrial materials laboratories and in commercial analytical houses consistently suggested there is a wide variety of work that either does not get done, because of lack of adequate spatial resolution coupled with chemical speciation information (i.e. suitable for micro-XANES or micro-XPS), or gets shunted to electron beam techniques which have the required spatial resolution, but have other severe drawbacks. When asked whether they would consider synchrotron facilities to solve some of these problems, reliability (i.e. continuous availability) and turn-around time were always the issues. One analyst summarized aptly with the following two statements: "A turn-around time of a few days is acceptable" and "Product cycle times in the high-tech materials industries are shorter than DOE proposal times." It is these aspects, not just technical issues, that need to be faced if DOE facilities, such as synchrotrons, are to impact U.S. high-technology manufacturing competitiveness.

Polymer Microscopy: About Balls, Rocks and Other "Stuff"

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During the last few years x-ray microscopy has made significant contributions to the characterization of polymeric materials. Much of these contributions are based on the chemical and orientational sensitivity of XANES¹ and linear dichroism² microscopy, and the associated low radiation damage with the use of x-rays. In several of these materials, certain morphological features could be visualized with other microscopies, but due to a lack of understanding of their composition, these features have been described as balls, rocks, and lumps - a characterization based purely on morphological "impressions". The submicron analytical capabilities of x-ray microscopy could be successfully utilized to provide a chemical understanding of these morphological features. One of these mystery morphological features are so-called "balls" in various polyurethane foams (Fig. 1). They are roundish looking precipitates which affect the material properties, and were thought to be polyurea in nature. Carbon XANES microscopy showed that these precipitates are high in aromatic content and have the spectral signature reminiscent of urethane rather than urea molecular analogues (Fig. 2). We are in the process of acquiring confirming spectra at the oxygen K-edge.

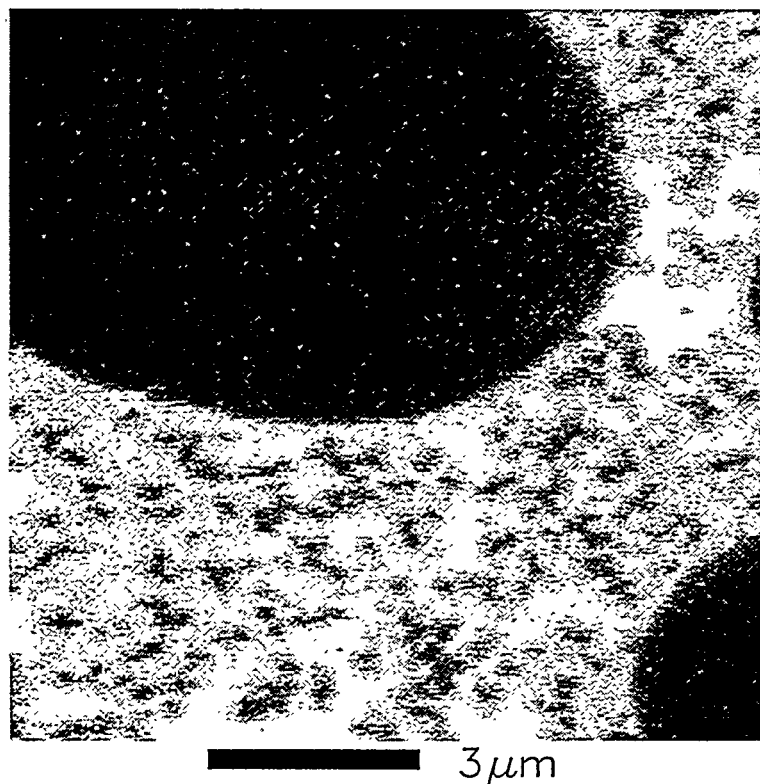


Fig. 1: Micrograph of 200 nm thick section of a MDI-based polyurethane foam acquired at a photon energy of 285.5 eV where aromatic groups are highly absorbing. The dark precipitates, present in two different sizes have thus a higher aromatic content than the matrix. We are in the process to determine the chemical difference between the large and small precipitates. (Sample courtesy of E. Rightor, Dow Chemical)

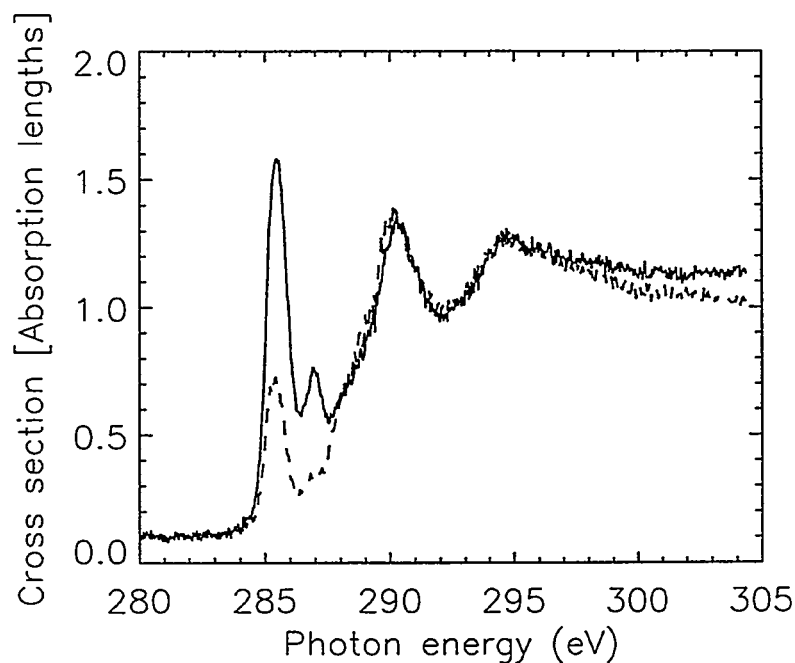


Fig. 2: Spectra from a big precipitate (solid) and the matrix (dash) of the MDI based polyurethane foam show in Fig. 1.

Another example of the contributions of x-ray microscopy is the characterization of the phase morphology of a liquid crystalline polyester based on several aromatic monomers. Electron microscopy and differential scanning calorimetry indicated the existence of phase separation in materials based on this polyester. The features observed in the electron micrographs have typically been referred to as "rocks", and the various characterization techniques utilized could not determine whether the phases are chemically distinct - and if so how many phases there are - or whether the "observed" phase separation is solely due to differences in crystallinity. We therefore employed XANES microscopy to investigate and compare two materials, a melt-screened and un-screened sample, based on this polymer. We unexpectedly found that the un-screened sample had in fact four chemically distinct phases: a discontinuous, highly aromatic phase with domains smaller than 100 nm, a discontinuous phase high in oxygen content with features on the order of 500 nm, as well as two continuous phases with dimensions of a few microns. None of the phases observed in the un-screened material was detected in the screened material. This illuminates and illustrates the structural dependence of the materials on the processing route. As an example of some of the morphology observed in the un-screened materials we show micrographs acquired at 285.0 eV and 286.8 eV in Fig. 3A and B, respectively. The former emphasizes aromatic content and the small, discontinuous phase as well as the two continuous phases with dimensions of a few microns are clearly discernible. In Fig. 3B, the contrast is reversed between the continuous phases, while there is virtually no contrast between the small features. The dark round features are the oxygen rich domains. This relative contrast change demonstrates the different chemical character of these domains (We assume that there are no orientational differences² for which we did not test explicitly).

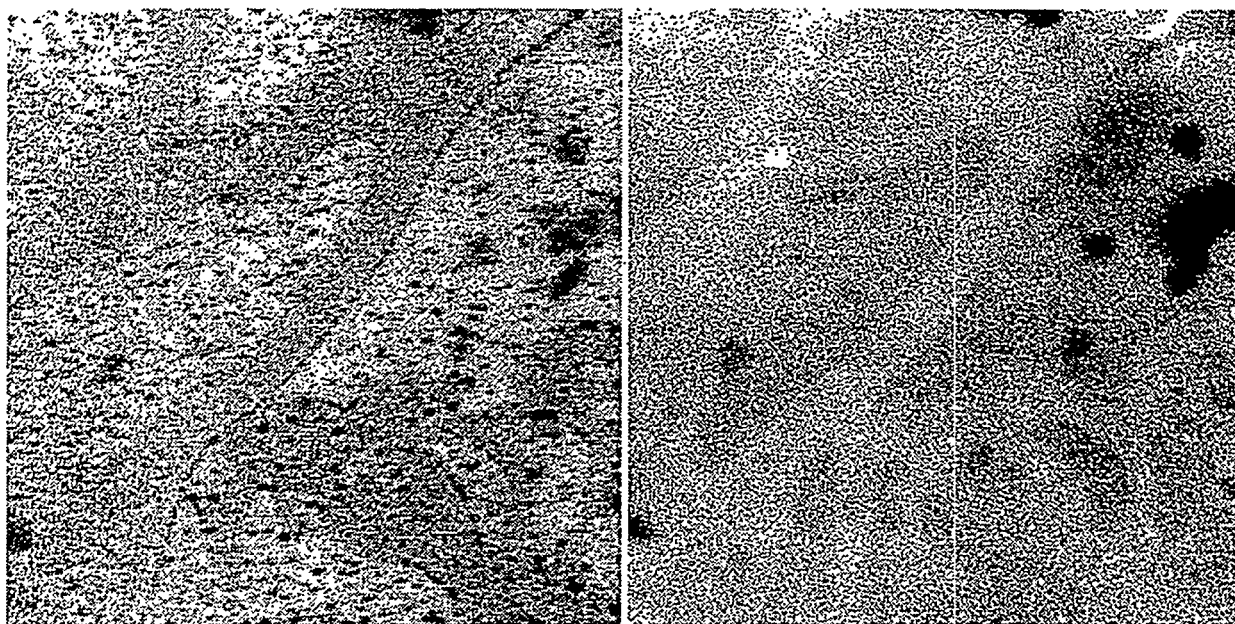


Fig. 3A

Fig. 3B

Fig. 3. Micrographs of the same region of a thin section of an aromatic liquid crystalline polyester imaged at (A) 285.0 and (B) 286.8 eV. Field of view is $9 \times 9 \mu\text{m}$. (Sample courtesy of B. Wood and I. Plotzker, DuPont).

Besides the chemical sensitivity, XANES is polarization dependent. The resulting x-ray linear dichroism can be used to image the orientation of specific bonds in (partially) ordered samples. Ade and Hsiao demonstrated this effect by investigating the structure of thin sections of poly(p-phenylene terephthalamide) (PPTA) fibers (Kevlar), particularly Kevlar 149. Subsequently, three types of Kevlar fibers were investigated more completely: Kevlar 29, 49 and 149, which differ from each other in the degree of crystallinity (Kevlar 149 is the most and Kevlar 29 the least crystalline) and the crystalline orientation along the fiber axis. The transverse structure of the fibers has been significantly less well characterized so far, and prior to x-ray linear dichroism microscopy there was no technique that could quantify the degree of orientation. X-ray micrographs of thin sectioned fibers cut at 45° exhibit a "butterfly" pattern when imaged at a photon energy corresponding to an absorption resonance. This pattern is strongest for Kevlar 149, intermediate for Kevlar 49, and weakest for Kevlar 29. Micrographs of Kevlar 149, 49, and 29 are shown in Fig. 4 as an example, although the precise relative contrast has not been preserved during image processing. The contrast of this pattern can be reversed as the sample is rotated by 90° about the optical axis². Our observations are consistent with radially symmetric structures and the average aromatic ring plane pointing radially outwards. The observed radial ordering is quite small for Kevlar 29, and prior to this study it has been questioned whether Kevlar 29 has indeed any radial symmetry at all. By analyzing pairs of images obtained at 285.3 eV and 283.0 eV respectively, we will be able to not only qualitatively, but quantitatively determine the degree of orientational ordering of the aromatic groups in the various fiber grades.

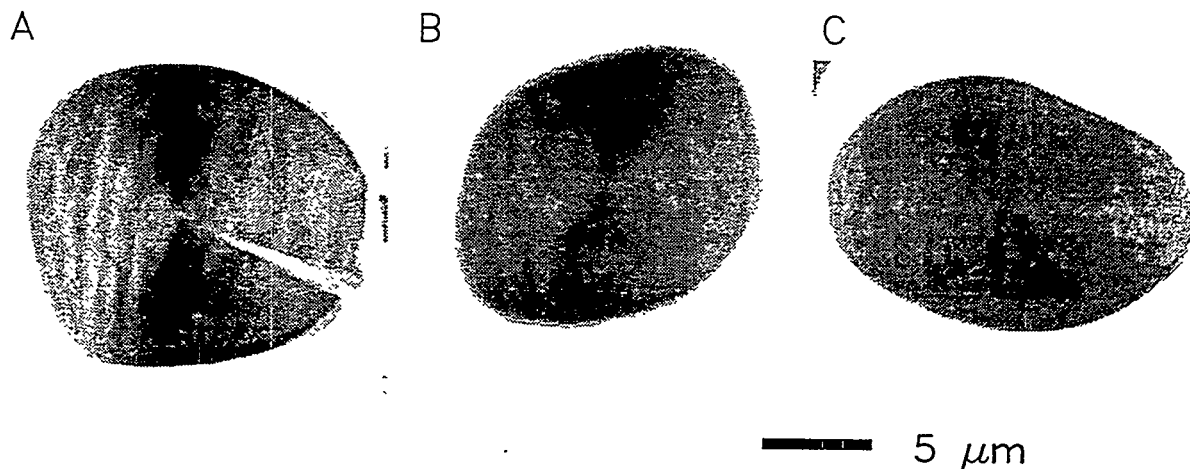


Fig. 4: Micrographs of 200 nm thick sections (cut at 45° relative to fiber axis) of (A) Kevlar 149, (B) Kevlar 49, and (C) Kevlar 29, imaged at a photon energy of 285.5 eV. This energy selects the aromatic groups of the fiber polymer and the butterfly patterns observed in all three grades of Kevlar fibers are due to the radial symmetry and orientational order of the fiber. (Sample courtesy of B. Hsiao and S. Subramoney, DuPont).

We also acquired NEXAFS spectra in the more transparent (horizontal) and more absorbing (vertical) directions of the fibers as exhibited in the micrographs, which will allow an independent measure of the degree of radial order. The most prominent peaks are associated with aromatic (285.3, 286.5 eV) and carbonyl (288.2 eV) groups, and their intensities depend on the orientation of the excited bond relative to the linearly polarized x-ray beam. The difference between the grades is illustrated by the spectra in Fig. 5. The relative change in peak heights between the horizontal and vertical locations and between the two grades of fibers is most apparent for the carbonyl π^* resonance (288.1 eV) and the sigma resonances (296 and 304 eV). By extracting these peak heights and their changes within the fibers through a spectral fitting procedure, we will obtain quantitative information for the degree of radial orientation in these fibers. A rough estimate indicates that the degree of radial orientation for carbonyl is about 0.3 for Kevlar 149, and about 0.15 for Kevlar 49, where perfect radial orientation corresponds to 1.0. The observation that the intensity changes are smaller for the aromatic groups than for the carbonyl groups can be explained by a torsion of the two inequivalent aromatic groups of the polymer with respect to each other.

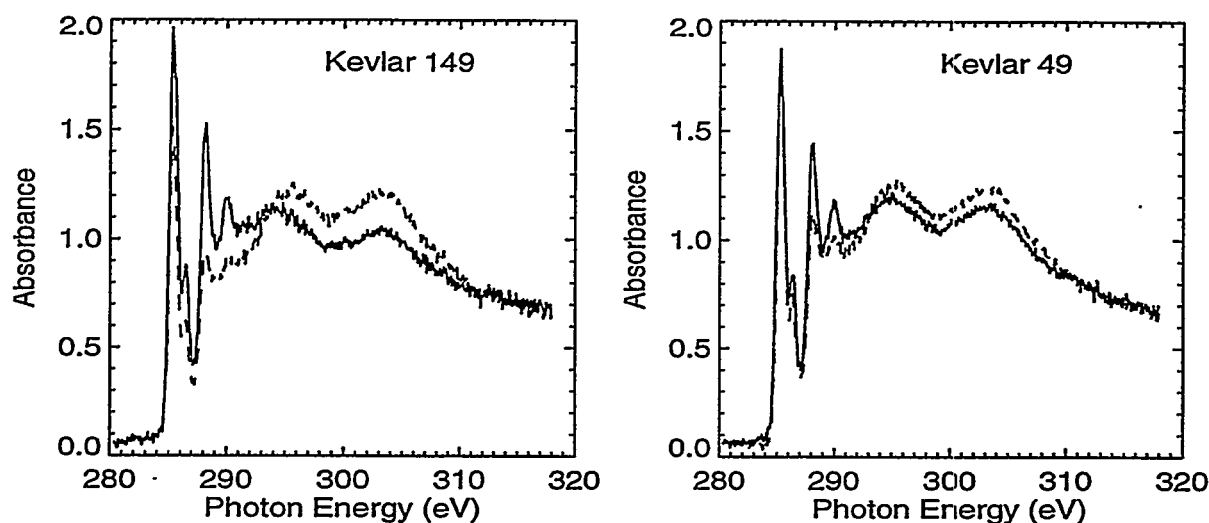


Fig 5: NEXAFS spectra of $0.1 \mu\text{m}^2$ area of $0.1 \mu\text{m}$ thick sections of Kevlar 149 and Kevlar 49 fibers obtained from the relative transparent areas (dash) and relative absorbing areas (solid) of an image acquired at 285.5 eV (see Fig. 4). The prominent spectral features at 285.3 eV, 286.5 eV, and 288.2 eV are π^* resonances associated with unsaturated bonding in the aromatic and carbonyl groups, respectively. The energies are characteristic and provide for bond selective imaging, whereas the intensity depends on polarization and the geometrical orientation of the bonds. The broader features at about 295 and 303 eV are σ^* resonances. The differences in the peak intensities is an indication of the degree of radial order in the fiber. Note, that the peak intensity differences are much smaller in Kevlar 49 than in Kevlar 149. By fitting the intensities of these spectral differences, quantitative information of the degree of order can be obtained.

All of the work reported and shown has been acquired with the NSLS X1-STXM. I thank J. Kirz, C. Jacobsen, S. Williams, X. Zhang, S. Wirick, H. Rarback, C. Buckley, and M. Rivers, as well as the many others who have been instrumental, for the development and maintenance of this instrument. J. Kirz, C. Jacobsen, S. Williams, X. Zhang, and M. Rivers have furthermore significantly participated in implementing the XANES imaging mode. The zone plates utilized in the X1-STXM have been provided through an IBM-LBL collaboration between E. Anderson, D. Attwood, and D. Kern. The author would also like to thank B. Hsiao, B. Wood, I. Plotzker and S. Subramoney from DuPont, G. Mitchell and E. Rightor from Dow Chemical, for providing samples and assisting in numerous ways, and S. Cameron, C. Costello, E. Berluche, J. Chudzinsky, and S. Behal from Exxon Research for their participation in the initial XANES imaging experiments. This work was performed at the NSLS, which is supported by the Department of Energy, Office of Basic Energy Sciences. I also gratefully acknowledge support from the NSF, grant DMR-9458060, Dow Chemical, and EXXON, and a DuPont Young Professor Grant.

- 1 "X-ray Linear Dichroism Microscopy", H. Ade and B. Hsiao, *Science* **262**, 1427-1429 (1993).
- 2 "Chemical contrast in x-ray microscopy and spatially resolved XANES spectroscopy of organic specimens", H. Ade, X. Zhang, S. Cameron, C. Costello, J. Kirz, and S. Williams, *Science* **258**, 972-975 (1992).

Infrared Research and Applications

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Synchrotron radiation is intrinsically very bright in the infrared spectral region, with a brightness advantage of 2 to 3 orders of magnitude over conventional black body sources. This puts it midway towards lasers which are around 6-7 orders of magnitude brighter than conventional sources. Synchrotron radiation does not compete with lasers, but rather is complementary. It is a white source and in this spectral range interferometers can readily be used, offering considerable advantages for spectroscopy.

While all synchrotron sources emit copious quantities of infrared radiation, it is not easy to extract, especially from rings of larger radii. At the NSLS, Brookhaven we have built a special high-aperture port which allows the extraction of light into an angle of 90 by 90 milliradians. This allows us to utilize radiation into the sub-millimeter wavelength region without severe diffraction problems. At the ALS special high-aperture ports were built into the ring vacuum chambers during the initial construction, and they will allow light out to 20-micrometer wavelengths to be utilized.

Infrared synchrotron radiation has seen extensive use primarily at the NSLS for several major programs, all of them involving considerable industrial collaboration. In surface science, bonding of adsorbates to surfaces has been studied by observations of the adsorbate-substrate vibrations. Studies of the dynamics in this region have led to new models and understanding of the role of the substrate in chemisorption and physisorption which is critical in catalysis, corrosion and epitaxial growth. The work has been done in conjunction with scientists from Bell Labs and Exxon Corporation. In studies of superconductors, the low frequency dynamics of small crystals of new materials provided insight into mechanisms of conductivity. This work has involved University groups from SUNY, Stony Brook, U. Florida and Lausanne in Switzerland, and industrial partners from the Grumman Corporation and Bell Labs. For hydrogen, which should be a good metal considering its place in the periodic table, high pressure studies at pressures approaching the point at which metallic behavior is speculated to exist, have revealed evidence of large behavioral changes which will provide theorists with valuable information on this fundamental material. Of considerable interest is the understanding of planetary interiors. The work has been done by the Carnegie Geophysical Institution in Washington, DC.

Recently, however, a new program has been instituted at Brookhaven in conjunction with Northrop-Grumman and Spectra-Tech Inc. involving infrared microspectroscopy. A conventional IR μ s microspectrometer modified for use with synchrotron radiation has demonstrated diffraction-limited spatial resolution in the micron regime, and superior spectral quality than that obtained with a conventional source. The instrument has a built-in Michelson interferom-

eter and at each point on the sample quickly generates a spectrum either in reflectance or transmittance covering the so-called chemical fingerprint regime, from 600–4000 cm^{-1} . Samples can be scanned to produce a map. Even with the preliminary set-up at Brookhaven, the spatial resolution is roughly an order of magnitude better in each dimension than that obtained with the conventional source. With a 3-micron pinhole, we have demonstrated sub-diffraction limited resolution. The microscope has been used by Grumman Corporation, Polaroid Corporation, General Electric, Hewlett-Packard, and the FBI for forensic studies. Samples have included photographic film, thin films on surfaces, and several small scale electronic devices, including some of the new wide band-gap materials destined to provide blue lasers for example. Defects, impurities and non-uniformities can readily be detected.

Plans are now being developed to establish an infrared beamline on one of the IR ports at the ALS. The beamline would be equipped with a Michelson interferometer and a microscope, thus providing unique analytical capability to the Bay Area. Several industries already use microscopes of lower spatial resolution and are interested in the considerably improved capability. They include Intel, Hewlett-Packard and IBM.

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ALS User Program

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The talks given yesterday and this morning have provided an excellent overview of the type and variety of science being done at the ALS, and a look at the present and future opportunities the facility offers. Now excited about the research possibilities, you may well ask, How do I become a user of the ALS? My presentation will address that subject as well as answer some other commonly asked questions such as: Where do I park? and Where can I stay? Much of what I will cover today, as well as information to help you prepare for your visit to the ALS and to assist you during your stay, is contained in our new *ALS Users' Handbook*, which I encourage anyone interested in doing research here to read.

Let me begin with the bottom line for the ALS user program: Happy Users. The photographs in Figures 1 and 2 show a few of our recent users at work on the ALS experiment floor, and they represent the customers of the ALS. The goal of our user program is to make access to the ALS as convenient as possible, and to ensure that our users experience an efficient and safe work environment when they arrive. We encourage feedback from our users on their experiences at the ALS, so that we can continue to tailor our program and facilities to meet their needs.

AVAILABILITY TO RESEARCHERS

There are three ways to become an ALS user:

- Join or collaborate with an existing participating research team (PRT)
- Submit a proposal as an independent investigator
- Form your own PRT.

If you already know the beamline you are interested in and some of the people associated with that beamline, the best place to start is by making direct contact with them, or contact me and I will be happy to make the necessary introductions. You can also submit a proposal as an independent investigator. Independent investigators' proposals for beam use are reviewed twice a year. The first deadline for submissions is December 1994; the next is June 1995. The third option, forming your own PRT, typically requires having enough resources to build and pay for a beamline. In all cases, please don't hesitate to contact us if you are interested in doing research here whether you are familiar with the methods described above or not; we will try to get you on the experiment floor within a reasonable period if possible.

HOUSING AND PARKING

The Laboratory and the ALS recognize the importance of helping users with the logistics of finding reasonable housing during their stay and providing access to parking on site. For short-term visitors, local Bed and Breakfast accommodations seem to be working out well, and we have a list of these with approximate cost, phone number, etc., which we can provide upon request. Most of the researchers or research teams anticipating a long-term stay have chosen

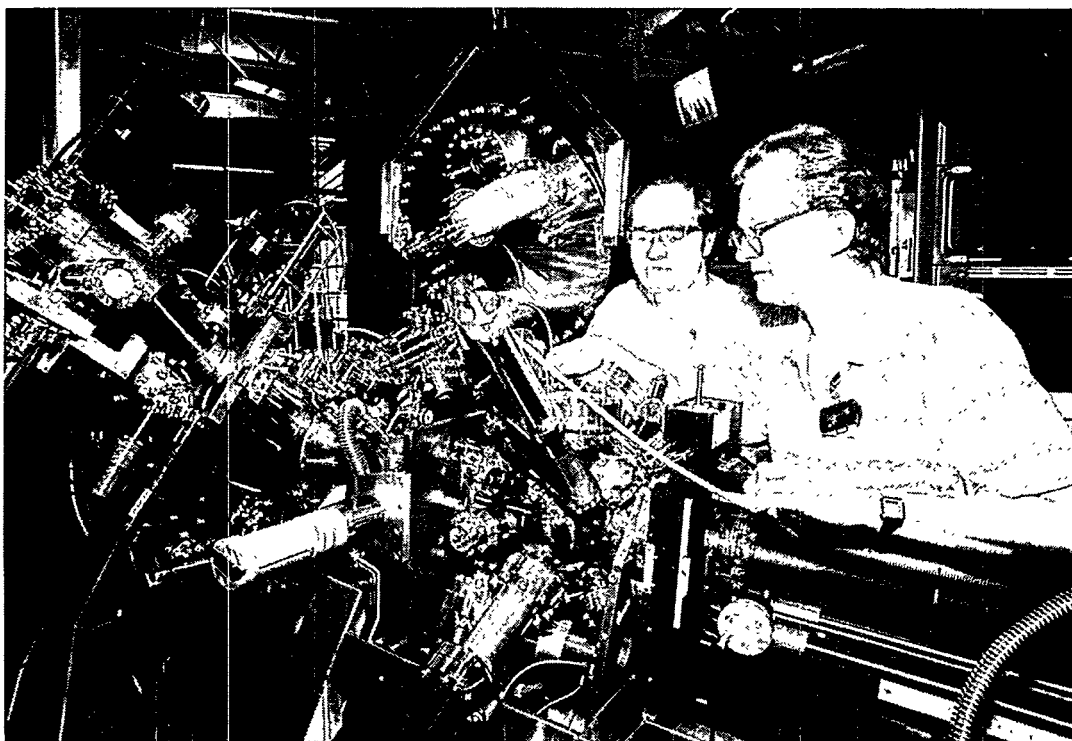


Figure 1. The ALS user services program is designed to assist researchers through the entire process of becoming a user of the ALS, from proposing an experiment through to the completion of their visit. This includes international visitors as well, such as Anders Nilsson (right) and Martin Weinelt from Uppsala University shown with their MAXII endstation on Beamline 8.0.



Figure 2. Recent "happy users" of the ALS include Nora Berrah, Burkhard Langer, and Ovidiu Toader from Western Michigan University working on Beamline 9.0.1.

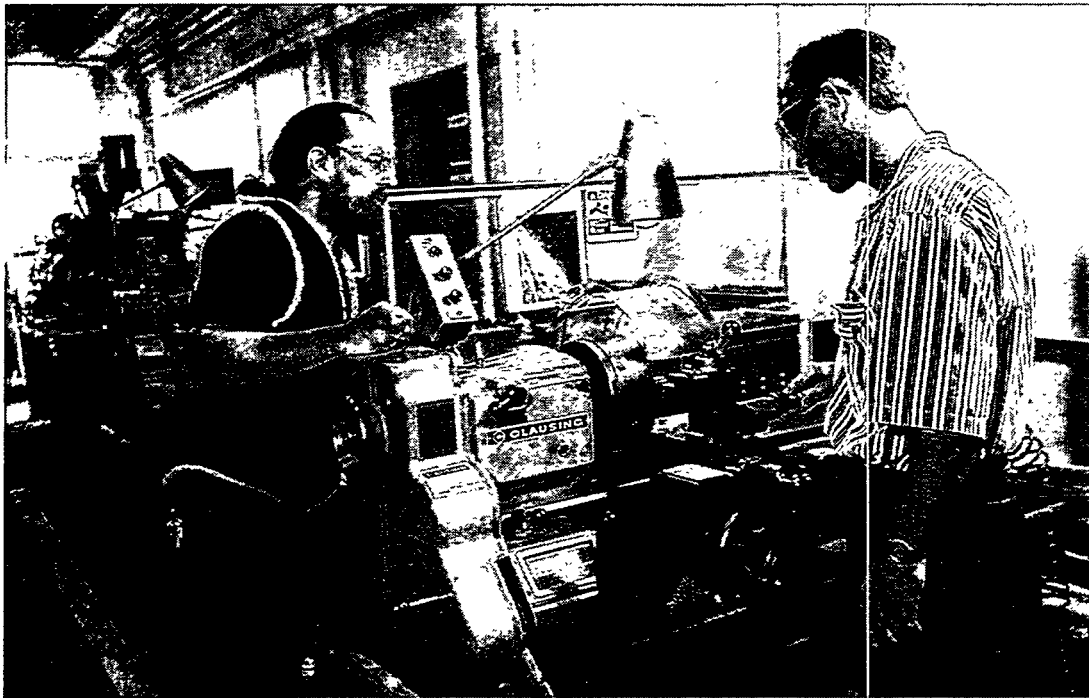


Figure 3. ALS Mechanical Technician Wayne Oglesby (left) demonstrates some of the ALS user machine shop equipment to researcher Reid Brennen from the Jet Propulsion Laboratory.

to rent an apartment or room locally, and again we can provide some assistance with this process. The parking situation at the ALS continues to improve. We now have some reserved parking spaces specifically for ALS users plus there is better availability of parking at the Laboratory. Free shuttle service from downtown Berkeley is also available.

USER SERVICES AND SAFETY

Some of the user services areas recently completed include a clean assembly area and a user machine shop (Figure 3). These facilities are exclusively for ALS users and are conveniently located next to the ALS experiment floor. Additional facilities underway include a chemistry laboratory and a gas storage room.

Ensuring a safe work environment continues to be a high priority and we have made several significant steps in the last year to guarantee that our users are adequately informed about safety issues at the ALS, and to streamline the experiment review process. Our objective in all cases is to facilitate research, not stand in the way of research, so users can work in a safe manner and get the research done. For example, to provide site-specific training for the ALS, we produced a new safety video and *ALS Safety Handbook*. We have worked to improve the experiment review process so that users can begin their research as scheduled, and so that we can accommodate requests that require timely approval such as in the case of the curium experiment described earlier by Brian Tonner.

MEETING THE USERS NEEDS

The ALS is here to serve the needs of the user community and spends considerable effort to keep in touch with what those needs are. Much of this communication revolves around the short- and long-term operations schedules so that users can provide input to us on what type of operating conditions they require. Though the ALS storage ring is optimized to run at an energy of 1.5 GeV, it can run from 1.0 to 1.9 GeV, which allows flexibility for user operations. The normal maximum operating current is 400 mA in multibunch operation; but other filling patterns are available upon request. For example, a user group requested and received two-weeks of 2-bunch operation for time-of-flight experiments referred to earlier this morning.

To keep everyone informed about the short-term schedule, Bob Miller, head of the Beamline Operations Group, conducts weekly scheduling meetings attended by accelerator operations personnel and users to review the upcoming operations schedule. We are in the process of developing a long-term schedule for most of 1995 based on user requests for specific operating conditions during that period that will be reviewed by all beamline spokespersons, the Users' Executive Committee, and ALS management.

We are also working to reduce the length of future shutdowns, thus maximizing beam delivery to ALS users. A new "dry tent" (or dry hood) technique developed by ALS mechanical technicians substantially reduced the length of the May-June shutdown by obviating the need for baking out *in situ* sections of accelerator and beamline vacuum chamber that had been brought up to atmospheric pressure, a 1-2 week process formerly necessary during shutdown time to remove contaminants from ultra-high vacuum components (see Figure 4).

In summary, it is our goal to establish the best possible environment for research at the ALS, and we encourage you, members of the ALS user community, to work with us to make your stay at the ALS an enjoyable and productive one.

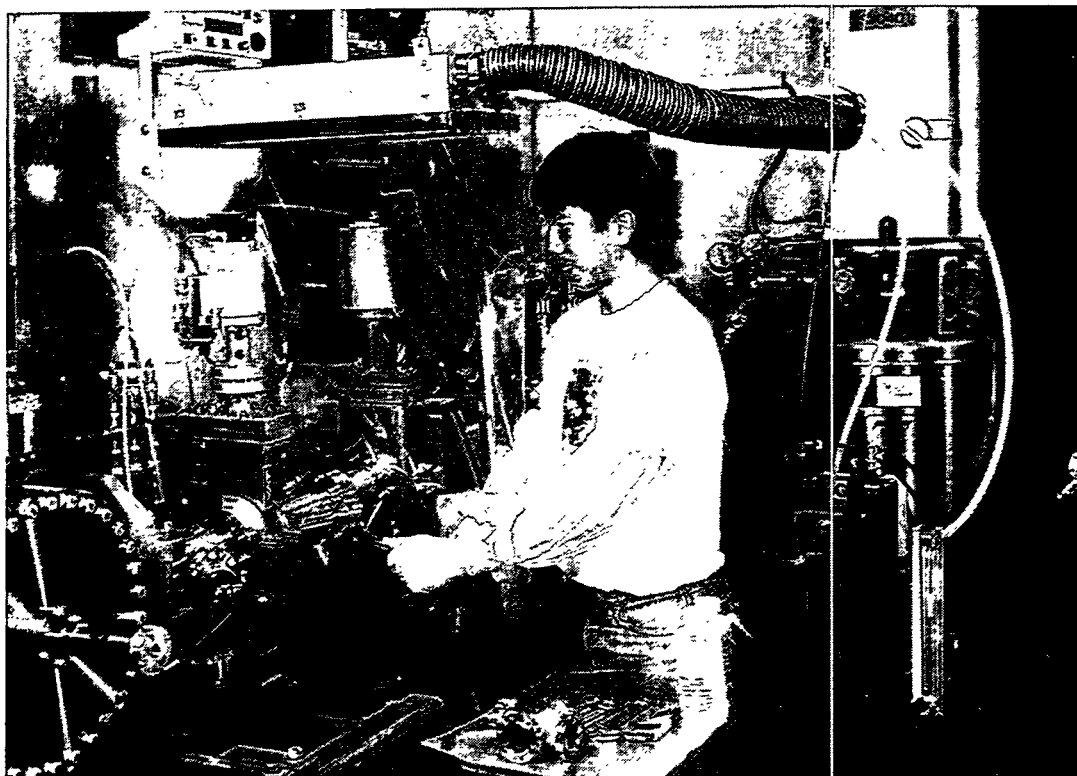


Figure 4. Technician Ed Wong demonstrates the dry tent technique, developed at the ALS by John Thomson. When workers are ready to connect a new UHV section to an existing UHV chamber, they erect a dry tent made of clear vinyl over the point of connection. Dry, filtered air is forced down inside the tent over the union point from above. Dry nitrogen, cleaned by a gas purifier, is introduced into both the new pre-baked UHV component and the existing UHV chamber, and end caps are then removed from the UHV components, allowing the nitrogen to flow out. All work inside the dry tent is carried out through penetration ports by technicians wearing double surgical gloves.

Appendix A

Program
ALS Users' Association Annual Meeting
Lawrence Berkeley Laboratory
October 20-21, 1994

Thursday, October 20

7:30 – 9:00

Registration and Coffee

Building 50, Auditorium Lobby

Recent Highlights from the ALS

(Chair: Michael White, Brookhaven National Laboratory)

8:30 – 8:45	Welcome	B. Kincaid, Director, ALS
8:45 – 9:00	Report from the DOE	W. Oosterhuis, DOE/BES
9:00 – 10:00	Overview of the ALS	B. Kincaid, Director, ALS
10:00 – 10:30	<i>BREAK</i>	
10:30 – 11:00	Accelerator Performance	A. Jackson, Leader of Accelerator Group, ALS
11:00 – 11:30	Experimental Systems	H. Padmore, Leader of Experimental Systems Group, ALS
11:30 – 12:00	Scientific Program	N. Smith, Scientific Program Head, ALS
12:00 – 14:00	Box Lunch and Vendor Exhibit	Building 6

Recent Results from User Beamlines at the ALS

(Chair: Nora Berrah, Western Michigan University)

14:00 – 14:30	Report on First Results from the Beamline 7.0 Spectromicroscopy Facility	B. Tonner, U. of Wisconsin, Milwaukee
14:30 – 15:00	Soft X-Ray Fluorescence (SXF) Studies on the Tennessee/Tulane Endstation on Beamline 8.0	T. Callcott, U. of Tennessee
15:00 – 15:30	<i>BREAK</i>	
15:30 – 16:00	SXF from C ₆₀ and Other Systems	J. Nordgren, Uppsala U.
16:00 – 16:20	Micromachining	K. Jackson, Center for X-Ray Optics, LBL
16:20 – 16:40	High-Resolution Photoemission from Simple Atoms and Molecules	D. Caldwell, U. of Central Florida
16:40 – 17:00	Resonant Photoemission from Ni	A. Nilsson, Uppsala U.
17:00	Adjourn	
18:30 – 19:00	Reception	Hong Kong East Ocean Seafood Restaurant
19:00	Conference Banquet	Speaker: Dr. Jay C. Davis Subject: "Nuclear Non-Proliferation: The United Nations Nuclear Inspections of Iraq"

Friday, October 21

New Opportunities at the ALS

(Chair: Steve Cramer, U.C. Davis)

8:30 – 9:00	Atomic Physics	F. Wuilleumier, University of Paris
9:00 – 9:30	Protein Crystallography	R. Stroud, U.C. San Francisco
9:30 – 10:00	Surface Analysis	R. Brundle, Consultant
10:00 – 10:30	<i>BREAK</i>	
10:30 – 11:00	Polymer Microscopy	H. Ade, North Carolina State U.
11:00 – 11:30	Infrared Microscopy	G. Williams, NSLS
11:30 – 11:50	How to Use the ALS	F. Schlachter, Manager of User Liaison Group
11:50 – 12:00	Users' Association Meeting	M. White, Chair UEC
12:00 – 15:00	Box Lunch, Vendor Exhibit, and Posters	Building 6
15:00	Adjourn	

Additional program

14:30 – 17:00	Workshop on Infrared Microscopy	G. Williams, NSLS, Chair
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Appendix B

List of Participants ALS Users' Association Annual Meeting

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