



## 50 Years of Science: Symposium Marks the Discovery of Synchrotron Radiation

The fiftieth anniversary of the discovery of synchrotron radiation was commemorated at a mini-symposium held April 16, 1997, during the Eighth Users Meeting for the APS. The guest speakers were John Blewett, Boris Batterman, Robert Madden, David Lynch, and Herman Winick. Dean Eastman, Director of Argonne National Laboratory, introduced each of the five speakers, who had been invited to share their personal insights into some of the key events during the early years of synchrotron radiation development.

John Blewett, who earned his Ph.D. in physics from Princeton University in 1936, began by providing a historical perspective on the first observation of synchrotron radiation. Blewett's involvement in the discovery of synchrotron radiation began after WW II when the General Electric Research Laboratory turned its efforts toward the construction of electron accelerators. Their 130-ton, 100-MeV betatron operated with a deafening roar at 60 cycles per second. The Russians, who were also looking at accelerators, warned about radiation emissions from high-energy accelerators. Blewett reviewed past works and concluded that the Russians were perhaps correct, but the opaque vacuum chamber on the betatron prevented any visual confirmation. Later, a 70-MeV synchrotron was built with a clear vacuum chamber, and on April 24, 1947, using a mirror to view the chamber, researchers observed a very bright, bluish-white spot, confirming the presence of synchrotron radiation. Blewett

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*Ernest Charlton and William Westendorp stand near General Electric's 100-MeV betatron. (photo courtesy of John Blewett)*

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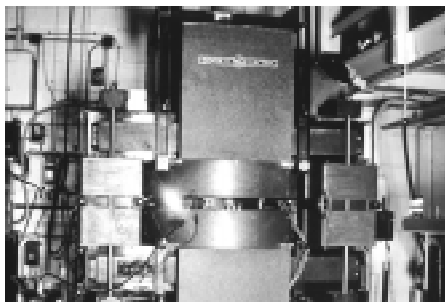
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recalled that the operators of the machine initially thought they were viewing an arc but soon realized that the light being emitted was different colors at different electron energies. The discovery was an “immediate sensation”; six Nobel prize winners were brought to G.E. to see the phenomenon. Yet, as Blewett noted, “In those days, synchrotron radiation was a nuisance.”

Dr. Boris Batterman continued the story by relating how synchrotron radiation came to be used for research purposes. Batterman, the Director of Cornell’s High Energy Synchrotron Source, and Walter S. Carpenter, Jr., Professor of Engineering at Cornell University, spoke about Cornell’s 300-MeV synchrotron, which was built by scientists from the Manhattan Project who went to Cornell after the war to do high-energy physics. The synchrotron was initially used to study pi-meson photoproduction. Paul Hartman, who was at Cornell doing solid-state research, was referred to Diron Tomboullian to discuss the possibility of using synchrotron radiation in his experiments. This partnership resulted in the first real beamline used to pull tangential radiation from a synchrotron. Batterman presented experimental results and techniques from Hartman and Tomboullian that represented the first quantitative work done using synchrotron radiation, including a system they developed to measure mono-energetic electron spectra. In the early 1950s, they conducted spectroscopic studies in the VUV region. Batterman quoted a paper by Hartman and Tomboullian describing the physical characteristics of the radiation: “The light intensity may be so bright as to illuminate a wall, opposite the exit port, in daylight.”

The story was carried into the 1960s by Dr. Robert Madden, Director of SURF II. Madden described the first synchrotron at the National Bureau of Standards (NBS), now the

National Institute of Standards and Technology. SURF I was created when the G.E. 100-MeV betatron was converted to a 180-MeV synchrotron, which was delivered and set up at the NBS. Madden gave a detailed description



*Synchrotron light (bright spot on side of vacuum chamber) visible as electrons travel toward the observer. (photo courtesy of John Blewett)*

of the components and construction of the machine, which was used for studies in high-energy physics until 1960 when synchrotron research began. At this time, the NBS had no VUV radiometric standards. Madden was asked to come to the NBS to carry out this research. Madden and fellow researcher Keith Codling conducted many

experiments to characterize the radiation obtained from the synchrotron using both photographic and photoelectric techniques. Madden stated that the flux had to be monitored constantly due to a 50% variability in the quantity of electrons per pulse! The early experimental results agreed with the theoretical curves, and the performance of the machine was predictable. Spectroscopy work on rare gases required Madden and Codling to design and build their own spectrometer, which was

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focused to within a couple 1,000ths of an inch and remained in place for more than 20 years, achieving 0.06-angstrom resolution. The machine was used until 1974 when it changed sites and was converted to a storage ring.

The first storage ring for UV radiation encountered so many construction difficulties that it was given the name "Tantalus" from a Greek myth in which a king was condemned to be able to see water and fruit that he wanted but could never reach. Professor David Lynch, currently from the Department of Energy's Ames Laboratory and a professor at Iowa State University, talked about how the project began with the construction of a model 50-MeV synchrotron on land south of Madison, Wisconsin, with plans for a larger unit to be built later on that same site. Funding difficulties ended the synchrotron project, and instead a 240-MeV storage ring was designed (using the 50-MeV unit as the injector) to study bunch phenomena. Construction of what would eventually become Tantalus began in 1965 but was halted, again, due to lack of funding. The ring was finally completed in early 1968 after three users signed on and additional funding was obtained. Lynch described how the machine (only 3 meters in diameter) was located in a very small building, partially underground. Initially, Lynch and the other users worked with less than 1 mA of beam current and often only had two to three hours of beam in a 40-hour week! Improvements were made including the addition of more ports and an upgrade to a 40-MeV microtron injector, increasing the available current and the hours of beam available per week. Lynch reviewed the development of the photo-emission technique at Tantalus, as well as angle-resolved photoemission work (including work done by Dean Eastman at IBM) pointing out where the use of synchrotron radiation offered improvement over standard techniques. Tantalus was decommissioned in 1987 and eventually disassembled in 1995, with one of the quadrants of

the machine going to the Smithsonian as a historical archive of the first storage ring.

The final speaker, Dr. Herman Winick, Deputy Director of the Stanford Synchrotron Radiation Laboratory, spoke about the Cambridge Electron Accelerator (CEA), which began operation in 1962 as a 6-GeV electron synchrotron. Designed by Stanley Livingston, it was the first multi-GeV accelerator built on the strong focusing principle. The machine was converted from a 60-Hz cyclic synchrotron to a dc colliding beam storage ring in 1970 using damping magnet technology. Winick showed a photograph of the urban setting of the machine in Cambridge and described its layout: 48 bending magnets with alternating gradients and 16 rf cavities. The facility suffered a disastrous explosion approximately two years into operation when 500 liters of liquid hydrogen ignited, destroying the experiment hall, breaking water mains, and blowing the roof from the building. The lab was rebuilt and went on to produce many significant advancements in the area of accelerators, including Robinson wigglers, rf quadrupoles, and external electron beams by resonance extraction. Winick stated that the beamline used for synchrotron radiation research was unique in that it was the first to make use of a collection and condensing mirror, which increased the flux density at the focus by

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*Five speakers gave presentations at the Mini-Symposium. Shown L to R: David Lynch, Robert Madden, Herman Winick, Boris Batterman, and John Blewett.*

## APSUO Business Meeting and Steering Committee Election

Elections for the APS Users Organization (APSUO) Steering Committee were held on Thursday, April 17, 1997, during the APSUO business meeting. At each meeting, six members are elected to serve three-year terms as specified in the APSUO bylaws.

The newly elected members are:

**Celerino Abad-Zapatero** - Abbott Labs

**Randy Alkire** - Argonne National Lab.

**Peter Eng** - The University of Chicago

**Eric Isaacs** - Lucent Technologies

**Denis Keane** - Northwestern University

**George Srajer** - Argonne National Lab.

The six continuing members are:

**Mark Beno** - Argonne National Lab.

**Robert Bubeck** - Dow Chemical Company

**Steve Dierker** - University of Michigan

**Lynda Soderholm** - Argonne National Lab.

**Steve Sutton** - The University of Chicago

**Jon Tischler** - Oak Ridge National Lab.

The retiring chair, Mark Sutton, will continue on the committee as an *ex officio* member. Jon Tischler assumes the role of Chair. A listing of APSUO Steering Committee members with contact information is available on the Web at the APSUO home page ([http://www.aps.anl.gov/apsuo/apsuo\\_home.html](http://www.aps.anl.gov/apsuo/apsuo_home.html)). The APSUO by-laws, employment registry, and posting of job openings can also be found there.

In other business, an informal poll was taken to determine the frequency with which User meeting participants would like to have the meetings held. The time interval of 18 months was strongly favored.

At the Thursday, August 14, 1997, APSUO Steering Committee Meeting, Steve Dierker was elected as Vice-Chair. 🐱

*The APSUO Steering Committee met on August 14, 1997. Back row (L to R): Denis Keane, George Srajer, Bob Bubeck, Celerino Abad-Zapatero, Jon Tischler, Eric Isaacs, and Randy Alkire. Front row (L to R): Susan Barr Strasser, Peter Eng, Steve Dierker, Mark Beno, Steve Sutton, and Connie Pittroff. Not pictured: Lynda Soderholm*



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a factor of almost 1000. When the National Science Foundation called for a review of x-ray synchrotron radiation facilities in the U.S., CEA's was one of three proposals submitted. Their proposed experimental program was, as Winick put it, "a very rich proposal," but CEA was passed over due to funding issues in favor of Stanford's parasitic set-up. CEA was shut down in May, 1973, after 11 years of operation.

These five men provided symposium attendees a first-hand look at some of the significant milestones in the evolution of the science of synchrotron radiation during the first 26 years. Their insights and contributions are valuable to the continued growth and development of the field. 🐱



*David Moncton, Associate Laboratory Director for the APS (far right), and Mark Sutton, Chair of the APSUO (far left), congratulate the 1997 Compton Award winners Phil Platzman (center left) and Peter Eisenberger (center right).*

## **Presentation of the 1997 Advanced Photon Source Compton Award**

The 1997 Advanced Photon Source Arthur H. Compton Award was presented on April 16, 1997, to Philip M. Platzman and Peter M. Eisenberger in conjunction with the Eighth Users Meeting held at Argonne National Laboratory. The award was established in 1995 by the APS Users Organization (APSUO) to recognize an important technical or scientific accomplishment at, or beneficial to, the Advanced Photon Source. Each recipient of the award receives a check for \$2500 and a certificate acknowledging his or her contributions to synchrotron radiation research or technology. This is the second presentation of the award by the APS; the first was given in 1995 to Klaus Halbach and Nikolai Vinokurov for their work in developing hybrid undulator x-ray sources. The achievements of Platzman and Eisenberger were summarized in the citation on the plaques presented to them at the ceremony. They were recognized for "their pioneering theoretical and experimental contributions to many of the fields of x-ray scattering." These advances provide the foundation on which planned scientific research at the APS is based.

David Moncton, Associate Laboratory Director for the APS, in his introduction, commented that the APS owes its existence to two main forces that have shaped the field of synchrotron radiation and x-ray research. The first is the technological potential to generate high-brilliance x-ray beams using undulators on storage rings. The second is the recognition of the scientific potential of such x-ray beams. The presentation of the Compton award is a celebration of Platzman's and Eisenberger's scientific contributions and "their enormous individual and combined impact on synchrotron radiation research." That impact began in the early 1970s after synchrotron radiation had been discovered and characterized. Moncton described how Platzman and Eisenberger began to think about the physics that could be done using synchrotron radiation, how to optimize instrumentation, and how to get others in the scientific community excited and committed to the effort to develop the field by pioneering research areas like inelastic x-ray scattering, surface scattering, and magnetic scattering.

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# CAT Stats

At its May 30, 1997, meeting the APS Program Evaluation Board recommended approval of scientific proposals from two prospective CATs, as well as approval of a fourth sector for SRI-CAT. The new CATs—Commercial CAT (COM-CAT) and Structural Genomics CAT (SG-CAT)—have each requested one sector, bringing the total number of sectors approved by the APS to 23. The current CAT breakdown is shown below.

<u>CAT Acronym</u>	<u>No. of Sectors</u>	<u>Sector Location</u>	<u>Research Focus</u>
BESSRC-CAT	2	11, 12	Basic energy sciences
Bio-CAT	1	18	Biophysics
CARS-CAT	3	13-15	Structural biology; geo, chemical, material, and soil/environmental sciences
COM-CAT	1	Unassigned	“Fee-for-service” x-ray analysis
CMC-CAT	1	9	Structural characterization of materials
DND-CAT	1	5	Surface and interface science, polymer science and technology, materials science
IMCA-CAT	1	17	Structural biology, macromolecular crystallography
IMM-CAT	1	8	Dynamic phenomena in materials science and physics
MHATT-CAT	1	7	Physics, real-time structural studies, chemical sciences
MR-CAT	1	10	Materials research
MU-CAT	1	6	Materials research
PNC-CAT	1	20	Environmental analysis, materials research, macromolecular crystallography
SBC-CAT	1	19	Structural crystallography
SG-CAT	1	Unassigned	Structural genomics
SRI-CAT	4	1-4	Strategic instrumentation development
UNI-CAT	2	33, 34	Materials science, structural crystallography, condensed matter physics, time-resolved studies, microprobe analysis

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In accepting the award, Phil Platzman related that he had a “special attachment” to the award since one of his first areas of study as a student at Cal Tech was Compton scattering and x-ray physics. He talked about his years at Bell Laboratories, which he described as a “fantastically stimulating place,” and how his “roots” in x-ray physics served him well in his research there. He expressed his pleasure for having the opportunity to interact with many prominent researchers in the field, including Peter Eisenberger. Platzman emphasized that Compton scattering is a “very active and still emerging area of research” and that he expects this field to enjoy a “rebirth” at the APS.

Peter Eisenberger recognized Phil Platzman as someone who has played a critical role in the

shaping of his scientific career. Despite only very minor involvement with x-rays throughout the course of his education, Eisenberger’s career took a dramatic turn when he joined Platzman at Bell Laboratories and began conducting the research in Compton scattering and inelastic scattering that eventually lead him to more powerful x-ray sources and synchrotron radiation research. Eisenberger related a story about one particular experiment involving the parametric conversion of x-rays which taught him that the passage of time and scientific progress can make even a seemingly unimportant result evolve into a powerful research tool. He stated that he will cherish the award for the reminder it will provide for him of all the people who have played such important roles in his career. 🐣

# Highlights from the Eighth Users Meeting

Approximately 500 people representing more than 35 universities, 45 industrial companies, 15 research institutions, and 8 foreign countries attended the Eighth Users Meeting for the Advanced Photon Source on April 15-17, 1997. The meeting opened on Tuesday, April 15, with a series of six workshops covering a diverse range of technical areas. The day ended with poster presentations and a reception held on the experiment hall floor. The first session of the Eighth Users Meeting on Wednesday, April 16, began with a welcome by ANL's Director Dean Eastman, who challenged the User community "to aggressively demonstrate the value of the APS by fully exploiting its potential." Pat Dehmer, Director, Office of Basic Energy Sciences (BES), Department of Energy, provided an overview of the BES interspersed with some personal reflections on her 23 years at Argonne, including her visit to the APS construction site. David Moncton, Associate Laboratory Director for the APS, reviewed the status of the APS facility, reiterating that the highest APS priorities are beam stability and reliability. John Galayda, Director of the Accelerator Systems Division, continued that theme by describing APS progress toward improved beam performance. The afternoon session was devoted to a mini-symposium celebrating the 50th anniversary of the



*From L to R, Dean Eastman, David Moncton, and Pat Dehmer share a moment at the opening session.*



*The Stanley Field Hall of Chicago's Field Museum was the site of the conference reception and banquet.*

discovery of synchrotron radiation (see related story, page 1). Five prominent physicists with close ties to the discovery and development of synchrotron research described early work in the field. The reception and banquet for the conference were held at the Chicago Field Museum of Natural History in the spacious Stanley Field Hall. After dinner, Egyptologist Frank Yurco spoke about applications for x-ray technology in the field of Egyptology.

Thursday, April 17, began with an APS Users Organization business meeting and Steering Committee election (see related story, page 4). Four speakers then presented technical overviews of current User science topics. Ercan Alp began by providing results of sub-MeV

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**Users Meeting...***Continued from p. 7* resolution x-ray scattering work to perform phonon spectroscopy and reviewed the strengths and limitations of this technique. A talk on a powerful tool for material characterization, the x-ray microprobe (XMP), was given by Eric Isaacs of Bell Laboratories. The XMP is proving to be a valuable resource for materials characterization work in a variety of disciplines. Wayne Hendrickson of Columbia University discussed the applicability of multi-wavelength anomalous diffraction (MAD) to structural determination of biological macromolecules. He reviewed the successful structure elucidation of human FHIT with data collected on the APS Structural Biology Center-CAT beamline. This is the first MAD work performed using undulator radiation. The session concluded with Carol Thompson of NIU presenting results of experiments on ferroelectric domains using x-ray interference on epitaxial films.

Throughout the three days, a vendor exhibit was held in the Gallery of the APS Conference Center. Twenty-nine vendors from various industries were present, representing services and products ranging from component design and fabrication, to detectors and vacuum systems. In addition, four Argonne service groups: Electronics and Computing Technologies Division, Central Shops, Technical Information Services, and the Experimental Facilities Division's Optics Fabrication & Metrology Laboratory (see related story,



*Jon Tischler and Gene Ice inspect the double crystal monochromator at UNI-CAT's 33-ID.*

page 11), were also present throughout the conference to describe on-site services available to Users. Visitors took advantage of a self-guided walking tour of the User beamlines on the APS experiment hall floor, where posters described the types of research conducted there.

The next Users Meeting for the Advanced Photon Source is planned for October 13-15, 1998. 🐱

## **On the Horizon: Electronic APS User News**

A new vehicle for communication is being developed for APS Users. An electronic "What's New" column available at the APS User Office home page will provide timely and up-to-date reports on issues critical to researchers and staff. This format can be used to communicate a wide range of information tailored to meet the needs of the User community. Facility scheduling timetables, meeting announcements, listings of symposia and workshops, and other issues relevant to the synchrotron radiation research community will be highlighted. In addition to current information, this vehicle will provide useful links to other on-line sources of information available from both the APS and ANL. 🐱



# Dr. Sunil Sinha Presented with Lawrence Award

By Catherine Foster

Argonne Senior Scientist Sunil K. Sinha, a member of the Experimental Facilities Division (XFD), has won the 1996 E.O. Lawrence Award in the Materials Research category, one of the highest awards presented by the U.S. Department of Energy. Sinha won his award “for his wide ranging theoretical and experimental contributions to neutron and x-ray scattering in materials physics.” Sinha, Associate Director of XFD, has developed new techniques for using x-rays and neutron scattering to learn new details about the structure of many materials.

In particular, he and his coworkers developed a general theory of x-ray and neutron scattering from “rough,” or real-world surfaces, and then validated the theory by carrying out an imaginative series of experiments. Sinha’s development of the “off-specular surface scattering” method has opened a wide range of important phenomena to experimental study. He has led the field in applying this technique in many areas, including growth of thin films and multilayers; structure of liquid surfaces; corrosion; surface phase transitions of biological interest; and “surface freezing” of long chain molecules such as those in lubricants.

“Every physical surface has some degree of roughness or imperfection,” Sinha explains. “Just as the topography of a landscape may be determined by sweeping a radar beam across it and reconstructing the image from that part of

the signal that is scattered back, the method I helped to develop provides a similar method for reconstructing the topography of the surface of a material – by analyzing the distribution of

scattered radiation when the surface is interrogated (scanned) with a powerful beam of x-rays or neutrons.

“Because of the extremely small wavelengths of such radiation, we are able to resolve features on length scales from atoms to thousands of atoms,” Sinha said. “And because of the pen-

etrating power of the radiation, interfaces buried beneath the visible surface can be accessed as easily and non-destructively.”

The technique has led to better material surfaces and thin films for technologies ranging from semiconductors and magnetic recording devices to coatings and adhesives. Details of how “pitting corrosion” occurs on metal surfaces are also being studied.

Sinha has been one of the leaders in using neutron and x-ray scattering to determine important properties of solids such as the role of lattice vibrations, the magnetic properties of metals whose behavior depends on certain kinds of electrons, the coexistence of ferromagnetism and superconductivity, high-temperature superconductivity, the behavior of liquids in porous media, and the structure and



*Dr. Sunil Sinha (right) is presented the E.O. Lawrence Award by Acting Secretary of Energy Charles B. Curtis.*

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# What's New on the Web?

## On-Line Registration for APS Users

New Users must register with the APS User Office and complete the orientation process prior to beginning any work in the experiment hall. Now the first step in the orientation process can be conducted on-line by accessing the User Office home page: (<http://www.aps.anl.gov/xfd/www/xfd/communicator/useroffice/homepage.html>).

The form, which became available on the Web the first week in May, 1997, can be reached from the home page by clicking first on Access, then on On-Line User Registration Form. It can then be completed and submitted electronically to the User Office. Receipt of the form initiates the mailing of a package of written materials that need to be read to complete the orientation process. (These materials, the APS User Guide, the APS User Safety Guide, and a General Employee Radiation Training (GERT) Study Guide are also available on the Web at the APS User Office home page.) The packet

of orientation materials includes a copy of the submitted registration information for review by the prospective User. Also provided is a cover letter explaining how to schedule the initial visit to the APS and outlining the orientation process. During the initial visit, the required orientation course can be completed on-line. Additional training required to complete orientation includes sector specific orientation. The User Office staff will also check to see that the sponsor institution for the prospective User has a User Agreement (which covers liability, funding, and intellectual property issues) in place with the APS. New Users are required to sign an acknowledgment form recognizing the presence of this agreement, as well as an additional form which stipulates that the new User has completed orientation, understands APS policies and requirements, and agrees to follow them. When this process is complete, the User will be issued a Cardkey® photo-I.D. badge, which can be used for site and building access. 📌

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### **Sinha...***Continued from p. 9*

kinetics of phenomena occurring in polymers and complex liquids.

“Dr. Sinha’s contributions to materials science have spanned a wide range from fundamental physics to technology,” said David E. Moncton, Argonne’s Associate Laboratory Director for the Advanced Photon Source. “He is one of the rare scientists equally at home in theory and experiment, and it is this combination of theory and experiment that most distinguishes Dr. Sinha’s contributions.”

Throughout his career in university, industrial, and government laboratory environments, Sinha has also acted as a mentor for several generations of scientists, serving both as a

teacher and a role model. “His evident love of condensed matter research, his skill, his breadth of interest, his integrity, and his generosity to young colleagues have been an inspiration to many,” Moncton said.

Sinha began his career as a Visiting Scientist at the Bhabha Atomic Research Centre in Bombay, India and then moved to Iowa State University in 1965, where he became Professor of Physics in 1972. In 1975, he became Senior Physicist at Argonne. From 1983-1995, he was a Senior Research Associate at Exxon Research and Engineering Company, and returned to Argonne in 1995, joining the staff at the Advanced Photon Source. 📌

## Optics Fabrication and Metrology Group: Status and Capabilities

The Optics Fabrication and Metrology (OFM) Group was formed on October 1, 1996, and has since evolved into a fully functional and highly productive group with facilities and instruments selected to meet the needs of CAT members. The objectives of the group are:

- development and operation of instruments for x-ray metrology and optics fabrication to support the APS User community;
- characterization of optics used on CAT beamlines and assurance of their performance;
- joint R&D programs with the User community on the development of new optics for special beamline applications, and;
- joint R&D with ESRF & SPring-8 on unique optics common to future needs of the Users.

The group is organized around the following areas: metrology, crystal fabrication, and deposition. There is also expertise available for designing optical components that must withstand high-powered synchrotron beams. Two examples of high-heat-load-related projects are designs for white-beam mirrors used for undulator radiation at the APS and masks for x-ray lithography.

### METROLOGY

The APS Metrology Laboratory (APS-ML) can provide a valuable, independent quality control service for Users at the APS. For example, Users who utilize mirrors for harmonic rejection, focusing, power filtering, or beamline branching can have the slope error and roughness of their mirrors measured. Mirror quality can be affected by the substrate used and the vendor's polishing technique and expertise. The laboratory offers three different non-contact optical instruments that are used for the measurement of figure and finish on



*The Long Trace Profiler (LTP) is used to measure slope and curvature of optical surfaces.*

mirrors. Slope errors of 1  $\mu$ rad and surface microroughness on the order of 1  $\text{\AA}$  rms can be measured.

A necessary condition for precise metrology is a well-controlled environment for the instruments. Metrology measurements are carried out in a stringently controlled clean room, rated class-10,000, located in the experiment hall of the APS. Special clean-room clothing and shoe covers are required for all mirror handling. During measurements, access to the room is restricted, and the room temperature stability is controlled to  $\pm 0.5$   $^{\circ}\text{C}$  to ensure instrument accuracy and repeatability. All of the instruments are mounted on air tables to minimize vibrational interference and can handle optics up to 2 m in length and 90 kg in weight.

A wide range of spatial periods can be evaluated in the APS-ML, ranging from a few microns up to 2 m, utilizing the lab's three primary instruments:

- The Long Trace Profiler (LTP) is designed to measure slope error and curvature of optical surfaces up to 2 m in length with good

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### **Metrology...Continued from p. 11**

sensitivity and reproducibility. Measuring in either the horizontal or vertical direction, it provides a rapid and accurate evaluation of a mirror bending mechanism.

- Whereas the LTP provides only 2D data (i.e., slope error along a line), the WYKO 6000 interferometrically measures the flatness of surfaces. Optical surfaces up to 6 in. in diameter (or larger optics at grazing incidence angles) can be probed. Data can be reported as 3D profiles of optical surfaces, 2D slices, or Seidel aberration coefficients.
- The TOPO Surface Profiler is a microscope-based instrument that uses visible-light interferometry to measure surface roughness. The TOPO system can provide output data such as contour maps, 3D maps, 2D data slices, and step height analysis, among others. The maximum length of an optic that can be measured on the TOPO is 1.5 m.

The APS Metrology Laboratory has evaluated a dozen major beamline mirrors, some up to 1.5-m long. Most of the mirror substrates have been either silicon or Zerodur; others tested have been Glidcop and float glass. The surfaces have had at least one reflective coating, typically platinum or rhodium. To avoid any bias, vendor data is typically obtained only after all metrology measurements have been made and analyzed. For slope error measurements, several lines across the surface of the mirrors are scanned at least five times each, and the scans averaged to minimize instrumental noise. Optical surface roughness is checked at several random locations on the mirrors to avoid any periodicity in the surface quality induced by the manufacturer's polishing technique. The TOPO is calibrated prior to use, and each site is measured for roughness multiple times. The results are averaged to minimize error due to noise. Major mirrors have been characterized for the SRI-CAT, SBC-CAT, DND-CAT, CHESS at Cornell University, and CAMD at Louisiana State University.

Suppliers have included Rockwell, Continental Optical, General Optics, SESO, PSI, and Zeiss.

Future developments in the Metrology Lab include the planned installation of a fourth instrument having both a scanning probe microscope and a near-field scanning optical microscope. This instrumentation can be used to obtain topographic maps of surfaces with fraction-of-an-angstrom resolution, which will be of great utility for surface roughness measurements. The lab is also currently working to refine mirror measurement procedures and prepare documentation of standardized results. A mirror measurement database is also being developed as a reference tool for future projects.

### **FABRICATION**

Crystal elements in use on x-ray beamlines at the APS are manufactured in the Fabrication Laboratory. The lab has a Meyer-Burger TS121 crystal cutter with a CNC-controlled saw that can accommodate diamond blades up to 16 in. in diameter. Three different lapper/polishers are available for the coarse, fine, and final polishing of optical components. An oven is available for the attachment of crystals to the substrates used during fabrication operations, and a second oven is utilized for the annealing processes. Two chemical hoods are available for etching and solvent handling. A crystal direct bonding setup, used for experiments on silicon-to-silicon direct bonding, can handle wafers up to 4 in. in diameter. These fabrication facilities have been used to manufacture more than 50 optical elements that have been used in monochromators, analyzers, and interferometers by various CATs at the APS.

The Fabrication Lab is supported by the X-ray Laboratory, which is equipped with three different x-ray generators. A 2 kW maximum power Spellman x-ray generator has two available horizontal beamlines. One of the beams

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### **Metrology...Continued from p. 12**

can be used in a large enclosure attached to the generator tower shielding. This beam has primarily been used to test high-heat-load monochromator prototypes that were later installed on the APS ring. The second beam of the Spellman generator can be used for two types of experiments; backscattering work using a Laue camera, and double-crystal reflection experiments using highly collimated primary beam.

One Rigaku x-ray generator (maximum power about 2 kW), producing x-rays in conventional tubes, has two horizontal beamlines available. One beam is collimated and used for the orientation of ingots or precut crystal pieces. The second beam is used for work with a double-axis diffractometer and is most often utilized for testing of samples, interferometers, and area detectors.

A second Rigaku x-ray generator is a rotating-anode type with a maximum power of 18 kW. One beam from this generator is employed for work with a triple axis diffractometer and has recently been used for reflectivity measurements on multilayers. Beam from the second port is transported to a double-axis x-ray diffractometer called the Topo Test Unit (TTU), designed and built in-house for topographic

testing of x-ray optics elements. The TTU is predominantly used for testing prototype monochromators and single crystals of Si, Ge, and diamond to be used at the APS by measuring rocking curves in double-crystal geometry and taking crystal images. High-heat-load monochromator testing has also been performed. The TTU has been used to test more than 50 optical elements over the past year for the CATs.

### **COATING**

The APS deposition facilities can provide Users with practical and cost-effective services for mirror preparation, substrate cleaning, and thin film/multilayer coatings. The development of reflective optical elements and experimental samples has led to the establishment of two sputter deposition facilities and an ultrahigh vacuum (UHV) surface analysis/evaporation facility to serve APS Users.

The 1.5 m sputter-deposition facility is a large mirror coating system that can handle substrates up to 150-cm long, 20-cm wide, and 14-cm high. Located in a class-10,000 clean room, substrate loading is done in a class-100 clean hood. Substrates can be outgassed *in situ* in the deposition chamber using a UV lamp.

*Continued on p. 14*



*The 1.5m sputter-deposition facility in the APS-ML.*

### **Metrology...Continued from p. 13**

The deposition chamber vacuum is less than  $\sim 1 \times 10^{-8}$  Torr. Sputtering is done in an upward direction (substrate face down) for better contamination control, while video cameras monitor the movement of the substrate during deposition. A rate-controlling aperture over the target source and the linear motion of the substrate over the sputter guns has resulted in a coating uniformity of better than 5% over a 4 in.-width. Comparable uniformity over an 8 in.-width can be achieved by using two specially arranged sputter guns.

The small sputter-deposition system is designed to handle smaller substrates, and is especially useful for the growth of multilayers on substrates up to 4 in. in diameter. Multilayers are grown by alternately passing the substrate over two sputter guns.

The evaporation facility, an ultrahigh vacuum system, is equipped with an electron energy analyzer, an electron gun, and a surface-cleaning sputter. The system is suitable for molecular beam epitaxy (MBE)-type sample preparation and for surface analysis. Miniature e-beam and precision temperature-controlled thermal evaporators have been designed and produced in-house for physical vapor deposition of thin and ultrathin films. The composition and uniformity of alloy films produced can be analyzed *in situ* using Auger electron spectroscopy.

Major applications of these deposition systems include reflecting x-ray mirrors, multilayer x-ray mirrors, and thin film/multilayer samples. The APS coating facility has produced a 56 in.-long reflecting x-ray mirror coated with 10 nm Ti and 30 nm Au. The float glass substrate was cleaned in a sophisticated ultrasonic aqueous cleaning facility at the APS. The end result was a mirror with excellent flatness with an average RMS roughness of 0.19 nm. Smaller Au/Cr-coated float glass mirrors made by the APS coating facility for the CARS-CAT/University of Chicago team, used for

microfocusing hard x-rays, produced a focus of  $6 \times 8$  square microns for 60-keV x-rays. The gold coating has remained intact without discoloration, clustering, or cracking.

Multilayer x-ray mirrors are grown in the deposition lab with the aid of a computer program developed in-house which controls the speed of a substrate's linear travel over two sputter guns. Layer thicknesses can be determined by the speed of substrate motion when the rate of deposition is known. Using the program, the speed can be changed from region to region, controlling the thickness of individual layers. Other applications include multilayer elements for multi-element detectors, high-heat-load multilayer monochromator crystals, coherent reflecting optics, and supermirrors.

State-of-the-art MBE-type samples can be prepared in the UHV facility. Substrates can be cleaned *in situ* using ion bombardment and subsequently annealed. Cooling during deposition helps avoid interdiffusion and segregation at the interface, which can be critical when two elements with significantly different surface free energies, such as Fe and Sn, are brought together. An isotopically enriched alloy film of  $\text{Fe}^{57}\text{Sn}^{119}$  was recently produced using the MBE-type growth. The deposition rates of each element can be stoichiometrically controlled using the precision temperature-controlled thermal evaporators. The facility and cleaning and masking techniques can be used to provide APS Users with a variety of physical deposition services, including protective coatings, laser mirror coatings, and metal coatings for x-ray lithography work.

In summary, the OFM group within XFD provides APS Users with the ability to:

- measure 2D figure for 2-m long mirrors
- measure 3D figure for 150 mm optics
- measure 2D & 3D roughness
- perform DC sputtering for coating of 1.5 m mirrors & multilayers

*Continued on p. 15*

**Metrology...Continued from p. 14**

- perform UHV evaporation of metal thin films
- consult on the design of high-heat-load mirrors
- characterize crystals with x-ray double crystal diffraction
- characterize crystals with x-ray double crystal topography
- characterize thin films and multilayers with x-ray triple-axis diffraction
- orient crystals with x-ray Laue diffraction
- slice large boules
- polish large crystals
- perform wet chemical etching
- cure bonding agents

These facilities are a very powerful combination of resources for the benefit of APS Users. The APS Optics Fabrication and Metrology group is dedicated to providing the APS User community with practical, cost-effective, and high-quality products in the areas of metrology, crystal fabrication, and coating of reflective

optical elements. The following staff would be glad to discuss how the lab's capabilities can complement the research being conducted by the CATs at the APS.

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<b>1997-1998 CAT Calendar</b>	
<b>Date</b>	<b>Event</b>
October 13-17, 1997	Fifth International Workshop on Accelerator Alignment
October 27-29, 1997	Fourth Generation Light Source Workshop
October 30, 1997	APS/USO Steering Committee Meeting
October 31, 1997	Research Directorate Meeting
August 4-8, 1998	6th International Conference on Biophysics and Synchrotron Radiation
August 10-14, 1998	10th International Conference on X-ray Absorption Fine Structure
October 13-14, 1998	Ninth Users Meeting for the Advanced Photon Source
October 14-15, 1998	APS/USO Workshops

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# CAT News

The *CAT News* column is designed to provide Users with quick updates on current CAT events, as well as highlight new scientific and technical developments taking place around the ring.

**BESSRC-CAT:** Installations on sector 12 have been completed, and BESSRC-CAT is expecting the entire section to be fully operational in September. A mirror has been ordered for sector 11 and will be installed on the beamline in 1998. In addition, during the December shutdown, a mirror will be installed on 12-BM. By spring of 1998, wiggler and FOE installations are expected to be complete.

**BIO-CAT:** Bio-CAT has a new staff scientist, Dr. Sergey Stepanov. White beam enclosure verification in hutches A-C has been completed, and the final monochromatic verification for enclosure D is planned for September. This will provide "first light." Bio-CAT is working with the Experimental Facilities Division Optics Fabrication and Metrology group to explore the feasibility of developing low-cost, multi-layer capabilities.

**CARS-CAT:** BioCARS has taken beneficial occupancy of the completed biohazard facility, which includes the FOEs on both the BM and ID beamlines. They are also currently using ANL's Electronics and Computing Technology (ECT) Division for assistance in the installation of beamline control and acquisition software. Funding for BioCARS for the next five years is anticipated from the National Institutes of Health, and the CAT is awaiting

official notification. GeoCARS is conducting experiments in the FOE using a small, prototype KB mirror. In sector 13, shielding verification on the ID line has been completed, and verification on the BM line is next. Shielding verification on the FOE in sector 14 is complete, and final verification has been done. Three mirrors have been ordered and received. The 14-ID mirror has been installed. Buildout of the LOM in sector 15 has been completed, and the PDR for that sector will likely be submitted to the APS by the end of the summer.

**CMC-CAT:** CMC-CAT currently has its components and hutches for the ID and BM beamlines in sector 9 in procurement.

**DND-CAT:** During the June run, mono-beam was taken all the way out to the last station of the ID. Optics are working well, and a vibration discovered in the monochromator is being investigated. Closed-gap, windowless beam was achieved without degradation. For phase two of the construction, the BM line will be completed during the end-of-the-year shutdown, completing all of DND-CAT's construction.

**IMCA-CAT:** Nine of the 12 IMCA companies currently have User agreements in place; 40 Users collected data on approximately 20 samples of 12 different proteins during the last run. This work was done on the sector 17-ID beamline using the CCD system on loan from Siemens. The Oxford Cryosystems cooling unit has been used on the ID beamline. Commissioning studies are planned for a sagittally bent

second crystal in the monochromator on the BM beamline. IMCA-CAT has found that their four-element Siemens CCD array on the BM beamline has yielded good diffraction data. Mirror systems have been ordered for the BM and ID beamlines. IMCA-CAT has submitted its final design report to the APS, and a four-CAT (IMCA, Bio, SBC, and PNC) LOM shop safety plan has recently been approved.

**IMM-CAT:** IMM-CAT has implemented a 0.25-mm mini aperture to carry out coherent x-ray scattering experiments in the 8-ID FOE. Preliminary measurements of x-ray speckle patterns, produced by partially-coherent illumination of static aerogel samples, indicate the measured coherence properties of the undulator beam (speckle contrast, intensity, and size) are within a few factors of those expected from the APS source size and brilliance. Future efforts will be directed at carrying out x-ray photon correlation spectroscopy studies of very low-energy dynamical processes.

**MHATT-CAT:** All six hutches have been assembled on the floor, and the utilities installed. The ID line components have been bolted into place. MHATT-CAT is developing new concepts in coherent beam optics for their ID line (compact pink beam filter). Gene Ice is working on a monochromator for the micro-focus project headed by Reinhard Pahl. A paper is available on the Web about the pink beam work. MHATT-CAT has also procured a multi-disk data acquisition system.

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**CAT News...Contd. from p. 16**

**MR-CAT:** MR-CAT is continuing with procurement and construction activities, and its final design report is written and will be submitted to the APS soon. The FOE is completed, certified, and instrumented, and includes a Daresbury-design monochromator fitted with a commissioning crystal cage that uses a cryogenically-cooled Si first crystal and a room-temperature, flat second crystal. The second-generation cage is currently in the design stage. The monochromatic-beam experiment hutch is completed (including utilities) and has passed shielding verification. The focus of MR-CAT for the upcoming year will be hutch instrumentation, particularly beam conditioning equipment, a new CCD system, and a Huber 8-circle diffractometer. Experiments are planned in the areas of scattering and spectroscopy. MR-CAT's current staff includes Paul Plag (beamline scientist/project manager), Nadia Leyarowska (beamline electronics engineer), Andrew King (mechanical engineer), and Bill Lavender (software engineer).

**MU-CAT:** Recent hires include new beamline scientist Doug Robinson and beamline technician Eric Zoellner. MU-CAT's enclosures are now fully constructed and are currently being painted. The D.I. water skid has been installed and MU-CAT is awaiting electrical utilities. Major beamline components, including the beam transfer pipes, have been purchased, and the LOM build out is beginning.

**PNC-CAT:** PNC-CAT has ordered all remaining hutches for their ID and BM beamlines in sector 20, and expects to build over the next couple months. The first test on PNC-CAT's monochromator was successfully

performed April 28, 1997, when a 140-eV scan was made, centered on a metallic Cu K-edge. Full XAFS scans on Cu have been run that have illustrated what is needed to optimize the spectra. During the last run period, commissioning tests on the monochromator in the FOE of the ID line were successfully completed. Measurements from several different imaging experiments conducted in the FOE have been collected and the data will be analyzed soon.

**SRI-CAT:** SRI-CAT's hard x-ray beamline stations have passed radiation verification tests and experiments are being run on all hard x-ray beamlines. Commissioning activities have been completed on 1-ID and it has been declared operational. Activities on 1-ID include experiments to determine the performance of silicon vs. diamond double-crystal monochromators, investigation of the nitrogen reliquifier system (difficulty with thermal trips on the compressors were resulting from vendor installation problems), and high-energy scattering experiments on confined fluids. On the hard x-ray branch of the 2-ID line, using radiation from undulator A, a beam focused to approximately 1/3 micron with a phase-zone plate is being used to carry out micro-diffraction and micro-spectroscopy work on a variety of samples (plants, roots, cells, computer chips, solid state laser modulators, etc.). The two soft x-ray lines on 2-ID received radiation in August from the 5.5-cm period soft x-ray undulator, which was recently installed on the 2-ID straight section along with the standard undulator A. Excellent progress is being made on the high-resolution program on 3-ID. A recently obtained sub-milli-eV resolution beam was used to measure the partial phonon density states in

iron and iron containing compounds. A double-crystal diamond monochromator, the first in routine operation at the APS, was recently installed on 3-ID.

**SBC-CAT:** SBC-CAT has completed shielding verification of all enclosures on both the BM and ID lines; the ID line can now be operated with the full range of undulator gap. In all other respects, both beamlines have been built, and officially, SBC-CAT is no longer a construction project of the DOE. It is now in its commissioning phase. Tests are being conducted on the CCD detector and monochromator. The CCD detector has been remounted on a more accurate and reproducible positioning system. Computer-controlled guard slits have been installed and tested. A kappa goniostat for sample crystal orientation has been built and is being tested. Sample crystal cooling tests suggest He cooling below 80° K will enhance sample viability in a focused undulator beam. The first new protein crystal structure from 19-ID (FHIT, a human tumor suppressor) has now been published (Lima et al., *Structure*, June 15, 1997).

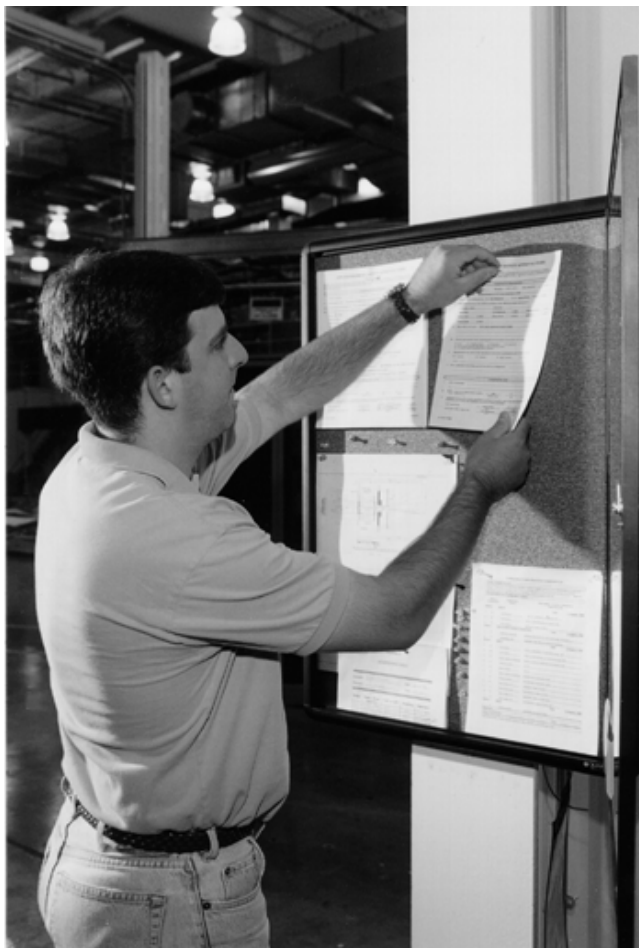
**UNI-CAT:** UNI-CAT received approval to merge with MICRO-CAT, combining sectors 33 and 34. Shielding verification of sector 33-ID has been completed. Procurement for sector 33-BM has begun, and construction is expected to start in 1998. Sector 34 is awaiting NSF funding approval. During the last run, UNI-CAT used its double-crystal monochromator and discovered a design flaw: not enough translational movement of the crystals is allowed. The UOP has added a new member from Dow, Simon Bare. David Robinson is returning to England, and UNI-CAT is in the process of hiring a research engineer. 🐣

# Safety Notes: Experiment Safety Review

An experiment safety review is the critical first step in beginning any experiment on the beamlines performed by CAT members, their collaborators, or Independent Investigators. An APS Experiment Safety Approval form is available at every Floor Coordinator's office, and is accessible through the APS User Office Home Page by clicking on [Technical Information](#) and then on [User Technical Interface Group Schedules, Logs, and Forms](#). The form has two main sections: The first part must be completed by the experimenter and provides important information about the experiment to be performed, such as any potential hazards, the staff involved, and materials and equipment used. The form is then submitted to designated CAT reviewers for approval, as described in each CAT's safety plan. The second section of the form documents the CAT's approval of the experimental design and indicates that User training and experiment safeguards have been verified by the CAT. The form requires evidence that the hazard mitigations are sufficient to allow the experiment to be conducted within the beamline operations safety envelope. The CATs are responsible for the actual scheduling of the experiments and assuring that the required hazard mitigation is in place before the start of the experiment.

Once the form is completed, it is returned to the appropriate Floor Coordinator who will post the completed and approved hard copy in the cabinet at the end of the designated beamline. Only Floor Coordinators are allowed to post the forms. (In order for a beamline to be operational, there *must* be either an APS Experiment Safety Approval Form or a commissioning form in place in the end cabinet.)

Each approved experiment safety form, covering a single experiment type, is valid for a six-month period. After six months, the experimenter must re-submit a new form for approval to the CAT in order to continue work on the beamline on that particular experiment. The CATs are responsible for tracking the six-month time period, with the Floor Coordinators functioning in an oversight capacity. Any questions regarding experiment safety issues should be directed to the specific CAT Safety Coordinator, the appropriate Floor Coordinator, or Bruce Glagola (User Technical Interface). 🐣



*Floor coordinator Steve Vanni posts an Experiment Safety Review form at the beamline in sector 19-ID.*

## Meet...

Connie Pittroff, the newest member of the User Administration staff, came to the APS from Radian International, where she served, most recently, as Quality Control Manager in an analytical laboratory. Connie, who received her B.S. and M.S. in organic chemistry from the University of Illinois, has worked the past eight years in the chemical industry in the areas of marketing, organic synthesis, and lab management, performing assignments requiring excellent communication skills.



**Connie Pittroff**

As Assistant User Program Administrator, Connie will be primarily responsible for various aspects of User communication, among which are producing *CAT Communicator*, developing electronic news reports, and assisting in the production of an annual User Activity report that describes the scientific accomplishments of APS Users.

Connie spends her leisure time rock climbing, playing softball with the APS Users team (called, appropriately, the Ringers), and doing needlework.

## Bruce Glagola



Bruce Glagola, the newest member of the User Technical Interface Group, came to the APS from ANL's Physics Division, where he served as the User Liaison for the ATLAS Heavy Ion Accelerator Facility. Bruce has a Ph.D. in Nuclear Chemistry from the University of Maryland. His research interests in the past involved nuclear astrophysics, heavy-ion-induced fission, transfer reactions, and accelerator mass spectrometry. He has spent the last 12 years in the role of User Liaison at ATLAS, which involved all aspects of User experimental support.

As a member of the User Technical Interface Group, Bruce will be primarily involved in beamline design reviews, experiment safety oversight, and technical support issues for the User community.

Bruce spends his leisure time golfing and working in his darkroom. 🐱

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