SRN

Synchrotron Radiation News
ISSN 0894-0886 is published bi-monthly.
Coden Code: SRN EFR

**Heather Wagner,** *Managing Editor*
**Maureen Williams,** *Advertising Manager*
**Ed Cilurso,** *Production Manager*
**Patrick Hufnagle,** *Designer*

**Editorial Services**
Synchrotron Radiation News
346 Meadowview Drive
Collegeville, PA 19426, USA
Tel/Fax: +1610 409 9082
E-mail: srnews@bnl.gov

**Advertising Services**
Maureen Williams
P.O. Box 1547
Surprise, AZ 85378-1547, USA
Tel: +1 623 544 1698
Fax: +1 623 544 1699
E-mail: mwilliams@nni.com

**Circulation and Subscriptions**
Taylor & Francis Inc.
325 Chestnut Street, 8th floor
Philadelphia, PA 19106, USA
Tel: +1 215 625 8900
Fax: +1 215 625 8914

*The following subscriptions are available:*
Vol. 15 (2002), six issues.
*Personal:* €64 EUR, $72 USD,
£48 GBP, $120 AUD, ¥13,500 JPY.
*Institution:* €480 EUR, $528 USD,
£360 GBP, $912 AUD, ¥100,800 JPY.

The opinions expressed in *Synchrotron Radiation News* are not necessarily those of the editors or publishers.

# CHESS 2002 Users' Meeting

CHESS, in its 22nd year of operations, conducted its 15th annual CHESS Users Meeting at Cornell University on June 18 and 19, 2002. The meeting highlighted first experimental X-ray data from the new G wiggler stations, first X-ray data on multilayers of unusually high energy resolution, and a new kind of video-based beam position monitoring system built upon helium optical luminescence. In addition, there were two concurrently running workshops, titled "New Technology and Innovative Applications of Small Angle X-ray Scattering" (Sol Gruner, organizer) and "High-Throughput Crystallography and Complementary Methods" (Quan Hao and Richard Gillilan, organizers). About 125 individuals attended the meeting. This article contains some of the highlights of the staff reports, invited talks and workshops (see Figures 1–4).

**Staff highlights**

Sol Gruner, CHESS director, reported that in the last four years, 1263 different researchers worked under 394 unique proposals. On the basis of hours of beam time, CHESS usage has been roughly equally divided between biological and non-biological science. Sol also reported that the organizations at CHESS are growing: there are 34 persons working at CHESS, 13 in MacCHESS, and three at G-line, plus about a dozen graduate students and approximately a dozen Cornell faculty members. A new five-year renewal proposal was recently reviewed for CHESS and a five-year renewal proposal has been submitted for MacCHESS.

Maury Tigner, director of the Laboratory for Elementary Particle Physics (formerly called the Laboratory for Nuclear Studies), described an anticipated year of activities during which the CESR storage ring would start to run, for the first time, in the 1.8 to 3.7 GeV range (per beam) for charmed quark studies. About 10 weeks of down time will be needed in early 2003 for the installation of five new superconducting wigglers into CESR. The resultant charm running will be at too low a beam energy for CHESS X-ray users, so periods of dedicated X-ray running and dedicated high-energy physics running are planned. The first dedicated X-ray runs are expected to begin in January 2003.

Joel Brock, director of the new faculty beamlines at CHESS known as G-line, spoke of the progress using the "backfire" beam from the A/G-line wiggler. Since there are both electrons and positrons simultaneously orbiting in opposite directions in the storage ring, the wiggler creates two oppositely directed X-ray beams. One of these beams feeds the CHESS A-stations and the other the G-stations. Ernie Fontes (CHESS Assistant Director) and Detlef Smilgies (G-line staff scientist) described the design of temporary multilayer optics for G-line and on commissioning experiments in each of the three hutches (G1, G2, and G3). A beamline was sketched out in January by Fontes, and with used CHESS components, publishable data from G1 were being obtained by April. The four-month plan was expedited by the safety committees and everyone at CHESS hustled to complete the setup in record time. In parallel, a new motion control and data acquisition system based on industrial PCs and PCI boards was commissioned by the G-line team and basic instrumentation was installed in all three G-line hutches. It is hoped that the final G-line optics designed by the G-line group will be fully procured and ready for installation during the planned CESR down period in 2003.

Quan Hao, associate director for MacCHESS, reviewed progress related to the protein crystallography beamlines. At monochromatic wiggler station F1, a pair of dual ADSC Quantum 4 CCD detectors has been installed (Figure 5). The monochromator has also been set to an energy 50 eV above the Br K edge, to allow fast SAD (Single-wavelength Anomalous Diffraction) experiments to be conducted

*Figure 1: At the outdoor CHESS banquet: Sol Gruner (CHESS director) and his wife Rosemarie Parker. In the background, Jun Wang and Rong Huang, both of Cornell.*

with crystals soaked with bromide salts. Liquid nitrogen has been piped to the three crystallography hutches for the on-line low temperature cryocoolers. A supercomputer consisting of a 128-processor Linux-based cluster for data analysis was recently commissioned. As a result of these and other improvements, the average time of each user visit has dropped to 40 hours and the time to collect a data set is typically less than six hours. A FedEx data collection system of mail-in crystallography is fully implemented and is in its first year of trial. There were about 140 MacCHESS-related publications in the last year, including a number of structures making the covers of top-ranked journals.

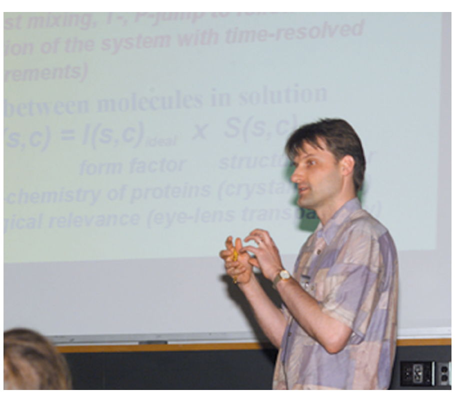


*Figure 2: Gunter Grossmann of Daresbury Laboratory speaking about SAXS for macromolecular studies.*

Alexander Kazimirov, CHESS staff scientist, spoke of progress with multilayer optics. He observed that CHESS has more beamlines running on multilayer optics than any other SR laboratory (5 of 12 stations or 42 percent). CHESS has separate collaborations with Al Macrander's group at APS and with Osmic to develop a number of specialized multilayer optics that are well matched to the CHESS source and its scientific interests. Kazimirov reported test results on low-contrast Osmic multilayers with the number of periods varying from 500 to 800. One particular sample, consisting of 500 bilayers of a 31.5 Å period on a glass substrate, gave a measured reflectivity of 50 percent at 8 keV and a bandwidth (fwhm) of 0.29 percent for deltaE/E (shown in Figure 6). Such high-resolution multilayers would be very useful for crystallography where more flux can be obtained than from a Si 111 type of optic, yet with enough resolution for protein crystallography and perhaps even useful enough for virus crystallography when the development work is finished.

Peter Revesz, CHESS' new instrumentation staff scientist, showed how visible light luminescence from helium gas can be imaged with a video camera and used for X-ray beam position monitoring with good vertical resolution (better than 5 microns). Developments are in progress to utilize the same method for horizontal beam position monitoring. Construction of new permanent monitors for the F and G wigglers and the F3 bending magnet line is underway for the summer shutdown.

Charles Sinclair, project manager for the Energy Recovery Linac project (recently arrived from the Thomas Jefferson National Accelerator Facility (Jefferson Laboratory), gave an update on the Cornell University plans, in collaboration with Jefferson Laboratory, to build a synchrotron light source based on the use of energy recovery [1]. An ERL light source would provide considerably higher average brilliance and far shorter duration X-ray pulses than is possible with a storage ring. However, many challenging accelerator physics and technology issues must be understood and resolved before a high-brilliance, full-energy ERL light source could be built. Cornell University is awaiting a final decision on a proposal to the NSF to build a prototype 100 MeV, 100 mA ERL to explore and develop ERL light source technology.

*Figure 3: Richard Gillilan and Michael Love, both of MacCHESS, discussing a crystallography poster.*

Staff reports were followed by invited talks from users. David Allara (Penn State) discussed molecules as electronic materials, the ultimate terminus of the race for electronic miniaturization. While single molecule transistors were very recently demonstrated, most molecular electronics involve studying many molecules configured as a monolayer on electrode surfaces by either self- or directed-assembly. Recent work has pointed to a strong relationship between the details of molecular organization (i.e. tilt, morphology, etc.) and the

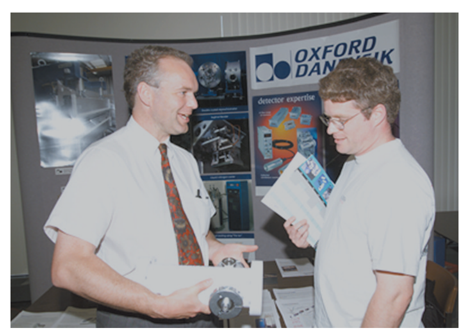


*Figure 4: Andy Broadbent (Oxford Danfysik) and Alan Pauling (Cornell) discussing ion chambers at the vendor's booth.*

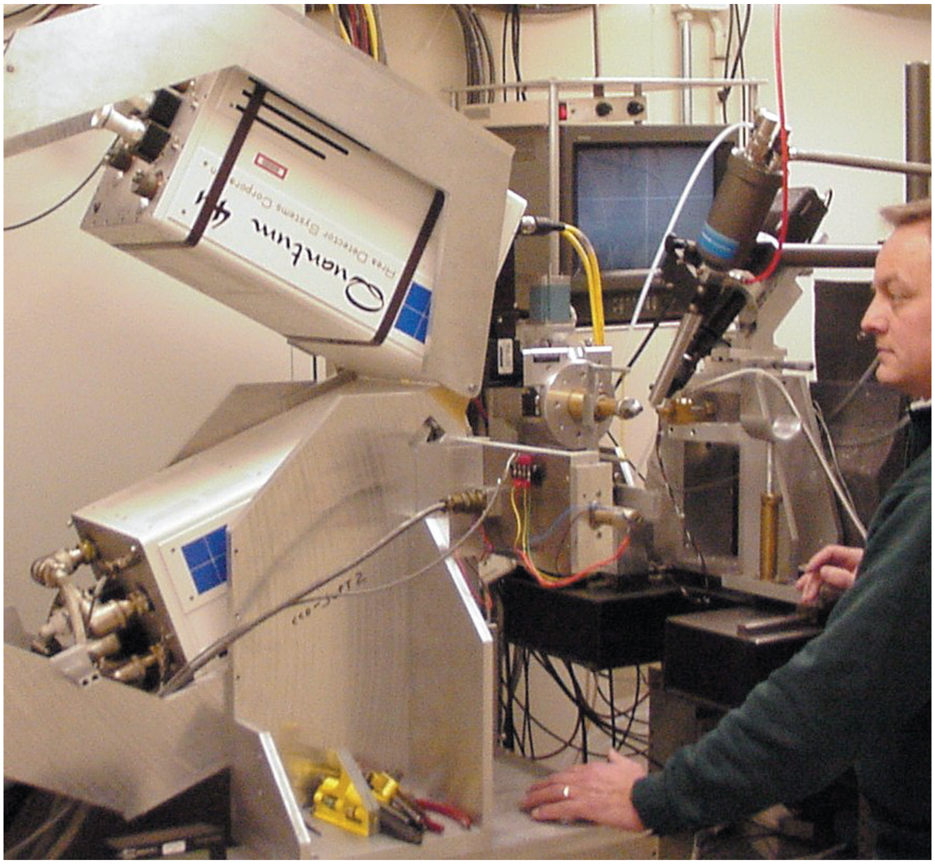

*Figure 5: Installation of a dual Quantum-4 detector system at F1 station by Bill Miller and other MacCHESS staff members. The two detector assembly, designed by Mike Cook, is capable of independent rotation by up to 30 degrees, effectively producing a detector with a total (V-shaped) surface area of 380 x 190 mm.*

operating parameters of the devices. Synchrotron radiation is a highly desirable tool for characterizing the structure and organization of these films.

Brian McClain (Harvard) presented the crystal structure of the rotavirus double-layered particle at 5.5 Å resolution (soon to be extended to 3.8 Å). The particle has an icosahedral shape, with 60-fold symmetry, and a total molecular weight of about 55 million Daltons, one of the largest high-resolution structures ever determined. McClain and co-workers are also determining the structure of the triple-layered rotavirus particle, which is the intact form; the outer layer is stripped off when the virus infects a cell. Rotaviruses include human pathogens and are a leading cause of diarrhea and death due to dehydration in the Third World. A vaccine has been developed, but is associated with adverse reactions when used in infants. Determination of the double- and triple-layered virus structures will illuminate the method of infection by rotavirus and may lead to the development of a better vaccine.

Paul Urayama (Dartmouth) spoke about probing protein substrates using high-pressure X-ray crystallography. Pressures in the range encountered in the biosphere are known to have myriad effects on proteins, but in most cases the molecular basis of these effects is not understood. Urayama and coworkers developed methods to perform protein crystallography at up to 2 kbar and then examined the effects of pressure on conformational substates in myoglobin. It was shown that crystallographic pressure studies can be used to understand the substates that occur upon ligand binding. The methods developed by Urayama and co-workers are relatively easy to use and generally applicable to other proteins. They may well catalyze the general study of the effects of pressure on proteins.

Olivier Thomas (Marseilles) presented his latest high-resolution X-ray diffraction study of 15 to 100 Å ultra-thin Fe films on GaAs (001) substrates. Such thin films have interesting magnetic spin properties and may prove useful for electronic and memory devices. Grazing-incidence reciprocal space maps around the (110) and (200) reflections of Fe, measured at CHESS F3 station, revealed unusual anisotropy in the in-plane domain size and in the in-plane lattice strain in thin films with two distinct directions, along [110] and [1–10]. Such anisotropies and their dependence on film thickness indicate that an interfacial magnetic effect is the only possible explanation for the uniaxial in-plane magnetic anisotropy measured in this system. This result resolves questions that have puzzled magnetic thin-film researchers for more than 15 years.

**Workshop on New Technology and Innovative Applications of Small Angle X-ray Scattering**

SAXS is the second most commonly used technique (after crystallography) at CHESS. The theme of the SAXS workshop was the demonstration of new techniques in the study of large-unit cell materials.

Dr. Adam Finnefrock (Penn) described work (in collaboration with the Wiesner and Gruner groups at Cornell) that used SAXS to determine the structure of novel nanoporous organic-inorganic composites. These materials are synthesized by using diblock copolymers as structure-directing agents. The copolymer phase is swollen by a small-molecule organometallic compound with a strong preference for one of the two polymers making up the copolymer. This provides an effective means

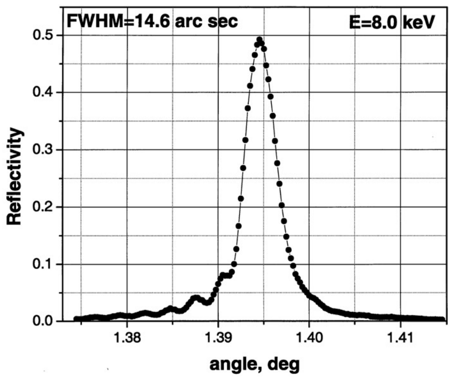


*Figure 6: Reflectivity curve from the Osmic low-contrast multilayer with 500 bi-layers and d = 31.5 Å. The width of the curve is 14.6 arc sec, bandwidth $\Delta E/E = 0.29\%$. Data taken at CHESS A2 station.*

of controlling the effective volume fraction of one of the blocks in the copolymer and allows fine tuning of the resultant phase. The resultant materials form beautiful three-dimensionally periodic structures of the "plumber's nightmare" morphology. The materials can be pyrolized to remove the organic parts, leaving a nanoporous metal oxide framework that is amorphous on atomic length scales and crystalline on the >100 Å scale. These are promising materials for applications ranging from battery electrodes to filtration matrices [2]. Finnefrock explained that the X-ray challenge was to determine the structure on the polycrystalline samples, even though each randomly oriented crystallite is uniquely deformed in a direction perpendicular to the plane of the thin cast-polymer samples. In order to accomplish this, Finnefrock had to invent new methods to analyze and understand the diffraction from strained, polycrystalline samples.

Detlef Smilgies (CHESS) described the use of Grazing Incidence SAXS (GISAXS) to probe the structure of thin polymer and copolymer films spin-coated onto silicon substrates. GISAXS is a very powerful and relatively easy to use method that is generally new to many in the polymer community. Using a wide-bandpass multilayer beam at the CHESS D-1 beamline in combination with a CCD area detector, Smilgies performed GISAXS studies on: (1) the dependence of the orientation of lamellae formed by symmetric diblock copolymers on increasing molecular weight; (2) nanoporous copolymer films formed by UV degradation of one of the blocks; and (3) the formation of a lattice of regular silica spheres in a copolymer in which an organic phase is enriched in one of the polymer components and the polymer is successively removed by thermal degradation. Smilgies showed that GISAXS can be obtained on low-contrast systems and he demonstrated that both lateral and perpendicular information about the thin film structure can be obtained. Even time-resolved studies on a time scale of minutes were possible and first results were presented on the dynamics of a lamellar film when exposed to solvent vapor.

Sol Gruner provided a tutorial on techniques for time-resolved SAXS (TR-SAXS), using numerous examples from the work of his group on polymers, solutions of biomolecules, lipid-water dispersions, and biomembranes. The theme of this talk was to present an outline of the options and decisions that need to be made to record TR-SAXS data in various regimes of scattering strength and time-resolution. Examples included when to use one-dimensional vs. two-dimensional detectors, when multilayer optics are particularly useful, how various custom clocking modes of a CCD detector can improve the signal strength and time resolution of the diffraction patterns, and the use of pixel X-ray detectors for microsecond time-resolved applications.

After lunch, the two workshops merged in order to hear Gunter Grossmann (CLRC Daresbury Lab) describe the use of SAXS for macromolecular studies. Dr. Grossmann's talk is further reported in the next workshop section of *High-Throughput Crystallography & Complementary Methods.*

The final speaker in the SAXS workshop was Ken Finkelstein (CHESS), who described the use of anomalous scattering in SAXS. He first summarized basic principles of the method, including how novel X-ray optics allow two beams of slightly different X-ray energies to cross at the sample and then to be separately collected with an area detector. By tuning the beam energies to span a region just below the absorption edge of a heavy atom in the sample, Finkelstein is able to measure very small difference signals arising from those atoms. He described using this method to study the size distribution of platinum clusters in a zeolite catalyst support, in collaboration with Harry Brumberger (now deceased), Douglas Hagrman, and Jerry Goodisman from Syracuse. He also described a study with Lois Pollack's group (Cornell) on the condensation of counter-ion clouds around nucleotide polymers in solution.

**Workshop on High-Throughput Crystallography and Complementary Methods**

This year's MacCHESS workshop focused on a variety of methods and technologies aimed at improving the overall throughput of X-ray crystallography. The opening speaker, Rob Thorne (Cornell), discussed his recent work at CHESS on understanding why flash-cooled protein crystals tend to have high mosaic spread. This phenomenon causes serious problems for viruses and other systems with large unit cells. The difference in thermal contraction between amorphous ice and protein results in ice pockets within the lattice that lead to the formation of microscopic domains. Thorne suggested that careful matching of the expansion coefficient of the cryoprotectant with that of the protein lattice could lead to better results. X-ray topography was also used to examine the process of crystal annealing. Microdomains caused by flash cooling can be annealed by warming the crystal to just above the water glass transition (150K). Thorne described a device invented in his lab that can warm and cool the cryostream rapidly for this purpose [3].

The second talk of the morning was given by Zbigniew Dauter (NCI and Brookhaven National Laboratory) on the use of Single-wavelength Anomalous Dispersion (SAD) for phasing protein diffraction data. Anomalous scatterers can either occur naturally in the protein (sulfur, phosphorous or transition metals) or can be introduced (selenium or certain halides). Recent advances in both hardware and software now allow structures to be solved on a single (peak) wavelength. High multiplicity in measurements, however, is a key factor for success. Dauter recommended collecting peak data first and attempting to solve the structure on the single wavelength while the next MAD wavelength is still being collected (the so-called 1-1/2 wavelength method). Reprocessing of historical data sets indicates that, in many cases, MAD is not needed. The method is well suited for use in high-throughput environments.

Martine Cadene (The Rockefeller University) introduced the many uses of mass spectrometry in protein crystallography. One of the outstanding barriers to high-throughput crystallography is obtaining crystals. When a protein fails to crystallize, it is often possible do a partial proteolysis to locate structurally compact domains. Matrix-Assisted Laser Desorption Ionization (MALDI) is an excellent tool for following the progress of proteolytic digestions. Proteins of up to 50,000 Daltons can be observed. Cadene presented a special thin layer method for sample spot preparation that enhances sensitivity [4]. MALDI mass spectrometry can also easily distinguish between native protein and selenomethionine derivatives.



Recent innovations in the reconstruction of biomolecular structures from small-angle solution scattering were discussed by J. Gunter Grossmann (CLRC Daresbury Laboratory) in a joint session between the macromolecular and SAXS workshops. X-ray scattering profiles in the 50 to 8 Å range yield information about the overall shape of a protein. Small volumes of protein solutions are used at concentrations similar to conventional crystallography (10 mg/ml or less). Grossmann presented examples with sufficient resolution to observe some conformational changes in proteins during complexation (nitrogenase and $\alpha$-crustacyanin). Molecular shapes can be reconstructed using spherical harmonic expansions or bead models, the latter being capable of resolving some internal structure. Low-resolution molecular shapes have also been used by Grossmann and Hao to help phase diffraction data.

Art Weaver (AJW Research) showcased the new Linux supercomputer he designed in collaboration with Frank Labonte (CHESS and MacCHESS). The 31 diskless dual-processor Athlon nodes are interconnected with a Myrinet 2 Gbit/sec low latency optical switch for high-speed distributed parallel computing. The nodes run the RedHat 7.1 SMP Linux kernel. Parallel tests were performed on SnB (Shake-and-Bake phasing), MPI_FSEARCH (molecular replacement) and WebXDS. Weaver phased a selenomethionine epimerase containing 70 Se sites in 39 minutes, a task that took 17 hours on two processors. This single rack-mounted system, nicknamed Sirius, weighs in at more than 450 Kg (1000 lbs). The power consumption is about 8 kW, with a cooling requirement of more than 27,000 BTU/hr. Weaver warned future cluster builders to always estimate power consumption and cooling requirements, and to seriously consider low-power alternatives such as Green Destiny developed by Los Alamos National Laboratory. Complete details on Sirius, including benchmarks, can be found on the web at staff.chess.cornell.edu/~weaver/sirius.html.

Poster presenters competed for the best posters in the categories of best science, best instrumentation and best graphic design. Members of the workshop executive committee evaluated the posters during the two meeting days and presented awards on the final afternoon. The awards for best science and best graphic design went to Peter Busch, Friedrich Kremer, Christine M. Papadakis (Leipzig), Dorthe Posselt (Roskilde) and Detlef Smilgies (CHESS) for a poster entitled “An X-ray Reflectivity and GISAXS Study of the Lamellar orientation in Thin Diblock Copolymer films.” Best instrumentation went to: “Pixel Array Detector for Microsecond Imaging” by Sol Gruner, Mark Tate, Matt Renzi, Alper Ercan of Cornell University; Sandor Barna and Giusseppe Rossi of Micron Technologies, Inc.; Eric Eikenberry, Paul Scherrer Institute; Bob Wixted; Don Bilderback and Ernie Fontes of CHESS; Jin Wang, Chris Powell, Andrew MacPhee, Yong Yue, and Suresh Narayanan, Advanced Photon Source. Matt Renzi and Alper Ercan received the award for the development team. ■

DONALD BILDERBACK, SOL GRUNER,
AND RICHARD GILLILAN
*Cornell University*

**References**

1. D. Bilderback, I. Bazarov, K. Finkelstein, S. Gruner, G. Krafft, L. Merminga, H. Padamsee, Q. Shen, C. Sinclair, M. Tigner, and R. Talman, *Synchrotron Radiation News,* **14,** May/June 2001, 12–21. Additional ERL information can be found posted at http://erl.chess.cornell.edu/.
2. A. Finnefrock, R. Ulrich, A. Du Chesne, C. Honeker, K. Schumacher, K. Unger, S. Gruner and U. Wiesner, *Angewandte Chemie Int. Ed.* **40** (2001) 1208–1211.
3. S. Kriminski, C. Caylor, M. Nonato, K. Finkelstein and R. Thorne, *Acta Cryst.* **D58** 459 (2002).
4. M. Cadene and B. T. Chait, *Anal. Chem.* **72,** (2000) 5655–5658.