User Facilities of the Office of Basic Energy Sciences

A National Resource for Scientific Research
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About the Cover
The cover depicts several of the scientific user facilities described in this document. The photographs show, in descending order, the Los Alamos National Laboratory Gateway to the Center for Integrated Nanotechnologies, the Spallation Neutron Source and Center for Nanophase Materials Sciences at Oak Ridge National Laboratory, the Center for Functional Nanomaterials at Brookhaven National Laboratory, the Advanced Photon Source and Center for Nanoscale Materials at Argonne National Laboratory, and the Molecular Foundry at Lawrence Berkeley National Laboratory. The pattern of purple and dark balls at the top of the page is an atomic force microscope image of self-assembled nanoscopic domains in a polymer structure. The light blue background is from an electron microscope image of metallic gold islands (blue) selectively decorating polystyrene domains of an ultrathin polymer film. Both of these science images were produced by Seth Darling of the Electronic and Magnetic Materials and Devices Group at the Center for Nanoscale Materials.
The Scientific User Facilities Division (SUFD) sponsors the operation of a number of facilities nationwide that offer the technical tools needed for 21st century science in energy-related disciplines. Thousands of scientists conduct their research at these facilities each year. These researchers experience first-hand the thrill of scientific discovery, as they develop sample materials of scientific interest, guide beams (e.g., of neutrons, synchrotron radiation, or electrons) onto these samples, and detect signals that reveal material properties.

This brochure overviews the scientific infrastructure that is constructed, maintained, and operated at DOE laboratories for the pursuit of energy-related research. Individually and collectively, these BES facilities comprise a user program that strives for excellence in world-leading science and provides innovative, state-of-the-art tools and capabilities to the broad scientific community.

Why Does BES Steward These User Facilities?

The reason for a suite of BES user facilities, and a user program at each, is to enable scientific discoveries in energy-related areas. This fulfills in part the BES mission, which is important not just for researchers, but also for society as a whole. That is, we all benefit from advances that impact our understanding of fundamental energetic processes within materials, because greater scientific understanding enables downstream technological developments for real-world applications.

The way in which scientific research is done in the 21st century involves specialized instrumentation and facilities that are on a larger scale than can typically fit inside an investigator’s own laboratory at his/her home institution. Therefore, user facilities such as those surveyed here are critical to the conduct of modern scientific studies. Indeed, these BES facilities constitute an infrastructure that supports a substantial research enterprise (conducted by over 10,000 users per year) for basic research in topics related to energy. Such research advances the frontiers of knowledge of underlying physical processes, chemical reactions, and material properties that comprise our basic scientific understanding of energetic phenomena.

Scientific Facility User Programs

The vast majority of facility users are granted access via a competitive peer review of their technical proposals in order to select those with the greatest scientific merit. After a research team’s proposal is accepted, that team is granted time at the facility to use its beam and/or specialized instrumentation for scientific experiments. Final research results are reported in the open scientific literature. Each facility has a user program to describe its specific offerings (e.g., characteristics of the specialized instruments available for use) and to administer the proposal submission and review process for prospective users.

Open To Broad Scientific Community Via User Programs

Each facility described in this publication operates as a national user facility, with capabilities (e.g., state-of-the-art equipment and technical expertise) that are available to prospective users from academia, industry, government agencies, and research institutions worldwide. Collectively, these users conduct research in a wide variety of disciplines. The vast majority of users gain access to BES user facilities via a merit-based peer review process.

1The SUFD is in the Office of Basic Energy Sciences (BES), part of the Office of Science (SC) in the U.S. Department of Energy (DOE).
The user program of each facility administers its peer review process to evaluate scientific proposals for access. Calls for these proposals take place at times indicated on each facility’s website. Scientific proposals submitted in response are typically quite brief and may propose independent or collaborative research. They are evaluated for scientific merit by independent proposal review committees or panels and for feasibility and safety by the facility, with those that are most compelling being accepted and allocated time. There is no charge for users who are doing non-proprietary work, with the understanding that they are expected to publish their results. Access is also available on a cost-recovery basis for proprietary research that is not intended for publication. After proposals are reviewed and time at the facility is awarded, the user program helps users prepare to visit the facility in order to conduct their research. Further information on proposal submission and other aspects of the user program is available on each facility’s website.

External Advisory Groups of Scientific User Facilities

In addition to the independent proposal review bodies mentioned above, each facility has other established ways to receive advice and input on its activities. Each facility Director receives input from a Scientific Advisory Committee. Each facility also has a Users’ Executive Committee, or equivalent, which fosters information exchange and communication on issues that are of interest to the user community. Each facility’s operations are independently reviewed as well by external experts convened by BES on a periodic basis.

BES Mission

The Office of Basic Energy Sciences (BES) supports fundamental research to understand, predict, and ultimately control matter and energy at the electronic, atomic, and molecular levels in order to provide the foundations for new energy technologies and to support DOE missions in energy, environment, and national security.

As part of its mission, BES conceives, plans, designs, constructs, and operates scientific user facilities to probe the most fundamental electronic and atomic properties of materials at extreme limits of time, space, and energy resolution through x-ray, neutron, and electron beam scattering and through coherent x-ray scattering. Properties of anticipated new x-ray sources include the ability to reach to the frontier of ultrafast timescales of electron motion around an atom, the spatial scale of the atomic bond, and the energy scale of the bond that holds electrons in correlated motion with near neighbors.

Plan, administer, and fund the design, construction, and operation of major scientific user facilities to serve researchers from the university, national laboratory, and industrial research communities; and

Act as a steward of human resources, essential scientific disciplines, institutions, and premier scientific user facilities.

www.sc.doe.gov/bes/

Dr. Pedro A. Montano
Director, Scientific User Facilities Division

Dr. Harriet Kung
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Unlocking the World of the Ultra Small and the Ultra Fast

X-rays are an essential tool for studying the structure of matter and have long been used to peer into dense material through which visible light cannot penetrate. For instance, the x-rays used in medicine and dentistry reveal bones and dense tissues, but use a broad, relatively weak pulse of x-rays. Today’s synchrotron “light-source” facilities produce x-rays that are millions of times brighter than medical x-rays. Scientists use these highly focused, intense beams of x-rays to reveal the identity and arrangement of atoms in a wide range of materials. The tiny wavelength of x-rays allows us to see things visible light cannot resolve, such as the arrangement of atoms in metals, semiconductors, ceramics, polymers, catalysts, plastics, and biological molecules.

The fundamental tenet of materials research is that structure determines function. The practical corollary that converts materials research from an intellectual exercise into a foundation of our modern technology-driven economy is that structure can be manipulated to construct materials with particular desired behaviors. To this end, synchrotron radiation has transformed the role of x-rays as a mainline tool for probing the atomic and electronic structure of materials and their surfaces. From their discovery in 1895, x-rays have tantalized scientists with their capability to reveal the interior structures of solid objects. For nearly a century, they have also been our principal means of unraveling the positions of atoms in crystallized solids, from the comparatively simple structures in metals and semiconductors to the highly complex arrangements in biological molecules, such as proteins and DNA. During the last four decades, however, the growth of synchrotron radiation with its bright, wavelength-selectable x-rays has markedly expanded the scope of investigation. The result for materials research is a tool that can probe in minute detail the interior and surface of all manner of samples, large and extremely small, including non-crystalline and inhomogeneous materials.

From its first systematic use as an experimental tool in the early 1960s, synchrotron radiation has vastly enhanced the utility of pre-existing and contemporary techniques, such as x-ray diffraction, x-ray spectroscopy, and imaging, and has given rise to scores of new ways to do experiments that would not otherwise be feasible, or even possible, with conventional x-ray sources. Synchrotron radiation is, in the newest facilities, 1 billion times brighter than the light from conventional x-ray tubes. Moreover, the wavelength can be selected over a broad range to match the needs of particular experiments. Together with additional features, such as controllable polarization (both linear and circular), coherence, and ultrafast pulsed time structure, these characteristics make synchrotron radiation the x-ray source of choice for a wide range of materials research.

The pages that follow describe a suite of light sources that generate synchrotron radiation using electron beams. Each operates as a user facility open to scientists for the conduct of their research.

Synchrotron Radiation Sources

Synchrotron Radiation is a Useful Probe of Material Properties

Synchrotron radiation is produced by acceleration of charged particles (e.g., electrons) that are traveling at speeds close to that of light. In the facilities discussed here, the synchrotron radiation comes from electrons in a storage ring. Application of a magnetic field (via a bending magnet, or a periodic array of magnets configured as an “insertion device” such as an undulator or wiggler) produces acceleration of the electrons (in directions transverse to their direction of motion) and concomitant emission of synchrotron radiation in a broad spectrum of energies (spanning the hard and soft x-ray regions as well as ultraviolet and infrared radiation). This radiation is captured and sent down beamlines where it impinges upon a material sample of interest. Signals such as the scattered photons are then detected and analyzed by researchers in order to deduce structural properties of their material.
The atomic arrangement of graphene (background) is a honeycomb lattice of carbon atoms arranged in a two-dimensional plane. Its electronic band structure consists of two bands (yellow) that intersect only at a few points at the corners of a hexagonal Brillouin zone (red).

The National School of Neutron and X-ray Scattering

The National School of Neutron and X-ray Scattering is an annual two-week course funded by BES. The idea for such a school was first proposed and organized by Argonne National Laboratory (ANL) in 1999. The school provides 45–60 graduate students attending U.S. universities a comprehensive introduction to the underlying scattering theory and experimental techniques that are available at DOE-funded neutron and x-ray facilities. The students receive a series of background lectures given by leading international experts (recruited from universities, national laboratories, and industry) and conduct hands-on experiments. For the first nine years, the school was hosted at ANL with experiments being performed using instruments at ANL’s user facilities. As of 2008, the school is being jointly hosted by ANL and Oak Ridge National Laboratory, enabling the use of High Flux Isotope Reactor and Spallation Neutron Source instruments for neutron scattering experiments. The school has been extremely successful in attracting high-quality applicants from universities with a wide geographical, academic, and demographic distribution across the United States, and consistently has 2–3 times more applicants than space available. Despite the heavy workload, student feedback has been uniformly positive and many students have applied as a result of word-of-mouth recommendations from alumni. The school provides a unique grounding in both neutron and x-ray scattering techniques, revealing the complementarity of the two, and as a result is highly valued by many university research groups. Most alumni have continued as users of the scientific user facilities when they move on to postdoctoral and permanent research positions. The school plays an important strategic role in preparing the scientific community to take full advantage of the exciting new research opportunities that can be pursued with neutron and synchrotron facilities.
The Advanced Light Source (ALS) is a national user facility that generates intense x-ray radiation for scientific and technological research. The ALS is operated by the Lawrence Berkeley National Laboratory (LBNL) of the University of California for the U.S. Department of Energy. Operation for users began in October 1993. The ALS synchrotron radiation source welcomes about 2000 scientists per year from universities, industries, and government laboratories around the world to use its portfolio of advanced insertion-device and bend-magnet beamlines for research broadly spanning the world of science and technology.

**ALS Capabilities**

The Advanced Light Source is optimized for high brightness at soft x-ray and ultraviolet photon energies with undulator sources. It also provides world-class performance in the intermediate-energy and hard x-ray regions with wiggler, superconducting bend-magnet (superbend), and normal bend-magnet sources. Infrared radiation is available, as well.

Soft x rays and VUV are ideal tools for probing with high resolution the electronic and magnetic structure of atoms, molecules, and solids, such as high-temperature superconductors, as well as for studying solid surfaces and the molecules adsorbed on them. The high brightness and coherence makes the ALS particularly useful for soft x-ray imaging (e.g., biological structures, environmental samples, polymers, magnetic nanostructures, and other inhomogeneous materials), coherent (lensless) imaging, holography, and interferometry. The pulsed nature of the ALS light also offers special opportunities for time-resolved research, from the dynamics of phase transitions between different states of matter to that of the electronic processes that give rise to the basic properties of matter.

In the hard x-ray range, experimental stations for structural molecular biology at the ALS provide world-class facilities for x-ray crystallography of proteins and other biological macromolecules. Facilities for x-ray microtomography and ultrahigh-pressure research are also available. Among new experimental facilities, one with state-of-the-art capabilities in the production and use of femtosecond x-ray pulses is now in operation, as are beamlines for studying magnetism and magnetization dynamics and for in situ studies of systems with photon-in/photon-out and photon-in/electron-out techniques.

Core capabilities in which the ALS is a world leader with multiple beamlines and end stations available for user research are grouped below, along with several examples of the science performed and possible measurements.

**Microscopy**

Imaging instruments at the ALS serve beamlines for scanning transmission x-ray microscopy (STXM), full-field transmission x-ray microscopy (TXM), photoemission electron microscopy (PEEM), and infrared microscopy. There is also a beamline for the growing field of coherent (diffractive) imaging.

PEEM images reveal how magnetic domains on interfaces between layers of magnetic nanostructures (on which computer hard disks store data) interact with each other and how these interactions are correlated with elemental and chemical composition of the materials—details critical to the continued development of next-generation data-storage devices. Installation of an aberration-corrected mirror in the new PEEM3 microscope will improve its spatial resolution from approximately 30 nm to less than 5 nm and will increase transmission by more than an order of magnitude.

A bend-magnet beamline with a transmission x-ray microscope (TXM) for the “National Center for X-Ray Tomography” is used for high-resolution soft x-ray microscopy and tomography of whole, hydrated biological cells. The research program at NCXT includes the development of novel methods for labeling proteins, both for x-ray and optical microscopy.

**Electron Spectroscopy**

Electron spectroscopy instruments serve beamlines for x-ray photoemission spectroscopy (XPS), angle-resolved photoemission spectroscopy (ARPES), and ambient pressure x-ray photoemission spectroscopy (APXPS).

Angle-resolved photoemission spectroscopy (ARPES) is one of the most direct methods of studying the electronic structure of solids and has emerged as a leading technique for understanding the electronic structure of high-temperature superconductors and other complex oxides. Ambient pressure x-ray photoemission spectroscopy (APXPS), available at several ALS beamlines, allows measurements of processes such as catalysis and corrosion and of environmental and biological systems at pressures up to 10 Torr.
Photon Spectroscopy and Scattering

Instrumentation for photon spectroscopy serves beamlines for resonant inelastic x-ray scattering (RIXS), small-angle/wide-angle x-ray scattering (SAX/WAX), x-ray absorption spectroscopy (XAS), and near edge x-ray absorption fine structure spectroscopy (NEXAFS).

X-ray absorption spectroscopy (XAS) combined with magnetic dichroism and the ability to modify the polarization of the x-rays and the angle of the applied magnetic fields is a powerful tool for understanding magnetic materials. A research team from Cambridge, UC Berkeley, and the ALS has now shown unambiguously that, contrary to common belief, the spectral shape and magnitude of x-ray magnetic linear dichroism (XMLD) signals are determined by not only the relative orientation of magnetic moments and x-ray polarization, but also their orientation relative to the crystallographic axes. Such complete orientation information is needed in order to accurately interpret the data.

Diffraction

Diffraction instruments serve beamlines for protein crystallography (PX), x-ray microdiffraction, and small-molecule x-ray diffraction.

Protein crystallography (PX) has proven critical to understanding the function of macromolecular biological mechanisms. Using PX at a number of ALS beamlines, a team of scientists from Washington, Indiana, and Cambridge has shown how Auxin, a plant hormone, acts as a ‘molecule glue’ to bring together two proteins to target the destruction of defective proteins.

X-ray microdiffraction, available on an ALS high-energy x-ray superconducting bend magnet beamline, uses white x-rays focused to a sub-micron spot. Phase, grain orientation, stress, defect density, and other parameters are derived from Laue diffraction patterns, and 2D maps are built up by sample scanning. This technique gives structural information on the microscopic spatial scale that is important for understanding materials properties such as strength and fatigue resistance.
Time-Resolved Spectroscopy
A number of ALS beamlines provide the capability of time-resolved spectroscopy, from a timescale of nanoseconds to femtoseconds. Researchers from Oxford and the ALS have measured the ultrafast dynamics of the photo-induced atomic rearrangements in vanadium dioxide using NEXAFS on the femtosecond timescale.

Accomplishments
Basic Research with Wide-Reaching Applications. Over the course of its first 15 years of operation, the ALS has matured and produced an enviable record of scientific output broadly spanning the physical sciences, energy sciences, biosciences, environmental sciences, and general sciences, often with implications for wide-reaching application in energy, environmental cleanup, health, and technology. The quality of the science is indicated by the recent awards received by its users, including a Nobel award in chemistry, two Davisson-Germer Prizes, an A.H. Compton Award, a Langmuir Prize, a MacArthur Foundation Genius Award, and a Priestly Award.

Facility Innovations. Such outstanding science depends on a continuous development and renewal of resources, ranging from the accelerator itself to research instrumentation and techniques. Some key innovations and accomplishments by ALS or Berkeley Lab partners include:

• Superconducting bend magnets (superbends) expand the spectral range of the ALS without using up straight sections best reserved for undulators. Superbends have made the ALS a world-class protein crystallography source and added high-pressure, microdiffraction, and tomography capabilities.
• X-ray pulses down to the 100-fs time scale are produced by the laser slicing technique. First developed in conjunction with Berkeley Lab’s Center for Beam Physics on a bend-magnet beamline, laser slicing is now implemented on undulator beamlines for time-resolved diffraction and spectroscopy processes at time scales at which atomic motion of atom occurs.
• Advanced x-ray optics research (also by Berkeley Lab’s Center for X-Ray Optics) has yielded world-leading high-resolution diffractive lenses for x-ray microscopy of solids and biological structures.
• Photoemission electron microscopy (PEEM) innovations include pioneering work both in development of PEEM instrumentation and its use to study magnetic nanostructures by x-ray magnetic circular and linear dichroism spectroscopic and time-resolved modes.
• Top-off (quasi-continuous beam injection) provides dramatic increases in brightness, stability, and effective lifetime that benefit users in all fields.

Remaining at the Forefront
The 21st century requires us to take science beyond mere observation. It is important to be able to design materials and processes that have the required properties to meet humanity’s needs. This requires an ability to see functionality at the relevant length, time, and energy scales and then to go beyond probing to controlling matter and energy at the molecular, atomic, and subatomic levels. Over the next 15 years, an ambitious upgrade program is planned to renew the facility and keep it at the cutting edge of synchrotron science with higher spatial, temporal, and spectral resolution than is now available.

Accelerator Upgrades
The ALS produces light over a wide spectral range for users. The quality of the photons is intimately connected to the performance of the accelerator complex. In addition to the imminent migration to top-off operation, proposed upgrades will involve the following:

• New instrumentation for improved photon-beam stability and control,
• Replacing outmoded components for enhanced reliability of the accelerator complex,
• A lattice upgrade for higher horizontal brightness, and
• A novel storage ring operational mode to serve multi- and single-bunch users simultaneously.

Advanced Light Source (cont.)
**Insertion Devices**

Of the ten straight sections available for insertion devices, five are occupied by the original full-length planar undulators. To enhance the utilization of the straight sections, future advanced insertion devices with polarization control, higher brightness, and extended spectral range will, like current EPUs, be short enough to permit two in each straight section. In addition to EPUs, other devices would include a short-period undulator for harder (12 keV) photons. Advanced insertion devices pose challenges. Those that we intend to explore include the following:

- Non-mechanically-moving EPUs and/or improved field shapes, and
- Superconducting undulators for achieving ultimate brightness in the soft x-ray range and for significantly higher brightness than achievable with wiggler sources at 12 keV.

**Bringing New Beamlines into Operation**

The ALS is a field-leader in core technology areas of spectroscopy, imaging, scattering, and ultrafast experimental techniques. Renewing and upgrading the key instruments in our core areas will allow the users to examine and characterize matter and energy at scales never before achieved. Among beamlines currently in the commissioning, construction, or advanced design stage are the four summarized below.

**The Ultrafast X-ray Facility (Beamlines 6.0.1 and 6.0.2).** This new facility consists of two undulator beamlines—one for hard x rays (6.0.1), the other for soft x rays (6.0.2). The facility is dedicated to time-resolved experiments on the femtosecond and picosecond time scales, increasing the flux of femtosecond x rays at the ALS a thousand-fold over an existing bend-magnet beamline. Many of the ultrashort pulse x-ray techniques to be developed on these beamlines will find direct applications in the experimental program at the Linac Coherent Light Source (LCLS) and will also provide unique capabilities in spectroscopy that will not be available at free-electron laser (FEL) sources.

**The Milli-Electron Resolution BeamLINE (MERLIN, Beamline 4.0.1).** This beamline provides high energy-resolution capabilities for photoemission and inelastic scattering experiments, which are crucial for understanding the behavior of strongly correlated systems such as conventional and high-temperature superconductors and magnetic systems. The MERLIN beamline has polarization control from a quasiperiodic elliptically polarizing undulator (EPU) in the VUV energy range below 150 eV.

**COherent Scattering and diffraction MICroscopy (COSMIC, Sector 7).** The COSMIC beamline will provide coherent light in the 0.5–3 keV range with polarization control. One branch of this beamline will serve the soft x-ray coherent scattering community, where much of the interest is in magnetic phenomena. The other branch will be devoted to diffraction microscopy. This new form of lensless imaging is designed to provide 3D structures to 10-nm resolution for frozen hydrated biological specimens and even higher resolution where radiation damage is not a limitation. This will be a major new venture in the use of soft x rays as a nanoscale probe.

**Microscopy And Electronic STRucture Observatory (MAESTRO, Sector 7).** The MAESTRO beamline will illuminate a new nanoARPES instrument specializing in valence photoemission spectroscopy in the energy range 20–600eV. Small probe sizes enable the study of systems such as isolated homogeneous regions within inhomogeneous samples, small specimens of materials that are manageable in minute quantities but hazardous in larger quantities (e.g., transuranics), surfaces and structures that cannot be prepared in larger formats, and nanostructures created in the Molecular Foundry.

**Contact**

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The National Synchrotron Light Source (NSLS) is one of the most prolific scientific user facilities in the world. Each year, about 2,200 scientists from more than 400 universities, government laboratories and companies use its bright beams of light for research in such diverse fields as biology, physics, chemistry, geology, medicine, and environmental and materials sciences. While the majority comes from academic institutions, about 7 percent of NSLS users are from industries.

The NSLS operates two electron storage rings: X-Ray (2.8 GeV) and Vacuum Ultraviolet-Infrared (VUV-IR) (800 MeV). These two rings provide intense light spanning the whole electromagnetic spectrum—from very long wavelength infrared radiation to ultraviolet light and super-short wavelength x-rays. Scientists use these intense light beams to study the electronic, chemical, and structural properties of materials at the atomic level. For example, researchers have used the NSLS to examine the minute details of computer chips, decipher the structures of viruses, probe the density of bone, determine the chemical composition of moon rocks, and reveal countless other fascinating aspects of science.

The facility operates 65 beamlines, with 52 beamlines on the x-ray ring and 13 beamlines on the VUV-IR ring. These beamlines are optimized for high-resolution atomic structure determination, two- and three-dimensional chemical and magnetic imaging with micron or sub-micron spatial resolution, as well as high-energy resolution spectroscopy. They are also equipped with state-of-the-art experimental apparatus to allow the study of materials under extreme temperatures, pressures, and magnetic fields. In addition, the intense synchrotron light enables the study of very small or very dilute samples and high-throughput measurements that cannot be carried out with laboratory-based light sources.

IBM researcher Simone Raoux with a wafer that contains millions of new computer memory switches. Raoux performs research partly with time-resolved x-ray diffraction at the NSLS to develop a material that could lead to a new kind of computer memory chip used for storing digital media. Photo courtesy of Noah Berger/The New York Times/Redux.
Summary of Experimental Techniques Available at the NSLS

The wealth and variety of experimental techniques available to NSLS users is characterized by the wide photon energy range covered by two storage rings, spanning the far infrared to the very hard x-ray region. Most techniques, and the instruments that enable their performance, are associated with a particular photon energy range. Thus, the wide energy range enables the NSLS to offer to its users a correspondingly wide variety of experimental techniques, which are grouped below into five major categories: diffraction and scattering, macromolecular crystallography, microspectroscopy and imaging, spectroscopy, and other. The figure is a graphical presentation of the distribution of experimental techniques provided at the 65 operating beamlines around the two NSLS storage rings.

Each of the five general categories listed above encompasses a number of more specific experimental methods. These specific methods restrict themselves to certain ranges of experimental parameter space and are generally named by this specialization, e.g. angle-resolved or time-resolved variants of the generic techniques. A description of the five general categories, including several specific techniques, is provided on the following page.
Diffraction and Scattering
This category consists of a large set of photon-in, photon-out experimental techniques, encompassing specific methods distinguished by momentum transfer, angular range, incident photon energy, surface vs. bulk, degree of sample order, and degree of coherence. Zero energy transfer techniques include most types of diffraction, elastic scattering, and reflectivity, whereas non-zero energy transfer techniques are usually labeled inelastic scattering. Wide-angle x-ray scattering or diffraction (WAXS, WAXD) and small-angle x-ray scattering (SAXS) are labeled by the angular range of the scattered x-rays relative to the incident x-ray direction. X-ray scattering of photons with energy selected at an absorption peak of the sample is referred to as resonant x-ray scattering, whereas scattering at other photon energies is called non-resonant x-ray scattering. Surface x-ray scattering or diffraction is confined to the surface and near-surface region, e.g. using grazing angle of incidence. Diffraction or scattering from single crystals is distinguished from powder diffraction from an ensemble of small crystals (powders). If the incident x-ray beam is spatially coherent in the transverse plane, scattering of this beam is called coherent x-ray scattering, as compared to incoherent x-ray scattering when the phase space of the incident beam is significantly larger. Other variants include time-resolved scattering or diffraction, magnetic scattering, and liquid scattering.

Macromolecular Crystallography
Macromolecular crystallography (MX) refers to x-ray diffraction from a crystallized array of macromolecules, where macromolecules have tens to hundreds of thousands of atoms in the unit cell. Macromolecules include proteins, viruses, and nucleic acids.

Microspectroscopy and Imaging
The techniques in this category either utilize (1) tiny, focused beams of synchrotron radiation, where the sample is raster-scanned through the beam to generate an image, or (2) “full-field” illumination where large beams of synchrotron radiation are used to flood a sample and a pixel-array detector is used to capture the image. Many of these techniques also permit imaging as a function of incident photon energy, i.e., as a spectroscopy, hence the term microspectroscopy. The specific techniques in this category include FTIR-based infrared microspectroscopy (FTIRM), soft x-ray scanning transmission x-ray microscopy (STXM), photoemission electron microscopy (PEEM), x-ray microtomography, micro-diffraction imaging, diffraction enhanced imaging (DEI), x-ray topography, and x-ray fluorescence microprobe (μXRF).

Spectroscopy
This category encompasses all techniques that vary the incident photon energy as the principal experimental variable, except microspectroscopy techniques. The largest specific technique in this category is x-ray absorption spectroscopy (XAS), which strictly refers to the transmission through the sample but is generally extended to include electron and fluorescence yield spectroscopies. Close variants of XAS include extended x-ray absorption fine structure (EXAFS) spectroscopy and near-edge EXAFS (NEXAFS). Electron spectroscopies are included in this category, such as ultraviolet photoelectron spectroscopy (UPS) and x-ray photoelectron spectroscopy (XPS), angle-resolved photoelectron spectroscopy (ARPES), and spin-resolved spectroscopy. Other specific techniques in this category include UV circular dichroism (UVCD) and soft x-ray magnetic circular dichroism (MCD), IR spectroscopy, time-resolved spectroscopy, magneto-spectroscopy, and diffraction anomalous fine structure spectroscopy (DAFS).

Other
A few specific techniques do not fall into any of the other four categories. At the NSLS, these techniques include metrology, radiometry, and x-ray footprinting.

Accomplishments
NSLS users and staff produce more than 900 publications per year. Of these, about 20 percent are published in premier scientific journals with high impact. The most notable example is work by Roderick MacKinnon, a visiting NSLS researcher from The Rockefeller University, who shared the 2003 Nobel Prize in Chemistry for explaining how one class of proteins helps to generate nerve impulses.

The NSLS also supported work linked to the 2008 Nobel Prize in Chemistry, which was awarded to two American scientists and a Japanese researcher for their discovery and
development of a protein called green fluorescent protein (GFP). GFP, which is found in a type of jellyfish and glows green under ultraviolet light, has become an extremely popular tool for probing the insides of living cells or whole animals and watching molecules interact in real time. The structure of GFP was first solved with the help of x-ray studies at NSLS beamline X4A and reported in the journal Science in 1996. Roger Tsien, who won a third of the Nobel, was an author on that seminal paper (Publication: Ormø, M., Cubitt, A.B., Kallio, K., Gross, L.A., Tsien, R.Y., and Remington, S.J., “Crystal Structure of Aequorea victoria Green Fluorescent Protein,” Science 273: 1392-1395 (1996)).

The NSLS also has won nine R&D 100 Awards for innovations including a closed-orbit feedback system, x-ray microscopes, and the first device able to focus a large spread of high-energy x-rays. Other significant contributions made to the field of synchrotron radiation include: the Chasman-Green Lattice, the storage ring design that is still used at light sources worldwide; resonant x-ray scattering techniques for probing magnetism; the development of coherent x-ray diffraction (or “lensless”) imaging, which allows the 3-D visualization of non-crystalline objects with nanometer-scale resolution; and pioneering free electron laser research.

In addition, scientists have used the NSLS to:

- develop a method for breast cancer detection that is more accurate than mammography
- study the structure and composition of plaques in Alzheimer’s disease using infrared and x-ray imaging techniques
- study the role of zinc in the development of age-related macular degeneration, the leading cause of blindness among elderly people in the developed world
- look for signs of life in comet and space dust collected by NASA’s Stardust spacecraft
- examine material dredged from the Port of New York/ New Jersey to determine the nature of pollutants in the sediment
- probe electrodes and electrolytes in lithium-ion batteries with the aim of improving their performance
- investigate magnetic materials to make better recording devices
- probe the properties of high-temperature superconductors, materials that conduct electricity with almost zero resistance
- explore new techniques for making denser, faster computer chips

The crystal structure of the potassium-ion channel, which was determined based on data partially collected at the NSLS. The structure, for which NSLS user Roderick MacKinnon received the 2003 Nobel Prize in Chemistry, is an important step in understanding how ion channels produce and control cellular electrical activity, which regulates hormone secretion, controls heartbeats, and drives the nervous system.

**Future Technology**

The NSLS has continually updated its technology and expanded its scientific capabilities since its first operations in 1982. As the boundaries of scientific discovery have been expanded, however, many researchers are looking for additional capabilities beyond those that can be provided by the NSLS or any other synchrotron in the world. As a result, Brookhaven has proposed building a replacement for the present facility – NSLS-II. The new facility will be a state-of-the-art, world-leading synchrotron that will produce x-rays up to 10,000 times brighter than those generated by the current NSLS. Its superior capabilities will reinforce U.S. scientific leadership, enabling scientists to explore the challenges they face in developing new materials with advanced properties.

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The Advanced Photon Source (APS) at Argonne National Laboratory is a third-generation storage ring source. This facility is optimized to produce radiation not from the bending magnets, which force the electrons to maintain their path on a closed orbit, but rather from insertion devices (IDs). The APS produces radiation in the hard x-ray range (from a few kiloelectron volts to 100’s of kiloelectron volts). The high-brightness x-rays in that energy range make the APS an ideal source for materials research because (1) the wavelength of the radiation is on the order of the interatomic distances of atoms in materials and, therefore, can be used for scattering experiments for the determination of the structure of materials; (2) the energy of the x-rays is in the right range to ionize (remove) core electrons from materials for spectroscopic studies of materials or to perform element-specific studies of materials; and (3) x-rays of this energy range are penetrating and so can be used to study bulk properties of materials, buried or liquid/solid interfaces, and/or materials in ovens, pressure cells, in situ reaction cells, etc. In addition, the x-ray beam is sufficiently intense that time-resolved studies can be carried out that follow the evolution of materials as they change under the influence of temperature, pressure, magnetic field, etc.

High-energy X-ray Scattering

High-energy x-rays (i.e., 50 keV to 120 keV) are extremely useful in the study of a variety of materials and a wide range of scientific areas, with the largest use being in applied materials science, in particular in the area of stress, strain, and texture measurements. Dedicated high-energy x-ray scattering capabilities at the APS include traditional aggregate strain/texture measurement capabilities, but also a three-dimensional x-ray diffraction microscope that is used to measure the diffraction properties of the individual single grains that make up polycrystalline materials. This is of great interest because the mechanical behavior of most structural materials is largely determined by a complex combination of the mechanical behavior of individual grains and the interaction between individual grains. Nearly all measurements of the mechanical properties of materials are made on bulk material as a whole (i.e., the aggregate of the single grains), and hence only average information is obtained. Following changes in materials grain-by-grain while they are exposed to physical transformations validates and extends our understanding of fundamental materials properties and has many implications for manufacturing.

The high-energy beamlines at the APS often utilize undulator radiation that is optimized for high brightness with full tunability in the high-energy x-ray regime. Special optics are available for a variety of focusing modes (down to a few microns if desired) and for high energy resolution. Primary areas of research are:

- Measurement of stress, strain, and texture from polycrystalline aggregates,
- High-energy diffraction microscopy to study single grains in polycrystals,
- Pair distribution function (PDF) measurements of non-crystalline materials, and
- Measurements of samples under extreme (high-pressure and/or high-temperature) conditions.

Facilities for high-energy imaging and high-energy small-angle scattering are also available for users.
Inelastic X-ray Scattering

Depending on the energy resolution, inelastic x-ray scattering (IXS) techniques can provide insight on vibrational and elastic properties (high-energy-resolution IXS), dynamical properties and elementary excitations of electrons (medium-energy-resolution IXS), and the chemical properties of low atomic number materials under extreme conditions of pressure and temperature: (low-energy-resolution IXS or x-ray Raman scattering).

If the sample has a specific type of nuclear isotope that can be excited with x-rays, nuclear resonant (Mossbauer) inelastic x-ray scattering measurements can be made that provide information on vibrational and elastic properties, such as the phonon density of states and sound velocities. All these experiments have become possible due to the characteristics of third-generation synchrotron radiation sources such as the APS.

At the APS, instruments have been specifically designed to perform these types of experiments. HERIX, a High-Energy-Resolution (1.5 meV at 23.7 keV) Inelastic X-ray scattering spectrometer, is used predominantly for studies of collective excitations in solids and liquids. Research areas include phonons in solids; sound modes in complex fluids; collective vibrational dynamics of polymer molecules; protein dynamics; and electron-phonon coupling, particularly in superconductors. MERIX, a Medium-Energy-Resolution (50-200 meV at 5 to 12 keV) Inelastic X-ray scattering spectrometer, is used for collective electronic and magnetic excitations (e.g., plasmons, charge-transfer excitations, d-d excitations, double magnon excitations, etc.). The targeted energy resolution is ~100 meV, covering the K-absorption edge of vanadium to zinc. MERIX is presently the best-in-class resonant inelastic x-ray scattering spectrometer regarding count-rates, energy resolution, and available analyzers. LERIX, the Lower-Energy-Resolution Inelastic X-ray scattering spectrometer, allows high-energy, nonresonant experiments to be performed. Numerous experiments have been carried out, including measurements on Li-metal oxide electrode materials in Li-ion batteries, on isomers of a carborane molecule (which may be a novel component in future cancer therapies), and on carbon “nano-onions” (which have been proposed as an important constituent of interstellar dust).
Imaging with Hard X-rays

X-ray imaging capabilities at the APS cover a wide range, from x-ray micro- and nano-probes (scanning techniques) to full-field techniques such as transmission x-ray microscopy and microtomography.

The hard x-ray nanoprobe at the APS utilizes x-rays with photon energies between 3 and 30 keV to study nanostructures materials and devices at the highest spatial resolution. The instrument takes advantage of the high brilliance at high photon energy provided by the APS to produce diffraction-limited focal spots for scanning probe microscopy. Photon energies in the hard x-ray portion of the electromagnetic spectrum are ideally suited for imaging with contrast mechanisms such as x-ray fluorescence and x-ray diffraction. These contrast mechanisms allow researchers to characterize and quantify elemental constituents of materials with sensitivity to individual nanoparticles, and to observe atomic-scale structural properties of nanoscale materials and systems, such as crystal structure and strain. Hard x-rays also allow penetration of overlayers and windows of environmental sample chambers (e.g., high-pressure diamond anvil cells and high-temperature furnaces). The nanoprobe will provide an initial spatial resolution of 30 nm—the size of 100 atoms. It is anticipated that significantly higher spatial resolution will be reached over the lifetime of the nanoprobe as x-ray optics continue to improve.

Full-field imaging allows for efficient two- and three-dimensional visualization of complex systems and devices. A recently completed, dedicated full-field imaging beamline was constructed at the APS to satisfy the rapidly growing need for advanced x-ray imaging investigations in materials science and biology, and to promote research and development to advance the cutting-edge in x-ray imaging. The facility currently supports research programs in static and dynamic phase-contrast imaging, ultra-small-angle-scattering imaging, and transmission x-ray microscopy. The high x-ray flux offered by the APS undulator allows high-definition, phase-enhanced imaging with exposure times as short as 200 μs in the monochromatic mode, and down to ultrafast single-pulse temporal resolution of ~150 ps in the white-beam mode, with simultaneous spatial resolution in the 1- to 5-μm range. These unique capabilities open new research areas in synchrotron x-ray imaging applications, from ultrafast imaging of materials processing and transient fluid dynamics to real-time and sub-video-rate imaging of biological functions in live insects and small animals.

The APS User Community

Given the vast range of capabilities of the APS, it is no wonder that the community of scientists who use the facility is large and varied. Nearly 75% of APS users come from the academic community and the user community is almost equally split between the physical and life sciences. Industrial users have obtained important and socially relevant results in pharmaceutical development, energy-related breakthroughs, and environmental remediation. One example is Suva®, a reliable, proven, and safe replacement for freon. Research at the APS helped DuPont researchers to characterize metal oxide contents of candidate substitute catalysts, aiding in the development of Suva®, which is used in refrigerators and freezers, fire extinguishers, propellants, and residential and light commercial air conditioning applications. Suva® has the potential for zero impact on ozone depletion while reducing energy consumption and waste products in the manufacturing process.

General users (users not seeking access as members of a collaborative access team) obtain beam time through a centralized, fully Web-based peer review proposal process. For researchers who publish their data in the open literature, there is no charge for beam time; however, scientists can perform proprietary work for a full cost recovery fee.
Accomplishments

**Facility Design:** The APS runs more than 75% of the scheduled time in top-up (constant-current) mode—an operational mode pioneered at the APS and now used at other synchrotron light source facilities to increase the number of ampere-hours delivered, facilitate beam stability, and permit ring operation at lower emittance.

**Science:** Fuel-injector efficiency and clean combustion are dependent on the best mixture of fuel and air. To improve injector design, it is critical to understand how fuel is atomized as it is injected. Scientists using extreme-brightness x-ray beams at the APS have developed a technique that peers through high-speed dense liquids, allowing the researchers to capture, for the first time, the internal structure of the jet and map its velocity with clarity and confidence.

The structure and behavior of one of the most common proteins in our bodies has been resolved at a level of detail never before seen. Researchers have examined the structure of collagen, a protein that composes more than a quarter of all proteins in the human body and forms the principal component of skin, teeth, ligaments, the heart, blood vessels, bones, and cartilage. Researchers never had the ability to study the structure of an entire fibril in the same way that they could study an individual collagen molecule.

**Iron and Neurodegenerative Disease:** Mark Davidson (left), University of Florida (UF), and Joanna Collingwood, Keele University in the United Kingdom (UK), who is supported by a UK Alzheimer’s Society Research Fellowship and Dunhill Medical Trust, align a sample of Alzheimer's brain tissue at the microfocus facility, beamline 10-ID at the APS.

A current problem of intense interest to the condensed matter physics community is the behavior of systems with strongly interacting electrons. Researchers using the APS have gained insight into this problem by uncovering a reconstruction of electronic orbitals confined at an interface between ferromagnetic and superconducting oxides. This insight has important implications for the behavior at interfaces between materials with strongly correlated electrons.

The application of intense, submicron-sized x-ray beams at the APS has made possible the discovery that deformed metals have large, variable internal stresses in opposing directions on very short (submicron or nanoscale) length scales. This result has profound implications for understanding the mechanical strength and behavior of metals.

**Looking Ahead**

As the APS looks to the future, we realize that revolutionary science requires a change in capabilities by a factor of at least 10. As a result, renewal planning for the APS has focused on approaches that will revolutionize APS experimental capabilities along these lines. In the short term (next 1–5 years), the APS is looking toward enhanced x-ray optics and detectors to achieve at least an order of magnitude improvement. One example is the innovation after construction of the Medium Energy Resolution Inelastic X-ray Scattering instrument, where an improvement in the energy resolution by a factor of 2, and the count rate improvement by a factor of more than 10, created a best-in-the-world facility.

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*Fuel injector efficiency and clean combustion are dependent on the best mixture of fuel and air. To improve injector design, it is critical to understand how fuel is atomized as it is injected. Scientists using extreme-brightness x-ray beams have developed a technique that peers through high-speed dense liquids. Shown here is the liquid breakup of a high-density stream from a fuel injector as imaged with ultrafast synchrotron x-ray full-field phase contrast imaging at X-ray Operations and Research beamline 32-ID. Other potential applications include the dynamics of material failure under explosive or ballistic impact, which is of major importance to transportation safety and national security, and material diffusion under intense heat.*
The Stanford Synchrotron Radiation Lightsource (SSRL) has provided synchrotron radiation to the scientific community for over 30 years. SSRL, a part of the SLAC National Accelerator Laboratory, utilizes x-rays produced by the Stanford Positron Electron Asymmetric Ring (SPEAR). The SPEAR accelerator was upgraded in 2004 to an intermediate energy (3-GeV), 500-mA capable, high-brightness “third-generation” storage ring, SPEAR3. This upgrade, jointly funded by the DOE and the National Institutes of Health (NIH), is an example of extraordinary teamwork and enables groundbreaking synchrotron science.

State-of-the-Art Experimental Facilities

In conjunction with the SPEAR3 upgrade, parallel upgrades of beam line optics and end station instrumentation were undertaken from 2004 through 2008, which provided 13 beam ports at SSRL serving 25 beamlines that can operate simultaneously. There are expansion opportunities for experimental stations to support additional techniques at numerous locations around the SPEAR3 ring.
Scientific Opportunities

As shown in the accompanying diagram, SSRL beamlines offer a variety of experimental techniques to users. Selected beamline capabilities are briefly mentioned below.

**High-Resolution Angle-Resolved Photoemission Spectroscopy (ARPES)** equipment at BL5-4 is used to reveal the electronic structure of materials such as high-temperature superconductors, and how electron structure changes due to the existence of charge density waves.

**Small-Angle X-ray Scattering (SAXS)**, a well-established characterization method for microstructure investigations, is applied to studies of various materials. It probes structure of inhomogeneities (essentially electron density differences) from the near atomic scale (1 nm) to the micron scale (1000 nm). Examples include dynamics of polymer structure, structure of teeth, nucleation/growth of nanoparticles on mineral surfaces, phase transitions of Langmuir monolayers of mixed fatty alcohols, characterization of porous metal films formed by dealloying.

**X-ray Microscopy and Microanalysis** – By tuning the x-ray microprobe beam at BL6-2 to a specific energy, researchers can scan a piece of material, measure the fluorescence that comes out, and produce a detailed map of where specific elements lie within that material. A new transmission x-ray microscope has demonstrated 40 nm resolution at 8 keV in both absorption and phase contrast as well as 3d imaging using tomography.

**X-ray Scanning Microprobe Fluorescence Techniques at BL6-2** have been used by an international collaboration to characterize the elemental chemistry of stardust samples from comet 81P/Wild-2 that was brought back to earth aboard the Stardust spacecraft in January 2007.

**Soft X-ray Spectroscopy** – In 2008, SSRL opened a soft x-ray facility (BL13) for coherent scattering, surface science spectroscopy, spectro, and STXM. Scientific foci include:

- Hydrogen Storage in Carbon Nanotubes - developing suitable storage media to realize hydrogen as an energy carrier,
- Energy Production and Utilization Science - understanding of electrolysis of water and fuel cell catalysis, and
- Molecular Environmental Science – probing chemical reactions at water/oxide interfaces.

**X-ray Absorption Spectroscopy (XAS), Photoemission Spectroscopy (PES), and X-ray Scattering (XS) Techniques** allow determination of the molecular structures and compositions of elements at mineral-water interfaces, in nanoparticles, bacteria and other living cells, amorphous and crystalline materials, liquids, and complex mixtures of these materials. The behavior of contaminants and nutrients in the environment is controlled by these properties. Therefore, these synchrotron-based techniques have become invaluable tools in environmental and interfacial science applications.

**Biological Small-Angle X-ray Scattering/Diffraction (SAXS/D)** is used to study largely non-crystalline biological macromolecular systems, including proteins in solution and biological fibers. SAXS/D techniques can often study specimens in conditions similar to the physiological environment. The ease of controlling sample conditions is also an advantage of SAXS/D techniques in physio-chemical characterization and for time-resolved studies which follow the response of a biological system to a perturbation in the physical or chemical environment.

**Biological X-ray Absorption Spectroscopy (XAS)** includes dilute metalloprotein XAS, polarized single crystal XAS studies, and microXAS imaging. Studies focus on understanding the electronic and geometric structure of active sites in enzymes, in order to understand the mechanism by which they perform their biological function. A wide array of beam line facilities and equipment are provided for XAS users.

- Detailed analysis of metal centers in proteins (non-crystalline conditions)
- Reaction intermediates in metalloproteins (trapped in frozen solution or crystals)
- Single crystal XAS
- XAS imaging and spectro-microscopy

**Macromolecular Crystallography** – Each of the SSRL crystallography beamlines is equipped with identical beam line hardware, computational environment and software interface for a highly standardized, user-friendly and robust environment. Highly efficient robots enable automated screening and data collection, and remote-access techniques enable the users to perform the experiments from their home institution. A significant amount of science is devoted to structure based drug design for medical therapeutics. Work by R. Kornberg and collaborators on understanding how DNA transcription works and is regulated was awarded
the 2006 Nobel Prize in Chemistry. In 2007, SSRL opened the Molecular Observatory, an undulator microfocus crystallography beam line (BL12), funded by the Gordon and Betty Moore Foundation. A new bend magnet crystallography beam line is under development (BL14), funded by Genentech and JCSG (NIH). All beamlines feature advanced detectors.

The Synchrotron Structural Biology Resource is supported by the NIH (NCRR & NIGMS) and DOE-BER to push the science envelope and develop technologies in macromolecular crystallography, x-ray absorption spectroscopy and small angle x-ray scattering/diffraction and to disseminate these methodologies for use in the biomedical research community. The Resource is advancing the scientific forefront with novel instrumentation, innovative software and automated/high-throughput systems for:

- studying high resolution structures of large, complex macromolecules, including molecular machines;
- investigating fundamental questions in biophysics such as protein and RNA folding;
- developing and improving methods for imaging non-crystalline biological materials at near atomic resolution to study the spatial distribution and chemical nature of the involved elements; and
- studying very fast time-resolved structural changes in chemical and biological systems with ultrafast absorption and scattering techniques.

This Resource serves ~1,300 investigators per year who publish ~200 publications annually. The Resource holds Summer Schools and workshops, and carries out extensive training programs for users.

Molecular Environmental and Interface Science (MEIS) research focuses on the fundamental interfacial, molecular- and nano-scale processes that control the behavior of contaminants and nutrients in the environment. This research makes significant use of the entire suite of SSRL-based synchrotron techniques to provide direct in-situ probes of physical and electronic structure. Basic research in these areas at SSRL contributes directly to important U.S. national priorities in the areas of energy, environment, and homeland security.

**User Operations**

SSRL operates about nine months each year, with beam line upgrades and facility maintenance scheduled during the summer shutdown. Over 1,000 scientists from a variety of national and international institutions annually benefit from utilizing SSRL beamlines.

SSRL users have published more than 8,500 papers since SSRL started, with the first one submitted in 1974. This total includes over 500 ‘high profile’ publications in journals such as PRL, Science and Nature. More than 850 graduate student theses have resulted from studies performed partially or fully at SSRL reflecting a significant impact on education of the scientific workforce.
Central Management Ensures Optimal Utilization

The experimental facilities at SSRL are all scheduled and managed centrally to ensure that the unique resources are most efficiently utilized and to ensure that all visiting scientific users have a productive experience. SSRL focuses on serving the general scientific user community, providing access to beamlines, instrumentation, ancillary equipment and dedicated staff scientists and technicians in numerous areas, such as:

- Correlated and Magnetic Materials – high $T_c$ materials and oxides with nanoscale ordering phenomena, magnetic materials and magnetic nanostructures
- Molecular Environmental and Interfacial Science – fundamental interfacial, molecular- and nano-scale processes that control contaminant and nutrient cycling in the biosphere with the goal of elucidating global elemental cycles and influences on the environment
- Structural Biology/Chemistry – local electronic and atomic structure and bonding, macromolecular crystal structure, biophysical approaches to static and time-resolved structure-function relationship
- Surface and Interface Science – surface reactions, catalysis, liquid-solid interfaces

Science Program Highlights

SSRL serves the research needs of a growing number of national and international scientists across a broad range of science, engineering, environmental and biomedical disciplines. Research results are widely disseminated through publications and presentations at scientific conferences and are also frequently recognized through international awards.

Future Opportunities in the Ultrafast and Ultrasmall

SPEAR3 offers high brightness x-ray radiation, which is emitted as ultrashort (about 30 ps) x-ray pulses. These capabilities allow the development of new instrumentation such as x-ray microscopy and scientific applications in new areas such as nanoscience. More generally, it will become increasingly important in the future to create micro- or nano-sized x-ray beams with high-intensity, well defined polarization and time structure. Such studies promise unique insight into materials such as the following:

- Materials under extreme conditions (e.g., pressure or high fields),
- Materials that exhibit nanoscale dynamics (i.e., respond to excitations on the second to picosecond time scale),
- Artificial nanostructures or those intrinsically inhomogeneous on the micro- and nanoscale, and
- Biological crystals which only exist on the microscale.

SSRL, well known for outstanding user support and important contributions to science and instrumentation, is well poised to continue to facilitate scientific discovery, in ways that also train the future technical workforce.

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Why This Facility?

The National Synchrotron Light Source (NSLS) at Brookhaven National Laboratory (BNL) provides essential scientific tools to about 2,200 users/year from more than 400 institutions, including universities, national laboratories, and industry. It has pioneered many advances in photon science and technology and is distinguished by providing a very broad range of photon energies that supports a diverse and productive scientific community, resulting in more than 900 publications per year and the 2003 Nobel Prize in Chemistry. Progress toward the development of more powerful microchips and more efficient hard drives, research toward reducing pollution by improving catalytic converters, new detection methods for breast cancer and osteoarthritis, and steps toward possible eventual development of a vaccine for HIV and AIDS are only a handful of the areas in which the NSLS has accelerated scientific progress.

Synchrotron light sources such as NSLS, which has operated since 1982, have launched many scientific revolutions but the capabilities of present facilities fall short of requirements. Major advances in energy technologies – such as the use of hydrogen as an energy carrier; the widespread, economical use of solar energy; or the development of the next generation of nuclear power systems – will require scientific breakthroughs in developing new materials with advanced properties. A key barrier to progress is the absence of non-destructive tools that will allow researchers to peer inside their samples and see the smallest and most subtle details. They need the ability to study the atomic and electronic structure, the chemical composition, and the magnetic properties of materials with nanoscale resolution, and to do so under real-world conditions of extreme temperatures and harsh environments, such as found in catalytic reactions in the chemical industry.

The discovery potential of the nation’s researchers is being greatly expanded by replacing NSLS with National Synchrotron Light Source–II (NSLS-II). NSLS-II will be a new synchrotron light source, highly optimized to deliver ultra-high brightness and flux and exceptional beam stability, which enable the study of material properties and functions with a spatial resolution of 1 nanometer, an energy resolution of 0.1 millielectron volt, and the ultra-high sensitivity required to probe atomic-scale features. It will deliver the world’s finest capabilities for both x-ray imaging and high-resolution energy analysis, using a beam designed to be approximately 10 times brighter than any other synchrotron worldwide that is now operating or under construction.

Scheduled for completion in 2015, NSLS-II will provide advanced tools for discovery-class science in condensed matter and materials physics, chemistry, and biology – science that ultimately will enhance national and energy security and help drive abundant, safe, and clean energy technologies. The resulting scientific advances will support technological and economic development in multiple sectors of the economy, with powerful applications to biotechnology, nanotechnology, and the study of materials under extreme conditions.
**Instrumentation**

NSLS-II will provide advanced insertion devices, optics, detectors, robotics, and an initial suite of scientific instruments. It will ultimately have the capacity for over 60 beamlines. These will serve a diverse range of scientific communities including energy sciences (improving efficiency and storage), materials science and engineering, environmental science, and bio-sciences. The beamlines will take advantage of the unprecedented performance of the storage ring to produce x-ray beams of world-leading flux and resolution. x-ray beams as small as 1 nanometer will be produced, and energy resolution as small as 0.1 millielectron volt will be achieved. In addition the experimental program will give users access to advanced sample environments allowing them to probe the nature of materials in extreme working conditions (for example, in high pressure or temperature, or surrounded by unusual gas compositions).

To enable such experimentation, the NSLS-II beamline quality places demands on the electron beam and synchrotron radiation properties in the NSLS-II design. To meet design goals, research and development activities are underway that include magnet measurement and alignment and the development of advanced optics. For example, aggressive x-ray optics research and development is underway to produce optics capable of delivering, with high efficiency, the very high spatial and energy resolutions required.

**Initial Beamlines**

Within the NSLS-II construction project scope, six insertion-device beamlines are being built. As detailed below, this initial suite of beamlines aims to achieve extraordinary capabilities in x-ray imaging, scattering, and spectroscopy, with many world-class characteristics that will meet the needs of the scientific community and enable new studies that are not possible to conduct at present-day facilities.

**Inelastic X-ray Scattering (IXS) Beamline**

Inelastic x-ray scattering allows studies of the dynamic behavior of various materials at molecular and atomic levels by detecting and analyzing the small energy differences between the incident and the scattered x-rays. Due to difficulties in high-resolution x-ray optics and limitations in x-ray flux, at present the best energy resolution available at other synchrotron facilities is about 1 meV. The IXS beamline aims to achieve world-leading ultra-high energy resolution of ~0.1 meV, thus opening up uncharted territories in studies of materials dynamics of disordered systems at high momentum transfers. Examples of scientific applications at ultra-high energy resolution include dynamic studies of liquids and disordered systems, vibration dynamics of materials under high pressure, relaxation dynamics of biomolecular assemblies in glassy states, and surface lattice dynamics in crystalline systems.

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*The conceptual layout of the IXS beamline incorporates a novel x-ray optics design based on the concept of asymmetric back-reflection wavelength dispersion proposed by Yuri Shvydko of the Advanced Photon Source. It consists of a standard high-heat-load pre-monochromator followed by an ultrahigh-resolution 0.1 meV monochromator. The incident beam is focused by a pair of Kirkpatrick–Baez mirrors to a 1 μm by 3 μm spot on the sample position. The scattered x-rays are collected by a pair of multilayer collimating mirrors and then analyzed by an identical 0.1 meV optical system to record the inelastically scattered x-ray spectrum.*
**Hard X-ray Nanoprobe (HXN) Beamline**

The HXN will be a state-of-the-art beamline that aims to achieve world-leading ~1 nm spatial resolution for x-ray fluorescence imaging and nanodiffraction, using advances in x-ray fluorescence, high-dynamic-range area detectors, and x-ray focusing optics such as multilayer Laue lenses. The HXN beamline will take advantage of the NSLS-II brightness and the penetration power of hard (2-30 keV energy) x-rays in order to enable x-ray structural and chemical imaging at nanometer scales, with an ultimate elemental sensitivity of a single atom.

The endstation instrument will incorporate cutting-edge focusing optics with specimen manipulation techniques. The instrument will be equipped with active and passive vibration controls as well as an active position feedback system for nanometer-scale positioning and stability between the optic and the specimen.

**Coherent Hard X-ray Scattering (CHX) Beamline**

The CHX scattering beamline will support x-ray photon correlation spectroscopy (XPCS) for microscopic dynamic studies of glassy and nonequilibrium systems, nanostructured fluids, nanoscale domain-walls in correlated condensed matter, and biological systems such as proteins in solution and bio-membranes. The beamline plans to use x-ray mirrors and focusing optics with ultralow distortions in order to guide a coherent x-ray flux onto the sample under investigation. The world-leading brightness of NSLS-II promises to deliver coherent flux to XPCS experiments that is one to two orders of magnitude higher than at any other facility worldwide, thus enabling new regimes of dynamic studies at two to four orders-of-magnitude faster time scales in intensity correlation experiments.

Besides XPCS, the CHX beamline offers the exciting potential to advance coherent diffraction imaging research as well as microbeam small-angle x-ray scattering studies on materials or biological samples. These techniques provide powerful ways to study the structure of noncrystalline materials.

**Coherent Soft X-ray Scattering and Polarization (CSX) Beamline**

The CSX beamline seeks to take advantage of the extraordinary brightness of soft x-rays at NSLS-II to facilitate coherent speckle imaging, metrology, and polarization switching experiments. The CSX beamline conceptual layout produces two beams in the 200-2200 eV soft x-ray range using two elliptically polarized undulators in a canted configuration. One beam is optimized for the highest possible degree of spatial coherence and the other is optimized for the highest-performance polarization-sensitive experiments with the capability to switch between opposite polarization states at a rate of up to 1 kHz. This high polarization switching rate achieves a high magnetic-moment sensitivity in x-ray magnetic circular dichroism experiments that is unmatched in other facilities. This beam is useful for scientific research on highly dilute magnetic materials, micrometer-size single crystals, magnetic nanoparticles, and interfaces.

The scientific motivation for the high-coherence beam is to provide world-leading high coherent flux to soft x-ray coherent scattering methods, including phase retrieval imaging and photon correlation spectroscopy, in order to transform these presently signal-limited methods into high-throughput, high productivity techniques for science-driven experiments. The design of the endstation instrument incorporates a Fresnel zone plate to allow studies of material inhomogeneities at better than 40 nm spatial resolution. Applications include the quest for better understanding of phase segregations in strongly correlated materials, such as ferromagnetic metal-
lic and antiferromagnetic insulating regions in manganites displaying colossal magneto resistance, and the antiferromagnetic insulating and superconducting regions in high-Tc superconducting cuprates.

**Submicron Resolution X-ray Spectroscopy (SRX) Beamline**

The SRX beamline provides a spectroscopic imaging capability. The conceptual layout of the beamline uses two beams from canted, high-brightness undulators. One beam uses a Kirkpatrick-Baez mirror system to focus 4-25 keV x-rays to 0.1 – 1 μm, and the other is based on a Fresnel zone plate to achieve a targeted high resolution of 30 nm in the 2-4 keV range. Both beams use high resolution x-ray fluorescence imaging and take advantage of the exceptional high brightness of NSLS-II to produce a world-leading scanning-probe spectroscopy and imaging tool.

The principal scientific objective of the SRX beamline is to conduct frontier research at important interfaces of the nanomaterials, environmental, and biological sciences. For example, elemental mapping by x-ray fluorescence nanoprobe helps to understand how semiconducting nanoparticles such as TiO$_3$ can be incorporated into different regions in biological cells for potential cancer therapy applications. Studies of metal distribution in micro-organisms (e.g., the subcellular localization of metals, as well as mechanisms relevant to nutrient distribution and clearance of excess (toxic) metals across membranes by active transporters) give insight into microbial pathogenesis and bioremediation. Nanoscale trace-metal element mapping of micro-organisms and plants helps to understand metal-binding mechanisms in diverse organisms and cells, and informs the development of new agricultural, pharmaceutical, and biofuel technologies. The SRX beamline will enable these frontier investigations with unprecedented sensitivity and data-collection rates, making many experiments possible that are impractical today.

**X-ray Powder Diffraction (XPD) Beamline**

The XPD beamline will be a state-of-the-art damping-wiggler based beamline equipped with high-energy powder diffraction and pair-distribution function (PDF) instrumentation with high-angular-resolution and time-resolved structural analysis capabilities. This beamline is designed to support measurements that (a) quantitatively characterize complex and nanoclustered material structures in their heterogeneous functional conditions and in extreme environments, (b) enable time-resolved in-situ studies of structure evolution under external excitation or during chemical reaction, and (c) provide critical structural information during the early stages of advanced material synthesis when single crystal specimens are often unavailable. Equipped with advanced powder diffraction capabilities, the XPD beamline will enable a wide range of scientific applications in areas such as the physics of complex materials, earth and geological sciences, and energy and catalysis research.

The conceptual layout of the XPD beamline consists of a main beam with a tunable energy range of 40-100 keV and a side beam for dedicated high-energy PDF studies at a fixed energy of 80 keV. The experimental station will house a heavy-duty diffractometer to accommodate specimen environmental cells for structural studies at extreme conditions of high temperature, high pressure, and high magnetic field. Advanced capabilities will include a multicrystal analyzer array for high-resolution powder research, a 7000-element strip detector for millisecond time-resolved studies, and a robotic sample mount for automated sample handling for high-throughput structural studies in chemical and material synthesis.

**Future Scientific User Program**

The NSLS-II Project is working closely with the user community to develop an overall scientific strategic plan that will guide the development of the experimental facilities for NSLS-II. The specific laboratory and experimental capabilities for particular beamlines are being designed in collaboration with Beamline Access Teams (BATs) consisting of a small number of experts in a specific technique.

Stakeholder interest in NSLS-II is strong as evidenced by a financial commitment by New York State to fund the construction of the Joint Photon Sciences Institute (JPSI) adjacent to the NSLS-II facility. The JPSI will serve as an intellectual center for the development and application of photon sciences and as a gateway for users of NSLS-II.

**Contact**

**National Synchrotron Light Source-II Project**
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For more than 40 years, the 3-km-long linear accelerator at the SLAC National Accelerator Laboratory has produced cutting edge science from high-energy electrons and positrons. Now, scientists will continue this tradition of discovery using part of the SLAC linac to create an entirely new kind of x-ray source. It will be used much like a super-high-speed strobe flash, enabling scientists to take stop-motion pictures of atoms and molecules in motion, illuminating the fundamental processes of physics, chemistry, and biology on unprecedented timescales.

An X-ray Laser

The Linac Coherent Light Source (LCLS) will produce ultra-fast pulses of x-rays billions of times brighter than synchrotron sources. LCLS will exploit the free-electron laser (FEL) process, in which a pulse of high-energy electrons traveling through a very long periodic magnet structure creates x-rays and then coherently amplifies their intensity by many orders of magnitude. The SLAC linac is uniquely capable of producing the intense, high-energy electrons required to drive such an x-ray source, and LCLS will be the first FEL facility operating in the hard x-ray spectral range, enabling frontier science.

Probing the Ultra-small, Capturing the Ultra-fast

Since their discovery, x-rays have been primarily used for studying structures—such as broken bones as well as the atomic arrangements in the proteins that drive biological processes. Their short wavelength and penetrating power make x-rays particularly useful for probing the ultra-small world of molecules and nanostructures. Increasingly, scientific attention is turning to the dynamics of these ultra-small materials—how molecules and nanoparticles change their structures under the influence of outside forces or their own interactions. Unfortunately, the time scale for these changes is incredibly fast, so standard x-ray studies catch only a blur. LCLS will be the first x-ray source to offer both the intensity necessary to probe complex ultra-small structures and the short pulse required to resolve their ultra-fast motions. It will capture images with a “shutter speed” of less than 100 femtoseconds. Photosynthesis is an example of an ultra-fast chemical process; x-ray pulses from LCLS could capture the details of the sequence of chemical structures involved in this reaction. A better understanding of photosynthesis—a highly efficient use of the sun’s energy—has implications for future energy sources and for agriculture.

Structure of a protein molecule imaged using x-ray diffraction at the SSRL synchrotron source. LCLS plans to not only increase the range of biological materials whose structures can be studied, but also to enable the study of ultra-fast dynamics at the atomic scale.
Construction

Groundbreaking for the LCLS took place in October of 2006. Transforming SLAC’s venerable linear accelerator into a next-generation x-ray light source has benefited from the efforts of hundreds of scientists from all over the world. The LCLS construction project involves a collaboration including three Department of Energy laboratories (SLAC National Accelerator Laboratory, Argonne National Laboratory, and Lawrence Livermore National Laboratory), and the University of California, Los Angeles. LCLS is currently completing construction of infrastructure for future experiments and support staff; the project will be completed by July 2010.

The SLAC linear accelerator will accelerate pulses of electrons to nearly the speed of light, and use them to create extremely intense, coherent pulses of x-rays through the free-electron laser process.

The free-electron laser itself was operated for the first time in April 2009 and was quickly brought to full power. Beginning in September 2009, scientists from around the world will come to SLAC to prepare for first use of the extraordinary capabilities of this research facility. The first experiments will employ the atomic/molecular/optical science (AMO) instrument, which has been constructed by the LCLS Project. The AMO instrument is the first of six instruments designed for LCLS science in the next three years. Two more instruments, for soft x-ray materials science and x-ray pump/probe studies, will be available in mid-2010.
Instruments

Several instruments which will use the LCLS beam for research are in various stages of design and development. Each instrument, described briefly below, is tailored to support a different type of scientific experiment. When completed, each instrument’s use will be managed by the LCLS User Program, which administers the peer review proposal process to grant access to those investigators with research proposals of greatest merit.

Atomic, Molecular & Optical Science (AMO)
This instrument will enable the study of the interaction between the extremely intense LCLS x-ray pulses and the basic constituents of matter: atoms and molecules. It could also be used to follow the evolution of chemical reactions in the gas phase on their natural time scales using powerful tools such as photoelectron spectroscopy. A suite of instrumentation including focusing optics, gas delivery systems, electron, ion and photon spectrometers and a synchronized high-power laser are currently being built.

Soft X-ray Materials Science (SXR)
This instrument will enable the high brightness and timing capability of the LCLS to be applied to scattering and imaging experiments that require the use of soft x-rays. Examples of such experiments are the dynamics of nanoscale magnetic domains in advanced data storage media and the dynamics of electron ordering in correlated-electron materials. The instrument will include an insertable grating monochromator so that the natural LCLS FEL bandwidth can be reduced for spectroscopic studies. A synchronized ultra-fast laser will be available for pump-probe experiments.

X-ray Pump Probe (XPP)
This instrument will use a fast optical laser to generate transient states of matter, whose subsequent dynamics will be probed by x-ray pulse from LCLS. The laser pump will have the ability to conduct precise optical manipulations, in order to create the desired excited states. The instrument design will emphasize versatility. To maximize the range of phenomena that can be excited, it will be necessary to be able to manipulate the laser pulse energy, frequency, and temporal profile. X-ray scattering will be the dominant tool for probing the laser-induced structural changes. These experiments require the union of four experimental capacities: the generation and delivery of x-ray and laser pulses to the sample, the preparation of the excited state in the sample, and the detection of the x-ray scattering pattern.

Coherent X-ray Imaging (CXI)
This instrument will take advantage of the extremely bright, ultra-short LCLS x-ray pulses to allow imaging of non-periodic nanoscale objects, including single or small clusters of biomolecules, at or near atomic resolution. The high coherence of the LCLS x-rays will allow single particles to be imaged at high resolution, while the short pulse duration will limit radiation damage during the measurement. The instrument will allow imaging of biological samples beyond the damage limit that cannot be overcome with synchrotron sources. Samples can be introduced to the x-ray beam either fixed on targets or using a particle injector that will deliver free particles to the beam. High-quality focusing optics will generate sub-micron foci, and will allow imaging of single nanoparticles of various sizes, pushing the limit down to single biomolecules.
**X-ray Correlation Spectroscopy (XCS)**
This instrument will observe dynamical changes of large groups of atoms in condensed matter systems over a wide range of time scales using x-ray correlation spectroscopy and coherent x-ray scattering. The XCS instrument allows the study of equilibrium- and non-equilibrium dynamics in disordered or modulated materials, such as glass-forming liquids and solids near phase transitions, on timescales ranging from 100 femtoseconds to several nanoseconds, and also from 10 milliseconds to thousands of seconds.

**Matter in Extreme Conditions**
This instrument will enable the detailed study of states of matter created when normal condensed matter is suddenly heated to very high temperatures, well above melting. Such matter occurs naturally in planetary cores, and briefly in materials during shock waves or implosions, but is extremely difficult to study in the laboratory. The instrument will include a powerful pulsed laser for creating extreme conditions in a sample.

*An aerial view of the 3-km-long linear accelerator at the SLAC National Accelerator Laboratory. LCLS in its initial phase utilizes one third of the linac to drive an x-ray laser. The LCLS experimental halls are located beyond the bottom edge of this photograph.*
Versatile Probes of Material Properties

Scientists worldwide use neutrons to study the arrangement, motion, and interaction of atoms in materials. Research using neutrons provides valuable information that often cannot be obtained using other techniques, such as optical spectroscopies, electron microscopy, and x-ray diffraction. Scientists need all of these techniques to provide the maximum amount of information on materials.

The inherent physical properties that govern how neutrons interact with other matter make neutrons versatile probes of material structure. Scientists guide neutron beams to material samples of interest, and control the neutron direction of travel, energy, and sometimes other properties (e.g., their spin). Detected signals come from neutrons that strike and scatter off of the material sample. These scattering signals are decipherable – when analyzed, they reveal information about the material under investigation.

The facilities described next produce neutrons for such scientific experiments. One way of generating neutrons is via a research reactor—the High Flux Isotope Reactor (HFIR) is this type of neutron source. The other way to generate neutrons is via an accelerator to generate protons that strike a target. As a result of the impact, neutrons are produced in a process known as spallation. The Manuel Lujan Jr. Neutron Scattering Center (Lujan Center) and the Spallation Neutron Source (SNS) each use this type of neutron source.

Jiandi Zhang of Florida International University works with the HB-3 Triple-Axis Spectrometer at HFIR.
For neutron scattering experiments, it is ideal to match the wavelength and energy of the neutron to the length and energy scales, respectively, of the materials under investigation. The thermal (room temperature) neutron spectrum of a reactor produces neutrons with wavelengths on the order of a few tenths of a nanometer, well matched to investigating atomic length scales and lattice vibrational energies. When thermal neutrons are passed through a container of low-temperature liquid hydrogen, they are slowed by inelastic collisions with the hydrogen. The process produces slower neutrons better suited for studies of soft matter. Because they reflect well from surfaces, cold neutrons can be transported over long distances with little loss. Neutrons with longer wavelengths and lower energies—cold neutrons—are better for studying large-scale structures (e.g., molecular organization, nanopore-size distributions, and aggregate size and shape) and low-energy excitations (e.g., excitations in frustrated systems and various problems in magnetism, superconductivity, and correlated electron systems).

**Outreach and Education**

Outreach and education activities help develop a user community and identify the resources needed to perform cutting-edge science. In response to requests from the user community for additional information, several means of communication have been established. These include workshops, progress reports that are distributed electronically, webpostings, fact sheets, technical journal articles, user newsletters, and annual reports.

*The building blocks of DNA direct the synthesis of proteins. Research using neutron scattering could help determine the shape and structure of those proteins.*

*Neutron scattering has guided the development of giant molecules that make up synthetic fibers for clothes.*
At the Los Alamos National Laboratory (LANL), academic researchers work side-by-side with national security researchers at the Los Alamos Neutron Science Center (LANSCE). Perched on its own mesa between steep canyons laden with archeological sites, the Manuel Lujan Jr. Neutron Scattering Center (Lujan for short) at LANSCE, provides state-of-the-art instrumentation and support for user-driven materials and nuclear research. The Lujan Center specializes in securely melding academic science and national security because the fundamental science is the same.

Neutron scattering is indispensable in today’s condensed matter and materials sciences. Neutrons are created by spallation – knocking them out of a tungsten nucleus after being hit by a speed-of-light proton from the LANSCE accelerator – and scatter from nuclei in solid and liquid matter, carrying away useful information about atomic spacing, vibrational motion, and internal magnetic fields. When combined with their high penetration through centimeters of heavy metal atoms, unmatched sensitivity to hydrogen and other light atoms, and their ability to distinguish isotopes of the same element, neutrons are uniquely suited to probe materials in special, powerful ways. Researchers measure the neutron’s time of flight after scattering to determine their energy (i.e. wavelength) accurately even after losing or gaining energy by scattering from a sample. Combining the energy with the angle at which neutrons scatter allows one to determine which atoms are where and how they move. No information is more fundamental to the properties of materials.

Neutrons are scarce even at a powerful spallation source like LANSCE. While the Lujan Center currently has a competitive peak flux—running at 100kW—it’s distinction is that it is one of the world’s most brilliant sources at long wavelengths, due in part to efficient, liquid-hydrogen moderators and a low repetition rate of 20 Hz between neutron pulses. The low repetition rate provides a large bandwidth between the fastest and the slowest scattered neutrons in a pulse and the long wavelengths match biological and soft matter well. Among recent scientific results, Lujan Center instruments have been used to determine the fundamental chemical function of xylose isomerase, the biocatalyst used to convert fructose, and to test the hypothesis that the Egyptian pyramids were made with the aid of an ancient form of concrete.

Classified experiments can also be performed at the Lujan Center in support of the national defense. Recent advances include studies of challenging materials such as cholera toxin, high explosives, and fundamental studies of excitations in plutonium relating to how it compresses in a nuclear weapon to reach critical mass.

R&D Areas of Emphasis
The Lujan Center emphasizes the following areas of science:

- National Security,
- Bioscience and nanoscience,
- Geoscience,
- Materials engineering and science,
- Condensed matter physics, and
- Advanced neutron instrumentation including spallation physics.

The first area is sponsored by the DOE National Nuclear Security Administration, whose programs at Los Alamos National Laboratory offer constructive synergy to the pursuit of the other areas, which are briefly discussed below.

Bioscience and Nanoscience
The Lujan Center’s bioscience activities are centered at the Protein Crystallography Station (PCS), the first dedicated neutron diffractometer for structural biology in North America and one of the first in the world. Because of its success since commissioning in 2002, the neutron science centers of ISIS (in the United Kingdom) and JPARC (in Japan) plan to build protein crystallography instruments.

Among the science of interest to neutron scatterers in nanoscience and technology are biomembranes, self-assembling materials, selective membranes, smart materials, and bio-inspired synthesis. In general, the high-performance density of biological systems is far from being matched by engineered systems, and mastery of the nanoscale is critical to developing that capability.

Other instruments at the Lujan Center typically subscribed by nanoscience users are SPEAR (reflectometer, biomembrane science), LQD (small-angle scattering, nanostructured materials), and NPDF (powder diffractometer for pair distribution analyses, nanoscale structure of materials).
**Geoscience**
With the commissioning of the High-Pressure Preferred Orientation (HIPPO) diffractometer, the geoscience community now has a fully instrumented center of excellence. A suite of sample environments, including a 100-position sample changer and texture goniometer, a stress-strain rig, and several options for high-pressure studies at high temperature, provides HIPPO users with new research opportunities. It is through fundamental materials science with geomaterials that huge resources such as methane hydrates (clathrates) may be accessible to meet future energy needs.

**Materials Engineering and Science**
A differentiating strength of neutrons for the study of the mechanics of hard materials is the ability to monitor strain in situ. In this area, the Lujan Center is a recognized world leader. The ability to stress a material at high or low temperature and to measure its response at the atomic scale is a unique strength of Lujan Center engineering science.

**Condensed Matter Physics**
Spurred by the completion and commissioning of Pharos, and the commissioning of an 11-Tesla magnet, the condensed matter physics community utilizes Lujan instruments for cutting-edge research in correlated electron systems, magnetism, and the physics of fundamental excitations.

**Advanced Neutron Instrumentation**
Staff at the Lujan Center have a long tradition of pushing the state of the neutron scattering art. From source to detection, LANL scientists and engineers have made substantial contributions to spallation neutron science, exemplified with the first side-coupled linac in 1972. The invention of partially coupled, cold moderators at the Lujan Center was put into practice in 1998, concurrently with the planning for IN500, a prototype beamline on FP13 designed to make optimum use of that new cold moderator design. The result of these efforts is a state-of-the-art chopper spectrometer with several innovations:
- Ballistic neutron guides that produce a flux advantage over long distances.
- “Event recording” data acquisition, a technique that involves recording the position and time of every arriving neutron. This method—enabled by high performance computing, DSPs, and massive storage—allows essentially full reconstruction of the scattering experiment.
- Repetition-Rate Multiplication, a complex Time-Of-Flight (TOF) scheme to handle frame overlap, to extend bandwidth dramatically while filling the counting frame efficiently.

User-driven science is the well-spring of innovative ideas for instrumentation. Recent experiments include:
- The first demonstration of pulse-echo encoding at a pulsed source,
- Magnetic neutron lenses, and
- Baez-style neutron focusing lenses, to name just a few areas.
Lujan Center Instrument Suite

Nine of Lujan Center’s seventeen flight paths were equipped in 2007 with new or upgraded instruments from an instrumentation campaign in the late 1990s. Twelve instruments are for materials research, three are devoted to fundamental nuclear physics, and two flight paths are empty. Six of the materials research instruments and two of the nuclear physics beamlines are considered world class. Owing to resource limitations and age, only an additional three neutron scattering instruments are competitive enough to be in a user program at some level.

Target

The Lujan Center’s unique split spallation target with its flux-trap moderators yield a higher peak neutron flux than any other spallation neutron source outside of the Spallation Neutron Source (SNS) at ORNL. Running routinely at 100 kW, the Lujan Center compares very favorably with the best pulsed spallation neutron source outside the United States: ISIS (in the United Kingdom) has a higher average (160 kW)—but a lower peak—neutron intensity. A significant strength of the Lujan Center is its high peak flux of cold neutrons and its low pulse repetition rate. At 20 Hz, the Lujan Center has wide time-of-flight bandwidth relative to other facilities (e.g., SNS at 60 Hz and ISIS at 50 Hz). Second target stations, proposed and in-progress at SNS and ISIS, will approximate Lujan Center’s repetition rate and flux. The Lujan Center strives to be complementary to SNS, the centerpiece of US neutron scattering strategy.

Floor Plan

For the national neutron scattering user program in materials and biological sciences, BES currently operates ten of the Lujan Center’s instruments (Flight Paths 1, 2, 4, 11a, 12, 14, and 15), upgrades to others (FPs 3, 9, 16), and one instrument under feasibility testing (FP 13).

In 2008, the Lujan Center experimental areas received new instruments (FPs 1, 2, 4, 11a, 12, 14, and 15), upgrades to others (FPs 3, 9, 16), and one instrument under feasibility testing (FP 13).
Sample Environment Capabilities
Continual efforts are made to enhance the availability and flexibility of the Lujan sample environment suite. A significant investment has been made in low and high temperature facilities particularly directed toward condensed matter physics users. Cryogenic temperatures are readily accessible at all instruments using closed-cycle refrigerators and cryostats. A dilution refrigerator capable of 15 mK was added in 2008. An 11-Tesla cryomagnet is available to provide a unique polarized-beam reflectivity capability over the temperature range 1.5-300K. For experiments above room temperature, there are two cryofurnaces, a heating stage, and a furnace. Additional effort has been directed toward liquid and gas cell pressure capabilities and toward anvil-type pressure cells. For example, a number of fluids-driven pressure cells (< 0.5 GPa) are available, which can be used on most beamlines.

Future Plans
The next phase of Lujan Center history is expected to be a period of significant growth for LANSCE and the Lujan Center in beam reliability, user support, and instrument capability. While the LANSCE Refurbishment Project will breathe new and longer life into the 30+ year-old accelerator, the Enhanced Lujan Project will more than double the utilization of the powerful beamlines on the neutron source. By 2013, with up to three new instruments, several upgraded ones, and more than twice the user support in the form of personnel and sample environments, the Lujan Center will be over 90% utilized. Although the core strength in diffraction will continue, much of the new instrumentation will be for inelastic scattering.

By 2010, the new Mark III target, which incorporates several innovations, will produce a 2-fold enhancement in cold neutron flux. The LANSCE accelerator has been a megawatt-class proton accelerator since 1986 and ran over 4,000 hours of production in 2005.

Starting in 2004 and lasting until the SNS comes to full power and instrumental strength, the Lujan Center leads as the DOE’s largest neutron scattering program. It serves the nation in a unique way by providing a place to perform national security research in a safe and secure manner without disrupting access by academic and industrial users.

A special treat for neutron facility staff and users is the annual LANSCE Neutron Scattering School. Serving 30 graduate students and postdocs per class, the School is one of the few topical scattering schools in the world, with a curriculum that includes concentrated subjects like magnetism, soft matter, and hydrogen materials. Students hear lectures by world-leading faculty followed by hands-on experience with actual neutron scattering experiments. Begun in 2003, alumni of the School are part of the worldwide neutron community.

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Centerpiece 11-tesla superconducting magnet gives users at the Lujan Center a wide regime in magnetic field studies.
The 85-MW High Flux Isotope Reactor (HFIR) provides one of the highest steady-state neutron fluxes of any research reactor in the world. HFIR fulfills three important missions: neutron scattering research, isotope production, and materials irradiation (e.g., for neutron activation analysis). The neutron scattering instruments installed on its neutron beamlines enable fundamental and applied research into molecular and magnetic structures and behavior of materials, including high-temperature superconductors, polymers, metals, and biological samples. The reactor is used to produce isotopes for medical, research, and industrial uses; to study damage resulting from bombardment of materials by neutrons; and to examine trace elements in the environment by neutron activation. A cold neutron source is installed that uses liquid hydrogen to slow the movement of neutrons, making them particularly useful for certain types of experiments. This cold source greatly enhances the reactor’s research capabilities, particularly in the biological sciences.

Description and Missions

The HFIR at the Oak Ridge National Laboratory is one of the highest flux reactor-based source of neutrons for condensed matter research in the world. Thermal and cold neutrons produced by HFIR are used for research probing the fundamental properties of condensed matter and for radioisotope production. The users of HFIR are from a wide range of U.S. and international university and industrial research and development organizations.

The HFIR has three main missions. The first is to generate neutrons for use in scattering experiments. Neutrons are an ideal probe for investigating the arrangements of and interactions between atoms and molecules. This activity has grown in both scientific and economic importance and today provides essential knowledge about the molecular and magnetic structures and behavior of materials, including high-temperature superconductors, polymers, metals, and biological samples. The neutron-scattering instruments installed on the HFIR horizontal beam tubes are used in fundamental studies of materials of interest to solid-state physicists, chemists, biologists, polymer scientists, metallurgists, and colloid scientists. These instruments are open to use by university and industrial researchers on the basis of peer-reviewed scientific merit.

The second key mission of HFIR is to produce radioisotopes, including californium and other transuranium isotopes, which are used for research, industrial, and medical applications. These materials are produced in the flux trap in the center of the HFIR fuel element where a working thermal-neutron flux of $2.0 \times 10^{15}$ neutrons/(cm²·s) is available to irradiate the target material. Additional irradiation facilities are also provided in the beryllium reflector. The HFIR is the western world’s sole supplier of californium-252, an isotope used in cancer therapy and in detection of environmental pollutants and explosives in luggage.
The third mission of HFIR is to provide unique capabilities for instrumented and un-instrumented materials irradiation studies (e.g., in neutron activation analysis). The HFIR provides for a variety of irradiation tests and experiments that benefit from the exceptionally high neutron flux available. In the fuel element flux trap, a hydraulic tube provides access to the high thermal-neutron flux in the reactor for short-term irradiations, and other positions are ideal for fast-neutron irradiation-damage studies. A modification of the flux trap experiment facilities in 1986 has provided two locations in the maximum flux region that can accommodate instrumented capsules and engineering loops. The beryllium reflector contains numerous experimental facilities with thermal-neutron fluxes up to $1.0 \times 10^{15}$ neutrons/(cm$^2$·s). These facilities can accommodate static experimental capsules, complex fuel-testing engineering loops, and special experimental isotope irradiations.

**Facilities and Capabilities – Instruments and Cold Source**

The HFIR is a world-class facility operating at a power level of 85MW. Major additions to the original facility are a guide hall for cold source instruments, a liquid hydrogen cold source, upgraded instruments, and new control systems. With these renovations and the successful commissioning of the cold source, HFIR has become a premier research and development facility. Fully instrumented, the HFIR can support 15 state-of-the-art neutron scattering instruments, several of which are designed exclusively for cold neutron experiments. The reactor is currently equipped with 10 neutron scattering instruments deployed across four horizontal beamlines.

The installation of the cold source is especially significant to prospective HFIR users. The cold source has a brightness comparable to the best in the world and is the first forced-circulation super-critical hydrogen cold source placed into a nuclear reactor. The neutron flux and the brightness of the cold neutron beams have been tested using time-of-flight methods, revealing the brightness to be significantly greater than predictions by computer simulations before installation. The cold source generates a substantial useful neutron flux in the 2- to 12-Å range. The cold neutrons from the source illuminate four neutron guides that bring beams to instrument positions in the cold neutrons guide hall.

Two of the guides provide beams to small-angle neutron scattering (SANS) instruments. One of these is a general-purpose SANS, designed for experimentation on hard and soft condensed matter and magnetic systems. The other, the Bio-SANS, is designed especially for studying the structure and function of biological materials and complexes, including biomacromolecules such as proteins and viruses, membranes, and biomimetic systems. These two capabilities are among the most advanced SANS instruments in the world.

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*Inelastic neutron scattering results revealed the Perovskite crystal structure, shown in this drawing, of a manganite phase that exhibits colossal magnetoresistance.*
High Flux Isotope Reactor (cont.)

Specific Instrumentation

**Diffractometers**

Recently upgraded, HB2A is a high-resolution neutron powder diffractometer. With a single wavelength, HB2A is particularly adapted for polarization analysis and hence the structural study of magnetic materials. Funded under a US-Japan collaboration, the WAND diffractometer (HB-2C) is a high-intensity instrument, with the ability to tilt either the sample or the detector plane. Applications of WAND include phase transformation kinetics, diffuse scattering in single crystals, and liquid and glass materials. The HB-2B is a residual stress diffractometer, particularly well suited for stress mapping in engineering materials. A motor-driven load-frame is also available for *insitu* deformation studies. High accuracy, small molecule crystallography can be done using the HB3A four-circle diffractometer with available sample temperatures of 10 to 300K.

**Spectrometers**

Triple-axis spectrometers are the most versatile instruments to study elementary excitations of solids, such as phonons and magnons. They have the ability to measure the scattering at any point in momentum-energy space that is physically accessible by the spectrometer. There are currently three operational thermal triple-axis spectrometers with complementary capabilities. The combination of the world’s highest continuous flux of thermal neutrons and a wide range of incident energies (between 5 and 120 meV) makes these instruments particularly well suited for the study of current problems in condensed matter physics such as magnetic excitations in high-Tc superconductors, multiferroic materials, colossal magnetoresistance manganites, heavy fermions, new classes of metals, quantum magnets, magnetic multilayers, and commercial magnetic alloys. The HB-1 triple-axis spectrometer is designed to operate as a polarized-beam instrument and as a general-purpose unpolarized-neutron spectrometer. The HB-3 triple-axis spectrometer is designed to operate as a versatile high-intensity unpolarized-neutron instrument. The availability of three monochromator crystal choices makes this spectrometer extremely versatile for a wide range of studies on material properties. The third thermal triple-axis spectrometer located at the HB-1A port is a fixed incident-energy (14.6 meV) instrument. This is an excellent instrument for measuring low-lying excitations, elastic studies of lattice or magnetic structures and transitions, and elastic studies on thin film or small samples where high flux on sample and very low higher order contamination of the beam are critical issues. A cold neutron triple-axis spectrometer (CG-4C, part of the US-Japan collaboration on neutron scattering) is under installation.

The United States’ highest flux reactor–based source of neutrons for condensed matter research.

The Scheduled Commissioning Date

- Operational 2008
- Future Development 2009

Low-energy excitations, magnetism, structural transitions

HB-1A

Polarized neutron studies of magnetic materials, low-energy excitations, structural transitions

HB-1

Structural studies, magnetic structures, texture and phase analysis

HB-2A

Diffuse-scattering studies of single crystals and time-resolved phase transitions

HB-2C

Strain and phase mapping in engineering materials

HB-2B

Medium- and high-resolution inelastic scattering at thermal energies

HB-3

Small unit cell crystal structural studies, particularly H-bonding

HB-3A

Cold Neutron Source
The two Small-Angle Neutron-Scattering instruments at the HFIR receive neutrons from the brightest cold source in the world, with integrated fluxes comparable to the best facilities worldwide. Each instrument has a large-area detector (1 m²), uses wavelengths from $\lambda_{\text{min}}$ (= 4.8Å and 6Å) to $\lambda_{\text{max}}$ (~30 Å), and provides extended Q-ranges (0.002-1.0 Å⁻¹) which enable a wide variety of research opportunities. The General Purpose SANS instrument (CG-2) is optimized for studies of soft condensed matter including the exploration of in-situ processing of soft materials to follow structural evolution in industrially relevant equipment (e.g., during extrusion or stretching). On this instrument, it is also possible to study small (~1 mm³) crystals of high-Tc superconductors to probe flux line lattice melting processes and to undertake kinetic studies (e.g., investigations of phase-separation kinetics in polymer blends and metallic alloys). On the Bio-SANS instrument (CG-3, operated in cooperation with DOE’s Office of Biological and Environmental Research), effective studies of biological materials, which are often weakly scattering (with cross sections ~ $10^{-1}$ to $10^{-3}$ cm⁻¹), are possible. Bio-SANS can probe small samples (~1 mg). Sample size is an important consideration for biological materials, which are often difficult to prepare in bulk, and for developmental polymers and pharmaceuticals, where only small amounts are initially available. High fluxes on both SANS instruments allow experiments to be performed close to the contrast “match point” and allow examination of the finer details of the structure (e.g., adsorbed surface layers on polymer latexes, core-shell colloids, and micelles).
The Spallation Neutron Source (SNS) is an accelerator-based neutron source that provides the most intense pulsed neutron beams in the world for scientific and industrial research and development. This powerful scientific tool gives researchers detailed snapshots of minute samples of physical and biological materials. The diverse applications of neutron scattering research provides opportunities for experts in practically every scientific and technical field. With the eventual SNS suite of best-in-class instruments located on 18 beamlines, scientists will be able to count scattered neutrons, measure their energies and the angles at which they scatter, and map their final positions. These instruments will allow measurements of greater sensitivity, higher speed, higher resolution, and in more complex sample environments than ever before.

SNS Facility in Brief
The SNS at the Oak Ridge National Laboratory (ORNL) is a powerful pulsed neutron source that ORNL and its partners built under sponsorship from DOE. Hydrogen ions (H-) are produced in an ion source, accelerated in a radiofrequency (rf) linear accelerator (linac), sent through a stripping foil to convert them to protons, and stored in an accumulator ring that then delivers them as a pulsed proton beam onto a mercury target, in order to generate spallation neutrons. These neutrons are moderated and sent to distinct beamline locations in the SNS Target Building. At these beamlines, some instruments are already available for scientific research, and other instruments are under assembly or planned for future development. The ORNL operates the SNS as a user facility, granting scientists access to specific instruments for experiments using neutrons, based on peer review of their scientific research proposals that request such access.

Instruments at the SNS
The Backscattering Spectrometer, Liquids Reflectometer, and Magnetism Reflectometer are the initial three instruments that have been available to scientific users since their commissioning in the Spring of 2006. Over time, other instruments are added as a result of ongoing instrument development and construction activities that are projected to continue through at least 2013.
The World’s Most Powerful Pulsed Spallation Neutron Source

Construction of the Spallation Neutron Source Project was formally completed in June 2006. In 2007, SNS became the world’s most powerful pulsed spallation neutron source. This banner year brought one celebration after another as achievements in accelerator operations far exceeded the goals that were set. For example, on August 11, 2007, a world record for pulsed sources was set with operation at 183 kW for more than one day. Another world record of $1.1 \times 10^{14}$ protons per pulse was set on September 21, 2007. Facility plans are to offer users thousands of hours of beam time each year, and to ramp up to the full operating power of 1.4MW.
Specific Instrumentation

**Diffractometers**

The diffraction instrument suite includes powder and single-crystal diffractometers, and the SNAP instrument which is optimized for the study of powder and single crystal samples under extreme conditions of pressure (50 to 100 GPa) and temperature. SNAP is being commissioned and will be available to the user program in 2009.

Two initial instruments in the powder diffraction suite was made available to the user program in 2009. POWGEN is designed for solving the structure of complex crystalline materials. With resolution matched to the lattice spacing, it produces refinnable diffraction data in a matter of minutes, enabling a wide range of parametric studies. VULCAN is designed for materials science and engineering studies with an emphasis on the mechanical behavior of materials. Applications include stress mapping in engineering components, fundamental aspects of deformation under multi-axial loading, and use of the high neutron flux to probe transient behavior during in-situ synthesis and processing. Scheduled for completion in 2010, NOMAD is optimized for the study of disordered materials such as liquids, glasses, and nanostructured materials.

The suite of single-crystal diffractometers at the SNS will enable neutron crystallography for unit cell sizes from small molecules to macromolecules, with higher flux and higher throughput than is possible elsewhere. The atomic arrangements of crystals will be studied using the diffraction instruments TOPAZ and MaNDi, planned to be completed in 2009 and 2012, respectively. Disorder will be the specific focus of the diffuse scattering instrument CORELLI, planned for completion in 2013.

**Spectrometers**

Seven beamlines are dedicated for inelastic neutron scattering (spectroscopy), providing the capability to measure excitations ranging in energy from micro-electron Volts up to an electron Volt. BASIS is an instrument in the user program that is optimized for the measurement of the “quasi-elastic” scattering that results from the diffusional motion of atoms, ions, and molecules in materials or the quantum tunneling of molecules between different states. Three chopper spectrometers – CNCS, ARCS, and SEQUOIA – provide complementary capabilities. ARCS is optimized for measuring lattice dynamical excitations in materials such as ferroelectrics, thermo-electrics, superconductors, metals and alloys, and elementary excitations in quantum liquids. CNCS is designed for the measurement of low-energy excitations in these materials as well as in soft materials such as polymers and biological samples. SEQUOIA is optimized for the measurement of excitations in magnetic and superconducting materials. HYSPEC is anticipated to begin commissioning in mid-2010 and will use polarized neutrons to distinguish...
between lattice dynamical and magnetic excitations from small single crystal samples. VISION, planned to be commissioned in 2011, is optimized for the measuring molecular vibrations in materials such as catalysts and hydrates. Finally, the Neutron Spin Echo spectrometer will provide access to an unprecedented dynamical range of six decades in time from 1ps to 1μs. It is especially well-suited for investigations of slow dynamical processes in soft-matter.

**Large Scale Structures**

The suite of instruments for probing structures on large length scales includes two reflectometers that are currently available in the user program. The Liquids Reflectometer is designed to illuminate horizontal surfaces (including liquid surfaces) and is useful for a wide range of science, including interfacial studies in polymers and surface chemistry involving thin layers of surfactants or other materials deposited on liquid surfaces. The Magnetism Reflectometer is optimized for studies of magnetic thin films and interfaces using polarized neutrons. Both reflectometers can obtain reflectivity curves approximately six times faster than the next best instruments in the U.S. and are able to reach minimum reflectivity values that are best-in-class worldwide. The combination of the high-power neutron source and the use of advanced neutron optics also enables studies of in-plane structures using off-specular scattering. The EQ-SANS instrument provides access to a wide range of length scales from less than 0.1 to 150 nm, with applications in diverse fields including the life sciences, polymer and colloidal structures, porous materials, and nanomaterials. Planned for completion in 2012, the ultra small angle SANS instrument (TOF-USANS) will extend the length scale of traditional SANS to the μm range.
Nanoscale Science Research Centers

Exploring New Realms in Understanding Matter and Controlling its Behavior

Nanoscience is the study of materials and their behaviors at a nanometer (nm) scale—probing single atoms, clusters of atoms, and molecular structures. The scientific quest is to observe and understand how these systems function, including how they interact with their environment. The nanoscale world is often complex and not yet fully known—and scientists are working hard to understand these complexities and unknowns, because within them lie many of nature’s important secrets, such as how photosynthesis really works. Scientific advances lead to engineering applications such as the fabrication of nanomaterials with superior properties (e.g., tailored carbon nanotubes), or the purposeful control of a chemical reaction so as to direct its outcome to a useful end product (e.g., as with synthetic catalysts made from nanoparticles).

Developments at the nanoscale have the potential to make major contributions to meeting the U.S. DOE’s mission (discovering the solutions to power and secure America’s future) and the DOE’s Office of Science mission (...to deliver the remarkable discoveries and scientific tools that transform our understanding of energy and matter and advance the national, economic, and energy security of the United States).

The DOE joins other Federal agencies in sponsoring nanoscience research. The five nanoscience centers featured here are the cornerstone of the BES effort to create a vibrant research community working at the research frontier. As described in the pages that follow, each center has premier expertise in several technical theme areas, with state-of-the-art facilities staffed by laboratory scientists. This research is inherently multidisciplinary.

Researchers seek advances in nanoscience that will enhance scientific understanding in challenging technical areas. Some sample topics are as follows:
- inventing both top-down and bottom-up assembly methods to fabricate nanoscale systems,
- assessing interfaces and energy transfers between nanoscale systems, and
- determining the design principles that govern how nanoscale system properties lead to larger-scale collective properties and functionalities.

Scientific progress in these and related topics creates the potential for engineering opportunities — exploiting the gain in scientific knowledge in order to tailor, fabricate, or otherwise control a system to serve a useful purpose. In particular, the study of nanostructured materials and their behaviors may enable advances in energy-efficient processes and devices such as more efficient catalysts, fuel cells, photovoltaics, or solid-state lighting.

Molecular model of a covalent carbon nanotube network that uses nonhexagonal rings such as heptagons and octagons as linking units at the nodes. The specific network structure shown is known as superdiamond, in which four armchair nanotubes are linked at each node by octagons. (Courtesy of J.M. Romo-Herrera, M. Terrones, H. Terrones of IPICyT, Mexico, and S. Dag, V. Meunier of CNMS, Oak Ridge National Laboratory.)
Perspective on the size of various natural and man-made objects. Probing nano-scale phenomena enables scientists to understand larger-scale structures and how they function.

## Micro-World
- **Human hair**: ~50-150 μm wide
- **Dust mite**: 200 μm
- **Red blood cells**: ~2-5 μm
- **DNA**: ~2.5 nm diameter
- **Head of a pin**: 1-2 mm

## Nanoworld
- **Ant**: ~5 mm
- **Fly ash**: ~10-30 μm
- **ATP synthase**: ~10 nm diameter
- **Nanotube electrode**: ~10-100 μm wide
- **Nanotube transistor**: ~10-100 μm wide
- **Quantum corral of 48 iron atoms**: Corral diameter 14 nm

## Visible Spectrum
- **Microworld**: ~0.1 mm
- **Nanoworld**: ~0.01 mm
- **Atoms of silicon**: ~10-100 nm
- **DNA**: ~2.5 nm diameter
- **Fly ash**: ~10-30 μm
- **Ant**: ~5 mm

Lights, Camera, Action! CFN user Professor Peter Bennett of Arizona State University (at right) and facility scientist Peter Sutter (at left) align the CFN’s low-energy electron microscope, a unique tool for capturing real-time movies of dynamic surface processes at the nanometer scale.
The Center for Functional Nanomaterials (CFN) at Brookhaven National Laboratory (BNL) is a user-oriented research center that provides external users broad access to state-of-the-art facilities for the preparation and study of advanced materials. The CFN staff carries out a scientific program in energy-related materials at the nanoscale and at the same time interacts closely with external users, in an environment that stimulates scientific exchange and collaboration.

**Scientific Themes**

The overarching theme of the CFN is the development and understanding of nanoscale materials that address the nation’s challenges in energy security. Nanostructured materials play a key role in processes and devices that are energy efficient or alternatives to fossil fuels, such as catalysis, solid-state lighting, fuel cells, or photovoltaic elements. Research programs in the CFN focus on three key areas:

**Nanocatalysis:** Nanocatalysis uses tiny structures, a few nanometers in size, to accelerate chemical reactions essential to modern life. Metal-containing nanoparticles are indispensable ingredients in industrial chemical production and energy-related processes. For instance, fuel cells for powering electric vehicles use bi-metallic particles of platinum and ruthenium to catalyze the conversion of chemical energy into stored electrical energy. These particles are less than 100 nanometers in size and comprise only a few percent of the catalyst’s weight, yet they provide the active sites where chemical reactions take place. CFN scientists are now developing new experimental and theoretical tools to image and understand chemical reactions activated by such nanoparticles.

**Biological and Soft Nanomaterials:** Soft nanomaterials include polymers, liquid crystals, and other relatively “squishy” materials that fall into a state between solid and liquid, whose properties can be engineered to replicate those of conventional “hard” materials, yet are lighter, transparent, cheaper, and, in some cases, biocompatible. Of current interest at the CFN is the development of methods that mimic nature to assemble novel nanomaterials by combining inorganic and biological components that maintain biofunctionality. Such nano-engineered systems will find applications in advanced devices that require the placement of nano-objects with high precision. To achieve this, CFN scientists are developing novel ways to biofunctionalize nanoparticles and nanotubes with DNA and proteins. Using the advanced facilities available at the CFN, scientists are devising ways to use biomolecules as scaffolds, or “guides,” to build two- or three-dimensional arrays of organized nano-objects, and they are learning how cooperative effects among those objects can be exploited in applications.
Electronic Nanomaterials: At the nanoscale, materials can exhibit electrical and optical characteristics that are quite different from those of macroscopic dimensions. Examples include large enhancement in electronic mobility and large changes in the emission or absorption of light. The CFN program emphasizes the preparation of nanomaterials and understanding of their optoelectronic properties. Scientists are using advanced synthesis and nanofabrication at the CFN to create both individual nanostructures and organized assemblies of them. With advanced electrical and optical probes and theory, the researchers seek to understand the emergent properties of such materials and assemblies, and to assess their potential for applications in energy conversion devices.

Facilities and Capabilities

The CFN is a hub of interdisciplinary nanoscience research in the northeastern United States and complements BNL's other facilities and programs whose missions include developing or understanding energy-related materials. In particular, the CFN is well paired with the National Synchrotron Light Source (NSLS), where intense light reveals the structure and function of a wide range of materials. A next-generation synchrotron facility (dubbed NSLS-II), 10,000 times brighter than NSLS, will be located adjacent to the CFN, and together will make BNL a laboratory unique in the world for nanoscale research.

The capabilities at the CFN available to staff and external users can be arranged into seven major facilities:

- **Materials Synthesis**: Chemical vapor deposition and other synthesis methods for the growth of nanowires and quantum dots; biofunctionalization of nano-objects and surfaces
- **Nanofabrication**: Nanopatterning via optical, electron-beam and nanoimprint lithography; wet or reactive-ion etching, focused ion-beam, thin-film deposition by evaporation and sputtering for materials processing and device fabrication in a class-100 facility
- **Proximal Probes**: An array of scanning probe tunneling and atomic force microscopies for advanced surface and interface analysis
- **Electron Microscopy**: Advanced transmission electron microscopy that, in addition to imaging, allows the study of electronic, magnetic and optical properties at the atomic level
- **Optical Spectroscopy**: CW and ultrafast spectroscopy tools for the study of optical processes, and their dynamics, in nanomaterials, down to single molecules
- **Dedicated Beamline at the NSLS**: Especially designed for small- and large-angle x-ray scattering and ideally suited for the study of soft materials and interfaces
- **Theory & Computation**: Staff and computational tools directed to understanding the formation and structure of nanoscale materials and associated electronic, optical and chemical phenomena

The CFN also offers access to BNL’s Laser Electron Accelerator Facility for studying the kinetics of radiation-induced reactions and especially suitable to understand charge injection into and charge transport between molecules.

**Contact**

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Scientists Mark Hybertsen and Eli Sutter discuss metal-carbon core-shell nanostructures prepared in-situ in an electron microscope.
The Center for Integrated Nanotechnologies (CINT) is a Department of Energy/Office of Science Nanoscale Science Research Center operating as a national user facility devoted to establishing the scientific principles that govern the design, performance, and integration of nanoscale materials. Through its Core Facility and Gateway to Los Alamos Facility, CINT provides access to tools and expertise to explore the continuum from scientific discovery to the integration of nanostructures into the micro and macro worlds.

**Scientific Themes**

To address the national grand challenges of nanoscience and technology, CINT supports four scientific thrusts that serve as synergistic building blocks for integration research available to the user community:

- **Nanophotonics & Optical Nanomaterials:** Synthesis, excitation and energy transformations of optically active nanomaterials and collective or emergent electromagnetic phenomena (plasmonics, metamaterials, photonic lattices).
- **Nanoscale Electronics, Mechanics & Systems:** Control of electronic transport and wavefunctions, and mechanical coupling and properties using nanomaterials and integrated nanosystems.
- **Soft, Biological & Composite Nanomaterials:** Solution-based materials synthesis and assembly of soft, composite and artificial bio-mimetic nanosystems.
- **Theory & Simulation of Nanoscale Phenomena:** Assembly, interfacial interactions, and emergent properties of nanoscale systems, including their electronic, magnetic, and optical properties.

**Facilities and Capabilities**

The CINT community can access dedicated research capabilities in the Core Facility (in Albuquerque) and the CINT Gateway to Los Alamos. Together, these facilities provide laboratory and office space for researchers to synthesize and characterize nanostructured materials, theoretically model and simulate their performance, and integrate nanoscale materials into larger-scale systems in a flexible, clean-room environment. These include:

- **Materials Synthesis:** Molecular beam epitaxy; physical and chemical vapor deposition; pulsed laser deposition; inorganic, organic and polymer synthesis; nanoparticles and nanowires; thin film deposition; membranes and monolayers; mesoporous materials; biomaterial synthesis and biomolecular recognition.
- **Characterization:** Scanning probe microscopies; electron microscopies; optical spectroscopy and microscopy; single molecule spectroscopy; ultrafast laser spectroscopies; nano/micro-mechanics; Raman microscopy; terahertz spectroscopy; transport.

*Inelastic scattering of a continuous wave on a femtosecond soliton in a nanostructured photonic crystal fiber, observed with simultaneous time and spectral resolution using cross-correlation frequency resolved optical gating techniques.*
• **Nano-Micro Integration**: Photon and electron beam lithography; soft nanolithography; focused ion beam; etching and deposition.

• **Theory and Simulation**: atomistic theory; predictive capability development; interpretation and design of experiments; multi-scale material modeling; and large scale computing.

• **Discovery Platforms™**: Modular, micro-laboratories designed and batch fabricated expressly for the purpose of integrating nano and micro length scales and for studying the physical and chemical properties of nanoscale material and devices.

CINT researchers also have access to selected Los Alamos and Sandia capabilities in biosciences, microsystems, nanofabrication and computing on a limited basis. Joint proposals involving other national user facilities at both locations, including the Los Alamos Neutron Science Center and the National High Magnetic Field Laboratory, are encouraged. CINT also welcomes user proposals related to the DOE National Laboratory Center for Solid-State Lighting Research and Development.

**Preparation of quantum dot attached microtubules for an atomic force microscopy experiment.**
The Center for Nanophase Materials Sciences (CNMS) at Oak Ridge National Laboratory (ORNL) integrates nanoscale science with neutron science; synthesis science; and theory, modeling, and simulation. Operating as a national user facility, the CNMS has a highly collaborative and multidisciplinary environment for research to understand nanoscale materials and phenomena.

**Scientific Themes**
Research at the CNMS focuses on understanding, designing and controlling the dynamics, spatial chemistry, and energetics of functionality and properties of nanoscale materials and architectures.

- **Origins of Functionality at the Nanoscale:** This theme focuses on the development of instrumentation to image functionality and couples research to develop understanding of the new emerging physics and chemistry at the nanoscale with ORNL’s expertise in developing tools and techniques for forefront research with scanning probes, neutron scattering, electron microscopy, and related techniques.

- **Functional Polymer Architectures:** This theme focuses on advancing our fundamental understanding and control of polymer structure, property and function that are controlled by weak forces and whose properties are largely dependent on interfacial phenomena. This theme is rooted in controlled synthesis of well-defined polymers and bio-inspired polymers, and in rigorous nanoscale characterization.

- **Emergent Behavior in Nanoscale Systems:** This theme builds on a strong theoretical effort, focusing on understanding the emergence of collective behavior at every scale from the electronic structure to the mesoscale and includes multiscale aspects of functionality in complex systems and assemblies of nanoscale materials such as oxides and bio-inspired nanomaterials.

**Research Capabilities**
The CNMS is housed in an 80,000-ft² facility on the Chestnut Ridge Campus of ORNL, adjacent to the Spallation Neutron Source. It is equipped with a wide range of specialized tools for synthesis, characterization, and integration of hard and soft materials. CNMS encompasses expertise and instrumentation for user research in a broad range of disciplines selected to address forefront research in nanoscience and nanotechnology. Many user projects take advantage of multiple capabilities in tackling research to understand complex nanoscale phenomena.

- **Macromolecular Nanomaterials:** Synthesis and molecular level characterization of small molecule building blocks, polymers, and polymer-modified interfaces, including biologically inspired systems, deuterated molecules and polymers for neutron scattering studies.

- **Multiscale Functionality:** Synthesis and characterization of inorganic and hybrid nanomaterials and assemblies with emphasis on functional performance for energy applications. Evolution of atomic-level and nanoscale structure and dynamics with varying environments; electron microscopy and laser spectroscopy at high resolution; neutron and x-ray characterization. Specialized laser, chemical, and CVD synthesis of catalysts, carbon nanostructures, oxide heterostructures, and related structures.
Scanning probe microscopy-based manipulation of local polarization in ferroelectric materials enables studies of bias–induced phase transitions on a single-defect level, and can also serve as the physical basis of ultrahigh-density information storage. Related techniques may be used to probe electromechanical energy conversion mechanisms and electrochemical reactions on the nanoscale. (Courtesy of Stephen Jesse and Sergei V. Kalinin, CNMS, Oak Ridge National Laboratory.)

- **Scanning Probes**: Imaging of functionality and dynamics in nanostructures including magnetic domains, electromechanics, energetics, chemical reactions and local phase transitions, mesoscale and quantum electrical transport, electronic, structural and spin phases, electro-optical and photovoltaics.

- **Nanomaterials Theory Institute**: Multiscale modeling, nanomaterials design, and virtual synthesis using ultrascale computing capabilities.

- **Nanofabrication Research Laboratory**: Controlled synthesis and directed assembly of nanomaterials in a 10,000-ft² cleanroom environment; chemical and biological functionalization of nanoscale materials.

CNMS has established partnerships with other ORNL user programs. The CNMS Nanomaterials Theory Institute provides collaborative workspaces, visualization equipment, and high-speed connections to the ultrascale computing facilities of ORNL’s National Center for Computational Sciences. The intense neutron beams from the Spallation Neutron Source and from the recently upgraded High Flux Isotope Reactor afford unique and expanding opportunities for fundamental studies of the structure and dynamics of nanomaterials. The CNMS also provides a gateway to electron microscopy, atom probe, nanoindentation and other capabilities in the Shared Research Equipment and High Temperature Materials Laboratory user programs.

**Contact**

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The Center for Nanoscale Materials (CNM) at Argonne National Laboratory is a national user facility that provides capabilities explicitly tailored to the creation and characterization of new functional materials on the nanoscale. The CNM mission includes supporting basic research and advanced instrumentation development, including a hard x-ray nanoprobe beamline at the Advanced Photon Source.

**Scientific Themes**

The CNM focuses on the self-assembly of nanostructures, exploring the nanoscale physics and chemistry of non-traditional electronic and magnetic materials, developing approaches to solving various energy issues, and creating new probes for elucidating nanoscale structure and phenomena. To address these grand challenges within nanoscience and nanotechnology, using both experiment and theory, the CNM is organized around six scientific themes:

- **Electronic & Magnetic Materials & Devices**: Discovers, understands, and uses new electron- and spin-based materials and phenomena in constrained geometries for reduced power dissipation, new medical imaging methods, improved efficiency of data storage by spin current and electrical field-assisted writing, and enhanced energy conversion in photovoltaic devices.

- **Nanobio Interfaces**: Integrates biological and organic molecular assemblies with inorganic nano-architectures to create functionally integrated biomolecule-inorganic hybrid conjugates and their assemblies, that are not found in nature but are guided by its principles, for energy and information transduction, advanced medical therapies, biosensors, and novel electronic devices.

- **Nanofabrication & Devices**: Fabricates new nanostructured materials, nanodevices, and nanosystems by advancing the state-of-the-art techniques in nanopatterning that incorporate both “top-down” and “bottom-up” approaches.

- **Nanophotonics**: Controls optical energy and its conversion on the nanoscale by combining the properties of metal, organic, semiconducting, and dielectric materials to create strongly coupled states of light and matter for enhanced chemical/catalytic reactivity, photonic circuits, new chemical and biological sensors, and enhanced optical non-linearities.

- **Theory & Modeling**: Develops the theory, modeling, and computational capabilities to establish a Virtual Fab Lab for nanoscience, with the ultimate goal of designing novel nanoscale materials with user-defined properties.

- **X-ray Microscopy**: Utilize x-rays to image new materials and novel phenomena at the nanoscale, with a particular emphasis on operation of the hard x-ray nanoprobe in collaboration with the Advanced Photon Source, and develop new concepts for nanofocusing x-ray optics.
Facilities and Capabilities

The CNM building, funded by the State of Illinois, is an 85,000 ft² facility encompassing 11,000 ft² of cleanroom environments, 13,000 ft² for conventional labs, as well as offices and public areas, and is equipped with a wide range of specialized tools. Many projects take advantage of multiple capabilities for engaging in research initiatives spanning the boundaries defined by the scientific themes above.

- **Materials Synthesis:** Synthetic techniques include, for example, hierarchical assembly using bottom-up polymeric and bio-templating, core-shell colloidal nanoparticle synthesis, peptide/DNA biosynthesis methods, complex oxide molecular beam epitaxy, and PECVD nanocrystalline diamond.

- **Nanofabrication Research:** Controlled synthesis and directed assembly of nanomaterials; lithographically assisted patterning of hybrid structures; chemical and biological functionalization of nanoscale materials; electron beam lithography, focused ion beams, and nano-imprint patterning methods.

- **Proximal Probes:** An array of scanning probe tunneling and atomic force microscopy capabilities for advanced surface, interface, and magnetic analysis; near-field scanning optical microscopy.

- **Dedicated Hard X-ray Beamline at the APS:** The nanoprobe beamline provides fluorescence, diffraction, and transmission imaging with a spatial resolution of 30 nm or better over a spectral range of 3-30 keV.

- **Computational Nanoscience:** Theory and multiscale computer simulations provide the interpretive and predictive framework for understanding fundamental studies and to aid in the design of new nanoscale functional systems. The CNM’s state-of-the-art 10TFlop supercomputer accommodates highly parallel applications for modeling, simulation, and visualization.

Facilitation to other Argonne User Facilities: The CNM also assists users with facilitated access to Argonne’s other major user facilities:

- The Advanced Photon Source, one of the world’s brightest sources of x-ray beams; and

- The Electron Microscopy Center, housing several of the world’s most advanced microscopes and microcharacterization tools.

Contact

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The Molecular Foundry at Lawrence Berkeley National Laboratory operates as a national user facility specializing in the synthesis of novel nanostructured building blocks, measurement and simulation of their properties, and their integration into complex assemblies. Utilization of these capabilities is enhanced through close ties to the other DOE User facilities at LBL, including the National Center for Electron Microscopy, the Advanced Light Source, and the National Energy Research Scientific Computing Center.

Research at the Molecular Foundry exploits the uniquely multi-disciplinary environment created through its six scientific facilities by integrating the research interests of our diverse scientific staff within four major themes:

- **Combinatorial synthesis of nanomaterials:** Using robotic synthesizers to create libraries of biological and inorganic nanostructures for self-assembly and functional selection of optical, electronic and thermal properties.

- **Multimodal in situ imaging and spectroscopy:** Applying scanned probe microscopy, nanophotonics and electron microscopy to investigation of dynamic nanoscale phenomena in liquid and vapor environments, with a strong emphasis on soft materials.

- **Interfaces in nanomaterials:** Engineering the mechanical and transport properties of hybrid nanomaterials through synthesis of heterostructures and interfaces, first-principles simulations, and characterization of function.

- **“Single digit” nanofabrication:** Utilizing biological and organic templates, advanced lithographies and probe-based surface modification to deterministically fabricate arbitrary structures with sub-10nm precision.

### Facilities and Capabilities

The Molecular Foundry is a six-story, 94,500 square-foot laboratory. Its six facilities are fully equipped with state-of-the-art, sometimes one-of-a-kind instruments. It includes approximately 4,800 square feet of modular cleanroom space comprised of Class 100 labs for nanofabrication/lithography and clean measurement, and a 5,500 square-foot low vibration, low-electromagnetic-field laboratory housing state-of-the-art imaging and manipulation tools. Space is allocated for equipment and staff dedicated to the preparation and characterization of inorganic, organic and biological nanostructures and for a theory group to collaborate with the experimentalists. Offices and laboratories are available for visiting scientists and resident technical user-support staff.

- **Inorganic Nanostructures Facility:** Synthesis and integration of diverse inorganic nanocrystals, nanotubes, and nanowires is pursued, driven by energy conversion and electronics applications. Advanced synthetic capabilities include a unique Workstation for Automated Nanocrystal Discovery and Analysis (WANDA) and two cold-wall Metal-Organic Chemical Vapour Deposition (MOCVD) reactors for nanowire growth. Synthetic development focuses on heterostructured nanocrystals and nanowires, as well as soluble metal-chalcogen clusters, and the integration of these building blocks into composite materials and functional systems. In addition to optical spectroscopies and x-ray diffraction, in house expertise includes the fabrication and characterization of transistors, thermoelectric devices, and photovoltaic cells based on nanomaterials.

- **Nanofabrication Facility:** This facility provides state-of-the-art lithographic and thin-film processing, including high-resolution electron-beam, focused ion beam and nano-imprint lithography. The research program emphasizes new lithographic, etching and resist technologies, their integration with chemical and biological nanosystems, and the development of nanoelectronic, nanomagnetic and nanophotonic devices.

In situ AFM image of S-layer proteins during self-assembly in fluid. Black (white) circles indicate newly attached (detached) proteins. Inset shows high-resolution image revealing tetrameric substructure of growth units.
• **Organic and Macromolecular Synthesis Facility:** This facility provides expertise and access to organic molecules, macromolecules and their assemblies, as well as components, polymers, hybrid materials and functional assemblies. In addition to self-assembly, techniques range from classical organic to combinatorial to polymerizations of all types. In addition to instrumentation supporting the synthetic methods, this facility has also capabilities enabling separation and purification of the molecules and assemblies and their detailed characterization. The in-house program of staff scientists focuses on nanoporous polymers for hydrogen storage and separations, microfluidic devices, polymers for fabrication of photovoltaic cells and light emitting diodes, organic polymer-inorganic nanocrystal hybrids, as well as functional supramolecular assemblies.

• **Biological Nanostructures Facility:** This facility studies the synthesis, analysis and mimicry of biological nanostructures. Expertise and capabilities are available to develop new materials based on the self-assembly of peptides, proteins, nucleic acids, and bio-inspired polymers. New bio-friendly imaging probes based on functionalized inorganic nanocrystals are being developed and are available to facilitate state-of-the-art bio-imaging studies. Synthetic biology techniques are used to re-engineer organisms and create hybrid biomolecules to interface with devices. Additional capabilities include synthesis of bio- and biomimetic polymers, bioconjugation, and combinatorial peptide and peptoid library synthesis and screening. Protein expression, microbial and eukaryotic cell culture, phage display, cellular engineering and biological microscopy, including deconvolution and single molecule imaging are offered.

• **Imaging and Manipulation Facility:** Facility staff work with users to develop and apply advanced imaging and characterization tools to a wide range of nanoscale materials and systems using electron, optical and scanning probe microscopes. Technical capabilities include analytical transmission electron microscopy, field-emission scanning electron microscopy, commercial and lab-built scanning probe microscopes operating in ambient, controlled gas environments, liquids and vacuum, and ultra-fast optical confocal and near-field spectro-microscopy, as well as conventional optical microscopy, x-ray photoemission spectroscopy and Auger electron microscopy. Research efforts include tip-enhanced Raman spectroscopy (TERS) and coherent anti-Stokes Raman spectroscopy (CARS) using plasmonic antennas, development of advanced cantilever probes, imaging of soft biological materials, in-situ SEM and TEM, and using cathodoluminescence to study nanoscale optical properties.

• **Theory of Nanostructured Materials Facility:** This Facility works with users and performs internal research to understand experiments, develop new theoretical and computational methods, and predict the behavior of new nanoscale materials and assemblies. Expertise is offered in a broad range of quantum and classical simulation techniques—from first-principles electronic excited state and transport methods, to coarse-grained statistical mechanical approaches—for the study of spectroscopy, charge dynamics, and self-assembly of hard and soft nanomaterials. Selected recent interest areas include charge transport in single-molecule junctions, new techniques for x-ray spectroscopy, nanoscale solar energy conversion and storage, and soft matter self-assembly dynamics.

• **Affiliated Laboratories:** A number of research laboratories at Lawrence Berkeley National Lab and the University of California at Berkeley, whose capabilities complement those of the Foundry facilities, are available to User Projects. Information on these labs can be found at the Molecular Foundry’s website.

• **Other User Facilities at LBL:** A single review process can be used for joint proposals to the Foundry and LBNL’s other User Facilities including: The Advanced Light Source, one of the world’s brightest sources of ultraviolet and soft x-ray beams; The National Center for Electron Microscopy, housing several of the world’s most advanced microscopes and microcharacterization tools; and The National Energy Research Scientific Computing Center, providing high-performance computing tools and expertise.
Electron Interactions with Matter Provide Atomic Scale Information

Electron beam characterization provides structural and chemical information about materials over critical length scales complementary to those probed by neutrons and photons. Today’s most advanced electron microscopes can produce beams of extremely high brightness that can be used to generate an image, spectrum, or diffraction pattern from sub-nanometer volumes of material. Consequently, electron beam methods play a key role in a wide variety of fields ranging from physics, chemistry, and materials science to biology and medicine. The information gained from such studies helps researchers understand how materials behave, leading to new breakthroughs in materials performance. For example, studies on materials used in buildings, aircraft, bridges, and other structures provide knowledge that can help researchers improve a material’s strength, durability, and/or resistance to corrosion. Electron beam studies of other materials, such as electronic or magnetic materials that are important in modern technologies, allow researchers to better understand how a material conducts electricity or changes its magnetic properties. In addition, electron beams can be focused to very tiny probes, and thus are ideally suited to studies of nanoscale materials. This capability allows scientists to explore new regimes of man-made materials with unique or tailored properties. Therefore, electron beam studies will be leading the way for technology that employs the next generation of advanced materials.

Electron scattering is a key tool for materials characterization because of its unique ability to obtain structural and spectroscopic information from nanoscale volumes of material and, in some cases, individual atoms. Electrons can be accelerated to nearly the speed of light and focused into atomic-resolution probes and images using electromagnetic lenses. Over the last 50 years, electron microscopy has contributed enormously to our understanding of materials and their properties—by helping engineers to analyze failure modes, developers to design stronger alloys, and explorers to discover new materials. Most of our knowledge about dislocations—the fundamental units of deformation in crystals—is based on diffraction contrast imaging in electron microscopy, and entirely new classes of materials such as nanotubes and quasicrystals were discovered by electron microscopy. Much of our understanding of how high-temperature superconductors work or how atomic-scale defects control oxides—with applications ranging from skin care products to recording media—has been learned from electron scattering. The exceptional ability of electron microscopy to correlate structure and properties on a nanometer scale have positioned the use of this tool at the core of materials science.

The BES electron beam microcharacterization centers are an important part of DOE’s arsenal of extraordinary facilities for scientific research that are made available as a shared resource to the scientific community. Collectively, these centers have made significant contributions to broad areas of materials science, including phase transformations, defects, deformation, radiation effects, science of interfaces, surfaces and thin films, alloys, ceramics and microelectronic materials. The instrumentation and associated expertise at the electron beam microcharacterization centers are playing a central role in the development of nanoscience. Understanding how the properties of a nanoparticle are related to its microstructure and processing is an essential part of the scientific process that underlies the development and discovery of new materials. The materials studied at these facilities find applications in advanced technologies that power economic growth, support national security, and foster the efficient generation and usage of energy.
Outreach Activities in the Scientific Community

Electron microcharacterization national user facilities play a role in expert training of scientists in the theory and practice of electron scattering, and in leading communications and educational events such as workshops, summer schools, discussion forums, special issues of journals, and symposia at scientific meetings. Such events are in support of outreach to the scientific community and help stimulate discussion on topical issues as well as training of the next generation of scientists.
The Electron Microscopy Center (EMC) at Argonne National Laboratory (ANL) provides scientific researchers with essential resources for electron beam characterization of a wide range of materials and through a suite of instrumentation that includes several of the world’s unique electron microscopes. The instrumentation available to investigators in the EMC provides a broad range of capabilities for imaging, diffraction, and spectroscopy that are essential for world-class materials characterization. The EMC also provides capabilities for in situ studies of materials using state-of-the-art microscopy. Traditionally strong research activities in the EMC include defect and transformation processes, microstructure and properties of complex oxides, physical behavior of nanoscale and magnetic materials, and the structure of amorphous materials through fluctuation electron microscopy. More recently, the EMC has developed programs in magnetic structures and domain dynamics. The EMC also provides expertise in aspects of fabrication and characterization of nanostructured materials based on focused electron and ion beams. These research activities have been enabled by the recent development within the EMC of electron beam–based probes of local properties, quantitative modeling and imaging of defect structures, and nanoscale dynamic phenomenon imaged and recorded in real time by advanced in situ techniques. At the same time, the EMC provides new scientific opportunities through the innovative development of techniques and instrumentation.

The EMC currently operates eight instruments for user research. EMC users come from universities, national laboratories, and industry and conduct studies ranging from imaging of electron-sensitive soft materials to in situ observations of phenomena at elevated and cryogenic temperatures in pure metals and alloys, semiconductors, and ceramics. Access to the EMC is through a peer-reviewed proposal process. The instrumentation in the EMC includes transmission electron microscopes, scanning electron microscopes, and ion beam–based instruments. Many of the instruments in the EMC are optimized for analytical electron microscopy, including state-of-the-art transmission and scanning transmission electron microscopes (TEM and STEM, respectively), high-resolution scanning electron microscopes (SEM), scanning electron microscopes with environmental capabilities (ESEM), and dual ion- and electron-beam instruments (FIB-SEM) that provide unique capability for nanoscale manipulation of materials. These instruments are utilized by users for a wide range of materials research. In addition, the EMC operates the Intermediate Voltage Electron Microscope (IVEM)-Accelerator, a facility provides capability for high-resolution imaging of a material under the influence of a high-energy ion beam. This unique capability is employed by investigators from around the world for a variety of in situ studies, especially for dynamic recording and structural characterization of the effects of ion irradiation.

Another important aspect of research conducted within the EMC is to drive important developments in the field of electron microscopy and scattering. For example, the EMC has led the development of aberration-correcting optics for use in the Transmission Electron Aberration-corrected Microscope (TEAM) instrument. The EMC is also developing unprecedented capabilities for in situ experiments that will be ideally suited to comprehensive studies of single nanoparticles in controlled environments, including gaseous and liquid environments. Complementing this work is the EMC’s research in the area of new technologies for high spatial resolution analytical spectroscopy and its application to computationally mediated experiments. Very high spatial resolution chemical imaging is being developed utilizing aberration-correction, and the EMC is working to make

A combined focused ion beam and scanning electron microscope (FIB-SEM) instrument provides researchers in the EMC with unique capabilities for nanoscale manipulation of materials.
those capabilities available to the broad scientific community. Together these efforts provide a balanced program that provides leading edge tools needed to understand and characterize current and future technologically advanced nanomaterials.

Through the resources within the EMC, researchers carry out world-class studies in a variety of areas. For example, using the EMC’s focused ion beam – SEM capability, researchers produced the first three-dimensional images of the interior of a fuel cell — providing a new tool for the study and development of fuel cells. These images provide a nanometer-scale view of the inside of a fuel cell, much like magnetic resonance imaging provides a view inside the human body, and are helping to unravel how fuel cells work so they can be improved and made more reliable.

Scientists are using the IVEM-Accelerator to provide a key experimental confirmation for the origin of interstellar material to improve our understanding of the processes that formed the materials of the early solar system. In this work, scientists used this EMC capability to make the first in-situ TEM ion radiation study of pyrrhotite, a mineral that is found in both stardust and in interplanetary dust particles. The results provided confirmation of the current working model for the formation of these nanophase aggregates, and have potential application for modeling the space radiation exposure histories of other extraterrestrial materials. In different experiments, in situ studies are carried out in the IVEM-Accelerator facility that are providing critical insight into the durability of nuclear reactor and waste storage materials that is an important part of the nation’s energy future.

The analytical capabilities of the EMC’s field-emission gun scanning transmission electron microscope have been used to establish a correlation between grain boundary solute segregation and the atomic structure of the grain boundary in an engineering alloy. High spatial resolution analysis of composition at various grain boundaries compared to the composition within grains was used to show that special, highly ordered grain boundaries exhibit less segregation than less well ordered boundaries in an aluminum-copper alloy. Improving our understanding of solute effects on microstructural evolution will greatly improve the control of industrial alloys used so widely in today’s electronics, aerospace and other industries. A new analytical technique, high-resolution momentum resolved spectroscopy, is being developed by EMC researchers to investigate the orientation dependence of the electronic properties of materials. This technique uses angular-dependent spectroscopy, measuring either x-ray or electron energy loss, to probe local structure on the length scale of a few Å. By varying the angle of incidence of the electron beam, a researcher can probe the orientation dependence of electronic and magnetic states. As an example, this approach has been used to measure the magnetic anisotropy in hematite, Fe_3O_4, using the electron energy loss variant technique known as HARECES (High Angular Resolution Electron Channeling Electron Spectroscopy).

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Since its inception in 1977, the Shared Research Equipment (SHaRE) user facility at the Oak Ridge National Laboratory (ORNL) has provided users with access to a suite of advanced electron microscopes and staff scientists with microscopy and materials expertise in order to collaborate on research projects. Transmission, scanning-transmission, scanning electron microscopes, and ancillary spectroscopic capabilities are all made available to U.S. researchers (including materials scientists, chemists, physicists, and biologists) for the structural and compositional characterization of materials (e.g., metals, ceramics, polymers, semiconductors, and composites) at the μm to atomic scales. The SHaRE program has 3 primary focused research areas:

- Transmission/scanning-transmission (TEM/STEM) and scanning electron microscopy (SEM), including aberration-corrected STEM and high-angle annular dark field (HAADF) imaging, equipped with spectroscopic techniques such as Electron Energy Loss Spectroscopy (EELS), Energy Dispersive Spectrometry (EDS), and Electron Backscatter Diffraction (EBSD)/orientation imaging microscopy (OIM).
- Atom probe tomography (APT) using a local electrode atom probe (LEAP); Atom probe tomography is an atomic spatial resolution microstructural technique for visualizing and quantifying the three-dimensional distribution of solute content in a material. The technique enables solute clusters to be detected and the size, number density, and composition of precipitates from their earliest stages of evolution to be determined.
- Surface science using X-ray Photoelectron Spectrometry (XPS) and Auger electron Spectroscopy (AES).

Faculty and students of U.S. accredited universities, industrial researchers, and scientists at national laboratories, including ORNL, may access SHaRE’s state-of-the-art microstructural characterization facilities by submitting a SHaRE research proposal. SHaRE staff will collaborate with users to conduct world-class materials characterization research and to develop new/improved analytical techniques, data analysis, and instrumentation. Access is granted free of charge, based on the scientific excellence of the proposed research and the ability to publish the scientific results in the open literature. Access may also be granted for proprietary research on a full-cost recovery basis.

Proposals to access the SHaRE User Facility are accepted at any time during the year and will be immediately assessed internally for feasibility and then sent for external review. Each proposal is reviewed by 3 members of the 50-member SHaRE Proposal Review Committee (PRC), where each member has expertise in one of the focus research areas supported within SHaRE. Once a proposal is reviewed and accepted, access to the requested SHaRE facilities is granted.

In the first image, single Pt atoms in fresh catalyst appear as white dots ~ 1Å in diameter. After reduction treatment to enhance catalytic activity, the second image shows these Pt atoms forming small (<2nm) clusters.
Accomplishment and Highlights

- Approximately a hundred SHaRE-supported journal publications are published each year.
- The SHaRE Facility collaborates with ORNL’s Center for Nanophase Materials Science (CNMS) to make advanced microscopy facilities available to CNMS users, thus becoming the microscopy research center for many nanoscience research projects both internal and external to ORNL.
- Internationally-recognized expertise in high spatial resolution STEM, Z-contrast imaging, probe aberration-correctors, and STEM-based analytical electron microscopy, including experience in both the practical application and the theoretical aspects of sub-Angstrom STEM imaging.
- The SHaRE User Program has traditionally focused on specific research topics that are unique to this facility, such as spectroscopy technique development and application to materials science, irradiated materials characterization, and atom probe tomography. Recently, SHaRE capabilities have also focused on the characterization of the atomic-structure of materials using aberration-corrected HAADF-STEM (especially related to catalysts, interfaces, nanostructures, etc.).
- SHaRE scientific research has focused on the development and application of “in-situ” microscopy for materials characterization. For example, TEM holders are being developed for high-resolution in-situ liquid STEM, atomic force microscopy, nanoindentation, electrochemistry, etc. In-situ characterization is certainly a large part of research and development efforts in SHaRE.
The National Center for Electron Microscopy (NCEM) develops and supports some of the world’s most powerful tools for atomic-scale characterization using electron scattering. Located at Lawrence Berkeley National Lab, the facility provides forefront resources and expertise to help researchers design, diagnose and discover new materials.

As a national user facility, NCEM is open to scientists from universities, government and industrial laboratories. Its distinguished scientific and technical staff assists and guides researchers in exploring the behavior of materials over length scales ranging from single atoms to hundreds of nanometers.

Based upon NCEM’s unique instrumentation, its infrastructure of user support, and the research expertise of its scientific staff, the facility has been a leading lab for atomic resolution electron microscopy for over two decades. The high-voltage Atomic Resolution Microscope (ARM) first brought atomic resolution electron microscopy to materials science. This instrument helped solve a number of important scientific puzzles such as the origin of diamond-hexagonal silicon, the atomic structure of strengthening particles in aluminum alloys, and the atomic arrangement at contacts in semiconductor devices. A second major advance was the achievement of 1 Angstrom resolution with the 300 kV One-Angstrom Microscope (OAM) in combination with computer processing. The OAM has been providing sub-Angstrom phase-contrast imaging for a decade, delivering the first images of nitrogen in gallium nitride, imaging sub-Angstrom atomic spacings of aluminum in sapphire, and reporting the direct observation of expanded crystal planes near the active surface of a catalytic platinum-iron particle.

NCEM’s experience in high-resolution electron microscopy served as the foundation of the Transmission Electron Aberration-Corrected Microscope (TEAM) project, a collaboration with other DOE-funded electron scattering efforts to redesign the electron microscope around aberration-correcting optics. The prototype double-corrected monochromated TEAM 0.5 microscope officially opened for user operations in the fall of 2008, one year before the dedication of the final TEAM instrument, which will feature the first corrector for chromatic as well as spherical aberration.

NCEM scientists conduct high-level research by applying new techniques to critical materials problems, and by collaborating with external research groups on the application of electron optical methods to materials characterization. Core areas of expertise include atomic resolution imaging of nanostructures, high resolution spectroscopy and in-situ observation of reaction mechanisms and dynamics. Advances in these scientific fields are supported by the development of techniques and instrumentation.
Instrumentation and Infrastructure

NCEM houses a full complement of highly specialized instruments designed to characterize the atomic structure and composition of materials. In addition to the TEAM microscope, the center features other advanced instruments such as two monochromated transmission electron microscopes and an aberration-corrected scanning transmission electron microscope. The facility also operates a unique instrument for imaging of magnetic surfaces with spin-polarized low-energy electrons, and develops techniques and instrumentation for dynamic in-situ experimentation. In support of its electron microscopes, NCEM maintains a state-of-the-art specimen preparation facility including a focused ion beam nanofabrication instrument, as well as advanced data analysis stations.

NCEM’s exceptional capabilities are making an important contribution to DOE’s scientific infrastructure. The center’s unique instrumentation and expertise, and its proximity to other LBNL user facilities such as the Molecular Foundry and the Advanced Light Source provide a strong platform for scientific discovery and innovation. With the growing sophistication of nanoscience, the need for nanoscale characterization will continue to increase rapidly. The future development of NCEM will complement Berkeley Lab’s strength in new energy technologies and fundamental science by further advancing electron optical instruments and methods to investigate the structure, composition and bonding of materials with extraordinary precision.

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In his famous 1959 lecture, now widely seen as the founding vision for nanoscience, Nobel laureate Richard Feynman said it would be easy to analyze any complicated chemical substance; “all one would have to do would be to look at it and see where the atoms are. The only trouble is that the electron microscope is one hundred times too poor … I put this out as a challenge: Is there no way to make the electron microscope more powerful?”

50 years later, a group of scientists prepared to meet the Feynman challenge by completing the TEAM microscope at LBNL’s National Center for Electron Microscopy.

With an image resolution of half an Angstrom the TEAM microscope will provide unprecedented opportunities to observe the atomic scale order, electronic structure and dynamics of individual nanostructures. This will allow scientists to address key issues in nanoscience that were identified in a series of workshops. Foremost among these are the observation of local electronic structure and bonding, single atom spectroscopy and atomic resolution tomography. Using unique aberration-corrected electron optics with an ultra-stable sample stage in a specially-designed “quiet” microscope lab it will become possible to record a series of real-time atomic resolution images, which can be used to assemble 3D tomographic images of individual nanocrystals.

The Transmission Electron Aberration-corrected Microscope (TEAM) project is 5-year collaborative effort amongst leading microscopy groups to design and construct the world’s most advanced electron microscope as a platform for a new generation of electron scattering instruments built around sophisticated aberration-correcting optics. The vision for the TEAM project is the idea of providing a sample space for electron scattering experiments in a tunable electron optical environment by removing some of the constraints that have limited electron microscopy until now. The TEAM microscope will feature unique corrector elements for spherical and chromatic aberrations, a novel Atomic Force Microscope-inspired specimen stage, a high brightness gun and numerous other innovations. The improvement in sensitivity, brightness, signal to noise and stability will make it possible to address major challenges in nanoscience.

The world-leading monochromated, aberration-corrected TEAM 0.5 microscope is capable of half-Angstrom imaging in both, broad-beam and scanning-probe modes of operation.
To meet its ambitious goals, the TEAM project utilized three test instruments to develop novel technologies at three different locations in a parallel process: optimization of aberration-corrected STEM imaging at ORNL, development of chromatic aberration correction for TEM imaging at ANL and double-corrected instrument integration for half-Angstrom image resolution at LBNL. The initial prototype instrument (TEAM 0.5) installed at LBNL’s National Center for Electron Microscopy, has demonstrated unprecedented performance, and was made available as a user instrument in October 2008. TEAM 0.5 will be surpassed only by the final TEAM I microscope, which will feature the first chromatic aberration corrector in a TEM, other sophisticated electron optics, an extra-high brightness source, a revolutionary new stage, high throughput electron detectors and integrated software to enable remote operation.

The unparalleled capabilities of the TEAM instrument enable the development of novel techniques to meet challenges such as atomic resolution tomography that were previously out of reach. Its high brightness gun, improved signal-to-noise ratio, exceptional resolution, chromatic aberration correction, and tunable electron optical environment help minimize the effect of radiation damage and optimize contrast and data collection.

One of the key scientific goals of the TEAM instrument is the experimental atomic-scale analysis of individual nanoparticles on the size scale that is just becoming accessible to first principles calculations. Combining the best experimental and computational capabilities presents new opportunities for exceptional insights into the behavior of materials.
Locations of the BES scientific user facilities discussed in this publication. For further information, contact each facility or BES at http://www.sc.doe.gov/bes/.
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Robert Winarkski (left) of the CNM X-ray Microscopy group explains the CNM x-ray nanoprobe beamline to a group of students from Notre Dame University.

The advanced target-moderator assembly at the Lujan Center. Lujan Center targets inaugurated flux-trap design and partially coupled moderators, now standard throughout the world.

Light-induced charge transport is possible in these dumbbell architectures of bridged donor-acceptor particles scaffolded by DNA (top) and protein (bottom), studied in the CNM’s Nanobio Interfaces group.

X-ray crystal structure of the plant TIR1-ASK1 complex bound to auxin and a substrate degron peptide. The auxin receptor TIR1 (blue) binds to ASK1 (magenta), together forming a mushroom-shaped protein complex. The plant hormone auxin (green and red spheres) and the substrate degron peptide (orange) occupy a single pocket on top of the TIR1 mushroom cap with auxin sitting at the bottom. A previously unknown inositol hexakisphosphate (IP₆) molecule is found in the middle of TIR1 right under the auxin-binding site (the red stick model in the center of the mushroom "cap").

Chemistry students Miranda Vanden Brink (undergraduate, foreground) and Saravuna Kumar Thauman Pillai (graduate student, background), from the University of South Dakota conduct experiments in the catalyst synthesis labs at the Center for Nanophase Materials Sciences in Oak Ridge, Tennessee, during an extended visit.
About the back cover:

SLAC National Accelerator Laboratory’s linear accelerator serves as the backbone of a new kind of x-ray laser, the Linac Coherent Light Source (LCLS). This photograph shows the construction of a transport hall to carry beams of electrons toward experimental facilities nearly half a mile away. The LCLS will allow scientists to make freeze-frame images of atoms and molecules as they vibrate and interact, revealing the chemistry of life as it happens.

The Advanced Light Source (ALS) is a national user facility that generates intense x-ray radiation for scientific and technological research. The ALS is operated by the Lawrence Berkeley National Laboratory (LBNL) of the University of California for the U.S. Department of Energy. Operation for users began in October 1993. As the world’s first third-generation synchrotron radiation source, the ALS welcomes researchers from universities, industries, and government laboratories around the world.

The luminous zone of the flame shown here (just to left of the glowing red sampling cone) has the typical blue-violet or blue-green color associated with chemiluminescence from electronically excited CH and C₂.

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