

The **ADVANCED Photon Source** at Argonne National Laboratory



**Lighting the Way
to a
Better Tomorrow**

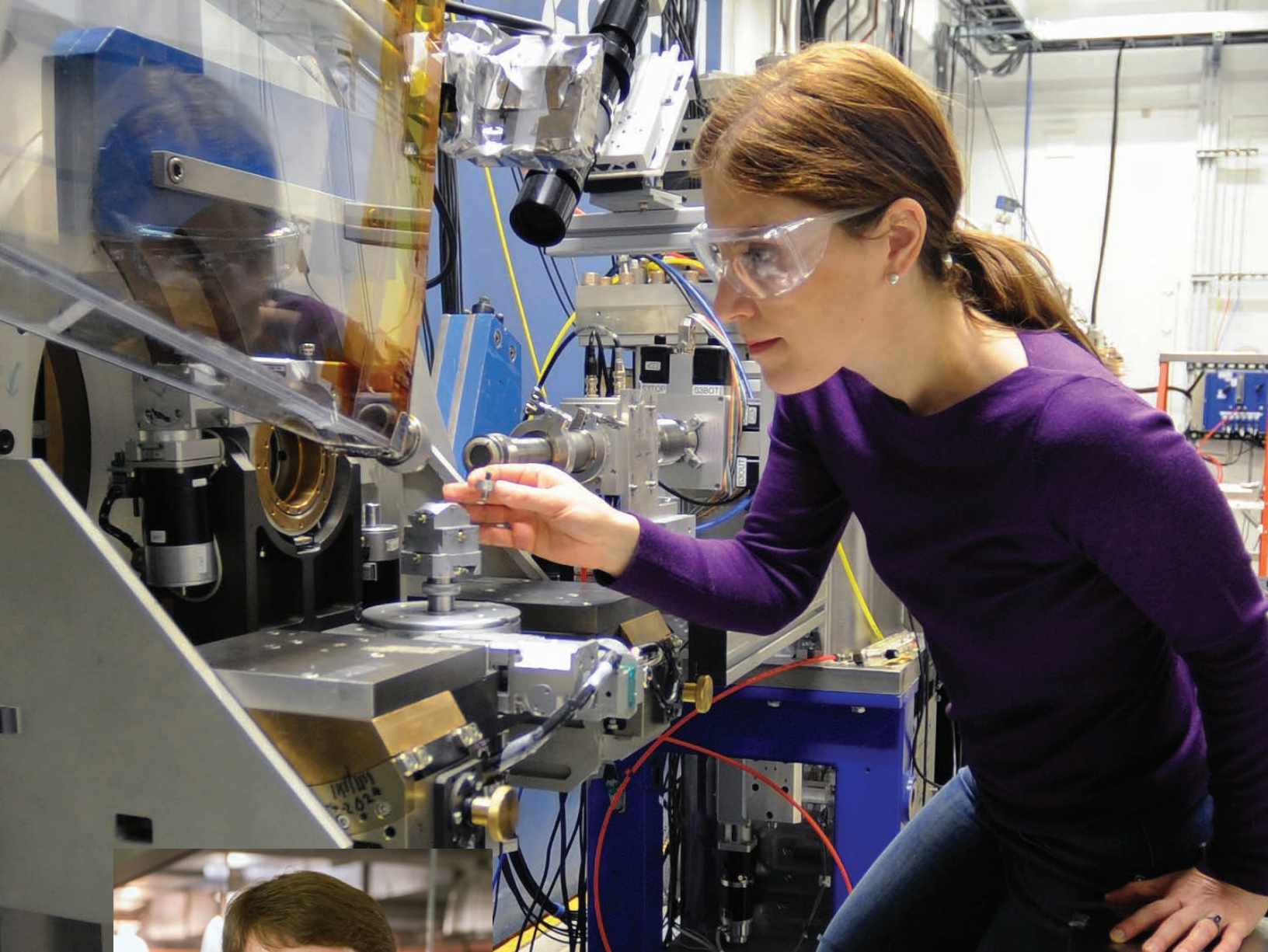
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On the front cover: Aerial view of the Argonne 400 area. The Advanced Photon Source facility is adjoined by the Advanced Protein Characterization Facility (the white building at the top of the circular APS experiment hall) and the Center for Nanoscale Materials (the white buildings to the right of the experiment hall).

Photo, top of page 1: Placing a sample in the resonant inelastic x-ray scattering spectrometer at the X-ray Science Division Sector 27 x-ray beamline. This instrument is used to measure the intrinsic properties of electronic excitations, which are key to understanding the electrical and magnetic behaviors of materials.

Inside back cover: The Argonne National Laboratory campus.



Stephen Streiffer

Welcome to the Advanced Photon Source!

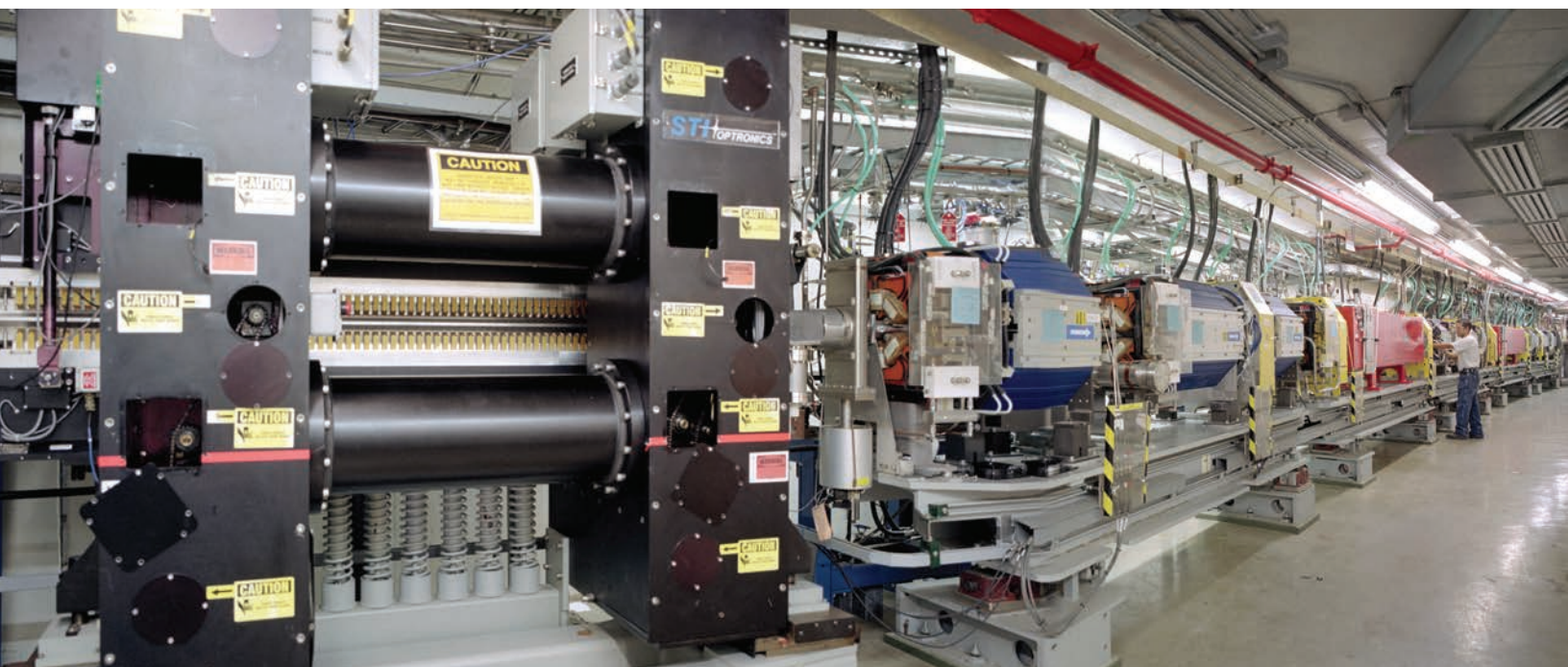
This brochure serves as an introduction to the Advanced Photon Source (APS). But these few pages can't do justice to the U.S. Department of Energy's (DOE's) largest x-ray light source research facility in terms of the number of researchers (or "users") who carry out experiments utilizing the extreme-brightness, highly energetic x-ray beams produced here, nor to the impact of the research carried out by our users.

Our users come from every U.S. state and Puerto Rico and Washington, D.C., and several foreign countries, and represent every major scientific discipline.

We like to think of our colleagues, the APS physicists, chemists, engineers, technicians, computer experts, and administrative and support personnel as the best of the best, all working toward the common goal of supporting our users and furthering the U.S. DOE Office of Science mission: "The delivery of scientific discoveries and major scientific tools to transform our understanding of nature and to advance the energy, economic, and national security of the United States."

Stephen Streiffer
*Argonne Associate Laboratory Director for Photon Sciences and
Director, Advanced Photon Source*

The Advanced Photon Source



Computer and smart-phone microprocessors, data storage devices and displays, next-generation energy storage, clean energy technologies, and drugs that improve our health are just a few of the by-products of scientific breakthroughs achieved by scientists using research facilities like the U.S. Department of Energy Office of Science's Advanced Photon Source (APS) at Argonne National Laboratory. Most of the technologies and pharmaceuticals we take for granted today are the end result of scientific discoveries made over a number of years by researchers who specialize in the study of physical and biological materials.

The APS is one of the world's most productive x-ray light source facilities. Each year, the APS provides high-brightness x-ray beams to a diverse community of more than 5,000 researchers in materials science, chemistry, condensed matter physics, the life and environmental sciences, and applied research. Researchers using the APS produce over 2,000 publications each year detailing impactful discoveries, and solve more vital biological protein structures than users of any other x-ray light source research facility. APS x-rays are ideally suited for explorations of materials and biological structures; elemental distribution; chemical, magnetic, electronic states; and a wide range of technologically important engineering systems from batteries to fuel injector sprays, all of which are the foundations of our nation's economic, technological, and physical well-being.

APS scientists and engineers innovate and share technology that is at the heart of advancing accelerator and

One sector of the 2/3-mile-circumference APS storage ring, where electrons orbit at nearly the speed of light to produce x-ray beams for research. The large instrument at the left is an undulator insertion device, the source of the APS high-energy, high-brightness, highly-penetrating x-rays.

light-source operations. This includes the insertion devices (arrays of permanent-magnet devices as seen at left in the photo above) that produce extreme-brightness x-rays prized by researchers, lenses that focus the x-rays down to a few nanometers, instrumentation that maximizes the way the x-rays interact with samples being studied, and software that gathers and manages the massive quantity of data resulting from discovery research at the APS.

The APS facility, which is large enough to encircle a major-league baseball stadium, houses a suite of particle accelerators that, along with myriad other equally sophisticated technical components, produces a beam of high-energy (relativistic) electrons. As the electrons pass through the powerful insertion devices they emit synchrotron radiation, which covers a broad segment of the electromagnetic spectrum with wavelengths that are shorter than visible light. The wavelengths of extreme ultraviolet and x-ray radiation match the corresponding features of atoms, molecules, crystals, and cells — just as the longer wavelengths of visible light match the sizes of the smallest things the human eye can see. These x-ray beams equip researchers to carry out cutting-edge experiments while training the next generation of scientists for continuing discovery.

Nobel Prize-Worthy Research

The three recipients of the 2009 Nobel Prize in Chemistry, working independently, published papers on their award-winning science based on data collected at the APS, the National Synchrotron Light Source at Brookhaven National Laboratory, and the Advanced Light Source at Lawrence Berkeley National Laboratory, all U.S. DOE x-ray light sources. Biochemists Venki Ramakrishnan of Cambridge, England's, Medical Research Center; the late Thomas Steitz of Yale University; and Ada Yonath of Israel's Weizmann Institute shared the award for their individual studies of the structure and function of the ribosome (right), which works as a protein factory in all organisms from humans to bacteria. Some of the Nobel-related studies were performed at the Structural Biology Center (SBC-XSD) macromolecular x-ray (MX) crystallography facility at APS Sector 19. Steitz and Yonath are also users of the National Institute of General Medical Sciences (NIGMS) and National Cancer Institute (GM/CA-XSD) structural biology facility at APS Sector 23, the Northeastern (NE-CAT) x-ray beamlines at APS Sector 24, and the BioCARS user facility at APS Sector 14.



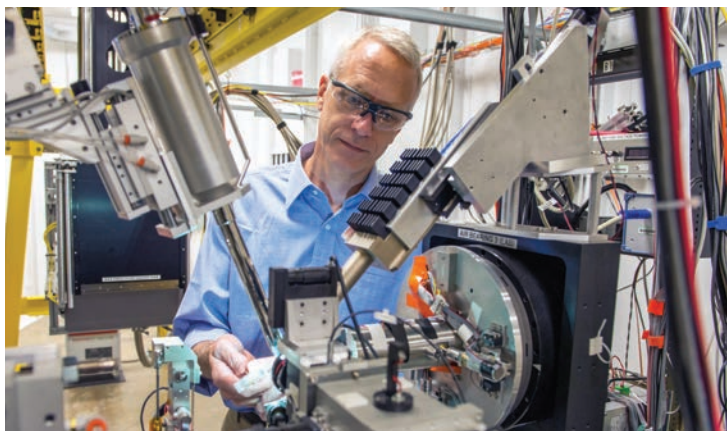
Venki Ramakrishnan



Thomas Steitz

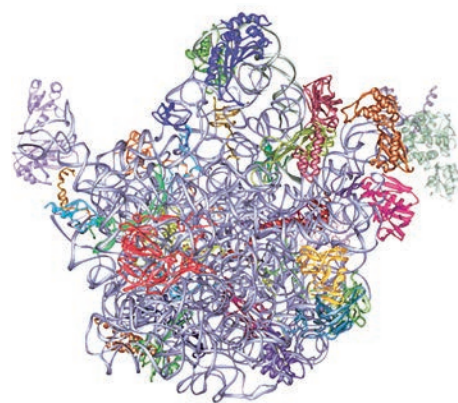


Ada Yonath



Brian Kobilka at the GM/CA-XSD beamline.

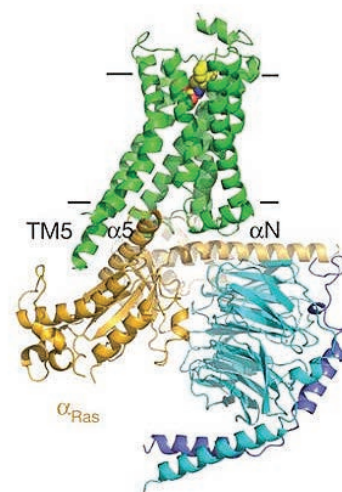
The 2012 Nobel Prize in Chemistry was awarded to Brian Kobilka (Stanford University) and Robert Lefkowitz (Howard Hughes Medical Institute and Duke University) for their work on G-protein-coupled receptors (GPCRs), thanks in large part to research performed at GM/CA-XSD. Kobilka and his colleagues made the first discovery of the structure of a human GPCR (right, bottom figure) that is responsible for a number of different biological responses, including breathing and dilating arteries. A second breakthrough occurred in 2011, when the Kobilka group again used Sector 23 to determine the structure of a GPCR at the exact moment that the protein-receptor complex signals across the membrane. This study represented the first time that a GPCR had been caught “in the act” of carrying out its biological mission, a discovery the Nobel Committee called “... the Holy Grail, a high-resolution structure of an active ternary complex.”



Ribosome structure



Robert Lefkowitz



G-protein-coupled receptor structure

Focus on the Future: The APS Upgrade

The APS Upgrade (APS-U) Project transforms today's APS into a powerful storage-ring-based, hard x-ray light source that equips scientists with a vastly more powerful tool for investigating and improving the materials and chemical processes that impact nearly every aspect of our lives. It affords the researchers in the synchrotron x-ray science community extraordinary new capabilities to fulfill U.S. research needs for decades to come.

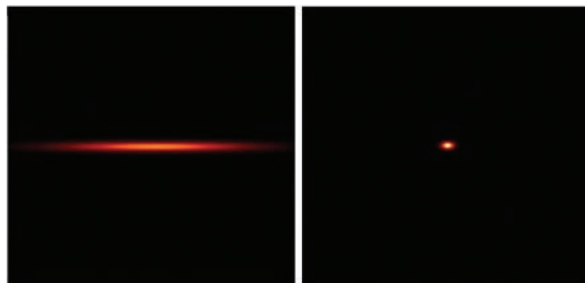
The APS is one of the most powerful synchrotron x-ray facilities in the world. The upgraded APS will ensure that the U.S. keeps this leadership position even as new or upgraded facilities similar to the APS-U are being planned or are under construction in France, Brazil, China, Japan, and other countries, threatening to supplant the historic U.S. leadership in synchrotron x-ray science.

The scientific opportunities enabled by the APS-U are seemingly boundless, empowering APS researchers to continue to seek answers to urgent societal and economic needs, including but certainly not limited to:

- New and improved materials for energy applications, tomorrow's technologies, and infrastructure improvements.
- Development of revolutionary technological breakthroughs.
- A deeper understanding of the molecular foundations of myriad diseases leading to new ways to treat, or even prevent, cancer and other deadly illnesses.

The APS-U Project entails removing the existing 2/3-mile-circumference electron storage ring equipment and replacing it with an entirely new, cutting-edge ring: a "multi-bend achromat" (MBA) lattice (sidebar) for the production of synchrotron x-rays, in addition to new and improved x-ray beamlines (see page 7). Thanks to the new MBA-based electron accelerator, the APS-U increases the brightness (focused intensity) of x-rays compared to the already extremely bright APS. This allows APS users to map an atom's position, identity, and movement, affording a view of matter at the atomic scale and in four dimensions far beyond capabilities available today.

The APS-U high-energy x-rays penetrate bulk materials and operating physical and organic systems, delivering macroscopic fields of view with nanometer-scale resolution and excellent time resolution in addition to the capability for lensless imaging with the highest spatial resolution. Together with rapid, ongoing advances in x-ray optics, insertion devices, detectors, computing, and theory the APS-U makes it possible to explore a new landscape of scientific problems that previously were completely inaccessible to researchers.



Shown above are simulated x-ray beam source profiles, comparing the beam from the APS today (left) to the beam from an upgraded APS storage ring (right) with an APS-U MBA lattice.

The term "lattice" refers to the sequencing and types of electromagnets in a particle accelerator like the APS electron storage ring. Unlike today's storage ring lattice segments, which contain a few strong two-(di)pole magnets, the MBA lattice incorporates a larger number of shorter dipole magnets.

The electromagnets in the new APS lattice have smaller centers to "compress" the electron beam, resulting in the spot simulated in the graphic at right above. Other components, such as the vacuum chambers threaded through the magnets to provide a racetrack for the electrons, are also scaled down from the current APS storage ring.

The MBA lattice reduces the current horizontal spread of the APS electron beam to a tightly focused spot resulting in a 2-to-3 orders-of-magnitude increase in brightness (the number of photons of a given wavelength and direction concentrated on a spot per unit of time) and coherence (a laser-like beam spot) relative to today's APS, depending on the photon energy.

Narrowing the APS x-ray beam to a spot as opposed to a "pancake" will allow more photons to be delivered to the sample being studied, which will greatly increase the amount of information obtained by experimenters and the speed at which that information is collected.

The APS-U Project includes the 6-gigaelectronvolt MBA storage ring and associated technical systems, new insertion devices such as permanent-magnet undulators or superconducting undulators for all insertion device beamlines, new x-ray beamlines, and enhancements to existing beamlines including improved optics and stability. The development, procurement, fabrication, and testing of these components are well under way.



Above: A computer-aided rendering of a typical APS-U storage ring sector showing the MBA-lattice configuration of red, blue, and purple electromagnets and associated components. Rendering by the AES Design and Drafting Group.



Left: APS-U technicians prepare an assortment of APS-U storage ring magnets for tests in the new, state-of-the-art APS-U Magnet Measurement Lab at Argonne.

Below: A monochromator from Instrument Design Technologies, Inc., designed for use with the upgraded APS, undergoes acceptance testing by an APS-U physicist following delivery to the APS. The monochromator is now installed and operating at the 2-ID beamline.



Above: Technicians from the APS Accelerator Systems Division Magnetic Devices Group and the APS Engineering Support Division Survey and Alignment Group working on a helical superconducting undulator insertion device. Super-conducting undulators are now in operation in the APS storage ring and are a key component of the APS-U. They produce stronger magnetic fields and thus higher-energy x-rays than conventional permanent magnetic undulators having the same period lengths.

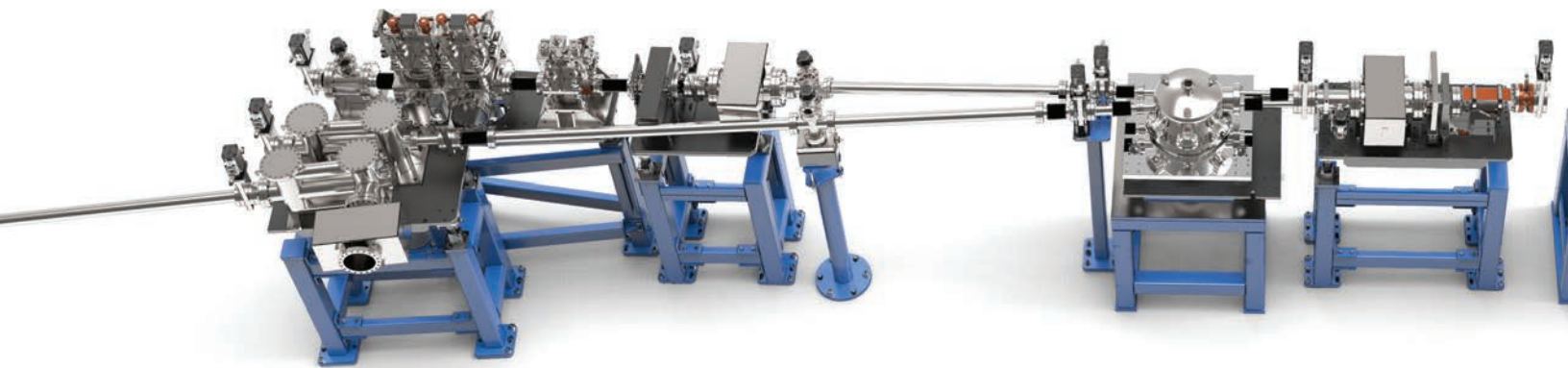
New Tools for High-Impact Science

The brightness of APS-U-delivered x-rays significantly enhances the majority of high-energy research techniques employed by APS users. The APS-U exceeds the capabilities of today's synchrotron light sources in brightness and coherent flux (photons on sample) in the hard (high-energy) x-ray range, enabling a transformational array of ways to study the structure, properties, and functionality of matter.

Here are a few examples:

- **Mesoscale Engineering and Advanced Materials:** Science is creating new and improved materials at an ever-increasing pace. The APS-U promises to accelerate this

- **Environmental Sciences and Geosciences:** Studying matter at extremes of pressure, temperature, and electromagnetic fields is transforming our understanding of a large array of fundamental problems in environmental and geoscience science. But this type of research is made difficult by the wide variety of materials that fall into this category. Today's most pressing challenge in environmental science and geoscience is to employ unified research techniques that permit studies of these complex, multi-component systems at a range of length scales, from atomic to meso- and macroscopic. The APS-U delivers unique x-ray brightness that provides for



critical evolutionary process by providing unique, non-destructive characterization tools to probe the structure, shape, electronic configuration, and evolution of matter under extreme and operational conditions.

- **Soft Matter:** Polymers, liquid crystals, colloidal suspensions, and much of living matter lie at the intersection of physics, chemistry, biology, and engineering. Soft matter possesses properties unlike any others seen in nature, which open the way to possibilities ranging from understanding diseases to development of next-generation microelectronics. The APS-U provides tremendous opportunities for soft-matter discovery by improving existing imaging techniques across unprecedented length scales and time scales.

- **Biological and Life Sciences:** Living systems with highly efficient and effective structures, materials, and processes are useful for innovations in engineering, materials science, medicine, and other fields. But living systems are typically highly complex. The APS-U equips researchers with many synchrotron x-ray techniques (diffraction, scattering, imaging, and spectroscopy) for studying biological systems across length scales from molecules to tissues. These new capabilities will revolutionize our understanding of biological systems and the structures of both healthy and diseased biological machinery.

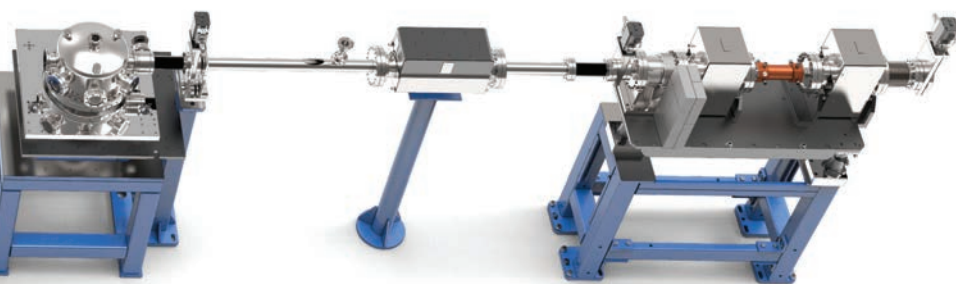
more than doubling the current range of pressure and temperature that can be accessed for probing materials at extremes of pressure and temperature; aiding our understanding of the conditions under which the solar system formed; imaging materials and interfaces in natural systems to link molecular to mesoscale (the scale between the nanoscale size of molecules and the size of materials measuring in micrometers) transport phenomena in the environment; understanding bacterial metabolic function; and evaluating the long-term environmental impacts of engineered nanomaterials.

- **Condensed Matter Physics:** The APS-U impacts the way we explore physical fundamentals that underpin the world around us, from creating new states of matter and exploring the properties that are revealed to understanding and potentially tailoring the way materials behave over a wide range of time and length scales. Examples of these include gaining new perspectives on quantum criticality, superconductivity, phases under extreme conditions, fluctuations in correlated electron systems, and dynamic manipulation of novel polarization phases.

Maximizing these impactful capabilities requires new and improved beamlines equipped to utilize the extreme brightness of APS x-rays, and new tools for APS researchers. The new beamlines, to be operated by the APS X-ray Science Division (XSD), are:

- **Polar: The Polarization Modulation Spectroscopy Beamline** enables studies of materials at the mesoscale in order to detect electronic and magnetic differences by means of tuning and controlling competing ground states under extreme high-pressure conditions (in the megabar range). Bright, polarized x-ray beams open remarkable opportunities for discovery of new phases and furthering our understanding of quantum matter.

- **The PtychoProbe Beamline** offers the highest possible spatial-resolution x-ray microscopy for obtaining structural and chemical information with a goal of focusing at or below a 5-nanometer spot, the capability of ultra-fast scanning of the beam across the sample, and lensless imaging to the nanometer level. These capabilities



Computer-aided rendering of a typical new APS-U beamline. (Rendering by Don Jensen and Dana Capatina, APS X-ray Science Division.)

accelerate the discovery of complex materials and establish the APS as the go-to place for hard x-ray, high-resolution microscopy.

- **The 3D Micro and Nano Diffraction Beamline** attacks many problems in materials science, physics, and geoscience. The new APS will provide small, intense x-ray spots to investigate spatial variations and correlations of strain and structure that define a wide range of scientifically and technologically important materials.

- **The ATOMIC Beamline** uses the enhanced coherence of the upgraded APS x-ray beam for high-resolution studies of the structural, chemical, and physical properties exhibited by advanced functional materials. There is a strong need for a single technique that can acquire atomistic structural information across many length scales in full three-dimensional detail. Coherent diffractive imaging at ATOMIC combines that capability with *in situ* and *operando* environmental cells for transformative impacts on many scientific disciplines.

- **The Coherent High Energy X-ray Beamlines** use coherent x-rays to advance the frontier for *in situ* real-time studies of advanced materials synthesis and chemical transformations in natural operating environments, addressing questions in condensed-matter physics and en-

vironmental science. This promises breakthroughs in discovering, developing, and understanding the materials and processes needed to address global challenges in energy, the environment, health, and national security.

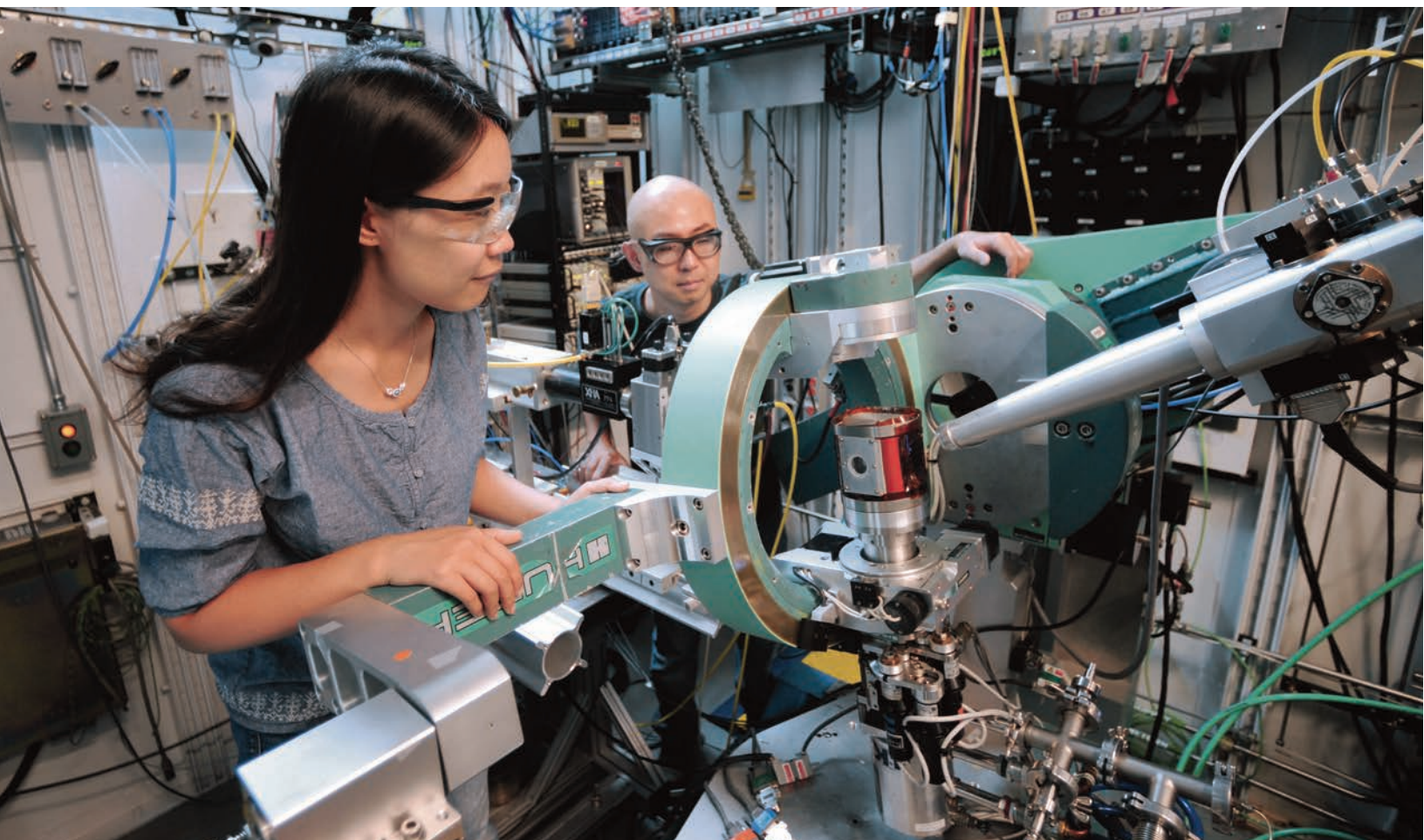
- **The High-Energy X-ray Microscope Beamline** is a long beamline that will allow users to investigate structure and structural evolution within bulk materials, often in extreme environments, with the high-energy x-ray scattering techniques and novel coherence-based techniques enabled by the upgraded APS. These types of materials are directly relevant in many areas, including mechanical engineering, biophysics, nuclear energy, energy storage, and advanced manufacturing.

- **The In Situ Nanoprobe Beamline** has a relatively large optical working distance. This enables (trace) elemental mapping and investigation of chemical states in complex functional materials and materials systems such as catalysts, batteries, photovoltaic systems, and nanoscale Earth and environmental samples during synthesis and operation under actual environmental conditions. This long beamline is designed to study these systems across many lengths scales, in two and three dimensions under *in situ* conditions.

- **The X-ray Photon Correlation Spectroscopy Beamlines** propel studies in key areas in physics and materials science and engineering, including the behavior of glass-forming materials when undergoing a phase transition from the liquid state to the glassy state, structural dynamics in super-cooled liquids, and fluctuations associated with competing mesoscale interactions in new materials. The beamline enables dynamics-related studies in areas as diverse as *in situ* rheometry (deformation and flow of materials), nano-fluidic flow, and high pressure; and advances potential applications in an array of technologies, from energy and transportation to health, agriculture, and national defense.

- **The Coherent Surface-Scattering Imaging Beamline** is ideal for probing and understanding mesoscale space-time correlations by combining a surface x-ray probe with state-of-the-art x-ray optics and detectors to study a range of materials surface and interface phenomena. These include the evolution of biological membranes and complex molecules in aqueous environments; thin-film and quantum-dot growth at surfaces and interfaces; assembly of polymer nanocomposites; and structural analysis of novel three-dimensional, nano-scaled electronic circuits in additive manufacturing.

Exploring the Physical Sciences Frontier



The 6-circle diffractometer in the research station at an XSD beamline in APS Sector 4.

Making Fuels from Shale Gas

By alloying just a small amount of platinum with copper, researchers made a new catalyst that prevents the well-known problem of coking: breaking hydrocarbons down into a carbon-rich soot called “coke,” which coats a catalyst surface and blocks reactions from happening. In research at XSD beamline 12-BM employing x-ray absorption spectroscopy, the structure of the single-atom alloy structure was shown to consist of highly active platinum atoms surrounded by coke-resistant copper. This new catalyst is a less expensive and more energy efficient way to transform shale gas into fuels and useful chemicals. Catalysts that suffer from coking must be cleaned periodically to continue working, sapping away time and energy precious to a large industrial plant. But with this discovery of coke-resistant, single-atom alloys, catalysts for small hydrocarbons become much easier to work with. The catalyst is not expensive; with relatively inexpensive copper as the main component, the relatively high price of platinum is mitigated.

How a Catalyst Makes Room for Hydrogen

Catalysts for the electrolysis of water are crucial to the development of cost-effective hydrogen generation systems for energy storage. Synthetic pentlandite shows considerable promise as a suitable catalyst, but the mechanism underlying its effectiveness has not been fully understood. Working at the XSD beamline 3-ID, researchers used nuclear resonance inelastic x-ray scattering to demonstrate that hydrogen atoms are efficiently captured into lattice sulfur vacancies at the surface of the mineral. The finding should be of value in the effort to refine pentlandite's performance.

Keeping Li-Ion Batteries from Fading Away

The rechargeable lithium-ion (Li-ion) batteries in our smart phones, laptops, and various other personal electronic devices make them completely portable, allowing us to unplug so long as the batteries are charged. But rechargeable batteries don't last forever, and the more times they're recharged, the less energy

they store. The question is: Why? Researchers used x-ray beams from two U.S. DOE-SC national user facilities, including the XSD 11-ID beamline (where they carried out *operando* x-ray diffraction studies) and a beamline at the Stanford Synchrotron Radiation Light-source, to reveal a major cause of charging capacity fading in Li-ion batteries: intergranular cracking in the interface between the electrode granules.

Developing New Materials for Nuclear Energy

The development and use of new materials have improved safety and affordability in the aerospace and automotive industries. Scientists are working to apply similar principles to the development of new materials for nuclear energy in order to extend reactor lifetimes and increase accident tolerance. Materials utilized within reactors face a variety of challenges, including the critical ability to withstand high radiation at high temperatures. A team of researchers used high-energy x-ray diffraction at the XSD 1-ID beamline and microbeam Laue diffraction at the XSD 34-ID beamline, as well as the Argonne Tandem Linear Accelerator System and the Argonne Intermediate Voltage Electron Microscope Tandem Facility, to demonstrate preliminary results for a new technique for evaluating fuel and reactor materials. This technique, which requires less time and expense than current techniques, involves irradiating potential materials with high-energy, atomic nucleus ions and then assessing the damage using synchrotron x-ray diffraction and electron microscopy.

Does Rock Water Loss Trigger Earthquakes?

Earthquakes can occur under the ocean at subduction zones, where two rigid plates in the Earth's crust meet and one is forced to sink beneath the other. Some quakes occur many kilometers below the surface in rock that is assumed to be too soft to allow fracturing. To help explain the existence of these intermediate-depth earthquakes, researchers carried out a study at the GeoSoil-EnviroCARS (GSECARS) 13-BM x-ray beamline. They placed rock samples under high pressure and high temperature, mimicking the relevant geological conditions. Using x-ray diffraction and acoustic energy measurements, they tracked the stress and mineralogical transformations within the rocks, showing that rock fracturing occurred as water was squeezed out of the rock. The connection to dehydration suggests that intermediate-depth earthquakes arise due to a sudden transfer of stress between different types of minerals in the rock.

Tracking the Currents of Memory

The experiences we live through are often etched into our minds. Something similar can happen for so-called “memory-resistors,” or memristors. These electronic devices can hold a memory of the past in their resistance, which can be in different states depending on the last electric current that passed through them. But the mechanism underlying this electronic memory has not been entirely clear. A new x-ray study performed at the XSD 2-ID x-ray beamline using multimodal *operando* x-ray microscopy charts the movements of atomic defects within a memristor. The results show that memristive behavior occurs when the defect concentration is near a threshold value. Guided by this information, engineers may be able to fabricate devices that operate consistently around this threshold concentration.

Seeing Elements Atom by Atom

What if you could identify elements atom by atom as your eyes scanned across any material? Researchers recently demonstrated an ability to do just that by imaging cobalt nanoclusters with a combination of scanning-tunneling microscopy and synchrotron x-ray absorption spectroscopy at the Center for Nanoscale Materials (CNM)/XSD x-ray beamline 26-ID. Their work can be extended to map any element with near-atomic resolution and investigate quantum effects within nanomaterials one particle at a time.

Weaving Gold and DNA into Metamaterials

Invisibility cloaks, super-speedy chemical reactions, and lenses that change their focus in response to environment are just a few of the technological tricks that could be performed by metamaterials, designed materials carefully structured to manipulate electromagnetic radiation. They are often made of several different components — for instance, two different metals, or DNA plus gold — and the most exciting metamaterials are structured at the nano level so that they can manipulate visible light. Metamaterials are a burgeoning field of research, but how to build specific metamaterials rapidly and at scale is still an unsolved problem for materials scientists. A team of researchers utilized high-brightness x-rays and the grazing-incidence small-angle x-ray scattering technique at the XSD 12-ID beamline to show that building metamaterials using templates that employ a combination of DNA and lithography to align the components is a viable method of creating custom materials at usable scales.

A Phase Change in Calcium Fluoride Crystals

Placing a crystal under extreme pressure can change its structure from one form, or phase, to another. Determining exactly how crystals change phase under compression is an important area of materials physics. The availability of x-ray diffraction at synchrotron facilities like the APS has allowed scientists to observe compression-driven phase changes in unprecedented detail. Most of this research focused on pressure-induced phase changes with slow (static) compression over minutes. Using diamond anvil cell compression tests at the High Pressure Collaborative Access Team (HPCAT-XSD) 16-ID beamline, and high-velocity impact x-ray diffraction studies at the Dynamic Compression Sector (DCS) 35-ID beamline, researchers saw microstructural phase changes over nanoseconds within a two-element (calcium fluoride) crystal subjected to extreme pressures achieved through high-velocity instantaneous shock compression and by statically squeezing the samples. Real-time observations of these phase transitions will provide a template for the phase transitions of similarly structured compounds. It is anticipated that the experimental methods and results of this study will lead to improved modeling of phase transitions over nanosecond time scales within a wide range of complex materials.

Why Zinc Makes Platinum a Better Catalyst

Many industrial processes rely on catalysts to increase the rate of a reaction and inhibit the formation of unwanted products. How catalysts work and how they can be improved are somewhat mysterious. Using x-ray absorption measurements and resonant inelastic x-ray spectroscopy at the Materials Research (MR)-CAT 10-BM beamline, and x-ray diffraction at the XSD 11-ID beamline, researchers identified two distinct mechanisms by which the addition of a zinc “promoter” to a platinum catalyst increases the selectivity of a catalytic reaction and the rate at which it works. The findings, backed up by theoretical calculations, should make it easier to design and refine catalysts in rational ways rather than relying on trial and error, which could pave the way for the design of still better catalysts that would increase the economic value of ethane, propane, and other light alkanes in shale gas.

A Low-Density Phase in Supercooled Water

To explain some of the puzzling properties of water, notably the fact that ice floats, theorists have embraced

a model in which the molecular structure of liquid water fluctuates between two phases with different densities. By means of high-resolution x-ray diffraction measurements on ice that was allowed to rapidly decompress, researchers working at the HPCAT-XSD 16-ID beamline found strong evidence for the existence of the hypothesized low-density liquid phase in supercooled water, suggesting that the relationship between the two phases may partly explain water’s unusual characteristics.

Putting a Little English on Antiferromagnets

Magnetic materials are everywhere, from the decorations on your refrigerator to the GPS in your phone. Ferromagnets, the most common kind, are tremendously useful because they are easy to control, but it also means that most ferromagnetic devices can be easily detected or destroyed using magnets available over the counter. Engineers would like to be able to build computers and other electronic devices out of a different family of magnetic materials, called “antiferromagnets.” Such devices would be magnetically invisible and thus much harder to detect than the typical ferromagnetic devices available today. They could make computers stealthier and much, much faster. However, that also means they would be more difficult to control. Researchers utilized the XSD 4-ID and 6-ID x-ray beamlines for magnetic circular dichroism and x-ray diffraction experiments, respectively, to demonstrate a super-thin antiferromagnet that is readable and controllable using outside magnetic fields. It’s the first step to building practical antiferromagnetic devices.

Confirming the $J_{\text{eff}}=3/2$ Ground State

The phenomenon of spin-orbit coupling in certain degenerate molecular systems can lead to unusual quantum phases, topological Mott states, and other interesting phenomena, some of which may even be important for spintronics and related applications. One such state arising from spin-orbit coupling is the molecular $J_{\text{eff}}=3/2$ ground state, which has been predicted by theory and modeling to be particularly exotic. A group of researchers using the resonant inelastic x-ray scattering spectrometer at the XSD 27-ID x-ray beamline has achieved the first experimental verification of this state in the lacunar spinel GaTa_4Se_8 .

Storing Data in 3-D with Light

An intensive research effort is under way worldwide to replace electrons with photons in solid-state switch-

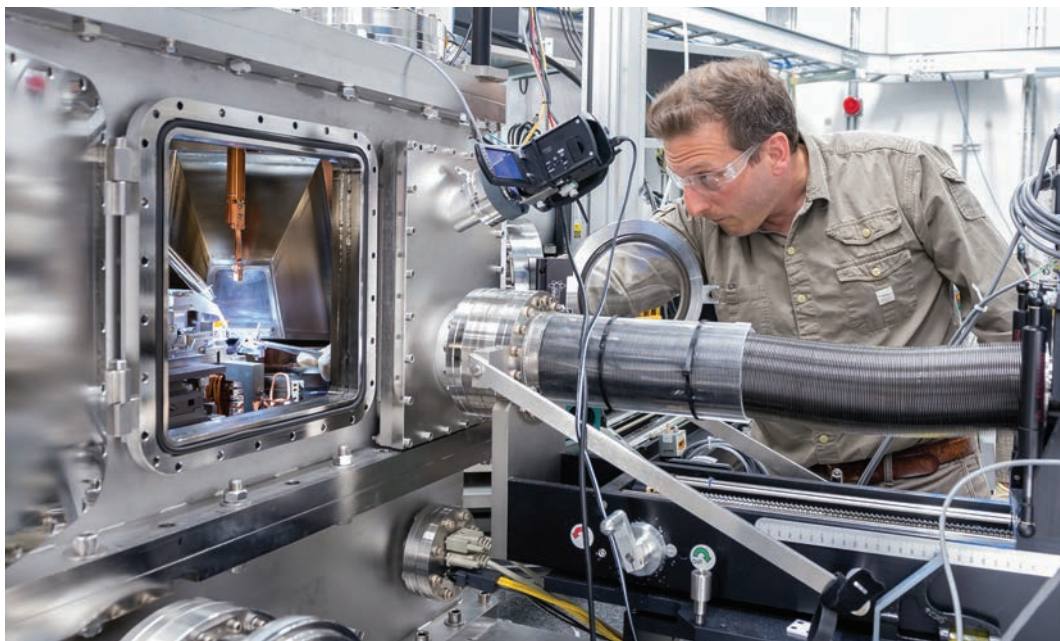
ing devices. Such switches have applications in optical data storage and quantum computing, but their adoption has been hampered by interactivity problems between light and the material. An international research team reported the outcome of x-ray diffraction experiments carried out at the ChemMat-CARS x-ray beamline 15-ID: the first-ever confirmed synthesis of an osmium sulfur dioxide ammine complex and the testing of its properties for solid-state optical switching. These results demonstrate that the osmium sulfur dioxide complex is a rare example of a material that can be developed into a promising osmium-based, solid-state optical switch.

Increasing the Solubility of Aerosol Iron

“Red tide” is the term applied to a harmful algal bloom. Red tides make the news for their striking visual effect and their ability to disturb ecosystems through the sudden die-off of fish. What may be less well known is that the phytoplankton stimulated by easy access to iron can sequester a great deal of carbon dioxide. But phytoplankton are only able to access iron when in solution, and the solubility of iron depends on many factors. Research carried out with micro x-ray fluorescence and x-ray absorption near-edge structure spectroscopy at the XSD 2-ID beamline examined the composition and oxidation states of aerosol iron and revealed that while many variables affect its solubility, including source and the path it travels, an increase in acidity increases the solubility of aerosol iron. A better understanding of the role of aerosol iron has implications for insights about sequestration of carbon dioxide by marine algae and other biogeochemical impacts.

Excitations from a Vibrating Crystal Lattice

The properties of materials crucially depend on the arrangement of the atoms that make up the material. For example, atoms that are farther apart will tend to vibrate more slowly and propagate sound waves more



Placing a sample in the Hard X-ray Nanoprobe operated by the CNM and XSD at Sector 26 of the APS. This instrument is used for imaging strain fields near defects and interfaces to understand structural correlations with chemical activity and dynamics in quantum and nanoscale materials for energy.

slowly. Researchers used the HERIX spectrometer and inelastic x-ray scattering at the XSD 30-ID beamline to discover “topological” vibrations in iron silicide (FeSi). These topological vibrations arise from a special symmetrical arrangement of the atoms in FeSi and endow the atomic vibrations with novel properties such as the potential to transmit sound waves along the edges of the materials without scattering and dissipation. Looking to the future, one might envisage using these modes to transfer energy or information within technological devices.

Inducing Polarization in Superlattices

The novel properties of superlattices make them ideal candidates for electronic applications that benefit from a separation of electrons and holes, such as generating electricity or photoelectrochemical hydrogen for fuel cells. While the abilities of superlattices composed of ferroelectric oxides have been previously examined, there has been little work done looking at non-ferroelectric oxides. Researchers used x-ray absorption near-edge spectroscopy at the XSD beamlines 20-BM and 20-ID to investigate a non-ferroelectric superlattice composed of two perovskite oxides — SrTiO_3 and LaCrO_3 — and characterize its properties. The results include strong polarization, comparable to that of ferroelectric systems, which opens up new material options for photochemical and photovoltaic applications.

Voids in Oxidizing Iron Nanoparticles

Anyone with a rusting vehicle has suffered the effects of iron oxidation. Iron nanoparticles can also “rust” to form an iron oxide compound, but this is not necessarily a bad thing, because these nanoparticles have important technological applications. To better understand this oxidation process, researchers used small- and wide-angle x-ray scattering at the XSD 12-ID beamline to watch the step-by-step geometrical transformation of iron nanoparticles into hollow iron-oxide shells due to oxidation, which involved the appearance and growth of numerous voids within the nanoparticles until the voids merged to form a hollow shell of iron oxide. Understanding this complex process offers hope for controlling the properties of these nanoshells and, for instance, allowing manipulation of nanoparticle porosity, nanoshell crystallinity, and their iron-to-oxygen ratio, which may lead to improvements in catalytic processes, electric batteries, and clean-fuel technologies.

Getting Into 3-D Printing

Additive manufacturing, also known as three-dimensional (3-D) printing, has long been employed to create prototypes and is increasingly being utilized to build commercial items, from sneakers to aircraft parts. Most 3-D printing with metal works by scanning a laser across a thin layer of metallic powder, then melting it to join with the layer below until a structure has been built up. Many such parts, however, suffer from mechanical weaknesses caused by defects in the layers, such as rough surfaces and large numbers of pores. It has been difficult to measure exactly how the defects form, in part because the process happens so quickly. Now scientists using the XSD 32-ID beamline for x-ray imaging studies have developed a technique to capture defects and better understand the processes involved in laser-bed powder fusion so that they will be able to adjust parameters such as laser power and scan speed to achieve the best microstructures. They say the technique will likely become critical in additive manufacturing, particularly as new materials and new printing processes are developed.

Carbon Fibers Built with Boron

Developing ultralight low-emission automobiles made from carbon fiber may come down to a question of cost. The current standard carbon fiber goes for around \$10/lb, a price that limits its use in industrial and automotive settings, and that is largely due to the expense of

the fiber's precursor, polyacrylonitrile. To lower the cost, scientists are exploring alternate precursor materials, including polyethylene. Carbon fibers developed from polyethylene, while less expensive, aren't as strong or stiff as those made from polyacrylonitrile, limiting their application. Graphitization at high temperatures improves the fiber properties, but the extra heat is expensive. The key to developing an economically friendly polyethylene-based carbon fiber at low cost is doping the polyethylene with boron, making it possible to catalyze the graphitization process and produce high-quality fibers at much lower temperatures. Researchers working at the DuPont-Northwestern-Dow (DND) CAT 5-ID beamline designed and built an apparatus that enabled them to use wide-angle x-ray diffraction to study the fibers as they were carbonized under tension, revealing the secrets of how boron boosts carbon fiber strength and stiffness on a microstructural level.

What Hath Iron (and Other Ions) Wrought?

Nearly all structural lumber in the Northern Hemisphere is composed of softwoods that are susceptible to attack from brown rot fungi, which degrades the structural components of wood and assimilates the nutrients via extracellular processes using both enzyme and non-enzyme mechanisms. Ion movement and translocation is an important aspect of brown rot and is implicated in both modes of decay. In addition to having important implications for building materials, a better understanding of the role of ions in the fungal decay process of wood and other biomaterials has significance for fields ranging from plant pathology and forest ecology to carbon sequestration and sustainable biorefinery applications. While scientists know that many ion species play important roles in fungal decay mechanisms, little is known about how abundant each species is, exactly where they accumulate within fungi, the wood they attack, and their movements throughout the decay process. Researchers used x-ray fluorescence microscopy at the XSD 8-BM beamline to map and quantify physiologically relevant ions in wood being decayed by the brown rot fungus *Serpula lacrymans* Wulfen. Their results show that the fungus actively transports some ions, such as iron, into the wood and controls the distribution of ions at both the bulk wood and cell wall length scales.

How Heat Moves

For tiny electronic devices, heat is the enemy. If the devices get too hot, they malfunction. The need to keep

the semiconductor brains of our devices cool has led to some impressive advances in the engineering of fans and airflow. But there's a limit to the cooling power of air-flow, especially as computer processors grow tinier. Now that processors are being made on the nanoscale — meaning individual components can be made from just a few atoms — we need to think differently about how to keep them cool. At its most basic, heat is movement. When atoms get hot, they jiggle, and the hotter they get, the more they jiggle. Researchers used the XSD 7-ID beamline, the only beamline in the world able to accommodate terahertz-field-pump and hard x-ray diffraction microscopy probe measurements, to track how far a single wave of jiggle, or “phonon,” could move through a material without bumping into a different phonon. Being able to visualize phonon movement so clearly will help researchers understand how heat moves through nanoscale computer processors, and lead to processors that can work faster without melting.

Carbon Clusters and High Explosives

The detonation process of high explosives is complex and difficult to understand. What is known is that a fast shockwave decomposes energetic material to produce carbon clusters, including graphite and nanodiamonds. Researchers used the DCS 35-ID beamline and time-resolved small-angle x-ray scattering for the first study in the U.S. to measure the formation and evolution of carbon clusters during the detonation of a plastic explosive containing the widely used insensitive high-explosive triamino-trinitrobenzene. Data obtained after a (controlled and safe) detonation revealed the time scales of carbon cluster formation and provided insights into the chemical and physical transformations that occurred. A better understanding of high-explosive detonation improves our scientific models underpinning the physics involved in the detonation process relevant to the U.S. nuclear deterrent.

Unconventional Battery Materials

Even as our electronic devices become ever more sophisticated and versatile, battery technology remains a stubborn bottleneck, preventing the full realization of promising applications such as electric vehicles and power-grid solar energy storage. Among the limitations of current materials are poor ionic and electron transport qualities. While strategies exist to improve these properties, and so reduce charging times and enhance storage capacity, they are often expensive, difficult to implement

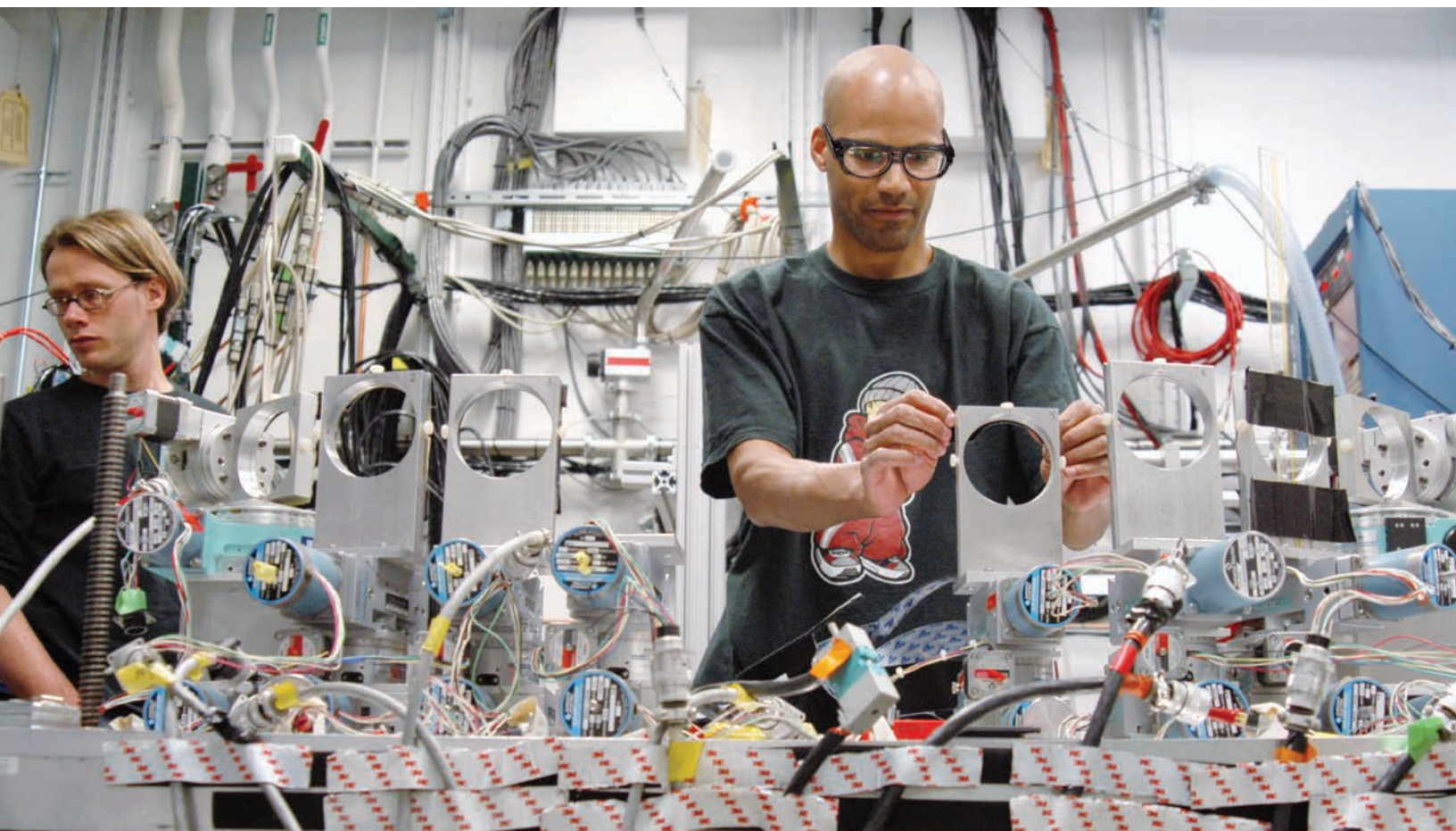
on a large scale, and of only limited effectiveness. An alternative solution lies in the search for new materials with the desired atomic structures and characteristics. This is the strategy of a group of researchers who, utilizing ultra-bright x-rays from the APS for *operando* powder x-ray diffraction studies at the XSD 9-BM beamline, and absorption spectroscopy measurements at the XSD 17-BM beamline, identified and characterized two niobium tungsten oxide materials that demonstrate much faster charging rates and power output than conventional lithium electrodes.

Metal Alloys Less Susceptible to Failure

Although crucial to modern life, metal alloys can fail, possibly leading to bridge collapse or other disasters. One cause of failure about which little has been understood is hydrogen embrittlement, which occurs when hydrogen from water propagates through a crack. Researchers used near-field, high-energy diffraction microscopy, and x-ray absorption tomography techniques at the XSD 1-ID beamline to analyze the microstructure of a preexisting crack in a nickel superalloy and determine the susceptibility of individual grain boundaries to hydrogen embrittlement. Understanding fracture behavior can better predict metal failure and how to design failure-resistant alloys.

Fine-Tuning Orbitals by Breaking Symmetry

Transition-metal oxides (TMOs) such as titanium oxide and nickel oxide are at the center of some of the most fascinating and important topics in solid-state physics, including superconductivity and the exploration of ferromagnetic and ferroelectric phenomena. By precisely “tuning” the structure and composition of TMOs, it is possible to achieve new insights into their behavior and potential for practical applications in electronics and materials science. Usually, such efforts rely on controlling mechanical properties of the crystalline lattice, such as by inducing epitaxial strain. Employing various x-ray absorption and diffraction studies at XSD beamline 33-ID at the APS and beamline U4B of the National Synchrotron Light Source at Brookhaven National Laboratory, experimenters have taken a more subtle and ambitious approach by actually controlling and manipulating the orbital properties of TMOs. Their work raises intriguing prospects for the development of useful techniques for the customization and fine-tuning of the electronic, magnetic, conductive, and even optical properties of various materials.



Readying a life-science experiment at BioCARS, Sector 14.

Solving Dilated Cardiomyopathy

Dilated cardiomyopathy (DCM) is a serious and progressive heart disease that affects over 1 million people in the United States annually. One hallmark of DCM is a reduced ability of the heart muscle to contract and adequately pump blood throughout the body. Research at a Biophysics (Bio) CAT x-ray beamline confirmed that a specific mutation in a particular gene likely has a direct role in the cause of DCM, paving the way for new DCM therapies.

Safer Opioids Taking Shape

Opioids play a crucial role in providing pain relief to millions of people through the activation of painkilling pathways found in cells throughout the brain and peripheral nervous system. But the primary opioid receptors — delta, kappa, and mu — are also tied to a variety of side effects, ranging from nausea and constipation to lethal respiratory problems. The kappa receptor also triggers non-lethal side effects. Knowing the exact structure of the kappa receptor can likely help researchers identify or

synthesize a safer pain-relief molecule. Researchers using a GM/CA-XSD x-ray beamline provided molecular insights with the potential to accelerate the structure-guided design of safer and more effective therapeutic compounds that target kappa opioid receptors.

Transforming Drug Development

Using x-ray diffraction data collected at a SBC-XSD x-ray beamline and at the Stanford Synchrotron Radiation Lightsource, researchers investigated the structure of the enzyme cysteine dioxygenase, which plays a critical role in regulating the level of thiols (compounds that contain a sulfur atom bonded to a hydrogen atom) in the body. They found (unexpectedly) that the enzyme could use oxygen to break down carbon-fluorine bonds, which are considered to be the strongest bonds in organic chemistry. This novel finding not only increases scientists' understanding about fluorine chemistry, but could also have important implications for drug development, given the important role that fluorine plays in pharmaceutical chemistry.

A New Treatment to Help Fight Parasites?

River blindness, a devastating, neglected disease that inflicts suffering on millions of people worldwide, especially in African countries, is caused by the parasitic roundworm *Onchocerca volvulus*, and is transmitted to humans by the bites of blackflies that carry the parasite. The results of a study carried out at the Industrial Macromolecular Crystallography Association (IMCA)-CAT x-ray beamline, which characterized the details of a molecular interaction that effectively blocks a critical drug-target enzyme from functioning, could eventually help scientists better treat diseases such as river blindness. They could also help guide discovery of new agents that can be used against targets for which conventional small-molecule drugs have failed.

Genome Mining in a Coal Mine

Biosynthetic engineering of novel molecules to create new medical treatments involves tinkering with existing natural molecules, such as antibiotics or peptides, to make them better. This can be done with standard synthetic chemical reactions, but sometimes it is easier to find an enzyme to do the work. The search for such an enzyme led a group of researchers to employ high-brightness x-rays at a Lilly Research Laboratories (LRL)-CAT x-ray beamline to study a strain of bacteria isolated from a coal mine fire in Appalachia. The team identified an enzyme that catalyzes a rare reaction and applied it to making a new, improved antibiotic.

A New Way to Kill Deadly Bacteria

According to the World Health Organization, antibiotic resistance is one of the biggest threats to health and a significant contributor to longer hospital stays, higher medical costs, and increased mortality. Scientists are continuously in search of new sources of antibiotics that disease-causing bacteria are unfamiliar with and so have not evolved defense mechanisms. A team of researchers identified a novel class of antibiotics produced by the nematode-symbiotic bacterium *Xenorhabdus nematophila*. Research with x-ray beams from a Northeastern (NE)-CAT beamline and other experiments led to clinical trials on one of these new antibiotics.

Pointing to a Human RSV Vaccine

Respiratory syncytial virus (RSV) is the dominant cause of severe respiratory infections in children and calves. However, no licensed human RSV vaccine is available, and bovine RSV vaccines suffer from issues

of effectiveness. Researchers set out to design, develop, and evaluate an investigational structure-based vaccine called “DS2.” They collected data at a Southeast Regional (SER)-CAT x-ray beamline to validate the structure-based design and showed that the novel vaccine generated high concentrations of neutralizing antibodies, protecting calves from bovine RSV infection. The results of this study could help to reduce the incidence of bovine RSV, and — by using a similar vaccine construct — could also help scientists develop an efficacious human RSV vaccine.

Solving the Riddle of Influenza Type A Virus

The flu virus comes in three types: A, B, and C. Type A accounts for all the virus pandemics in recorded history, resulting in many millions of deaths worldwide. Influenza pandemics occur when a new strain from other animal species overwhelms inter-species barriers and infects the human population by means of efficient airborne transmission of the virus, so a better understanding of the mutations that cause these viruses to become transmittable to humans through air is urgently needed. Work by researchers utilizing a Life Sciences (LS)-CAT x-ray beamline contributed to gaining a deeper understanding of the molecular mechanism behind the switch in receptor preference from avian to human, which could help in the timely detection of newly emerging influenza strains that pose a pandemic threat.

Beginning to See the Light

To make efficient use of sunlight as an energy source, photosynthetic organisms developed complex molecular machinery for detecting and orienting toward light. A key piece of this are phytochromes, proteins containing light-sensitive molecules, chromophores, which upon absorbing light deform in a certain way. These atomic-level structural changes are amplified to produce nanoscale changes to the structure of the surrounding phytochrome and lead to changes in gene expression that drive movement of the organism toward light. But the structure of photoactivated phytochrome and the nature of its conformational changes were unknown. Researchers using a BioCARS beamline to study the resting and active states of phytochrome found that light absorption induces a fluid corkscrew motion within the structure, thus filling critical knowledge gaps about the structural changes that phytochromes undergo during the detection of light that could lead to advances in medicine and agriculture.

Closer to an Effective HIV Vaccine

Macromolecular x-ray crystallography data collected at the SER-CAT beamlines were employed to investigate the development of VRC01, extraordinary antibodies that can neutralize about 90% of all strains of HIV. An understanding of how VRC01 antibodies are generated might ultimately lead to the ability to induce similar antibodies in HIV-infected people.

The researchers examined the crystal structures of several antibodies from the VRC01 lineage and identified significant differences in their amino acid sequences.

They also tracked the development of the VRC01 B cell lineage and found that it involved hundreds of generations of B cells that evolve faster than HIV-1 (the predominant strain of the virus), allowing the development of VRC01 antibodies that can neutralize the virus effectively. However, during natural HIV infection, VRC01 does not develop into its mature form quickly enough to protect people from the virus.

The results of this study have important implications in the development of a long-awaited HIV vaccine.

A Therapy for Improved Vision

A new therapeutic opportunity for modified retinals that helps improve vision, and offers a major improvement over current therapeutics designed to perturb cell signaling in the eye, was aided by research at a NE-CAT x-ray beamline.

A light-sensing pigment found in everything from bacteria to vertebrates can be biochemically manipulated to reset itself, an important therapeutic advantage.

Researchers used a modified form of vitamin A, called “locked retinal,” to induce the recycling mechanism and engage proteins central to human vision.

The targeted proteins include light-sensing rhodopsin, which belongs to the family of proteins — GPCRs — that sit in cell membranes and transmit external cellular cues into internal cell signaling pathways.

A Key Target for Cancer Drugs

Many approved cancer therapies target a protein called “epidermal growth factor receptor” (EGFR) that regulates many crucial cellular processes and — when mutated — can promote the proliferation of tumor cells. Scientists carrying out research at a GM/CA-XSD x-ray beamline made a fundamental discovery about EGFR signaling that may open the potential for new types of cancer drugs.

Scientists have long known that growth factors activate EGFR by “stitching” two receptor molecules together. This paradigm has suggested that the receptor must be either “off” or “on,” so all EGFR drugs have been designed to shut off the receptor and thus shut off proliferation. But a longstanding puzzle was that the EGFR is regulated by a total of seven growth factors, which can make the cell take different actions. How can those different actions be driven by a single binding (and activation) scenario?

The researchers found that EGFR signaling is not just an on/off process controlled by stitching two receptors together. Rather, the growth factors can turn on the receptor in a spectrum of different ways, depending on the strength of the stitch and the timing of this binding. Instead of therapeutics that just shut off EGFR, new ones might be designed that encourage it to give a beneficial signal.

The spectrum of effects from different EGFR binding mechanisms also might help shed light on other biological mysteries such as the causes of liver cancer, where pathways that work in ways similar to EGFR signaling play major roles that have not been well explained.

Building a Better Aspirin

Science has been working for decades to improve upon the first non-steroidal anti-inflammatory drug (NSAID), commonly known as aspirin. Newer versions of NSAIDs such as ibuprofen, naproxen, and celecoxib are the mainstays of treatment for pain, from minor bumps and bruises to more serious chronic pain and inflammatory conditions such as arthritis and cancer, but these drugs also have gastrointestinal side effects and are associated with rare fatal adverse reactions.

Researchers utilized the LRL-CAT x-ray beamline to study the structure of the microsomal prostaglandin synthase 1 (mPGES-1 enzyme) with the aim of providing a framework for the rational design of new molecules that can control inflammation and pain without troublesome side effects.

Their approach was to selectively inhibit an enzyme that generates a specific inflammatory lipid compound without blocking other lipids that are important in the regulation of blood clotting, blood pressure, and control of gastrointestinal integrity, thus eliminating the associated side effects that those lipids control.

These findings offer an opportunity for optimizing the interaction between the inhibitors and mPGES-1 to improve the action of potential drugs.

Life-Saving Research at the APS

A Drug that Fights Kidney Cancer

VOTRIENT®

The research that led to the development of VOTRIENT, a successful drug for fighting advanced kidney cancer, was carried out by scientists from GlaxoSmithKline (now GSK) using the IMCA-CAT x-ray beamline. Votrient is an angiogenesis inhibitor, which interferes with the growth of new blood vessels needed for solid cancer tumors to survive. It is used to treat advanced renal cell carcinoma (kidney cancer). Votrient is also used to treat soft tissue sarcoma, a tumor that can develop in or around muscles, tendons, joints, organs, or blood vessels.

A Drug that Fights AIDS

KALETRA®

KALETRA (lopinavir/ritonavir) is one of the most successful drugs used to stop the progression of the human immunodeficiency virus (HIV) into full-blown acquired immune deficiency syndrome (AIDS). KALETRA got its start at the APS when scientists from Abbott Laboratories used the IMCA-CAT x-ray beamline to discover a way to stop HIV from replicating in the body through the use of a protease inhibitor that blocks the breakdown of proteins. In 2002, KALETRA became the most prescribed drug in its class for AIDS therapy, and it remains widely used today, prolonging the lives of thousands of AIDS patients.

A Drug that Fights Leukemia

VENCLEXTA®

VENCLEXTA (venetoclax) is a small-molecule drug that treats chronic lymphocytic leukemia in those with a specific chromosomal abnormality. Chronic lymphocytic leukemia is one of the most common types of leukemia in adults; an estimated 20,940 cases are diagnosed in the U.S. each year. Researchers from AbbVie, Inc., developed the drug, in part, using data from the IMCA-CAT x-ray beamline. They studied the structure of a particular protein and how it interacted with potential inhibitors. In April 2016, VENCLEXTA was approved by the U.S. Food and Drug Administration (FDA).

A Drug that Fights Skin Cancer

ZELBORAF®

ZELBORAF (vemurafenib) halts the progression of malignant and inoperable skin cancer. Researchers from Plexxikon, Inc., and Genentech developed the drug using data obtained at three U.S. DOE-SC synchrotron x-ray research facilities including the SBC-XSD beamlines. ZELBORAF®, which was approved by the FDA in 2011, is the first drug to treat advanced melanoma by targeting a specific gene mutation.

Drugs that Fight Breast Cancer

KISQALI®

KISQALI (ribociclib) is an endocrine-based therapy for treating postmenopausal women who have hormone receptor-positive, human epidermal growth factor receptor-2 negative (HR+/HER2-) advanced or metastatic breast cancer in combination with any aromatase inhibitor. Novartis developed it with data from the IMCA-CAT x-ray beamline, and the FDA approved it for use in 2017.

Verzenio®

Verzenio (abemaciclib), a drug for the treatment of advanced or metastatic breast cancers, was developed by Eli Lilly using x-rays from the LRL-CAT beamline. It was designated as a breakthrough therapy for breast cancer by the FDA in October 2015 and approved for use in the U.S. in 2017 for the treatment of certain breast cancers.

A Drug that Fights Type 2 Diabetes

JANUVIA®

JANUVIA (sitagliptin) is a diabetes medication that researchers from Merck & Co. developed with data from the IMCA-CAT x-ray beamline. JANUVIA helps lower blood sugar levels in adults with type-2 diabetes. The FDA approved the drug in 2006, and it is now one of the most popular type 2 diabetes drugs on the market. In 2007, the FDA approved a variation of JANUVIA called JANUMET®, which is a combination of sitagliptin and metformin, and is also made by Merck & Co.

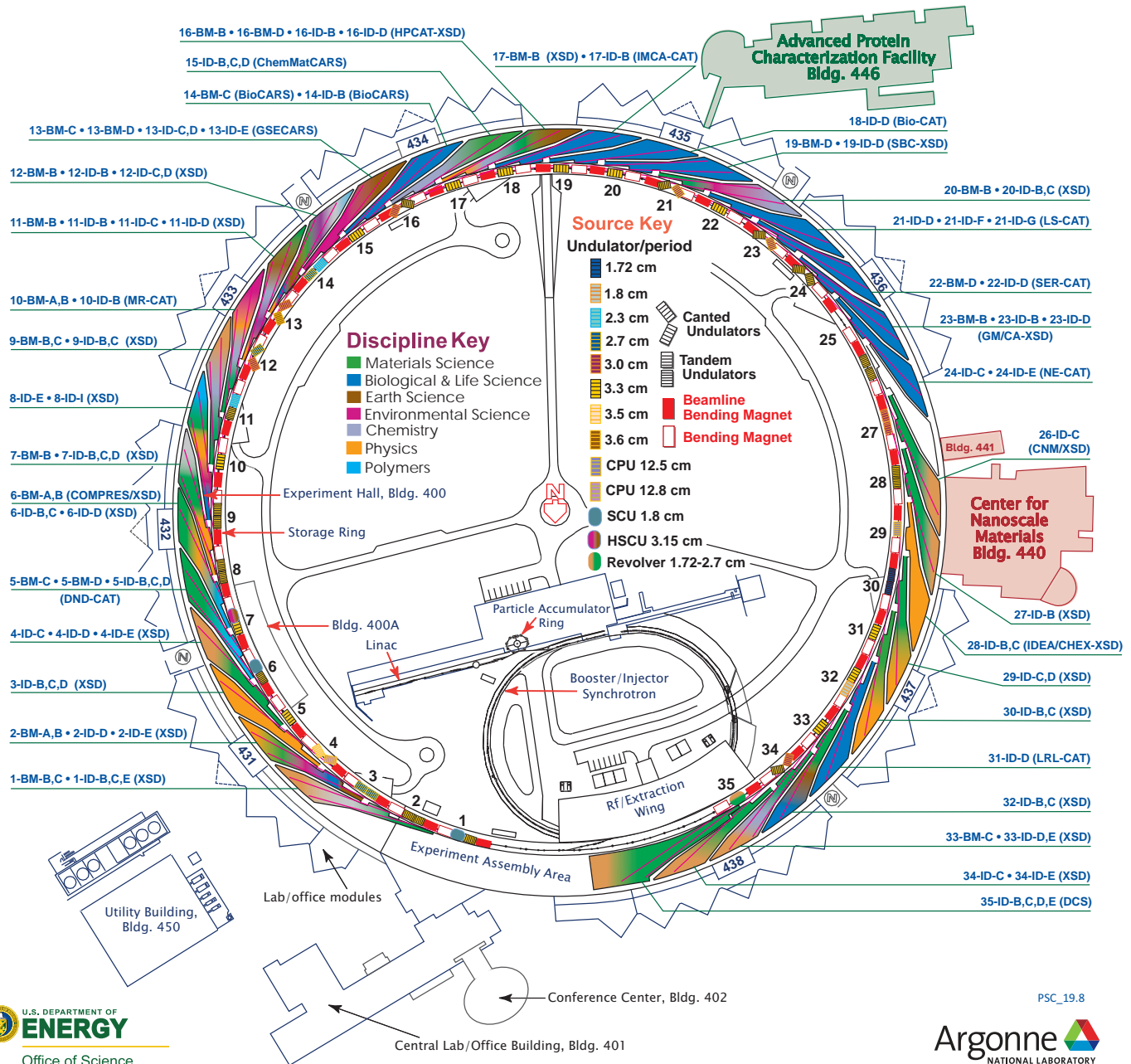
ARGONNE NATIONAL LABORATORY 400-AREA FACILITIES

ADVANCED PHOTON SOURCE

(Beamlines, Disciplines, and Source Configuration)

ADVANCED PROTEIN CHARACTERIZATION FACILITY

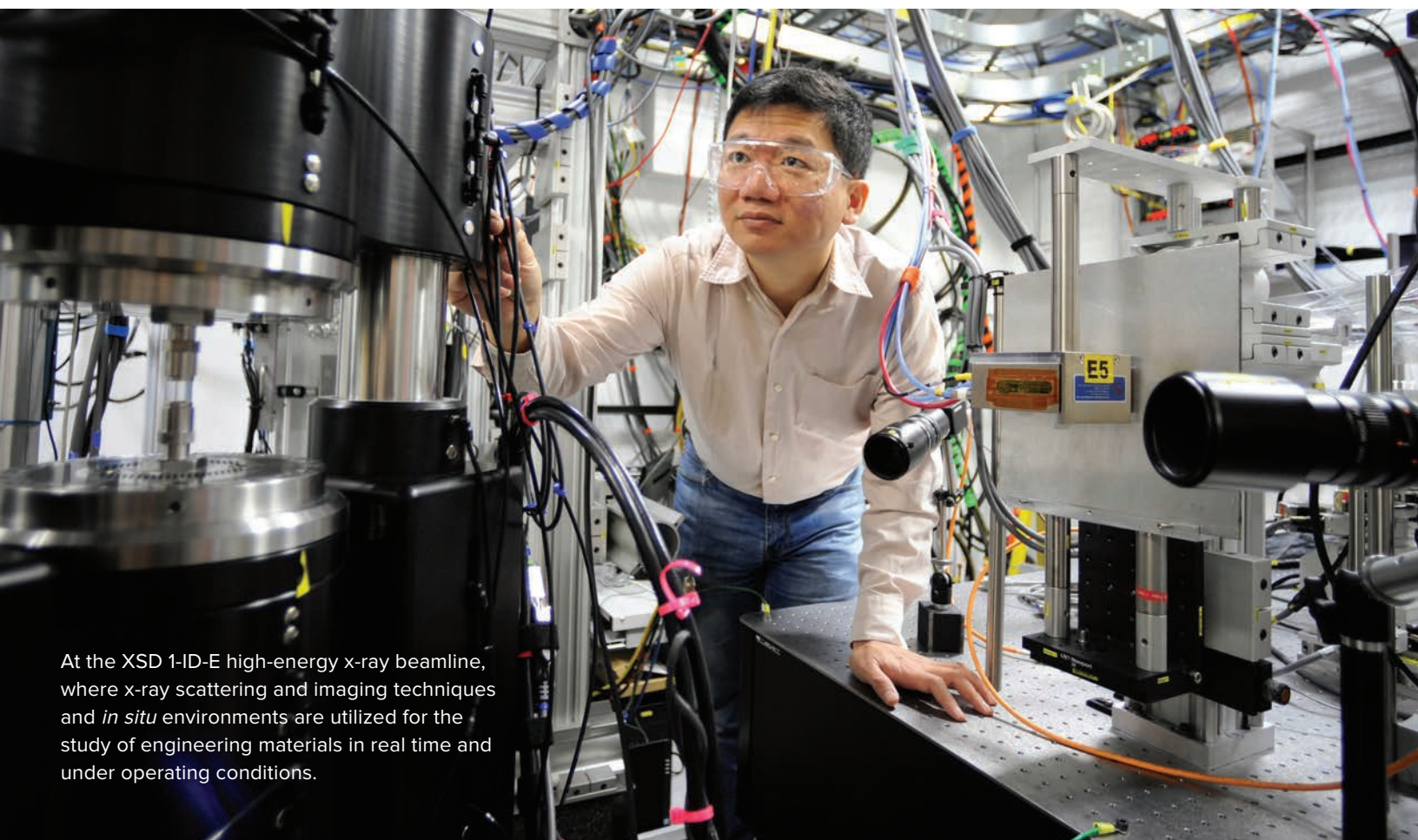
CENTER FOR NANOSCALE MATERIALS



PSC_19.8

Argonne
NATIONAL LABORATORY

This plan view of the APS facility shows locations of x-ray beamlines and beamline operator groups, scientific disciplines investigated at the APS, and locations and arrangements of x-ray producing electromagnets and insertion devices in the electron storage ring.



At the XSD 1-ID-E high-energy x-ray beamline, where x-ray scattering and imaging techniques and *in situ* environments are utilized for the study of engineering materials in real time and under operating conditions.

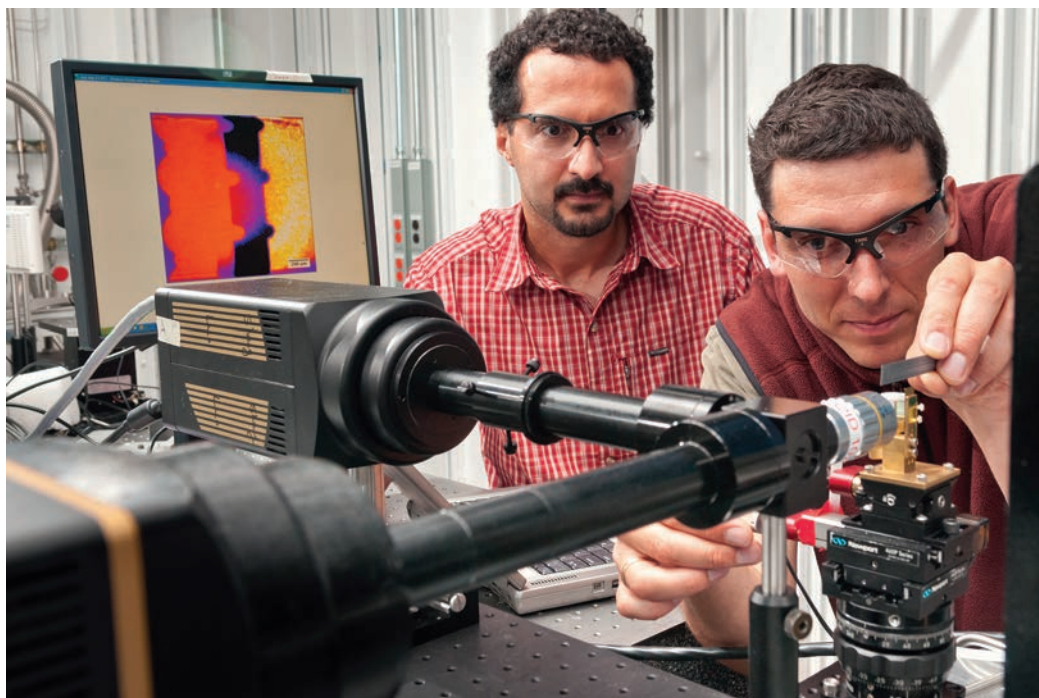
An APS sector comprises the radiation sources (insertion devices and bending magnets), x-ray beamlines, research stations, and instrumentation that are associated with a particular section of the electron storage ring and a particular research group. The APS has 40 sectors (diagram at left), 35 of which are dedicated to user science and experimental apparatus; the others are taken up with electron storage ring equipment. All of the user-science sectors can operate simultaneously and are available to the research community for experiments through an open, peer-reviewed proposal process.

X-ray Science Division sectors are operated by the APS and are primarily funded through U.S. DOE Basic Energy Sciences, while CAT sectors are built and operated by groups comprising scientists from universities, industries, and/or research laboratories. Some current XSD sectors have historic CAT origins.

Sectors 1-4, 6-9, 11, 12, 16, 19, 23, 29, 30, 32-34, and beamline 17-BM are operated by XSD. Beamline 6-ID is operated by XSD and COMPRES. Sector 20 is operated by XSD in partnership with the Canadian Light

Source. Sector 26 is operated by the CNM and XSD. Researchers using the beamlines in these sectors carry out experiments in materials and chemical science; environmental, geological, and planetary science; physics; polymer science; biological and life science; pharmaceutical research; atomic, molecular, and optical physics; and the properties of nanoscale materials, advancing our fundamental scientific understanding and the technologies that support a secure future for our nation.

Sector 5: DND-CAT is supported through Northwestern University, The Dow Chemical Co. and E. I. duPont de Nemours & Co. (the latter two are both part of DowDuPont). Additional support has been provided by the State of Illinois through the Department of Commerce and the Board of Education (HECA), the U.S. DOE Office of Energy Research, and the U.S. National Science Foundation (NSF) Division of Materials Research. It has as its scientific thrust the study of two-dimensional or quasi-two-dimensional atomic structures (surfaces, interfaces, and thin films) and polymer science and technology. All are of immense technological importance.



Studying crack formation in glass using a "shock gun" for high-pressure studies at the XSD 32-ID-B beamline. APS users carry out investigations that examine a wide range of critical materials subjected to extreme conditions. These experiments lead to stronger, safer, more durable materials.

Sector 10: MR-CAT, which is supported by the DOE and the MR-CAT member institutions, enables scientific work that is broadly materials oriented. The main emphasis is on *in situ* studies of materials by x-ray spectroscopy, scattering, and reflectivity. There are strong environmental science and catalysis components to this research, in addition to furthering materials-based technologies, such as photochemistry and x-ray lithography.

Sectors 13 through 15 are operated by the Center for Advanced Radiation Sources (CARS):

Sector 13: GSECARS is supported by the NSF-Earth Sciences and is dedicated to state-of-the-art research on Earth materials for a better understanding of our environment and planet.

Sector 14: BioCARS, supported by NIGMS of the National Institutes of Health (NIH), is dedicated to the development of resources and facilities to foster frontier research in time-resolved macromolecular x-ray (MX) crystallography and time-resolved biological small-angle and wide-angle scattering. Seeing macromolecules in action furthers our understanding of how they function and leads to advances in the cause, prevention, and treatment of diseases. Experiments in the physical sciences are conducted under the auspices of CARS, with additional support from the APS.

Sector 15: ChemMat-CARS is principally supported by the Divisions of Chemistry and Materials Research of the NSF and focuses on the study of surface and interfacial properties of liquids and solids as well as their bulk structure at atomic, molecular, and mesoscopic length scales with high-spatial and high-energy resolution to advance materials and chemical science. The APS ultra-small-angle x-ray scattering program has moved to Sector 15 under a collaboration between CARS and the APS.

Sector 16: HPCAT-XSD, whose operations are supported by the DOE-National Nuclear Security Administration (NNSA) Office of Experimental Science and is situated organizationally within XSD, advances cutting-edge, multi-disciplinary, high-pressure science and technology enabling myriad scientific breakthroughs in high-pressure physics, chemistry, materials, and the Earth and planetary sciences.

Sector 17: IMCA-CAT, dedicated to accelerating drug discovery research, operates a state-of-the-art structural biology synchrotron beamline for the pharmaceutical industry. The facility is optimized for high-quality, high-throughput macromolecular crystallography experiments, producing essential data for determining the structures of key components in structure-based drug design. The facility is funded by the pharmaceutical members of IMCA (Industrial Macromolecular Crystallography Association): AbbVie, Bristol-Myers Squibb, Merck, Novartis, and Pfizer, and operated through a contract with the Hauptman-Woodward Medical Research Institute. Data from IMCA-CAT have been crucial to the development of a significant number of therapeutics for the prevention and treatment of disease.

Sector 18: Bio-CAT, funded by the National Institute of General Medical Sciences of the NIH, develops and operates state-of-the-art facilities for studies of the structure and dynamics of biological systems under non-crystalline conditions that are similar to their functional states in living tissues to better understand human physiology.

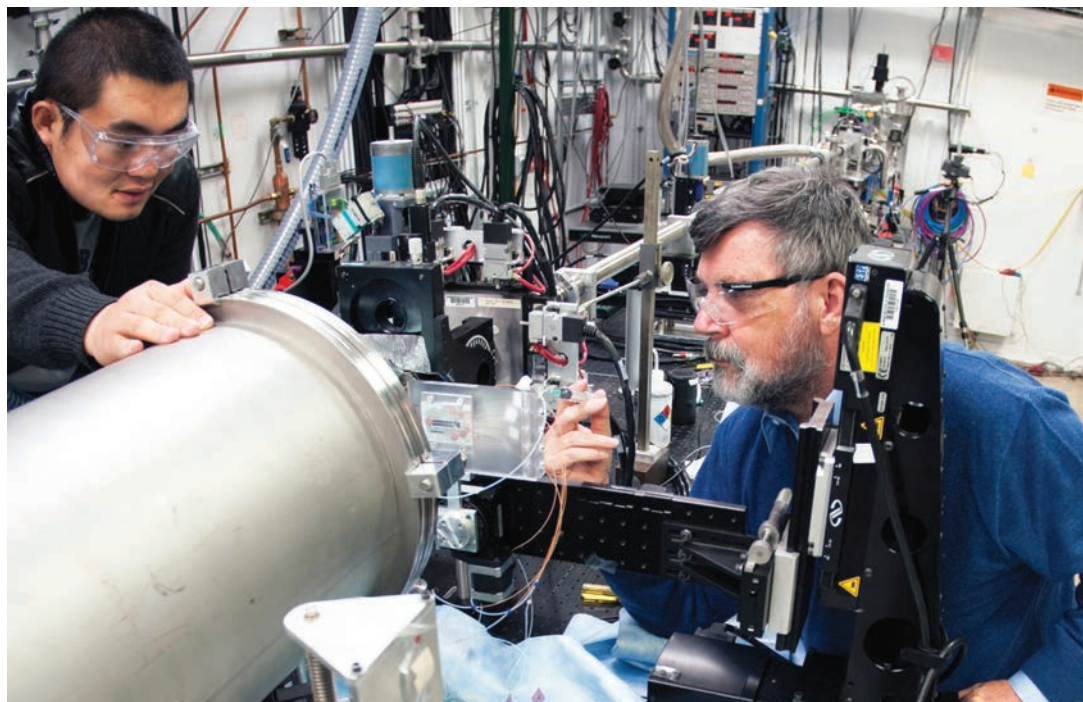
Sector 19: SBC-XSD, funded by the DOE Office of Biological and Environmental Research, operates a national user facility for macromolecular crystallography at the APS

in order to advance and promote scientific and technological innovation in support of the DOE mission by providing world-class scientific research and advancing scientific knowledge. SBC-XSD is an important component of integrated biosciences and contributes to the expansion of existing programs and exploration of new opportunities in structural biology, proteomics, and genomics with a major focus on medicine, bio-nanomachines, and biocatalysis that are highly relevant to energy resources, health, a clean environment, and national security.

Sector 21: LS-CAT is supported by the Michigan Economic Development Corporation and the Michigan Technology Tri-Corridor and provides macromolecular crystallography resources for those with a need to determine the structure of proteins. LS-CAT affords users access to state-of-the-art x-ray diffraction facilities at the APS.

Sector 22: SER-CAT, which is supported by its member institutions (see www.ser-cat.org/members.html) and equipment grants from the NIH, provides third-generation x-ray capabilities to macromolecular crystallographers and structural biologists in the southeastern region of the U.S. Emphasis is placed on structure determination, high-resolution structural analyses, large unit cells, drug design, structural genomics, soft x-ray data collection, and next-generation beamline automation.

Sector 23: GM/CA-XSD, organizationally within XSD, operates a national user facility for structural biology specializing in intense, tunable micro-beams for determining the structure of proteins and other macromolecules at the forefront of biological research, emphasizing problems in structural genomics and structure-based drug design. The scientific and technical goals of GM/CA-XSD are streamlined, efficient throughput for a variety of sample types, sizes, and qualities representing the cutting edge of research.



Adjusting a microfluidic mixer mounted in the small-angle scattering instrument on the Bio-CAT 18-ID x-ray beamline. This instrument is utilized for macromolecular small-angle x-ray scattering and fiber diffraction to probe the structure and dynamics of non-crystalline biological systems such as muscle, connective tissue, macromolecules, and macromolecular complexes in solution.

GMCA-XSD is funded in whole or in part with Federal funds from the National Cancer Institute and the NIGMS of the NIH.

Sector 24: NE-CAT, which is funded by the NIGMS of the NIH, operates an x-ray crystallographic research facility designed to address the most demanding and complex diffraction problems in structural biology and is organized to allow its operation to be driven by the requirements of its users.

Sector 31: LRL-CAT, operated by Eli Lilly and Company, is dedicated to the determination of protein structures and the analysis of the interactions between potential pharmaceutical compounds and a protein of interest to further research into the causes, prevention, and treatment of disease.

Sector 35: DCS is operated by Washington State University under the U.S. DOE-NNSA. The DCS integrates state-of-the-art dynamic compression platforms and drivers with an APS x-ray beamline to produce a first-of-a-kind experimental capability (worldwide) focusing on time-resolved x-ray diffraction and imaging measurements in dynamically compressed condensed matter.

The APS is Organized for Success

Any discussion of the APS is really a discussion about people: the researchers who use the facility and the scientists, engineers, technicians, and support staff who keep the facility at the forefront of synchrotron x-ray science.

As noted on page 19, XSD operates a number of APS beamlines and is partners in the operation of several others. Personnel in XSD support users of XSD beamlines while pursuing their own research interests and developing cutting edge x-ray beamline instrumentation and techniques that are utilized by scientists coming to the APS and at x-ray light sources around the world.

While XSD looks after the experimental side of the APS, the APS Engineering Support (AES) Division and the APS Accelerator Systems Division (ASD) see to the technical aspects of one of the world's most technologically complex machines. Their combined efforts result in an accelerator facility that has routinely delivered x-ray beams to users at a level of availability that typically exceeds 97% of scheduled user-beam hours.

The people in the AES Division provide reliable operations and technical support to the APS user community. They design and maintain most of the sophisticated mechanical and digital systems that comprise the APS electron accelerator and x-ray beamlines, including the

facility's accelerator complex, computing infrastructure, accelerator controls systems, and various personnel protection systems. The Design and Drafting Group in AES provides state-of-the-art renderings of future accelerator and beamline instrumentation, while the survey and alignment team makes sure that those components are installed to critical tolerances that can be equivalent to about 1/3 the thickness of the average human hair or sheet of paper.

The staff of the ASD operate the APS accelerator and are responsible for the maintenance and design of some critical accelerator systems. The accelerator complex consists of an S-band radio-frequency thermionic electron gun; a 400-megaelectronvolt pulsed linac; a 400-megaelectronvolt particle accumulator ring; a full-energy booster synchrotron; and a 7-gigaelectronvolt, 1.1-kilometer-circumference electron storage ring operating with a 100-milliamp beam. Groups within the ASD are dedicated to accelerator physics and operation; beam diagnostics; radio-frequency systems; magnet power systems; and the huge magnetic devices, both bending magnets and insertion devices (IDs), that produce x-rays for research.

A variety of innovative IDs designed by APS physicists and engineers are operating in the APS electron storage ring. Other ID technology, often designed in col-

The APS booster/injector synchrotron, where electrons are accelerated to nearly the speed of light before they are injected into the electron storage ring.



laboration with colleagues from other labs, can be found in storage rings and free-electron lasers at other x-ray light sources in our nation and around the world.

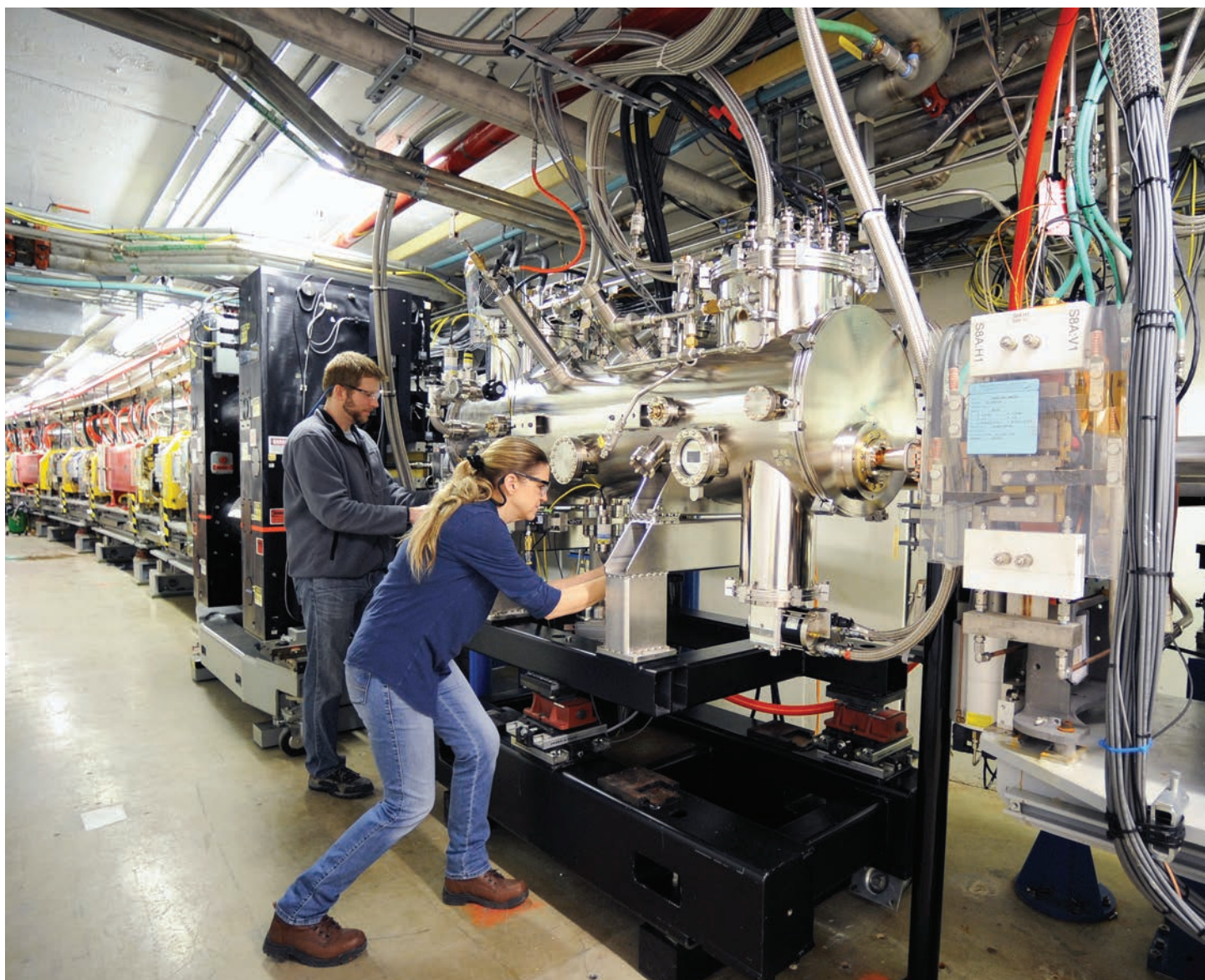
The newest ID technology at the APS is the superconducting undulator (SCU). These IDs will be the primary source of high-brightness, high-energy x-rays from the upgraded APS. The SCUs employ superconducting technology to deliver more photons at a wider photon energy range as compared to the permanent magnet undulators currently installed in the APS storage ring.

The first prototype APS superconducting undulator, SCU0, which was installed in Sector 6 of the APS storage ring in late December 2012-early January 2013, was the culmination of an 11-year effort, from R&D to design, fabrication, magnetic measurement, and pre-installation and testing. This was followed by installation of a second SCU in Sector 1, a replacement of the pro-

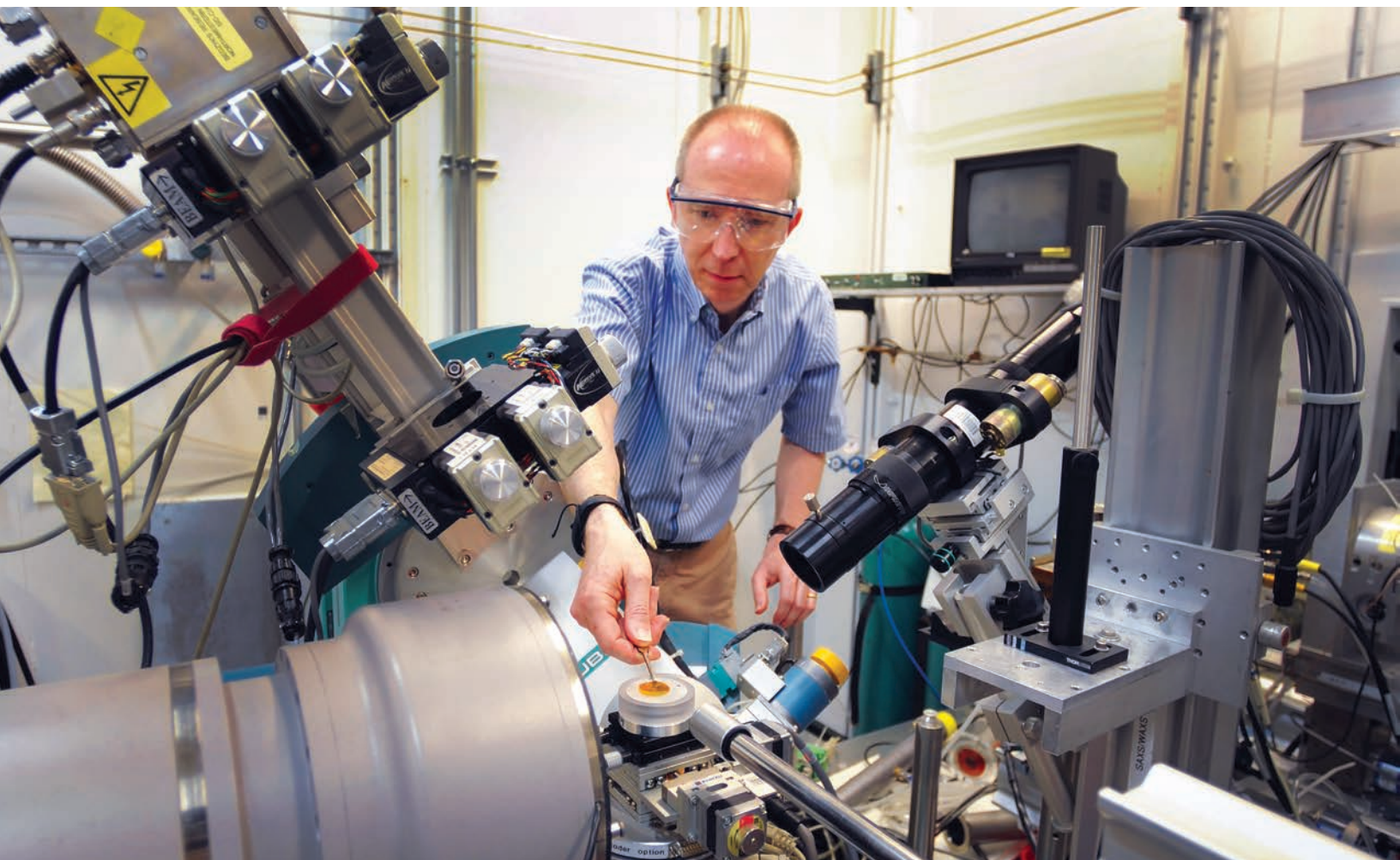
totype at Sector 6 with a full-length SCU, and a helical superconducting undulator (HSCU) installed in January of 2018 at Sector 7.

The HSCU is another quantum leap forward in ID design. The device has two primary advantages over other types of IDs for producing high-brightness x-rays: it allows researchers to select a single energy from the x-ray beam without using any x-ray optics, and it produces an x-ray beam with circular polarization. The HSCU provides researchers with a more intense x-ray beam that allows for faster data acquisition than conventional undulators, at time scales of a billionth of a second. The ability to produce circularly polarized radiation overcomes a limitation of linear, unpolarized light: circular light is sensitive to properties of a material such as magnetism and molecular chirality — or handedness — that linear or unpolarized light cannot see.

The helical superconducting undulator installed on Sector 7 of the APS storage ring.



Industrial Research at the APS



At DND-CAT, Sector 5: Placing a sample in the kappa geometry diffractometer used for measuring x-ray fluorescence, x-ray standing waves, and wide-angle x-ray scattering to determine the surface and bulk structure of thin films and other samples.

Scientists come to the APS to carry out investigations that increase our fundamental knowledge of processes and materials, which allows us to move beyond observation to control for a nearly endless array of technologically and economically important applications including advances in energy storage, manufacturing, information technology, nanotechnology, pharmaceuticals, biomedicine, oil and gas, transportation, agriculture, environment, and many other areas that are critical to our technologies, economy, and physical well-being.

More than 230 companies have research agreements with the APS, including (but not limited to) Chevron, 3M, Amoco, Bayer, Caterpillar, E.I. DuPont de Nemours & Co., Exxon, Ford Motor Co., GE Global Research Center, General Motors, Intel, Kraft Foods

Technology Center, Monsanto, Packer Engineering, Texas Instruments, Westinghouse Electric, and a comprehensive roster of leading pharmaceutical firms.

The APS welcomes industrial users doing both proprietary and non-proprietary research and considers requests for work ranging from short-term feasibility studies to long-term research projects, either on a stand-alone basis or in collaboration with facility or academic colleagues.

The DOE has some prerequisites for user research. The industry partner and Argonne must sign a legal agreement before experimental work at the APS can begin; the type of work being done will determine the appropriate agreement. For non-proprietary work published in the open literature, beam time is free; for proprietary work, users must pay the APS an hourly fee for operational costs.

For more information see <https://www.aps.anl.gov/Industry> or contact: apsuser@aps.anl.gov.

Scientific Disciplines Studied at the APS

- Materials and chemical science; environmental, geological, and planetary science; physics; polymer science; biological and life sciences; pharmaceutical research; atomic, molecular, and optical physics; properties of nanoscale materials.

APS users investigate:

- Better materials for batteries and other energy-related technologies
- More efficient designs for fuel-injection systems
- Clues to the causes of and treatments for a multitude of diseases including AIDS and toxic threats such as anthrax
- A greater understanding of human physiology
- Ways of eliminating and remediating environmental depredations
- Insights about conditions at the center of the Earth, the causes of earthquakes and volcanoes, and the composition of cosmic dust
- An endless array of new information about materials that support practical applications like advanced digital storage media, more efficient lighting, environmentally friendly refrigerants, methods for increasing the durability of man-made structures, and the characterization of nano-structures whose sizes are measured in atoms

The APS Facility

- Electrons cannot go faster than the speed of light, but the electrons in the APS storage ring have an energy of 7 gigaelectronvolts (7 billion electron volts), so the electrons are traveling at over 99.99999999% the speed of light
- There are more than 2000 conventional electromagnets and 16 pulsed electromagnets in the APS electron accelerators
- Over 400 beam-position monitors, 600 corrector electromagnets, and 80 computer systems monitor and correct

the electron orbit, steering x-ray beams to within a fraction of the width of a human hair

- More than 120 programmable logic controllers monitoring over 25,000 signals comprise radiation interlock systems



Part of the APS computer room, where servers for the electron storage ring and x-ray beamlines are located.

protecting APS personnel and equipment

- The APS has five 1-megawatt radio frequency (rf) power systems (the equivalent of 5000 microwave ovens) that accelerate and maintain the high-energy electron beams in the storage ring
- The storage ring rf systems contribute to a combined accelerating voltage equal to a 16-million-volt power supply
- APS rf systems produce more rf power than the combined output of every radio and television station in the city of Chicago
- The superconducting detectors that collect data from the interaction of high-brightness APS x-rays and the sample being studied operate at temperatures colder than outer space
- The outer diameter of the APS experiment hall is 1225 feet, slightly less than the height of the Willis Tower in Chicago (1454 feet)
- Experiment hall construction required 56,000 cubic yards of concrete (equal to a football-field-sized block 30 feet high); 5000 tons of structural steel (enough for 3500 mid-size cars);

2,000,000 linear feet (380 miles) of electrical wire; and 190,000 feet of pipe for water, steam, drainage, and HVAC

- Total floor space of all APS buildings is 1,042,811 feet²
- APS facility construction started in spring 1990; research started in the fall of 1996
- Total APS construction and project cost at completion in 1995: \$812 million USD (1995 dollars)
- APS employees at any one time: approximately 450

Other Argonne User Facilities

The Advanced Protein Characterization Facility (APCF) houses state-of-the-art labs for the production and characterization of proteins, protein complexes, and crystals, making it a unique resource for the MX user community. The facility provides know-how for high-throughput production, purification, characterization, and crystallization of proteins using highly parallel automated methods, efficiently integrating the production of recombinant proteins and their complexes from the genome sequence with accelerated determination of their three-dimensional biomolecule structures at the APS that can be further characterized using biophysical techniques available at the APS. The APCF is linked to the modern computing needed to support Argonne's emerging activities in systems and synthetic biology making the APCF a focal point for intellectual interactions, scientific exchange, and technological advances.

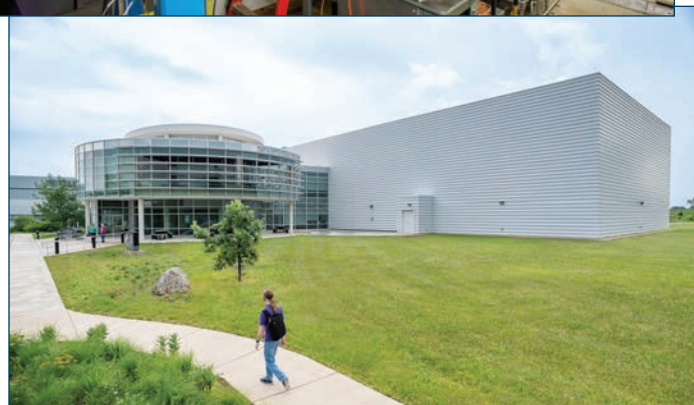
The prime national facility for nuclear structure research, the Argonne Tandem Linear Accelerator System (ATLAS) is the world's first superconducting linear accelerator for heavy ions at energies in the vicinity of the Coulomb barrier. This is the energy domain best suited to study the properties of the nucleus, the core of matter, and the fuel of stars. ATLAS can provide beams of essentially all stable isotopes from protons to uranium, and a variety of light radioactive beams through an in-flight production program and heavier neutron-rich

isotopes from CARIBU. ATLAS is a U.S. DOE user facility that hosts roughly 200 to 300 users each year from U.S. universities and national laboratories as well as from foreign institutions. The facility is also accessible to industrial users.

The Center for Nanoscale Materials is a premier user facility providing expertise, instruments, and infrastructure for interdisciplinary nanoscience and nanotechnology research to academic, industrial, and international scientists. The CNM is at the forefront of discovery science that addresses national grand challenges encompassing the topics of energy, information, materials and the environment. The scientific strategy of the CNM unites three crosscutting and interdependent scientific themes that collectively aim at the discovery and integration of materials across different length scales studied at the extremes of temporal, spatial and energy resolutions.

The Center for Transportation Research brings together scientists and engineers from many disciplines across Argonne to work with the U.S. DOE, automakers, and other industrial partners. The goal is to put new transportation technologies on the road that improve the way we live and contribute to a better, cleaner future for all.

Clockwise from top right: The ATLAS booster linac; the Center for Nanoscale Materials; and the Advanced Protein Characterization Facility.



The Office of Science X-ray Light Sources

The Scientific User Facilities Division of Basic Energy Sciences in the U.S. DOE Office of Science supports the operation of many major research facilities across our nation that provide open, peer-reviewed access to sophisticated research tools for scientists from academia, national laboratories, and industry carrying out research in all major scientific disciplines.

These facilities consist of a complementary set of intense x-ray sources, neutron scattering centers, electron beam characterization capabilities, and centers for nanoscale science. They allow scientists to probe materials in space, time, and energy with the appropriate resolutions that can interrogate the inner workings of matter — transport, reactivity, fields, excitations, and motion — to answer some of the most challenging science questions.

More than 10,000 scientists conduct experiments at these facilities each year. Thousands of other researchers collaborate with those users, analyze data from the experiments, and publish scientific findings in peer-reviewed journals.

The synchrotron x-ray light sources, including the APS, produce radiation over a wide range of photon energies from the infrared to hard x-rays. The Linac Coherent Light Source is a free-electron laser light source that fires pulses of light to illuminate fundamental aspects of the physical world for investigations of energy, momentum, and position using techniques including spectroscopy, scattering, and imaging applied over various time scales.

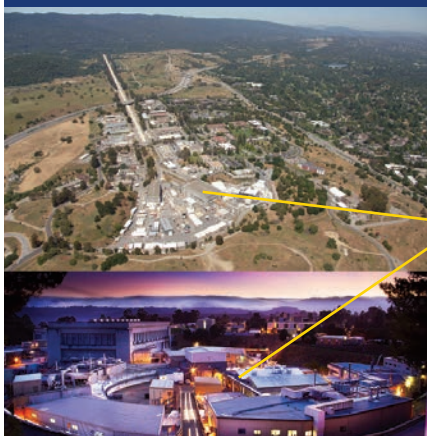
The U.S. DOE Office of Science x-ray light source facilities are:

- Advanced Light Source, Lawrence Berkeley National Laboratory
- Advanced Photon Source, Argonne National Laboratory
- Linac Coherent Light Source, SLAC National Accelerator Laboratory
- National Synchrotron Light Source II, Brookhaven National Laboratory
- Stanford Synchrotron Radiation Lightsources, SLAC National Accelerator Laboratory

See science.energy.gov/bes/suf/user-facilities/

Locations of the five DOE-BES x-ray light sources.

Top: Linac Coherent Light Source
Bottom: Stanford Synchrotron Radiation Lightsources
SLAC National Accelerator Laboratory



National Synchrotron Light Source II
Brookhaven National Laboratory



Advanced Light Source
Lawrence Berkeley National Laboratory



Argonne National Laboratory

The nation's first national laboratory, Argonne National Laboratory (photo below) is managed by UChicago Argonne, LLC, for the U.S. Department of Energy's Office of Science. The Office of Science is the single largest supporter of basic research in the physical sciences in the United States, and is working to address some of the most pressing challenges of our time. For more information, please visit: www.science.energy.gov.

Argonne is a multidisciplinary science and engineering research center where "dream teams" of world-class researchers work alongside experts from industry, academia, and other government laboratories to address vital national challenges in sustainable clean energy, the environment, technology, and national security. Researchers at Argonne and their collaborators around the world strive to discover new ways to develop energy innovations through science; create novel materials molecule-by-molecule; and gain a deeper understanding of our planet, our climate, and the cosmos. More than 6000 scientists conduct experiments at Argonne user facilities each year.

Surrounded by the highest concentration of top-tier research organizations in the world, Argonne leverages its Chicago-area location to lead discovery and to power innovation in a wide range of core scientific capabilities, from high-energy physics and materials science to structural biology and advanced computer science.

Argonne is the Midwest's largest federally-funded R&D center, employing some 3350 employees and more than 1250 scientists and engineers in dozens of fields. It is located in DuPage County, IL, about 25 miles southwest of Chicago, just south of Interstate 55.

Argonne's annual operating budget of around \$650 million supports more than 200 research projects.

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About Argonne National Laboratory

Argonne is a U.S. Department of Energy laboratory managed by UChicago Argonne, LLC under contract DE-AC02-06CH11357. The Laboratory's main facility is outside Chicago, at 9700 South Cass Avenue, Lemont, Illinois 60439. For information about Argonne and its pioneering science and technology programs, see www.anl.gov.

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To apply for Advanced Photon Source beam time: apsuser@aps.anl.gov | (630) 252-9090

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