

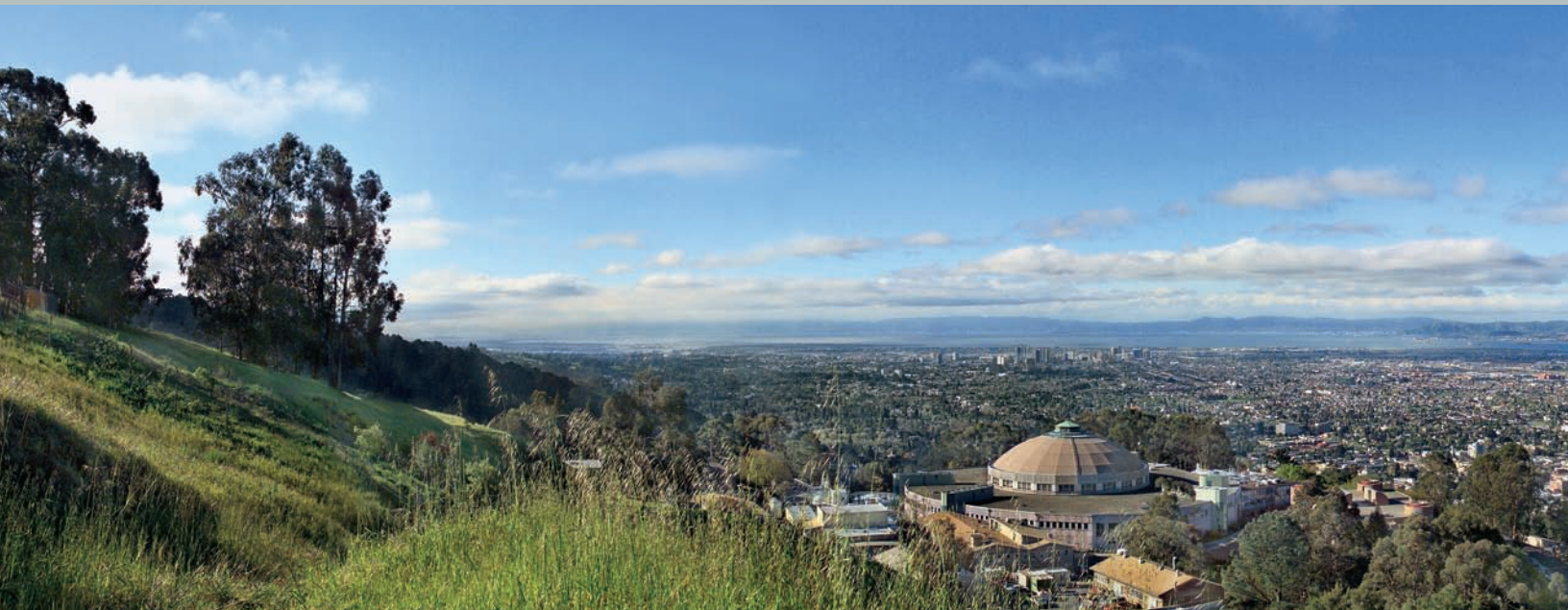
# PHOTON SCIENCE FOR RENEWABLE ENERGY

at Light-Source Facilities of Today and Tomorrow



Lawrence Berkeley National Laboratory

APRIL 2010



## TOOLS FOR JOULES

Our current fossil-fuel-based system is causing potentially catastrophic changes to our planet. The quest for renewable, nonpolluting sources of energy requires us to understand, predict, and ultimately control matter and energy at the electronic, atomic, and molecular levels.

Light-source facilities—the synchrotrons of today and the next-generation light sources of tomorrow—are the scientific tools of choice for exploring the electronic and atomic structure of matter. As such, these photon-science facilities are uniquely positioned to jump-start a global revolution in renewable and carbon-neutral energy technologies. In these pages, we outline and illustrate through examples from our nation's light sources possible scientific directions for addressing these profound yet urgent challenges.





# FUNDAMENTAL QUESTIONS

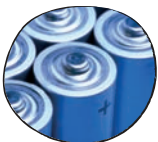
To establish the scientific foundations for the kind of transformative breakthroughs needed to build a 21st-century energy economy, we must address fundamental questions involving matter and energy. At right is a sampling of such questions that can be addressed by light-source facilities:



How can we modify the behavior of electrons and holes in semiconductors to more efficiently convert **solar energy** into electricity using earth-abundant materials?



How can we harness photosynthesis and efficiently transform abundant plant material into **biofuel**? Can we produce fuel directly from sunlight by developing **artificial photosynthesis**?



How can we increase the energy capacity and durability of lithium-ion **batteries** to maintain performance over hundreds to thousands of charge-discharge cycles?



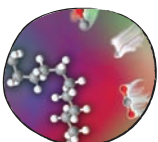
How can we understand the electrical and chemical properties of a working **electrochemical fuel cell** to tailor its properties for the production of fuel or electricity?



How does **carbon dioxide** interact with naturally occurring materials under ambient conditions during storage, and what can we learn about these materials to improve their **capture** capacity?



How can we identify **combustion** products at the parts-per-million level to improve efficiency and control pollution that results from the burning of fuel?



How do we tailor the electronic properties of nanostructured **catalysts** to achieve higher activity or selectivity with inexpensive materials?

# ABOUT LIGHT SOURCES: SYNCHROTRON RADIATION AND FREE-ELECTRON LASERS

Synchrotron radiation is emitted by electrons that are accelerated, generally in bunches through bending magnets around a storage ring with a circumference ranging from few hundred meters to a kilometer, to near the speed of light. This synchrotron “light,” which may be a million times more intense than that produced by x-ray tubes, covers the spectrum from infrared to ultraviolet and from soft (long-wavelength) x rays to hard (short-wavelength) x rays. With the use of special multipole magnetic devices known as wigglers and undulators, the brightness of the emitted radiation can be increased to achieve high resolving power for **spectroscopy/spectrometry**, high spatial resolution for **microscopy/spectromicroscopy/microtomography**, and high coherence for **diffraction/scattering**.

The radiation emitted by a synchrotron storage ring scales with the number of electrons ( $n$ ) per bunch for a bending magnet,  $nN$  for a wiggler, and  $nN^2$  for an undulator ( $N$  is the number of magnetic poles in the wiggler or undulator). Recent developments in linear accelerator technology using free-electron lasers have decreased the size of the electron bunches enough to emit coherent radiation in the soft to hard x-ray range with intensities that scale as  $n^2N^2$ , increasing brightness by ten orders of magnitude and timing resolution by five orders of magnitude.

## KEY



Sunlight to electricity



Sunlight to fuel



Batteries



Fuel cells



CO<sub>2</sub> capture & sequestration



Combustion



Catalysis

A survey of beamlines that have performed energy-related research at the Advanced Light Source (ALS) as of February 2010.

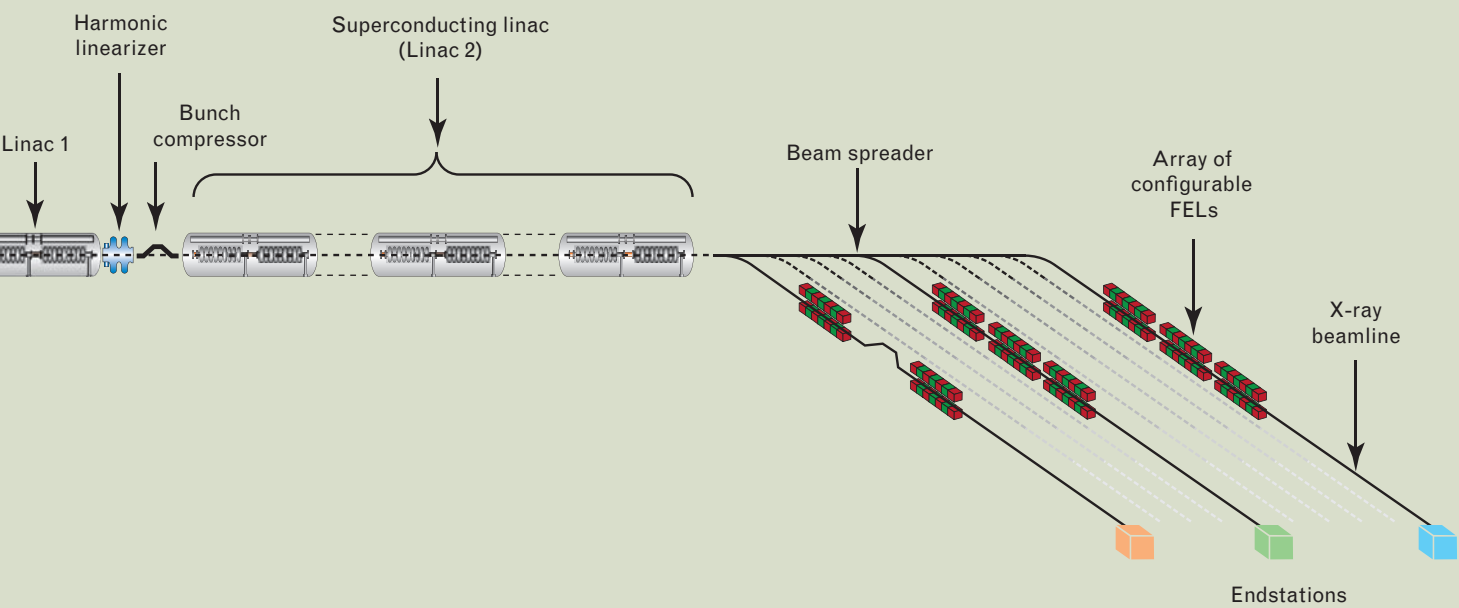
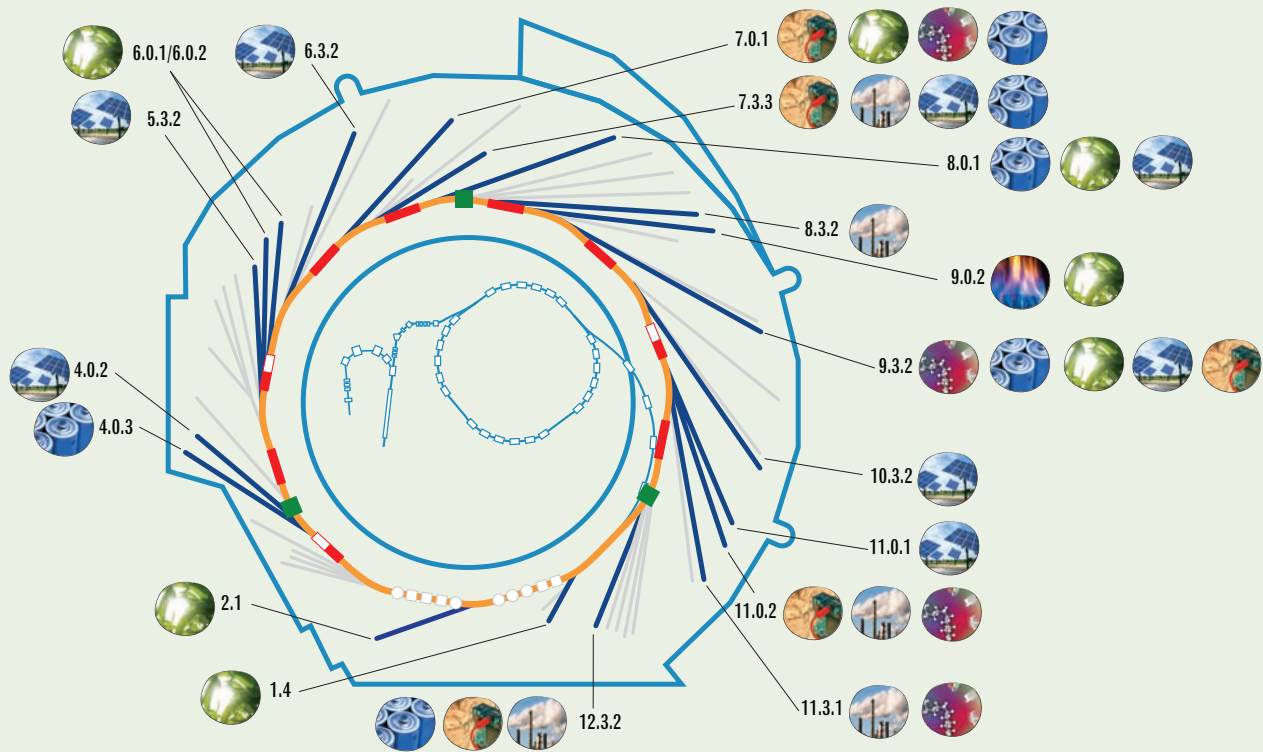
High-brightness,  
high-repetition-  
rate electron gun

Injector

Laser  
heater



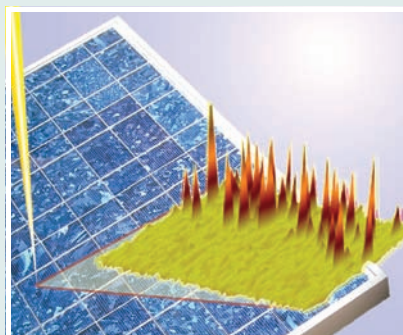
A proposed next-generation light source (NGLS) would consist of an array of soft x-ray free-electron lasers (FELs), each optimized for specific scientific needs. It would revolutionize our understanding of atoms, electrons, and the flow of energy on fundamental space and time scales. Courtesy of J. Corlett and P. Denes (Berkeley Lab).



# SUNLIGHT TO ELECTRICITY

The Sun delivers to Earth in one hour the amount of energy consumed globally in one year. However, solar power supplies just 1.5% of the world's demand, due to its higher cost relative to fossil fuels. To become competitive, rapid advances in converting solar energy into electricity are needed in order to produce low-cost, high-yield, and high-efficiency solar cells.

Metal impurities are among the main culprits in degrading the efficiency of multicrystalline solar cells. Scientists have combined x-ray emission (fluorescence) and absorption **spectroscopy** to study the distribution, chemical state, and electron-hole recombination activity of metal clusters in a variety of multicrystalline solar cells before and after processing. They discovered that the size, distribution, and chemical binding of metals within clusters matter as much as the total metal concentration in limiting performance. This suggested that, instead of trying to remove the impurities, we can manipulate them in a way that reduces their impact on efficiency ("defect engineering"). Further developments, such as high-temperature in situ measurements to probe defect states during processing, will provide deeper insight for additional optimization.



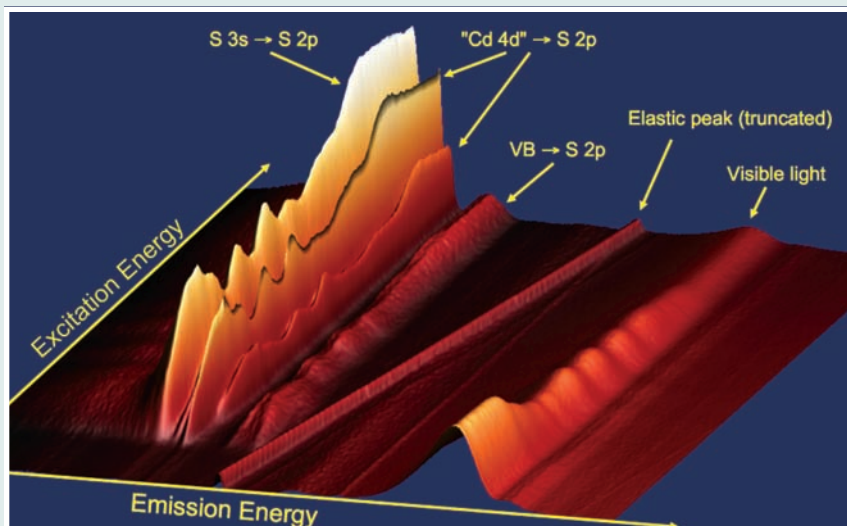
Artist's depiction of an intense beam of synchrotron light striking a solar cell and the resulting fluorescence image of the distribution of iron impurities. Courtesy of T. Buonassisi (MIT). [Buonassisi et al., *Nat. Mater.* **4**, 676 (2005); Buonassisi et al., *Appl. Phys. Lett.* **89**, 042102 (2006); Buonassisi et al., *Acta Mater.* **55**, 6119 (2007).]

Thin-film solar cells, widely considered the next generation of photovoltaic devices, have reached efficiencies of up to 20%. With high-brightness

undulator radiation and a high-transmission x-ray spectrograph, it is now possible to collect resonant inelastic soft x-ray **scattering** (RIXS) data in a record time (less than an hour). Together with sophisticated theoretical calculations, the observations allow a deeper understanding of the energies associated with occupied and unoccupied electronic states, giving insight into the band gap, an understanding of the excitation behavior, and element- and state-specific information about electronic wavefunctions, the most fundamental description of electrons in matter.

Small-angle x-ray **scattering** (SAXS) has been used to demonstrate that one can manipulate the spatial distribution of nanoparticles into specific regions of block copolymer structures using light-responsive and heat-responsive small molecules.





Resonant inelastic soft x-ray scattering map of CdS, a central material in a variety of thin-film solar-cell devices. The map, combining information from traditional x-ray absorption and emission spectra, gives complete information about the electronic states of a material. Courtesy of C. Heske (UNLV). [Weinhardt et al., *Phys. Rev. B* **79**, 165305 (2009).]

Precise ordering and manipulation of the positions of the conducting nanoparticles in the polymer matrix may afford solar energy and storage devices with high power-conversion efficiency. Both hard x-ray SAXS and grazing-incidence SAXS techniques have been used to probe the underlying structural properties of nanomaterials composed of block copolymers with or without inorganic nanoparticles dispersed in the matrix. In these cases, the candidate materials contain one or two conducting components—copolymer chains and nanoparticles—that are engineered with the goal of optimal and efficient charge separation for fabricating efficient solar energy devices.

# SUNLIGHT TO FUEL

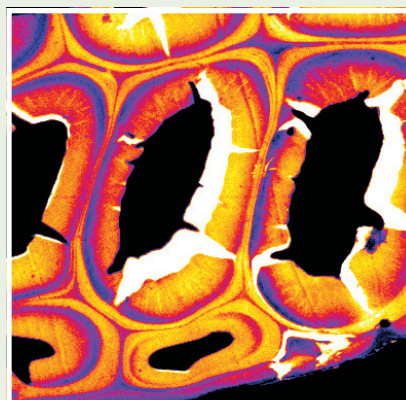
Nature has devised a remarkably diverse set of pathways to convert solar photons into chemical fuels. Learning from Nature, we hope to use plant matter as the raw material for transportation biofuels. Another avenue of research is to create an artificial photosynthetic device, made from earth-abundant materials, capable of efficiently converting sunlight and carbon dioxide into alcohol-based fuels.

Biofuels can be produced efficiently from crops with high sugar content (the sugar is converted into ethanol by yeast). Cellulose, a key component of plant cell walls, is far more abundant in plants than sugars, and plants that produce large volumes of cellulose, but no sugar (e.g., grasses) can be grown on poorer quality land, with much lower use of water and fertilizers. Cellulose is, in fact, a sugar polymer and can be broken down into sugar components, potentially providing a new, highly abundant biofuel source. The use of cellulose is hindered by its presence in cell walls as a complex mixture with hemicellulose and lignin, all of which are naturally resistant to breakdown.

Ionic liquids have emerged as promising new solvents for pretreating plant material, a process that disrupts the crystalline structure of

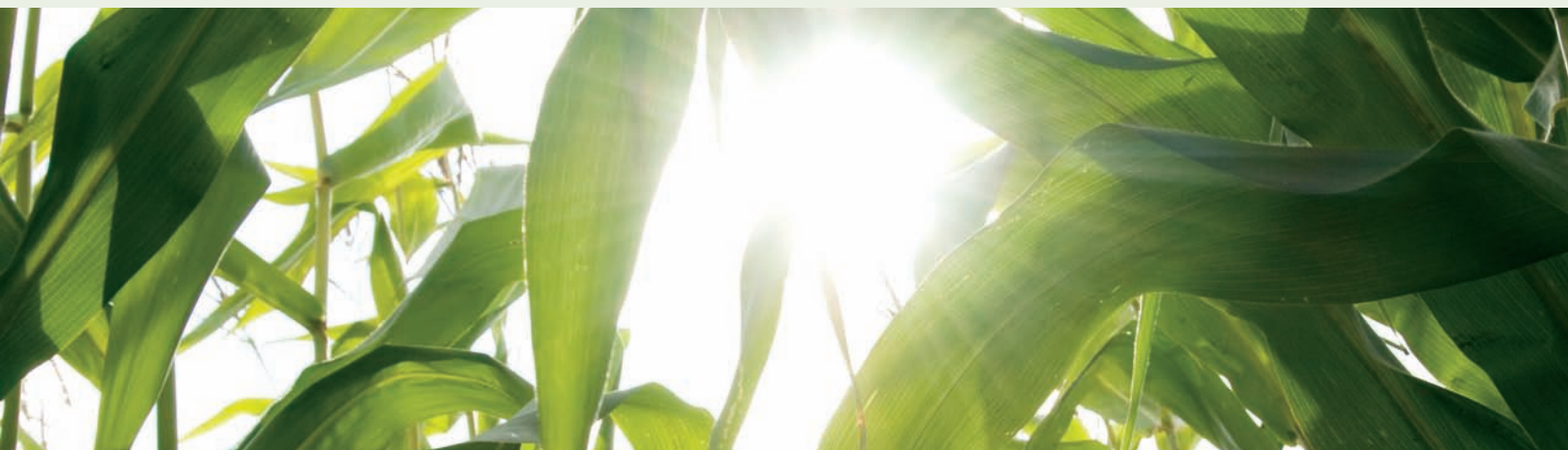
the cellulose. To study these processes, switchgrass and corn stalks and leaves are placed into capillaries containing an ionic liquid. X-ray **diffraction** is then used to monitor the real-time changes of the cellulose crystalline structure, and small-angle x-ray **scattering** measures changes in the cellulose microfiber size. These studies can be combined with infrared and Raman **spectroscopy** studies of changes in intermolecular hydrogen bonding. Together, these studies are designed to provide useful information on how to improve pretreatment of lignocellulosic biomass and thereby deliver cheaper biofuel.

Scientists are also exploring ways to use sunlight to convert carbon dioxide and water into fuel molecules (hydrogen, methane gas, or simple alcohols) in a single, integrated



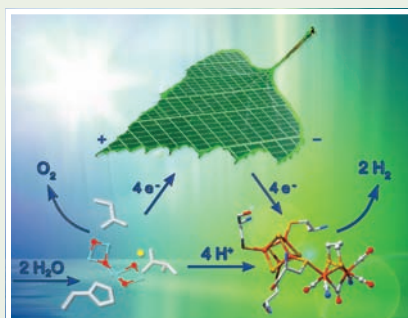
Soft x-ray microscopy image of cellular composition of compression wood from red spruce. The image is 40 x 40 microns with a spatial resolution of 100 nm; the acquisition energy of 285 eV highlights the spatial distribution of lignin (purple regions). Courtesy of G. Cody (Carnegie Inst. of Washington), recorded at the National Synchrotron Light Source, 1998.





device made from earth-abundant inorganic materials (artificial photosynthesis). The electronic structures of these materials are determined in part by using synchrotron-based near-edge and extended x-ray absorption **spectroscopy**. Recent soft x-ray developments involving high-efficiency emission spectrographs, ambient-pressure photoemission spectroscopy, and in situ measurement techniques provide a better indication of the oxidation state, symmetry, electronic structure, and charge-transfer processes in the complexes involved.

The development of light sources based on free-electron lasers is also providing new opportunities for time-resolved x-ray experiments that would make it possible to create movies of the breaking and formation of bonds as well as of charge-transfer



Schematic representation of solar splitting of water. Nanotubes embedded within a membrane could act like green leaves, using incident solar radiation to split water molecules ( $\text{H}_2\text{O}$ ), freeing up electrons, hydrogen ( $\text{H}_2$ ), and oxygen ( $\text{O}_2$ ) that then react with carbon dioxide ( $\text{CO}_2$ ) to produce a liquid fuel that can be used for vehicles. The result is a renewable green energy source that also helps scrub the atmosphere of excessive carbon dioxide from the burning of fossil fuels. Courtesy of W. Lubitz (Max Planck Institute). [Lubitz et al., *Energy Environ. Sci.* **1**, 15 (2008).]

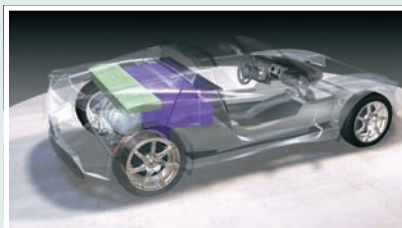
processes as they happen in time frames that range from picoseconds to femtoseconds to perhaps attoseconds. Such experiments would isolate the processes involved and reveal their evolution over time by combining the chemical and atomic specificity of x rays with the use of stroboscopic pump–probe techniques.

# ENERGY STORAGE: BATTERIES

Batteries give us the ability to store energy, which is important for portability (e.g., electric cars) as well as for the steady delivery of electricity using a smart grid from green but intermittent energy sources (e.g., sun and wind farms) to consumers when and where it is needed. These goals require a new generation of batteries that store more energy, recharge faster, and survive more duty cycles without degradation.

To develop better batteries, researchers must assess what is happening inside the battery materials as the batteries charge and discharge. For example, in lithium-ion batteries (used in everything from cell phones to state-of-the-art electric vehicles), researchers would like to observe how the lithium ions (which replace electrons as the charge carriers) interact with the batteries' cathodes and anodes. In the case of polymer-based batteries, researchers want to watch the block copolymers of the electrolyte change pattern or even heal themselves through annealing during heating and cooling. Problem areas also need to be addressed, such as the swelling of materials and growth of lithium dendrites into the electrolyte.

Fast small- and wide-angle x-ray **scattering** provides the metric needed to make nanoscale measurements

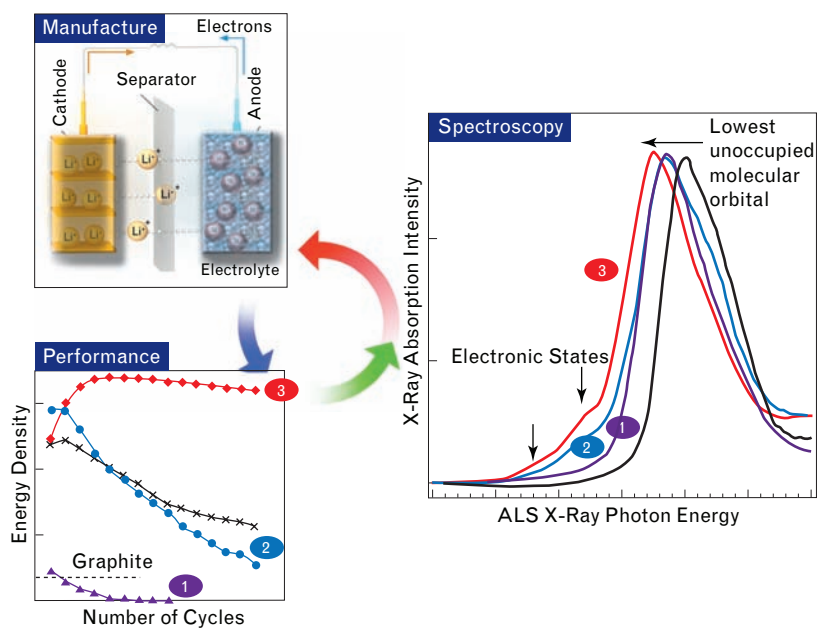


X-ray beamlines provide several tools and techniques necessary to keep up with the growth of the world's energy demands through rational design of new materials with real-world payoffs, such as car batteries with higher capacities and more duty cycles. Rendering courtesy of Tesla Motors, Inc.

of the structure of a rapidly changing battery system. Researchers have recently conducted an almost ideal experiment: entire lithium-ion batteries have been placed into the x-ray beam and charged and discharged in situ while SAXS measurements

were made. This allowed them to quantify the nanoscale changes that occur in electrode morphology during charging and discharging. This kind of information will be invaluable in developing the next generation of battery technology.

New conductive polymers have been designed and synthesized to greatly improve the energy density of lithium-ion batteries. The polymers, when made into negative electrodes with commercial silicon nanoparticles, provide both intimate contact with the nanoparticle surfaces for electrical conductivity and structural flexibility to accommodate large-volume changes during lithium-ion insertion and removal. Soft x-ray absorption **spectroscopy** provides direct information for a rational optimization of these materials. Synthesis of the polymers is fully compatible



X-ray absorption spectroscopy reveals the evolution of the electronic states around the band gap in polymers with different functional groups, providing a rational basis for the design of batteries that perform up to ten times better (red curve) than what's currently available (purple curve). [G. Liu and W.L. Yang et al., unpublished (2010).]

with current large-scale lithium-ion manufacturing processes, and x-ray spectroscopy was performed directly on real-world samples without any surface treatment. Silicon nanoparticle electrodes of such polymers have so far shown the best cycling behavior, with an energy capacity almost an order of magnitude higher than that of the widely used graphite-based electrodes on today's market (patent filed).

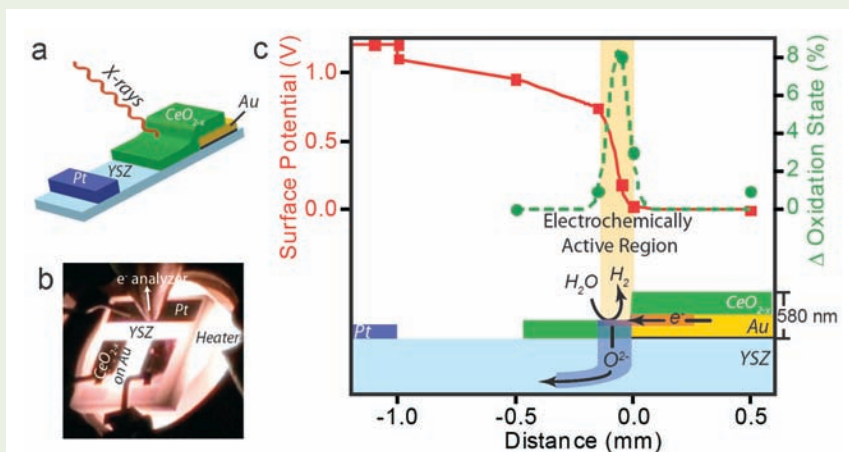


# ELECTROCHEMICAL FUEL CELLS

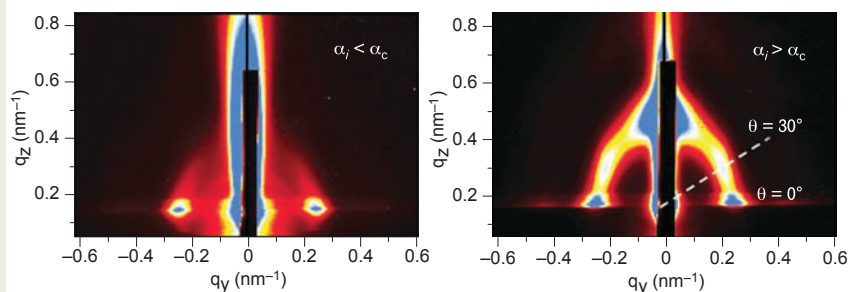
When hydrogen and air are pumped into a fuel cell, electricity is generated and air and water vapor leaves. A hydrogen fuel cell can thus be classed as a zero-emission energy source provided that the hydrogen is not generated via carbon-based fuels. In situ synchrotron-based techniques and nanofabrication are providing new insight into the operation of these electrochemical devices.

Whereas batteries store energy, fuel cells require a constant fuel supply—hydrogen, for example. The hydrogen's electrons and protons are catalytically separated, and an electrolytic membrane allows the protons to travel from the cell's anode to its cathode, while forcing the electrons through an external circuit, creating an electric current.

Fuel cells that employ solid oxide membranes typically require high operating temperatures (above 650 °C), limiting material choices. Yttria-stabilized zirconia (YSZ) membranes and cerium oxide (ceria) electrodes have received significant attention due to properties that can, in theory, expand the electrochemically active region. However, there is little direct knowledge of local overpotential and length scales of the electrochemically active regions of such materials in opera-



Electrochemical activity over 100  $\mu\text{m}$  of a ceria anode. (a) Schematic of solid oxide cell showing Pt counter electrode, Au current collector on top of a thin alumina film (black), and ceria working electrode patterned onto a YSZ single-crystal substrate. This geometry exposes the entire cell to the x-ray beam. (b) The view through the sample chamber window during cell operation at about 700 °C. (c) A ceria anode converts  $\text{H}_2\text{O}$  into  $\text{H}_2$  and  $\text{O}^{2-}$  in a narrow region at 1.2-V cell potential. In situ APXPS reveals gradients in surface potential (red) and the shift of Ce oxidation state out of equilibrium (green) in this region. [C. Zhang et al., submitted (2010).]



GISAXS patterns for PEMs composed of block copolymers supported by a silicon substrate. Data were obtained at two incident angles: below the critical angle (left) and above the critical angle (right). Courtesy of N.P. Balsara (UC Berkeley). [Park et al., *Adv. Mater.* **21**, 203 (2009).]

tional solid-state fuel cells. Measuring the overpotentials is quite challenging due to the high temperatures and the need to connect the components in series. The use of an ambient-pressure x-ray photoelectron **spectroscopy** (APXPS) instrument allowed, for the first time, direct, in situ study of an

operating solid-state fuel cell to determine the overpotential across each component interface as well as its chemical composition. Such measurements will allow further optimizing and tailoring of the performance of solid-state fuel cells.

Another type of fuel cell employs polymer electrolyte membranes (PEMs), with hydrophilic, proton-conducting channels embedded in a hydrophobic matrix. Grazing-incidence small-angle x-ray **scattering** (GISAXS) data provided information about the orientations of the channels near the air interface and through the interior of 180-nm-thick PEMs before and after exposure to humid air. Scattering at incident angles below the critical angle (and thus dominated by contributions from the PEM/air surface) contained well-defined spots, indicating the presence of hydrophilic channels oriented perpendicular to the surface. Scattering at incident angles above the critical angle (and thus containing contributions from the entire film), exhibited a scattering ring, indicating the presence of hydrophilic channels parallel to the plane of the film.

# CO<sub>2</sub> CAPTURE AND SEQUESTRATION

Capturing carbon dioxide (CO<sub>2</sub>) gas from stationary sources such as power stations, and then pumping it underground for storage in permeable rock is a potential way to avoid or mitigate “greenhouse gas” effects on climate. What materials are best at efficiently capturing the CO<sub>2</sub>? What happens to the CO<sub>2</sub> when underground?

The separation of CO<sub>2</sub> from other emitted gases requires materials exhibiting exactly the right adsorption or diffusion properties. Metal–organic frameworks (MOFs) are molecular cages with channels or pores whose sizes can be tailored to selectively trap CO<sub>2</sub> gas. Perhaps the most powerful tool available for analyzing the structure of MOFs is single-crystal x-ray **diffraction**. Combining this technique with an in situ gas environmental cell can reveal how porous MOFs store gas molecules such as CO<sub>2</sub>. Various soft x-ray **spectroscopies** can also reveal the electronic structures of MOFs and possibly of the gas species as well, with the aim of better understanding the interactions between these materials. This would complement studies of crystal structure and might ideally even look at the same materials. The combination of electronic and crystal

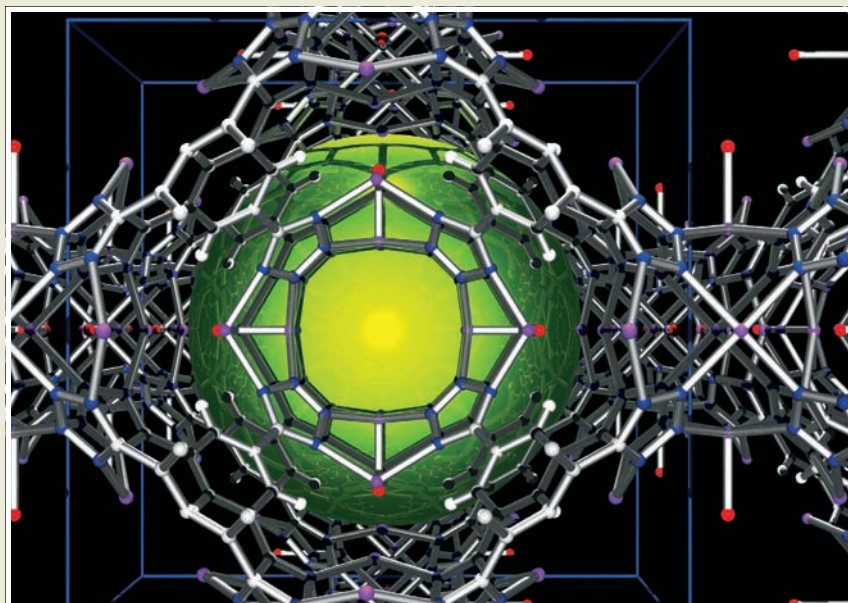
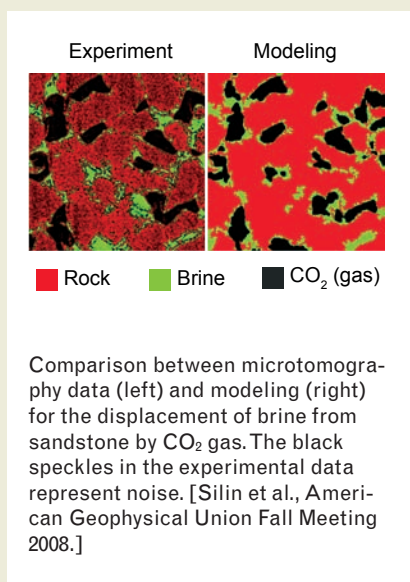


Illustration of a metal–organic framework (MOF) structure. Metal compounds are connected via organic-molecule bridges into a lightweight scaffolding (gray sticks) with large voids (yellow sphere) that can accommodate “guest” gas molecules such as CO<sub>2</sub>. Illustration by Christine Beavers (ALS). [Dinca et al., *J. Am. Chem. Soc.* **128**, 16876 (2006).]





structure information would provide more data to compare with theoretical models of gas interactions with porous host materials.

When injected into underground rock structures, the flow properties of CO<sub>2</sub> are dominated by the innumerable fluid/fluid exchange interactions within the micron-sized pores of the rock. Understanding these interactions is the key to predicting the behavior of underground CO<sub>2</sub> reservoirs. Using synchrotron light, we are able to look inside rocks with three-dimensional x-ray **microtomography** to see how fluids flow through permeable rock, displacing existing liquids. These experiments yield the data that will confirm the theoretical models being developed that will in turn be used to predict the properties of large CO<sub>2</sub> underground reservoirs.

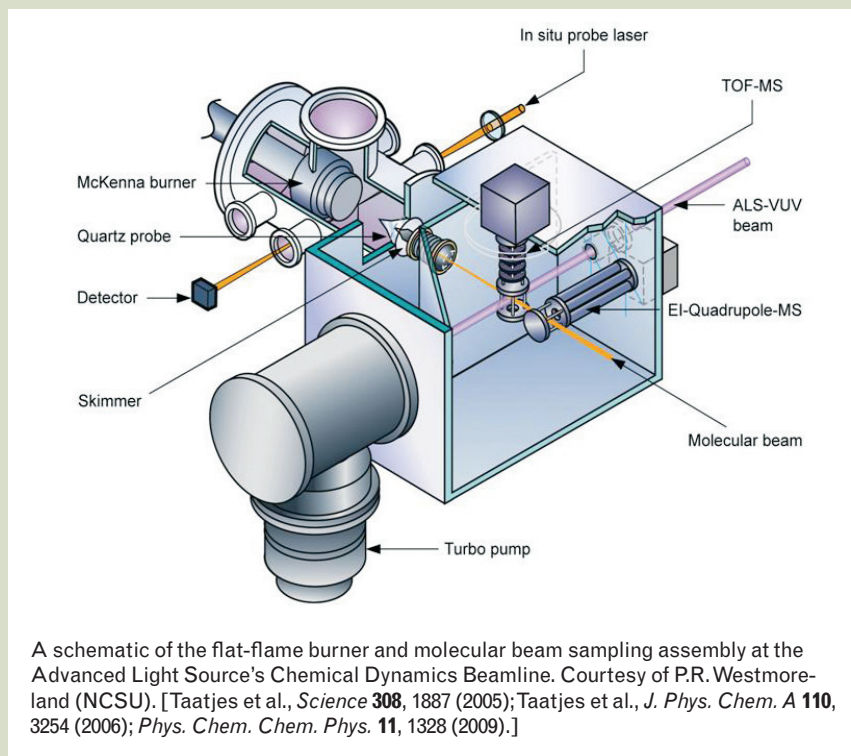
The interaction of CO<sub>2</sub> with rocks, soil, ice, sea water, and aerosols plays an important part in the CO<sub>2</sub> chemistry of Earth's atmosphere. In

particular, chemical processes at the interfaces between solid, liquid, and vapor determine the uptake and release of CO<sub>2</sub>. Ambient-pressure x-ray photoelectron **spectroscopy** (APXPS) allows the investigation of these interfaces under realistic conditions of relative humidity and CO<sub>2</sub> partial pressure. Current projects include the investigation of the uptake of CO<sub>2</sub> by mineral surfaces (e.g. MgO) as a function of relative humidity, where in particular the formation and possible removal of self-passivating carbonate layers (which limit CO<sub>2</sub> uptake by mineral surfaces) is of interest [Hendrik Blum (Berkeley Lab) et al.].

# COMBUSTION

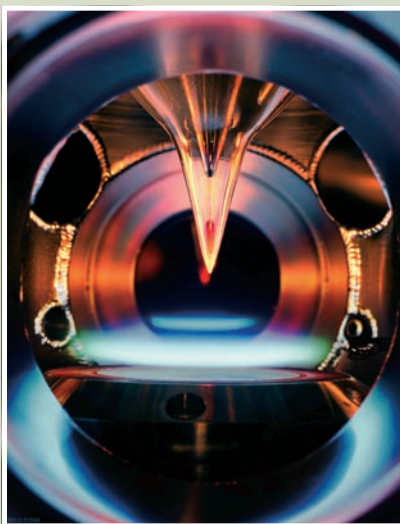
Fossil fuels, biofuels, and synthetic alcohol-based fuels all have different combustion processes. After more than 150 years of study, combustion is well understood in terms of average energy output, high-concentration intermediates, and major products. However, it is necessary to understand flame chemistry at the parts-per-million level if we are to further improve efficiency and control pollution.

As fuel burns and transforms into its final products, it goes through an intricate network of thousands of reaction pathways with hundreds of short-lived transient molecules (reaction intermediates). These often include lung-damaging particulates like soot, smog-causing nitrogen oxides, and other pollutants. Detailed mathematical models have been developed to help improve combustion efficiency and reduce pollution. However, the accuracy of these models depends on knowing all the chemical species involved and having quantitative values for the reaction constants that describe how the reactions proceed. As a result, many important rate constants have never been measured directly, nor have all the species included in mathematical flame models been directly observed.





For those studying flame chemistry and the properties of combustion intermediates by means of molecular beam mass **spectrometry**, the use of tunable vacuum ultraviolet light from a synchrotron to photoionize the beam for mass spectrometry makes for a powerful technique capable of differentiating between isomers with the same molecular weight and composition. Using such a technique, researchers discovered that a class of compounds known as enols (ethenol or vinyl alcohol is the simplest of these), only recently observed as a combustion intermediate, is in fact widely present in flames burning a variety of fuels, including commercial blends, such as gasoline. Combustion modelers will have to take this new finding to heart, as will those studying other forms of hydrocarbon oxidation, including those involving synthetic and biofuels.



The luminous zone of the flat flame has the typical blue-violet or blue-green color associated with chemiluminescence from electronically excited CH and C<sub>2</sub>. The gases produced by the flame are sampled through a small aperture in a quartz cone. These gases can then be analyzed by synchrotron-photoionization mass spectrometry, which has revealed previously undetected combustion intermediates. Photo by Linda Hadley (Sandia National Laboratories).

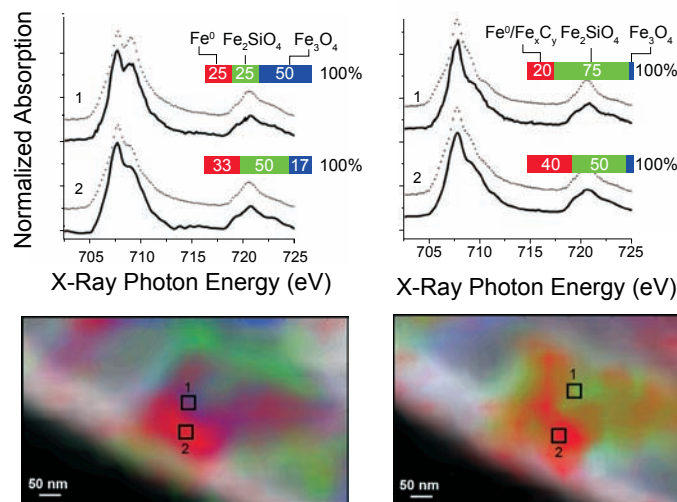


# CATALYSIS

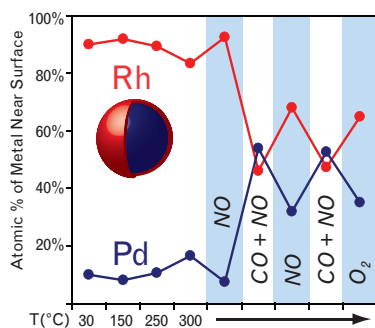
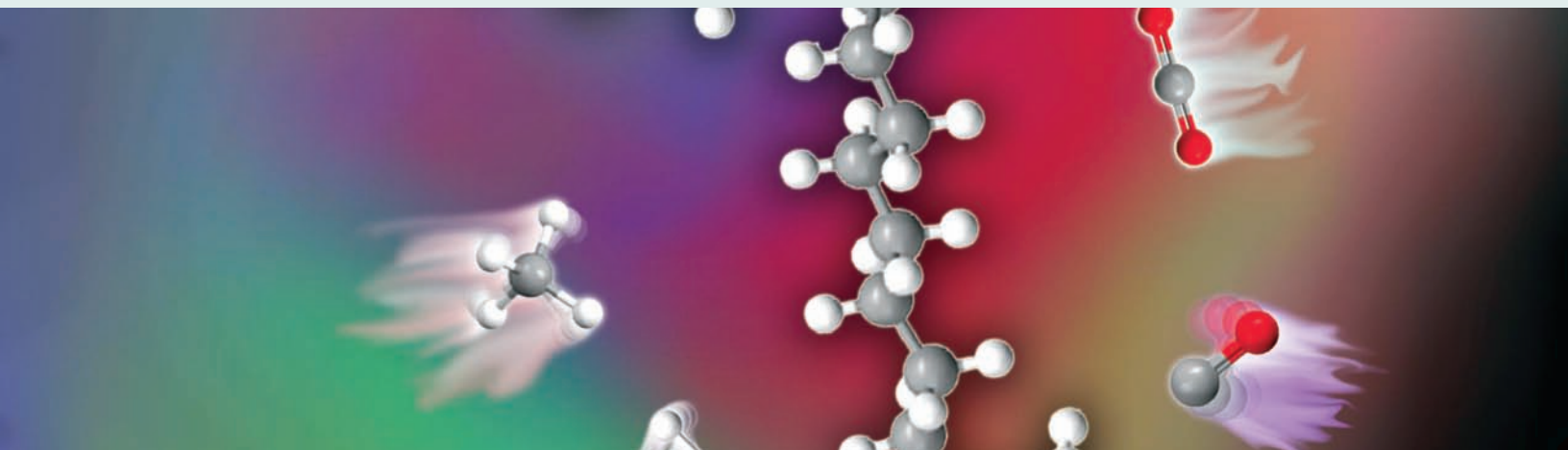
Catalysts—substances that speed up chemical reactions without being consumed—play a central role in technologies relevant to energy and the environment. Because of their importance, researchers are working to develop cheaper and smarter catalysts that are fine-tuned to accelerate reactions that, for example, drive fuel-refinement techniques, feed hydrogen fuel cells, or sweep toxins from emissions.

The catalysts used in chemical processes often consist of nanoscale metal or metal oxide particles dispersed on a surface. While these particles are the active elements of the catalyst, their overall performance depends not only on their size and composition but also on their interactions with the surface, reactants, products, and environment (gaseous atmospheres at high temperatures). Probing this chemical soup in real time under realistic reaction conditions is a tall order, and such catalysts could previously be observed only before and after, but not during, a reaction. In some cases even the catalytically active chemical species is not known.

By combining scanning transmission x-ray **microscopy** (STXM) with a reaction chamber adapted from electron microscopy, we can identify



Tracking changes in an iron-based catalyst. Top left: Iron spectra taken before catalysis from two regions (1 and 2) of the catalyst material. Top right: Spectra taken after catalysis. Dotted lines are calculated from reference spectra. Bar graphs show the relative contributions of different iron phases at the sampling points. Bottom: Iron species contour maps generated from the spectra taken at each pixel. The colors are keyed to the bar graphs above. Courtesy of F.M.F. de Groot (Utrecht University). [de Smit et al., *Nature* **456**, 222 (2008).]

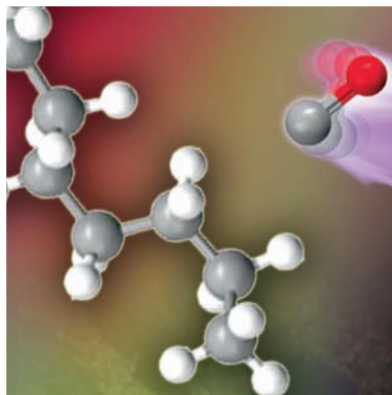
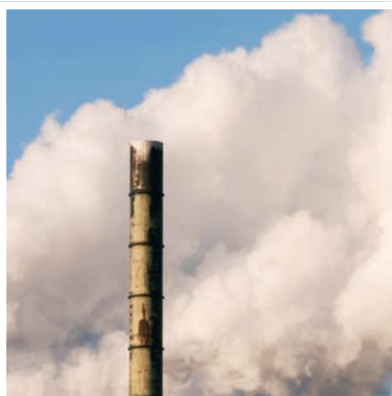
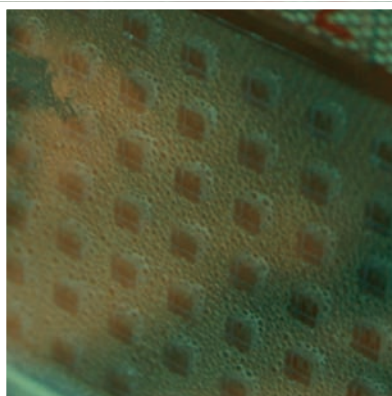


Bimetallic nanoparticles are synthesized with a rhodium-rich shell. Under reaction conditions, the composition of the outer shell is determined by the temperature and the composition of gases surrounding the nanoparticles. Courtesy of G.A. Somorjai (UC Berkeley). [Tao et al., *Science* **322**, 932 (2008).]

the chemical species present and see their distribution on the nanoscale. Such measurements demonstrate that STXM can provide details about the morphology and composition of complex catalytic systems under realistic conditions. When developed further, this new tool may give chemists the ability to design and tailor catalysts for maximum selectivity and efficiency in a wide range of chemical processes.

Some of the most active and selective catalysts are bimetallic nanoparticles, with the two metals variously

distributed between the particle core and shell. In situ studies using ambient-pressure x-ray photoelectron **spectroscopy** have been used to demonstrate that bimetallic catalysts can undergo profound structural and chemical changes in response to reactive environments at ambient pressures. With tunable synchrotron light, it is possible to create depth profiles of particle composition and chemical state. This work is a first step toward designing “smart” catalysts whose structures change advantageously depending on the reaction environment.



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## ADVANCED LIGHT SOURCE

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