

# Scientific Advances Made Possible by User Facilities

Gordon E. Brown Jr.,<sup>1,2</sup> Georges Calas,<sup>3</sup> and Russell J. Hemley<sup>4</sup>

**N**ational scientific user facilities are becoming increasingly available to many different scientific communities in a number of countries. There is a growing use of these facilities by Earth and environmental scientists to study a broad range of materials and processes under realistic P–T and environmental conditions at unprecedented levels of energy and spatial resolution and elemental and isotopic sensitivity. The results of these studies are providing new insights into biogeochemical processes operating at Earth's surface as well as petrological, geochemical, and geophysical processes in Earth's interior. The availability of national user facilities is changing scientific approaches and is leading to multidisciplinary studies that were not possible a decade ago.

KEYWORDS: national scientific user facilities, synchrotron radiation sources, neutron sources, Earth science research.

## INTRODUCTION

Discoveries in most areas of science come from observations at different spatial scales, ranging from astronomical to subatomic, using instrumentation that ranges in complexity from a simple reflecting telescope to a particle accelerator. In the Earth sciences, relevant spatial scales extend from atomic to global, and instrumentation can be as simple as a hand lens and Brunton compass or as complex as a synchrotron light source. The repertoire of available research instruments has expanded greatly during the past 20 to 30 years because of the development of large-scale national user facilities in many countries. These facilities, which include synchrotron light sources, neutron sources, particle accelerators, electron beam microcharacterization facilities, and nanoscale-science research centers, are having a major impact on research on materials of all types. Such facilities are being used to determine the structures, compositional variations, physical properties, and submicron-scale heterogeneities of Earth materials. Experiments are carried out under conditions mimicking those in key geological environments, such as in Earth's deep interior or at the interfaces between mineral particles and aqueous solutions in soils at Earth's surface, where much of the chemistry relevant to the environment occurs. Many of these advanced techniques and cutting-edge technologies are not

generally available in university or individual investigator laboratories, but user facilities make them available to many scientific communities. User facilities also make possible experimental studies that were impossible prior to their existence. This article focuses on the role of national user facilities, particularly synchrotron radiation and neutron sources, in Earth and environmental science research and highlights some of the discoveries such facilities have enabled in these fields over the past decade.

At the atomic scale, 3-D observations require the use of electromagnetic radiation of wavelengths

similar to the bond distances between atoms (0.1–0.5 nanometers) in the building blocks (unit cells) of crystals. Short-wavelength radiation, referred to as X-rays (with energies ranging from about 100 eV to greater than 100 keV), was first discovered in 1895 by Wilhelm Conrad Röntgen. Since that time, X-rays and X-ray diffraction (XRD) have revolutionized many areas of science, as demonstrated by the 18 Nobel Prizes in physics, chemistry, and medicine awarded from 1901 through 2005 that are directly linked to the usage of X-rays. XRD has had a profound impact on mineralogy, having been used over the past 90 years to determine the atomic structures of the 4000+ known minerals.

XRD has also had a major impact on our knowledge of the materials that comprise Earth's interior through studies of the stability and phase transformations of minerals at pressures and temperatures representative of Earth's mantle (~0 to 136 GPa and 1200 to 3600K). The high pressures and temperatures can be generated using diamond anvil cells (DAC) and laser heating, respectively, permitting XRD and spectroscopic studies of mineral phases under in situ conditions (Mao and Hemley 1998; Bass et al. 2004). Large volume presses provide access to somewhat lower temperatures and pressures, but the sample volume is larger than in a DAC. High-P–T studies using these devices have revealed phase transformations and changes in mineral densities at pressures and temperatures corresponding to the depths of major discontinuities in seismic wave velocities within the deep Earth.

XRD is not the only means of determining the atomic structure of matter. The elastic scattering of neutrons also provides this type of information, with some advantages and disadvantages. Advantages include the ability to distinguish between atoms with similar atomic numbers or between different isotopes of the same element. Disadvantages include the fact that larger samples are required in neutron

<sup>1</sup> Department of Geological & Environmental Sciences, Stanford University, Stanford, CA 94305-2115, USA  
E-mail: gordon@pangea.stanford.edu

<sup>2</sup> Stanford Synchrotron Radiation Laboratory, 2575 Sand Hill Road, MS 69, SLAC, Menlo Park, CA 94025, USA

<sup>3</sup> Institut de Minéralogie et de Physique des Milieux Condensés, UMR CNRS 7590, Universités de Paris 6 et 7, ICPG, 140 rue de Lourmel, 75015 Paris, France  
E-mail: calas@lmcp.jussieu.fr

<sup>4</sup> Geophysical Laboratory, Carnegie Institution of Washington, 5251 Broad Branch Rd NW, Washington, DC 20015, USA  
E-mail: r.hemley@gl.ciw.edu

scattering than in X-ray scattering experiments because the neutron scattering cross-sections of atoms are smaller than X-ray scattering interactions with atoms (see Sutton et al. 2006 for additional details).

In addition to XRD, various types of X-ray spectroscopy are also becoming important research tools of scientists from many disciplines, including Earth and environmental sciences. X-ray absorption fine-structure (XAFS) spectroscopy in particular has become the technique of choice for studies of heavy metal and radionuclide speciation at mineral-water interfaces and for characterization of the chemical forms of environmental pollutants in soils. The only practical way of conducting an XAFS study of environmental pollutants at trace concentration levels (<1000 ppm) is to utilize a synchrotron radiation source—the most common type of national user facility worldwide.

Since synchrotron light sources first became available to general users in 1974, they have had a growing impact on research in the Earth sciences. This research focuses on atomic to micron scales and includes trace element geochemistry, interface and surface geochemistry, mineralogy, mineral physics, and petrology. In addition, the new field of molecular environmental science (MES) has been a major beneficiary of synchrotron light sources because of the need to determine the molecular-level speciation, spatial distribution, and phase association of environmental pollutants at very low concentration levels (low parts per million) and spatial scales of nanometers to microns in highly heterogeneous Earth materials such as soils. Synchrotron light sources have also contributed to the development of a mechanistic understanding of the abiotic and biotic processes that can sequester or release pollutants or transform them into more (or less) toxic forms.

The techniques that synchrotron light sources make available include wide-angle and small-angle elastic X-ray scattering (WAXS and SAXS), anomalous X-ray scattering (AWAXS and ASAXS), surface X-ray scattering, nuclear resonant inelastic X-ray scattering (NRIXS), synchrotron X-ray Raman scattering (XRS), nuclear resonant forward scattering (NRFS, a measure of the Mössbauer effect), synchrotron X-ray fluorescence (SXRF) spectroscopy, X-ray absorption fine-structure (XAFS) spectroscopy, X-ray emission spectroscopy (XES), X-ray photoelectron spectroscopy (XPS), X-ray standing wave (XSW) spectroscopy, infrared (IR) spectroscopy, transmission X-ray microscopy (TXM), scanning transmission X-ray microscopy (STXM), X-ray photoelectron emission microscopy (XPEEM), X-ray tomography, and X-ray fluorescence tomography at both hard and soft X-ray energies. Some of these methods are surface sensitive (e.g. XPS and XPEEM) or can be utilized in a surface-sensitive fashion (e.g. grazing-incidence XAFS or regular XAFS when the element of interest is present only at a surface; crystal truncation rod diffraction). Most of these methods can also be made spatially sensitive by reducing the size of the X-ray beam using focusing mirrors ( $\mu$ SXRF,  $\mu$ XAFS,  $\mu$ XRD,  $\mu$ -X-ray fluorescence tomography) or Fresnel zone plates (TXM, STXM, XPEEM), resulting in spatial resolution down to 25 nm at low X-ray energies (e.g. the carbon *K* edge – 284 eV). Many of these X-ray methods and their scientific applications, particularly those involving focused X-ray beams, would not be possible without the extreme brightness of synchrotron light sources.

Developments in high-energy synchrotron techniques coupled with new high-pressure techniques allow inelastic scattering and spectroscopic measurements under extreme pressures down to 4 keV, which covers the *K* edges of first-row transition elements and *L* edges of rare-earth elements,

both central to geochemistry. These measurements allow atomic coordination and electronic and magnetic properties to be studied at extreme pressures.

The examples of scientific discoveries presented below are representative of different subdisciplines within the Earth sciences. Most of the examples come from the application of synchrotron radiation and neutron scattering techniques to Earth materials and processes. Additional examples of applications of neutron scattering and analytical methods, such as accelerator-based mass spectrometry, can be found in the article by Sutton et al. (2006) in this issue.

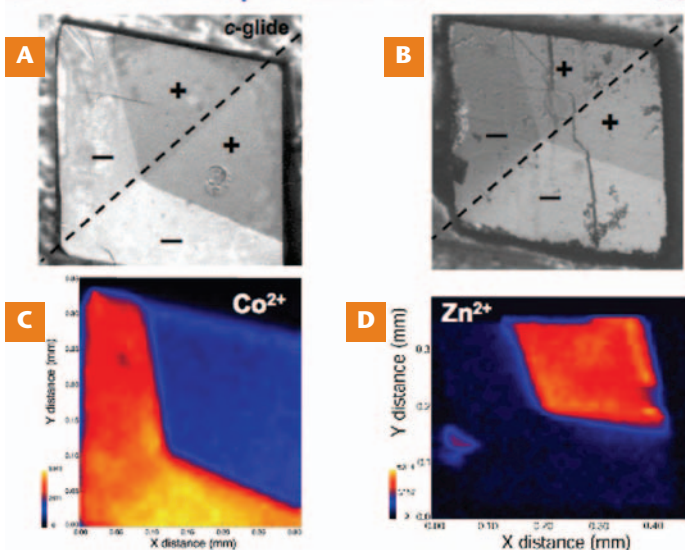
## EARTH AND ENVIRONMENTAL SCIENCE APPLICATIONS OF USER FACILITIES

### *Mineralogy, Petrology, and Trace Element Geochemistry*

Minerals are key components of rocks and provide unique information on their formation conditions. Their minor and trace element and isotope compositions provide important insights into the geochemical cycling of elements and can give us the absolute ages of rocks and the Earth. In addition, minerals are of great importance in modern society because of their many technological and industrial applications. Synchrotron-based methods such as XAFS spectroscopy provide unique information on the location of trace elements in mineral structures, which is key to understanding the controls that minerals exert on element partitioning (Blundy and Wood 2003). This information is also important for understanding charge-compensation processes in minerals and for deriving realistic activity-composition relations. Intersite trace element distributions in minerals can be uniquely provided by XAFS. For example, Quartieri et al. (1999) used XAFS to demonstrate that Nd enters the <sup>VIII</sup>X site in the X<sub>3</sub>Y<sub>2</sub>Si<sub>3</sub>O<sub>12</sub> garnet structure and does not substitute for Al at the <sup>VI</sup>Y site. Most recent XAFS determinations of trace element environments in minerals indicate a high degree of relaxation around substituting elements of different sizes in minerals, such as the substitution of Cu<sup>2+</sup> and Zn<sup>2+</sup> for Ca in calcite (Elzinga and Reeder 2002). The mismatch between the size of the relaxed substituted sites and the lattice site is accommodated by elastic strain. A similar XAFS investigation of substitutional Cr<sup>3+</sup> and Fe<sup>3+</sup> ions in corundum has also confirmed full site relaxation during substitution (Gaudry et al. 2003). The coordination shell relaxes to an arrangement similar to that for Cr in  $\alpha$ -Cr<sub>2</sub>O<sub>3</sub> or for Fe in  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>. This work shows that the red and green colors of ruby and  $\alpha$ -Cr<sub>2</sub>O<sub>3</sub>, respectively, do not originate from different Cr sites but from a modification of the electronic structure.

Petrological applications of synchrotron radiation and neutron scattering methods include XAFS studies of the structural environments of trace elements in silicate melts and glasses and neutron scattering studies of medium-range order in silicate glasses. Silicate melts play a major role in element and heat transfer within the Earth and are responsible for all igneous rocks. In addition, silicate glasses, which are produced by quenching of silicate melts, are used widely as industrial materials and as surrogates for silicate melts in experimental studies. Synchrotron X-ray and neutron scattering methods are unique in their ability to provide information on the structure of amorphous solids and liquids (oxidation states and coordination environments of elements, polymerization of silicate tetrahedra). Oxidation states may now be measured in  $\mu$ m-sized spots, as in glassy inclusions in volcanic minerals (Métrich et al. 2002). X-ray spectroscopic and scattering techniques have been used to determine the local structural environments of trace and major elements as well as the medium-range order in sili-

## Site-selective incorporation of metals at the calcite surface



**FIGURE 1** Differential incorporation of cation (DIC) images of as-grown (10 $\bar{1}$ 4) surfaces of Co<sup>2+</sup>-doped (A) and Zn<sup>2+</sup>-doped (B) calcite crystals. The c-glide plane is vertical and the c-axis points at 45° to the upper right. Polygonal shading patterns reveal two pairs of vicinal surfaces, labeled + and -. They represent regions on the surface having growth steps oriented in different directions. Growth steps in the + vicinals, parallel to [441] and [48 $\bar{1}$ ], are symmetrically equivalent, but are not equivalent to those in the - vicinals. Individual growth steps are not resolved. (C and D) Synchrotron  $\mu$ XRF maps corresponding to crystals in the DIC images. Raw counts are plotted, and light shading indicates highest counts. Corresponding Ca maps are featureless. Co<sup>2+</sup> is preferentially incorporated at steps in the - vicinals, whereas Zn<sup>2+</sup> is preferentially incorporated at steps in the + vicinals. FIGURE COURTESY OF RICH REEDER; AFTER ELZINGA AND REEDER 2002.

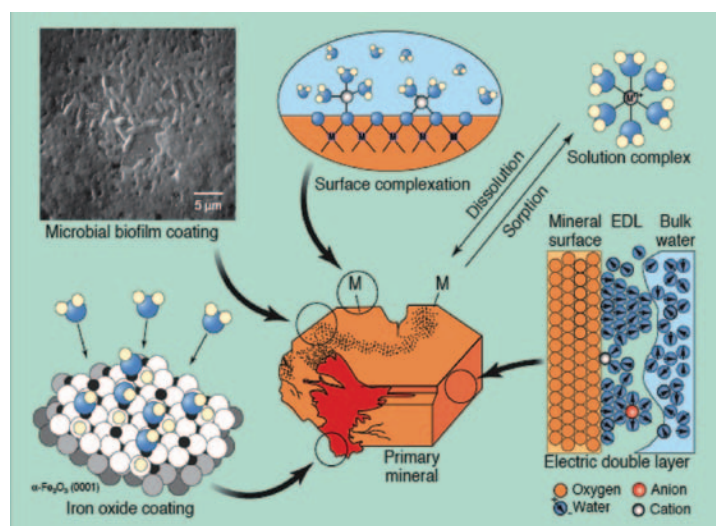
cate glasses and melts (Brown et al. 1995). This work provides unique insights into trace element partitioning and crystal nucleation. For example, XAFS studies of silicate glasses provided the first direct evidence for 5- and 4-coordinated Ni<sup>2+</sup> (Galoisy and Calas 1993) and Fe<sup>2+</sup> (Jackson et al. 2005) and for two-dimensional cation-rich regions extending over 15–20 Å (Cormier et al. 2001). This inhomogeneous distribution of cations in glasses, distinct from precursors of mineral nuclei, may be related to the progressive organization of supercooled liquids as temperature decreases. The use of high-energy X-ray scattering, which is only possible using synchrotron radiation, allows resolution of Si–O and Al–O contributions in aluminosilicate glassy networks, giving an accurate description of Si/Al disorder (Petkov et al. 2000). Structural changes in glasses and melts may be investigated as a function of temperature using in situ X-ray and neutron diffraction and XAFS (Brown et al. 1995). Disorder and thermal expansion of cationic sites occur without discontinuity through the glass transition (Majérus et al. 2004a). Above the glass transition, a relaxation of the silicate network occurs on a medium-range scale (6–7 Å) as a result of a higher cation motional rate relative to the network. Large user facilities have also been used to study processes governing the modification of glass and melt structures at high pressure (polyamorphism). Determination of the structure of high-density glasses requires in situ studies since coordination changes are reversible (Majérus et al. 2004b).

## Interface and Surface Geochemistry

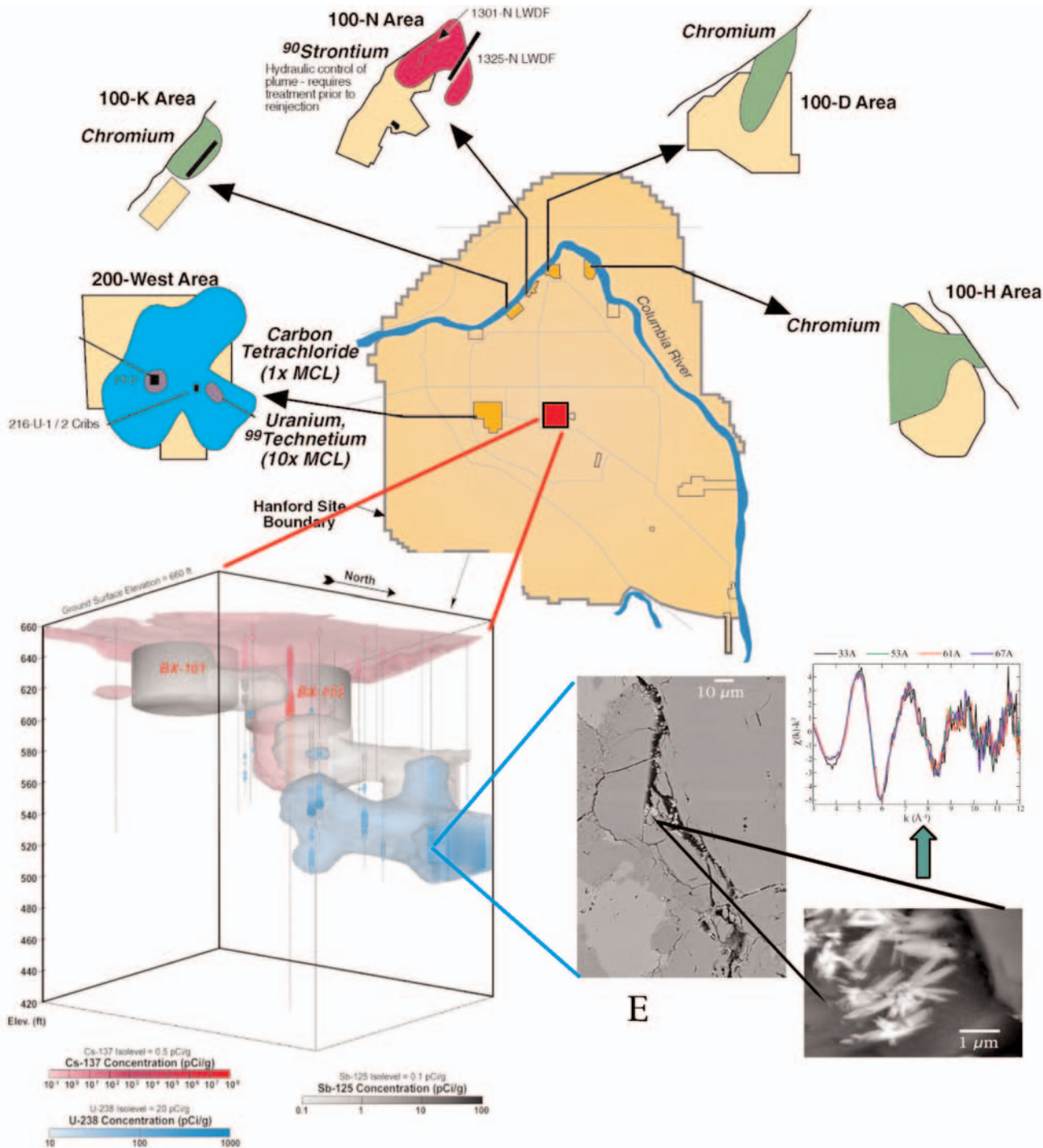
Recognition of the importance of surface chemical reactions in geochemical and environmental contexts (Fig. 2) can be traced back to the pioneering work by early soil chemists on soil–fluid interactions, particularly the paper by J. Thomas Way (1850). More than 150 years later, we

have a better understanding of the sorption and exchange phenomena that account for Way's observation that the filtration of "liquid manure" through a loamy soil resulted in the manure being "deprived of colour and smell." Much of this molecular-level understanding has come from synchrotron radiation studies, particularly XAFS studies of ion sorption reactions at mineral–water interfaces (Brown and Parks 2001). For example, Hayes et al. (1987) used XAFS under in situ conditions to show that aqueous selenate ions (SeO<sub>4</sub><sup>2-</sup>) form dominantly outer-sphere complexes on goethite ( $\alpha$ -FeOOH) surfaces, whereas aqueous selenite ions (SeO<sub>3</sub><sup>2-</sup>) form dominantly bidentate-binuclear inner-sphere complexes on these surfaces. Since this study, hundreds of similar XAFS experiments on many different adsorbed cations and anions have been performed, providing much-needed molecular-level insights into ion sorption processes. An important question raised by these studies is how the structures of mineral surfaces in contact with water differ from simple terminations of the bulk crystal structure, which are typically assumed to accurately represent the structures of wet mineral surfaces (Brown and Sturchio 2002). This question has recently been addressed for the hydrated  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> (0001) (Eng et al. 2000) and  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> (0001) (Trainor et al. 2004) surfaces using synchrotron-based surface scattering known as crystal truncation rod (CTR) diffraction. These CTR diffraction studies show that the structures of the hydrated  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> and  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> (0001) surfaces are significantly different from each other, which helps explain their differences in reactivity to water (Liu et al. 1998) and aqueous metal ions such as Pb<sup>2+</sup><sub>aq</sub> (Bargar et al. 2004). They also show that these hydrated surfaces are not well represented by simple terminations of the bulk corundum and hematite structures along the (0001) plane. An excellent review of synchrotron-based X-ray scattering studies of these and other mineral surfaces is provided by Fenter and Sturchio (2004). This approach is providing the first detailed molecular-level views of mineral–water interface structures and interfacial water under environmental conditions, and shows that surface hydration layers are a general characteristic of mineral–water and mineral–water vapor interfaces.

X-ray scattering and other synchrotron-based methods, such as scanning transmission X-ray microscopy, are also being used to provide unique insights into the changes in



**FIGURE 2** Example of a complex mineral–water interface, including both iron oxide and microbial biofilm coatings, illustrating some of the molecular-scale processes occurring at these interfaces, including interaction with water, sorption or surface complexation of aqueous metal ions, and dissolution. Also shown is a model of the electrical double layer at the mineral–aqueous solution interface. From BROWN 2001.



**FIGURE 3** (Top) Map (not to scale) of the Hanford Site, Washington State, showing contaminant plumes in the vadose zone beneath various Hanford Tank Farms. (Lower left) 3-D nature of  $^{137}\text{Cs}$ ,  $^{125}\text{Sb}$ , and  $^{238}\text{U}$  plumes beneath Tank BX-102 in the 200-East Area, where 300,000 liters of waste containing 7–8 metric tons of uranium were spilled in 1951. (Lower right) Backscattered electron images of vadose-zone plagioclase grains containing Na-boltwoodite in cracks, and  $\text{U L}_3$ -XAFS spectra of Na-boltwoodite grains. TOP FIGURE FROM RILEY AND ZACHARA 1992; LOWER LEFT FIGURE FROM PEARSONS 2000; LOWER RIGHT FIGURE FROM JIM MCKINLEY, PERS. COM.; XAFS SPECTRA FROM CATALANO ET AL. 2004)

mineral surfaces resulting from abiotic and biotic reactions, which cause oxidation and dissolution of mineral surfaces. For example, the oxidation and dissolution of pyrite surfaces by *Acidithiobacillus* sp. have been studied by oxygen *K* edge and iron *L* edge XAFS (pers. com., B.C. Bostick 2004), and the oxidation and dissolution of pyroxene surfaces have been studied by STXM spectromicroscopy (Benzerara et al. 2005). Another example of the unique information provided by synchrotron radiation studies is the use of long-period X-ray standing wave fluorescence yield spectroscopy to study the partitioning of heavy metals such as  $\text{Pb}^{2+}$  between  $\alpha\text{-Al}_2\text{O}_3$  (and  $\alpha\text{-Fe}_2\text{O}_3$ ) single-crystal surfaces and *Burkholderia cepacia* biofilms grown on these surfaces (Templeton et al. 2001). In addition, XAFS and synchrotron-based XRD methods are providing important insights into the formation and structure of nanominerals resulting

from bacteriogenic processes (e.g. Suzuki et al. 2002). The study of mineral–bacteria interactions using synchrotron radiation methods is anticipated to grow significantly in coming years and to yield important new information on biomineralization, bioweathering, and the biogeochemical cycling of elements (Kemner et al. 2005).

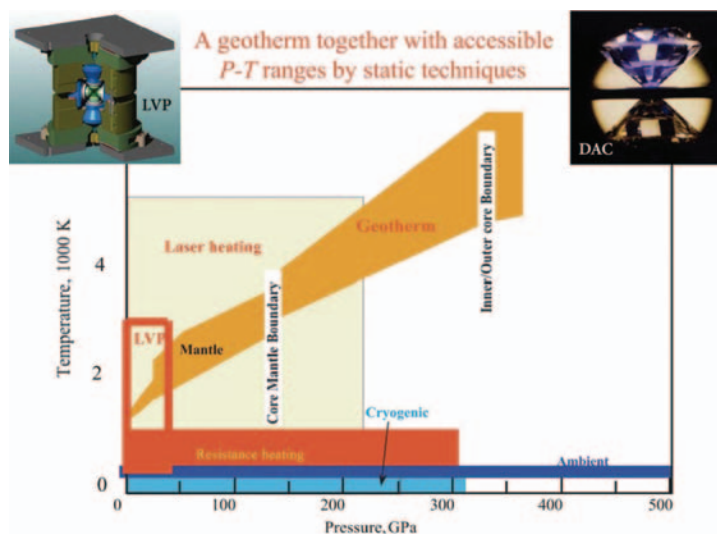
## Environmental Mineralogy and Geochemistry (Molecular Environmental Science)

Synchrotron radiation methods have played a major role in studies of the chemical forms, spatial distribution, and phase association of contaminant elements in environmental samples. These materials range from mine wastes and soils contaminated by various heavy metals, radionuclides, and xenobiotic organics to plants that hyperaccumulate specific elements and fish that contain high concentrations of methyl mercury. The new field of *molecular environmental science* has developed in response to the need for such information for understanding the transport behavior, toxicity, and potential bioavailability of contaminants (Brown et al. 1999). A great deal of attention in this new field has focused on contaminant speciation in soils, which are the products of chemical and biological reactions at the interfaces among the solid Earth (lithosphere), atmosphere, hydrosphere, and biosphere. Soils host crops and support most human activities. Their high reactivity makes them sensitive to environmental modifications and contamination by heavy elements and xenobiotic organics. The finely divided texture of soils, the importance of surface reactions, and the need to determine the speciation of inorganic, biological, and organic components explain the growing number of soil-related studies at national user facilities such as synchrotron light sources.

Lead and other heavy metals are major contaminants in some soils, raising environmental concerns; however, the bioavailability and chemical lability of these contaminants often show little correlation with their bulk concentrations. The key to understanding this observation is the molecular-level speciation of contaminants (i.e. their chemical form). For example, mineralogical and X-ray spectroscopic studies have shown that in smelter-contaminated soils from northern France, lead is sorbed to humic material as well as to both manganese and iron oxides, oxyhydroxides, and hydroxides (Morin et al. 1999). In mine tailings from Colorado, USA, carbonate-buffered tailings with near-neutral pH contain up to 50% of the total lead sorbed on iron-(oxyhydr)oxides, whereas Pb-bearing jarosites  $[\text{KFe}_3(\text{SO}_4)_2(\text{OH})_6]$  dominate in sulfide-rich low-pH samples (Ostergren et al. 1999). In both cases, sorbed Pb may become potentially bioavailable if the local Eh and pH conditions change. Another example is the speciation of uranium in the vadose zone beneath Tank BX-102 in the 200-East Area Tank Farm at Hanford, Washington. This problem has also been addressed with synchrotron radiation methods (Catalano et al. 2004) (Fig. 3). Using a combination of XAFS and  $\mu\text{XRD}$ , this study showed that  $\text{U}^{6+}$  occurs in fractures in plagioclase feldspar grains dominantly as the uranophane group mineral Na-boltwoodite  $[\text{Na}(\text{UO}_2)(\text{SiO}_3\text{OH}) \cdot 1.5\text{H}_2\text{O}]$ , which is moderately soluble in the interparticle pore waters.

However, the location of this phase in feldspar grain fractures and the low moisture content in the Hanford vadose zone make it unlikely that commonly used remediation methods will be successful in removing this phase or that this phase will release  $\text{U}^{6+}$ . These types of studies of heavy metal and radionuclide contaminants are providing much-needed understanding of their phase association, potential bioavailability, and environmental fate.

Although not strictly in the field of molecular environmental science, recent studies of the structure of water using synchrotron radiation methods (O K-edge XAFS and X-ray Raman scattering) (Wernet et al. 2004) have led to the revolutionary suggestion that the 4-hydrogen bonded structure of water that has heretofore been assumed is not correct. Instead, compelling experimental and theoretical evidence has been found for about 80% of water molecules in



**FIGURE 4** Temperature and pressure range accessible using laser-heated diamond anvil cells (DAC) (light tan area), resistance-heated DAC (reddish brown area), and DAC at ambient (dark blue area) and cryogenic temperatures (light blue area). Also shown is the P-T range accessible using large volume presses (LVP) (area outlined by reddish brown boundary). Earth's geotherm is shown as the light brown area. Also shown are a schematic diagram of a large volume press (upper left) and a photograph of a diamond anvil cell (upper right). FROM BASS 2004. PICTURE AND DIAGRAM AT THE TOP OF THIS FIGURE ARE FROM THE GEOPHYSICAL LABORATORY, CARNEGIE INSTITUTION OF WASHINGTON

liquid water having an asymmetric 2-hydrogen bonded structure. The implications of this discovery in aqueous geochemistry have yet to be detailed, but it is likely that this new structure of water, if correct, will lead to revisions in our thinking about its structure-property relationships and the hydration of ions in aqueous solutions.

## Mineral Physics

Recent years have witnessed many advances in the use of synchrotron techniques in high-pressure mineral physics, including XRD, spectroscopy, inelastic scattering, and radiography, as well as infrared spectroscopy using laser-heated diamond anvil cells, which currently permit access to simultaneous temperatures and pressures of 5000K and 225 GPa, respectively (Fig. 4). Polycrystalline XRD includes simultaneous high P-T diffraction using double-sided laser heating methods, and newly designed cells allow determination of stress-strain conditions and the measurement of second-order elastic tensors at ultrahigh P-T conditions. Single-crystal methods have been extended to above 100 GPa using high-brightness X-ray sources. These developments have also opened a window for studying local chemical environments, including atomic coordination, structures, and bonding character with a diversity of X-ray spectroscopies and scattering techniques. Other experiments involve the use of X-ray radiography over a range of pressures. The enormous flux advantage of infrared synchrotron radiation relative to conventional thermal IR sources provides a significant improvement in spectroscopic studies of microscopic samples at very high pressures. The following examples illustrate the application of multiple techniques to Earth and planetary materials under extreme conditions. Specifically, a growing number of studies utilize the complementary roles of synchrotron X-ray and neutron scattering methods.

One of the major developments in mineral physics of the last several decades has been the discovery of the post-perovskite phase of  $\text{MgSiO}_3$  above 100 GPa with synchrotron radiation and laser-heated diamond cell techniques (Murakami et al. 2004). The post-perovskite phase can take

up to 80% iron in the Mg site and exists in the low-spin (i.e. magnetic spin-paired) state at conditions of the D'' region at the base of the Earth's mantle, as shown by subsequent synchrotron X-ray and emission experiments (Mao et al. 2004). Such high P-T diffraction measurements have been complemented by an array of high-pressure X-ray spectroscopic techniques that have now come on line. Most recently, the X-ray emission technique has been applied to more chemically complex deep-Earth minerals such as (Mg,Fe)O and (Mg,Fe,Al)SiO<sub>3</sub> perovskite to identify high-spin/low-spin transitions of iron, which are expected to have profound implications for compositional layering in the lower mantle (Badro et al. 2004; Lin et al. 2005a).

Studies of the high P-T behavior of iron represent an excellent example of the use of multiple synchrotron radiation techniques (Hemley and Mao 2001). In situ high P-T synchrotron XRD studies demonstrate that the hexagonal closest-packed phase ( $\epsilon$ -Fe) has a wide stability field extending from deep mantle to core conditions. Seismological observations of inner core anisotropy and structure present new questions about the dynamics and magnetism of the core. The aggregate longitudinal velocity ( $V_p$ ) and shear velocity ( $V_s$ ) at high P and T are the primary data obtained from seismic observations. Radial diffraction measurements also provide information on single-crystal elasticity, deformation mechanisms, preferred orientation, slip systems, plasticity, failure, and shear strength (Wenk et al. 2000). The nuclear resonant inelastic X-ray scattering (NRIXS) technique has been developed for the measurement of phonon densities of states and elasticity of materials containing <sup>57</sup>Fe. The observed compressional and shear wave velocities of  $\epsilon$ -Fe decrease with increasing temperature at high pressures, and a strong temperature effect on the linear sound velocity-density relations (Birch's law) was found (Lin et al. 2005b). High-pressure phonon dispersion measurements have been carried out to megabar pressures (Fiquet et al. 2001). The structure of molten Fe has been determined over a range of P-T conditions generated using both large volume and diamond cell methods (Sanloup et al. 2000; Shen et al. 2004).

Detailed information on crystal structures is an essential starting point for understanding the origin of the chemical and physical properties of materials that comprise not only the Earth but also other planets. The single-crystal X-ray diffraction method first used for H<sub>2</sub> (Mao et al. 1988) has been extended to megabar pressures (Loubeyre et al. 1996). Integration of new synchrotron X-ray microdiffraction methods is opening up new fields of micro- to nanomineralogy, as shown by the refinement of the crystal structures of new Fe-Si phases identified in a lunar meteorite (Anand et al. 2004).

Continued advances in high-pressure synchrotron infrared spectroscopy have led to a series of studies of materials at ultrahigh pressures, including the discovery of a number of unexpected phenomena in dense hydrogen. Synchrotron infrared reflectivity spectra of H<sub>2</sub>O to 210 GPa showed that the transition of ice to the long-sought, non-molecular, symmetric hydrogen-bonded structure occurs at 60 GPa (Goncharov et al. 1996). A particularly important new development has been the extension and improvement of far-IR measurements in ice (Klug et al. 2004) and biomolecules (Klug et al. 2002). The inelastic K-edge scattering has been used to probe bonding changes in materials, including most recently in ice, providing evidence for a new low P-T phase (Cai et al. 2005).

Finally, it is useful to point out the complementarity between X-ray and neutron experiments. Recently, the evolution of the structure of amorphous GeO<sub>2</sub> at high pressure was determined by combining high-energy synchrotron

X-ray and neutron diffraction techniques (Guthrie et al. 2004). Variable P-T neutron diffraction techniques reveal new findings concerning the magnetic properties of the high-pressure rhombohedral phase of Fe<sub>1-x</sub>O (wüstite) (Ding et al. 2005b), whereas zone-axis synchrotron XRD experiments on single crystals reveal that Fe<sub>1-x</sub>O has a long-range defect-cluster order-disorder transition (Ding et al. 2005a). Recent neutron and X-ray diffraction studies of hydrogen isotopes provide a direct probe of pressure-induced transformations in this important planetary material (Goncharenko and Loubeyre 2005). Another example where both techniques have been exploited is H<sub>2</sub>O-H<sub>2</sub> clathrate, which is important for diverse problems ranging from condensation in solar nebulae to use as a hydrogen storage material for automobiles (Mao et al. 2002).

## CONCLUDING REMARKS

Molecular-level studies of the type discussed above provide the main intellectual interfaces between Earth sciences and disciplines such as chemistry, materials science, microbiology, physics, and structural molecular biology. They are also one of the main conduits for students from these other disciplines to enter the Earth sciences and make truly interdisciplinary contributions. Moreover, the analytical instrumentation and techniques developed in other disciplines, such as transmission electron microscopy, electron microprobe analysis, mass spectrometry, X-ray absorption spectroscopy, NMR spectroscopy, SQUID magnetometry, and X-ray and neutron diffraction, have had and will continue to have enormous impacts on Earth sciences research. Without this cross-fertilization of ideas and techniques from other disciplines, vital Earth sciences research areas such as isotope geochemistry, geomicrobiology, surface and interface geochemistry, mineral physics, and paleomagnetism would have taken longer to develop. Breakthroughs in most areas of science depend strongly on the development of new experimental methods and the new observations and new theories they provide, which guide our thinking and lead to new paradigms. National user facilities of the type highlighted in this issue of *Elements* are providing new experimental capabilities that are making new discoveries about Earth materials and processes possible. Such facilities also play another important role in Earth sciences. They are fertile ground for new ideas because of the mix of scientists from many different disciplines and different countries they attract and the many chances for young Earth scientists to interact with young scientists from other fields. They are well worth the investment because of the advances in science and technology they enable as well as the cross-disciplinary collaborations they stimulate.

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