

*30<sup>th</sup> Annual*

# *SSRL Users' Meeting*

*October 8-10, 2003*



SSRL 30

Stanford Synchrotron Radiation Laboratory  
30<sup>th</sup> Annual Users' Meeting



***PROGRAM & ABSTRACTS***

**OCTOBER 8 - 10, 2003**

**Stanford Synchrotron Radiation Laboratory  
SLAC, Stanford University  
Stanford, CA 94309**

**Organizers: Benjamin Bostick, Dartmouth College  
Timothy McPhillips, SSRL**

SSRL is funded by the Department of Energy, Offices of Basic Energy Sciences and Biological and Environmental Research; the National Institutes of Health, National Center for Research Resources, Biomedical Technology Program, and the National Institute of General Medical Sciences.



## Acknowledgments

### **SSRL Users' Organization Executive Committee**

Uwe Bergmann, *Chairman*, Stanford Synchrotron Radiation Laboratory  
Corwin Booth, *ex-officio*, Lawrence Berkeley National Laboratory  
Benjamin Bostick, *Vice-Chairman*, Dartmouth College  
Richard Brennan, Oregon Health Sciences University  
Jane DeWitt, California State University, San Francisco  
Andrew Fisher, University of California, Davis  
Richard Lee, Lawrence Livermore National Laboratory  
Anneli Munkholm, Lumileds Lighting  
Erik Nelson, Lawrence Livermore National Laboratory  
Cathy Knotts, *SSRL Liaison*  
Nicholas Pingitore, University of Texas at El Paso  
Martina Ralle, Oregon Health Sciences University  
Deanne Jackson Rudd, Stanford University  
C. David Stout, The Scripps Research Institute

### **30<sup>th</sup> Annual SSRL Users' Meeting Committee**

Benjamin Bostick, *Scientific Program Chair*  
Timothy McPhillips, *Scientific Program Chair*  
Cathy Knotts  
Stephanie Carlson  
Lisa Dunn  
Michelle Montalvo  
Ann Mueller  
Jennifer Peck  
Amanda Prado  
Jackie Robleto  
Amy Rutherford  
Todd Slater  
Michelle Steger



## Contents

- 1 Comprehensive Program
- 5 Users' Meeting Speaker Abstracts
- 19 Workshop: Crystallography Beam Line Automation: Work Smarter Not Harder
- 20 Workshop: Inelastic X-ray Scattering and Advanced Spectroscopy Facility for SPEAR3
- 21 Workshop: Synchrotron Techniques for Environmental Microbiology and Biogeochemistry
- 23 Workshop: Probing Mechanical Deformation and Failure via Synchrotron X-rays
- 24 Workshop: Recent Advances in Soft- and Hard X-ray Microscopy
- 27 Poster Index
- 29 Poster Abstracts



## Comprehensive Program

### Wednesday, October 8, 2003

7:30–8:00 am

*Workshop Registration*

8:00 am–5:00 pm

### Concurrent Workshops

- Crystallography Beam Line Automation: Work Smarter Not Harder
- Inelastic X-ray Scattering and Advanced Spectroscopy Facility with SPEAR3
- Synchrotron Techniques for Environmental Microbiology and Biogeochemistry
- Probing Mechanical Deformation and Failure via Synchrotron X-rays
- Recent Advances in Synchrotron-Based Microscopy

*(Note: some of the joint SSRL-ALS workshops take place at SSRL and/or at ALS.)*

3:00–5:00 pm

*Vendor Exhibit Set-up*

5:00–7:00 pm

*Workshop Reception / Users' Meeting Registration*

### Thursday, October 9, 2003

7:30–8:00 am

*Registration and Continental Breakfast*

*Vendor Exhibits*

### Session I

### Users' Meeting Opening Remarks

8:00–8:15 am

SSRLUO-EC Welcome

Benjamin Bostick, Dartmouth College (Session Chair)

8:15–8:30 am

View from Washington

Mike Lubell, Director of Public Affairs, American Physical Society;  
Chairman, Department of Physics, CUNY

8:30–8:45 am

Welcome Remarks

Jonathan Dorfman, SLAC Director

8:45–9:15 am

DOE BES Perspective

Patricia Dehmer, Associate Director of Science for BES, DOE

9:15–9:45 am

SSRL Director's Report

Keith Hodgson, SLAC Associate Director for SSRL

9:45–10:10 am

*Break (Vendor Exhibits / Poster Set-up / Deadline for SSRLUO-EC Voting)*

**Session 2****Perspectives on the UTEP/SSRL Gateway Program**

In tribute to the late Iran Thomas of the U.S. Department of Energy's Office of Science, a special session has been organized to honor Thomas' interest in education and diversity. Thomas, who passed away on February 28, 2003, was a long-time director in the Office of Basic Energy Sciences (BES) who supported and enabled the development of new and innovative research programs, including the University of Texas El Paso (UTEP) and SSRL Gateway program.

10:10–10:15 am

Introduction  
Russ Chianelli, UTEP (Session Chair)

10:15–10:30 am

Overview Remarks  
Patricia Dehmer, Associate Director of Science for BES, DOE

10:30–10:45 am

The UTEP/SSRL Gateway Program  
Russ Chianelli, UTEP and Arthur Bienenstock, Stanford University

10:45–11:00 am

Synchrotron Radiation Study of Pb and As in Air Filter Particulate Matter  
Xiomara C. Kretschmer, UTEP

11:00–11:15 am

Cadmium Uptake and Translocation in Tumbleweed (*Salsola kali*), a Potential Cd-Hyperaccumulator Desert Plant Species: ICP/OES and XAS Studies  
Guadalupe de la Rosa, UTEP

11:15–11:45 am

HRTEM, Synchrotron, and Simulation Techniques Applied to Activity and Selectivity Correlation in Hydrosulfurization Catalysts  
Myriam Perez De la Rosa, UTEP

11:45 am–12:30 pm

*Lunch / Vendor Exhibits*

**Session 3****Micro X-ray Beam Spectroscopy and Diffraction**

12:30–12:35 pm

Introduction of Technique and Speakers  
John Bargar, SSRL (Session Chair)

12:35–1:00 pm

Examples of the Application of Mirror-based Hard X-ray Microprobe Techniques in the Earth and Environmental Sciences  
Antonio Lanzirrotti, University of Chicago

1:00–1:25 pm

Biological X-ray Absorption Spectroscopy Imaging  
Ingrid Pickering

1:25–1:50 pm

X-ray Spectroscopic and Diffraction Study of the Speciation of Uranium in Contaminated Sediments from the DOE's Hanford Site  
Jeffrey Catalano, Stanford University

1:50–2:15 pm

Microbial Polymer Templatation of Iron Oxyhydroxides  
Clara Chan, University of California at Berkeley

2:15–3:40 pm

*Break / Vendor Exhibits*

**Session 4**

2:15–3:40 pm

**Session 5**

3:40–3:45 pm

3:45–4:20 pm

4:20–4:45 pm

4:45–5:10 pm

5:10–5:35 pm

5:35–6:00 pm

6:00–6:30 pm

6:30 pm

**Friday, October 10, 2003**

7:45–8:15 am

**Session 6**

8:15–8:20 am

8:20–8:55 am

8:55–9:20 am

9:20–9:45 am

9:45–10:10 am

10:10–10:20 am

**Poster Session**

User Research Poster Session, Tent

**Spectroscopy and Crystallography of Macromolecules**

Introduction of Techniques and Speakers  
Timothy McPhillips, SSRL (Session Chair)

A Conformational Mimic of the MgATP bound “On State” of the Nitrogenase Fe Protein  
John Peters, Montana State University

The Structure of an Electron Transfer Complex Consisting of a Water-Soluble and Integral Membrane Protein from a Photosynthetic Bacterium  
Herbert Axelrod, University of California at San Diego

Instrumentation for Polarized XAS of Protein and Inorganic Single Crystals at SSRL  
Matthew Latimer, SSRL

X-ray Spectroscopy of Photosystem II Single Crystals  
Junko Yano, LBNL

Low-resolution Phasing by Se-MAD in the Presence of Radiation Damage  
Constantina Bakolitsa, The Burnham Institute

*Reception*

*Dinner*

*Refreshments / Vendor Exhibits*

**Small Angle X-ray Scattering**

Introduction of Technique and Speakers  
Hiro Tsuruta, SSRL (Session Chair)

*In situ* SAXS/WAXS Studies of the Precipitation, Crystallization and High Temperature Phase Transitions of Minerals  
Sam Shaw, University of Oxford

Counterion Behavior on the Surfaces of Condensed Biopolymers  
Thomas Angelini, University of Illinois

Probing the Counterion Atmosphere around DNA by Anomalous Small-angle X-ray Scattering  
Rhiju Das, Stanford University

Closing the Folding Chamber of the Eukaryotic Chaperonin Requires the Transition State of ATP Hydrolysis  
Anne Meyer, Stanford University

*Break / Vendor Exhibits*

**Session 7**

10:20–10:25 am

**Surface Spectroscopy**

Introduction of Technique and Speakers  
Piero Pianetta, SSRL (Session Chair)

10:25–11:00 am

Investigations of the Structural Properties of Aqueous Geochemical Interfaces by the Use of X-ray Diffraction, Standing Waves and Spectroscopy  
Peter Eng, University of Chicago

11:00–11:25 am

Investigations into the Surface Structure and Chemistry of ZnS Nanoparticles  
Benjamin Gilbert, University of California at Berkeley

11:25–11:50 am

Rapid and Heterogeneous Oxidation of Pyrite Surfaces by *Thiobacillus ferrooxidans* and *T. thiooxidans*  
Benjamin Bostick, Dartmouth College

11:50 am–12:15 pm

The Structure of Ni-based Refractory Metal Glasses  
John Bilello, University of Michigan

12:15–1:00 pm

*Lunch / Vendor Exhibits*

**Session 8**

**SSRL Reports**

1:00–1:05 pm

Introduction  
Timothy McPhillips, SSRL (Session Chair)

1:05–1:40 pm

SSRL Beam Line Development  
Tom Rabedeau, SSRL

1:40–2:05 pm

SPEAR3 Status Report  
Robert Hettel, SSRL

2:05–2:30 pm

SPEAR3 Commissioning Plans  
James Safranek, SSRL

2:30–2:55 pm

LCLS Update  
John Galayda, SSRL

2:55–3:15 pm

SPPS Update  
Jerry Hastings, SSRL

*Users' Meeting Adjourns*

3:30 pm

---

**SSRLUO-EC Meeting**

Introduce Newly Elected Members; Elect Vice Chair for next year

Stanford Synchrotron Radiation Laboratory  
30<sup>th</sup> Annual Users' Meeting



***USERS' MEETING SPEAKER  
ABSTRACTS***



## **Perspectives on the UTEP/SSRL "Gateway Program"**

### **The UTEP/SSRL "Gateway Program"**

#### **Synchrotron-based Studies in the Environmental, Geological, Materials and Life Sciences: Enhancing Interdisciplinary Graduate Research, Education, and Training at UTEP**

R. R. Chianelli,<sup>1</sup> N. Pingitore,<sup>1</sup> G. Meitzner,<sup>1</sup> J. Gardea-Torresday,<sup>1</sup> and  
Arthur Bienenstock,<sup>2</sup> John Pople,<sup>2</sup> and Apurva Mehta<sup>2</sup>

<sup>1</sup>University of Texas at El Paso (UTEP), El Paso, TX 79968, USA

<sup>2</sup>Stanford Synchrotron Radiation Laboratory, SLAC, Stanford University, Stanford, CA 94309, USA

Synchrotron-based research represents the cutting edge of many scientific sub-disciplines and interdisciplinary areas. In 1999 a program was initiated with the support and assistance of Iran Thomas to bring training in synchrotron research to students and faculty on the United States/Mexican border. This program involves scientific collaborators at SSRL/Stanford and UTEP. The population represented in this program is largely Hispanic and is rapidly growing. The objective of the program is to bring members of this group in to the mainstream of synchrotron research. In this session we report progress in training students from the United States/Mexican border. The session will also "showcase" students now entering the world of synchrotron research and the first-class research that they are performing in areas of materials and environmental science and engineering geology and the life sciences.



Gateway Students, L. Polette, C. Gutierrez Wing and N. Ugarte (University of Texas), working on a powder diffraction experiment on BL2-1.

## **Synchrotron Radiation Study of Pb and As in Air Filter Particulate Matter**

X. C. Kretschmer and N. E. Pingitore, Jr.

The Department of Geological Sciences, The University of Texas at El Paso, El Paso, TX 79968, USA

El Paso and Ciudad Juarez are neighboring cities on the U.S.--Mexico border that historically have experienced particulate matter (PM) pollution of their shared airshed. Pb and As are important pollutants present in the particulate matter fraction trapped by air sampling filters. We examined air filters from as early as 1977 to as recently as 2001 to determine the oxidation state and speciation of As and Pb present on these filters. We believe that the lead found on these air filters has three possible major sources: a local copper smelter, leaded gasoline (in use until approximately 1990 in Mexico), and a mixed general urban lead input. Information on the chemical speciation of Pb and As is important in determining bioavailability, fate, and possible sources of these contaminants.

## **Cadmium Uptake and Translocation in Tumbleweed (*Salsola kali*), a Potential Cd-Hyperaccumulator Desert Plant Species: ICP/OES and XAS Studies**

G. de la Rosa,<sup>1</sup> J. R. Peralta-Videa,<sup>1</sup> M. Montes,<sup>2</sup> J. G. Parsons,<sup>1</sup> and J. L. Gardea-Torresdey<sup>1,2</sup>

<sup>1</sup>Environmental Science & Engineering Ph.D. Program, The University of Texas at El Paso, El Paso, TX 79968, USA

<sup>2</sup>Chemistry Department, The University of Texas at El Paso, El Paso, TX 79968, USA

Cadmium is a heavy metal, which, even in low concentrations, can be highly toxic to the growth and development of both plants and animals. Plant species vary extensively in their tolerance to excess cadmium in a growth medium and very few cadmium-tolerant species have been identified. In this study, tumbleweed plants (*Salsola kali*) grown in an agar-based medium with 20 mg l<sup>-1</sup> of Cd(II) did not show phytotoxicity, and their roots had the most biomass (4.5 mg) (P<0.05) compared to the control plants (2.7 mg) as well as other treated plants. These plants accumulated 2,696, 2,075, and 2,016 mg of Cd per kg of dry roots, stems, and leaves, respectively. The results suggest that there is no restricted cadmium movement in tumbleweed plants. In addition, the amount of Cd found in the dry leaf tissue suggests that tumbleweed could be considered as potential cadmium hyperaccumulating species. X-ray absorption spectroscopy studies demonstrated that in roots, Cd was bound to oxygen while in stems and leaves the metal was attached to oxygen and sulfur groups. This might imply that some small organic acids are responsible for Cd transport from roots to stems and leaves. In addition, it might be possible that the plant synthesizes phytochelatins in the stems, later coordinating the absorbed cadmium for transport and storage in cell structures. Thus, it is possible that in the leaves, Cd either exists as a Cd-phytochelatin complex or bound to cell wall structures. Current studies are being performed in order to elucidate the proposed hypothesis.

**HRTEM, Synchrotron, and Simulation Techniques Applied to Activity and Selectivity Correlation in Hydrodesulfurization Catalysts**

M. Perez De la Rosa,<sup>1</sup> G. Berhault,<sup>2</sup> M. J. Yácaman,<sup>3</sup> A. Mehta,<sup>4</sup> S. Fuentes,<sup>5</sup> and R. R. Chianelli<sup>1</sup>

<sup>1</sup>M.R.T.I., University of Texas at El Paso, El Paso, TX 79968, USA

<sup>2</sup>LACCO-CNRS, 86022 Poitiers, Cedex, France

<sup>3</sup>Department of Chemical Engineering, University of Texas at Austin, Austin, TX 78712, USA

<sup>4</sup>Stanford Synchrotron Radiation Laboratory, SLAC, Stanford University, Stanford, CA 94309, USA

<sup>5</sup>Centro de Ciencias de la Materia Condensada, Universidad Nacional Autónoma de México, 22800, Ensenada, B.C., México

The highly anisotropic character and inherent disorder in the structure of supported MoS<sub>2</sub>-based catalysts, used extensively to perform hydrotreating reactions for the removal of heteroatoms (S, N, and O), aromatics, and metals, makes characterization of the active catalyst a difficult challenge. XAS (x-ray adsorption spectroscopy), XRD (X-ray diffraction) and HRTEM (high resolution transmission electron microscopy) have been widely used to study these catalysts in an attempt to understand the structure and origin active phases in these catalysts. However, all these techniques have limitations in determining the structure of the active MoS<sub>2</sub> phase and the associated Co promoter when used individually. Current techniques are not able to provide information of both lateral dimensions along the basal direction and of stacking height of MoS<sub>2</sub> slabs without ambiguity. We report here the use of a synchrotron source for X-ray scattering measurements of supported MoS<sub>2</sub> and cobalt-promoted MoS<sub>2</sub> catalysts that strongly increases the signal to noise ratio resulting in the detection of diffraction features providing information on the dispersion of the active phase in combination with HRTEM giving a more complete picture of catalyst structure and the of the active phases present. Furthermore supported industrial catalysts have been studied that have operated under refinery conditions for more than four years leading to new understanding of the catalytic phase that are stabilized under these conditions. Industrial hydrotreating conditions induce a “destacking” process resulting in the stabilization of single-layered nanoparticles with the MoS<sub>2</sub> structure. This effect has been confirmed on a freshly sulfided model CoMo/Al<sub>2</sub>O<sub>3</sub> catalyst that underwent substantial morphological change leading to the formation of single slabs under HDS conditions as determined by XRD. Other structural effects are also reported. This study emphasizes the importance of determining the catalytically stabilized phases under operating hydrotreating conditions as a basis for understanding the activity and selectivity of this class of catalysts.

## Micro X-ray Beam Spectroscopy and Diffraction

### Examples of the Application of Mirror-based Hard X-ray Microprobe Techniques in the Earth and Environmental Sciences

A. Lanzirotti and S. R. Sutton

Consortium for Advanced Radiation Sources, The University of Chicago, Chicago, IL 60637, USA

The ability of high-energy synchrotrons to produce highly collimated, intense x-ray radiation makes them ideal sources for x-ray microprobe analysis of earth and environmental materials. Such synchrotron based x-ray microprobes allow for trace element quantification (x-ray fluorescence), chemical speciation determination (x-ray absorption spectroscopy), and phase identification (x-ray diffraction). In the design of such instruments Kirkpatrick-Baez mirrors have proved particularly useful for producing x-ray microbeams because of their achromaticity, photon density gains in excess of  $10^4$ , and long working distances (centimeters). In the earth and environmental sciences, instrument development and utilization has been driven by the need for the determination of material compositions, structures, oxidation states, and bonding characteristics with trace element sensitivity and micrometer spatial resolution. A number of examples will be discussed that show flexibility of this technique, but two particular examples will be highlighted. These include the use of x-ray fluorescence analysis and x-ray absorption spectroscopy in evaluating the contaminant history of Ni, U, and other metals within annual rings of willows (*Salix nigra*L.) from a former de facto radiological settling basin (Punshon, *et al.*, 2003). We'll also examine how x-ray microbeam compositional imaging, absorption spectroscopy, and diffraction techniques have been used to quantify the co-existence of As<sup>3+</sup> and As<sup>5+</sup> in oxidized rims of roaster iron oxide grains in mine tailings from gold mining activities in the Yellowknife area (Canada).

### Biological X-ray Absorption Spectroscopy Imaging

I. J. Pickering,<sup>1</sup> G. Hirsch,<sup>2</sup> H. H. Harris,<sup>3</sup> M. J. George,<sup>4</sup> E. Y. Sneed,<sup>4</sup> D. Durkin,<sup>4</sup> R. C. Prince,<sup>5</sup> and G. N. George<sup>1</sup>

<sup>1</sup>Department of Geological Sciences, University of Saskatchewan, Saskatoon, SK, S7N 4R5, Canada

<sup>2</sup>Hirsch Scientific, Pacifica, CA 94044, USA

<sup>3</sup>School of Chemistry, University of Sydney, Sydney, NSW 2006, Australia

<sup>4</sup>Stanford Synchrotron Radiation Laboratory, SLAC, Stanford University, Stanford, CA 94309, USA

<sup>5</sup>ExxonMobil Research and Engineering Company, Annandale, NJ 08801, USA

Micro X-ray beam studies of biological systems such as cell cultures, intact tissues or whole organisms pose special experimental challenges. While such studies can provide unprecedented spatial and chemical information, the specimens are unusually delicate and great care must be taken to ensure the measurement is of a living system. Beam damage is of critical concern. Additionally, the elements of interest are often present at such low concentrations that they are close to the limit of fluorescence detection.

We have developed protocols at SSRL's beam lines 9-3 and 6-2 to ensure minimum exposure of the sample. We use the technique of X-ray absorption spectroscopy imaging, in which fluorescence maps are collected at two or more energies each sensitive to a different chemical species of the element, and from these quantitative concentration maps of each species are deduced. An advantage of this method over microprobe coupled with micro-XAS is that chemical contrast is obtained for the entire scanned area, rather than from selected pixels, and the dwell time at each pixel is very small. The sample is maintained under ambient conditions and additional external checks on its integrity are also applied. Tapered metal monocapillaries provide the microfocusing capabilities. Results of XAS-imaging on arsenic in a hyperaccumulating fern, on sulfur in whole cells, and on mercury in zebrafish larvae will be presented.

**X-ray Spectroscopic and Diffraction Study of the Speciation of Uranium in Contaminated Sediments from the DOE's Hanford Site**

J. G. Catalano,<sup>1</sup> J. M. Zachara,<sup>2</sup> S. M. Heald,<sup>3</sup> and G. E. Brown, Jr.<sup>1,4</sup>

<sup>1</sup>Surface & Aqueous Geochemistry Group, Department of Geological & Environmental Sciences, Stanford University, Stanford, CA 94305-2115, USA

<sup>2</sup>Pacific Northwest National Laboratory, Environmental Dynamics & Simulation, P.O. Box 999, MS K8-96, Richland, WA 99352, USA

<sup>3</sup>PNC-CAT, Advanced Photon Source, Argonne National Laboratory, Argonne, IL 60439, USA

<sup>4</sup>Stanford Synchrotron Radiation Laboratory, SLAC, Stanford University, Stanford, CA 94309, USA

Uranium contamination of the subsurface at numerous locations across the DOE's Hanford site has occurred through the leakage of high-level nuclear waste. At one site of particular concern, the BX Tank Farm, leakage from the overfilling of tank BX-102 released approximately 7.5 metric tons of uranium dissolved in caustic aqueous sludge to the vadose zone. Assessment of the potential hazards posed by this leakage, as well as the development and application of accurate contaminant transport models, requires an understanding of the speciation of uranium in the sediments under tank BX-102. We have applied advanced, synchrotron-based X-ray spectroscopic and diffraction techniques to characterize the distribution, phase associations, and chemical form of uranium in samples from these sediments. X-ray absorption fine structure (XAFS) spectroscopic studies demonstrate that >95% of the uranium in all samples occurs as uranium(VI), and that the primary uranium species is likely a uranium(VI) silicate from the uranophane group of minerals. Unfortunately, XAFS cannot distinguish between the members of this group due to near identical local coordination environments of uranium in these phases. Combined micro-scanning x-ray fluorescence ( $\mu$ SXRF) and electron microprobe analyses show uranium distributed heterogeneously in the sediments, occurring as micron-sized particles inside cracks in feldspar grains. X-ray microdiffraction ( $\mu$ XRD) studies reveal the presence of sodium-boltwoodite,  $\text{Na}(\text{UO}_2)(\text{SiO}_3\text{OH}) \cdot 1.5\text{H}_2\text{O}$ ; no other uranophane group mineral was observed.  $\mu$ XRD studies also confirm the association of uranium with feldspar. Future release of uranium from these sediments will occur through dissolution of sodium-boltwoodite, which has the lowest expected solubility of the uranophane group minerals.

## **Microbial Polymer Templatation of Iron Oxyhydroxides**

C. S. Chan,<sup>1</sup> S. Fakra,<sup>2</sup> G. De Stasio,<sup>3</sup> S. A. Welch,<sup>4</sup> and J. F. Banfield<sup>1</sup>

<sup>1</sup>Department of Earth and Planetary Science, University of California at Berkeley, Berkeley, CA 94720, USA

<sup>2</sup>Advanced Light Source, Lawrence Berkeley National Laboratory, Berkeley, CA 94720, USA

<sup>3</sup>Department of Physics, University of Wisconsin, Madison, WI 53706, USA

<sup>4</sup>CRC-LEME, The Australian National University, Canberra ACT 0200, Australia

Biopolymers have been shown to control the nucleation, orientation, and growth of crystals. Microbial mineralization can have a significant effect on environmental chemistry by producing minerals with phases, morphologies, and reactivities that differ from inorganically precipitated minerals.

We have found evidence of a novel form of polymer-assisted biomineralization: extremely thin (few-unit cell wide), microns-long akaganeite ( $\beta$ -FeOOH) filaments that are templated on natural microbial polymer fibrils. These filaments were identified in iron oxide-encrusted biofilm samples in the flooded Piquette Mine, WI, USA using high resolution transmission electron microscopy. These pseudo-single crystals are composed of assembled elongate nanocrystals oriented end-to-end in the [010] direction. The presence of akaganeite is unusual, since it is generally considered to form in high salinity environments, suggesting that polymer templatation has altered iron oxide phase stability.

In order to verify the presence of the polymer, filaments in biofilm samples were imaged by synchrotron X-ray photoelectron emission microscopy (PEEM; performed at the Synchrotron Radiation Center, University of Wisconsin) and scanning transmission X-ray microscopy (STXM; performed at the Advanced Light Source, Lawrence Berkeley National Laboratory). The carbon 1s X-ray absorption near edge spectra of the filaments confirm the presence of organic polymer, including polysaccharide and possibly protein. To test the hypothesis that polymer fibrils can template akaganeite filament formation, we have been performing experiments to simulate the postulated mineralization pathway. Initial analyses of these samples by STXM has yielded preliminary information about the molecular interactions between polysaccharides and iron.

These experiments show the utility of synchrotron-based X-ray spectromicroscopy in biomineralization studies.

## Spectroscopy and Crystallography of Macromolecules

### A Conformational Mimic of the MgATP Bound "On State" of the Nitrogenase Fe Protein

J. W. Peters,<sup>1</sup> S. Sen,<sup>1</sup> R. Igarashi,<sup>2</sup> A. Smith,<sup>1</sup> M. K. Johnson,<sup>3</sup> and L. C. Seefeldt<sup>2</sup>

<sup>1</sup>Department of Chemistry and Biochemistry, Montana State University, Bozeman, MT 59717, USA

<sup>2</sup>Department of Chemistry and Biochemistry, Utah State University, Logan, UT 84322, USA

<sup>3</sup>Department of Chemistry and Center for Metalloenzyme Studies, University of Georgia, Athens, GA 30602, USA

Biological nitrogen fixation is catalyzed by the complex metal containing enzyme termed nitrogenase. This enzyme is composed of two separable metal-containing enzyme components that interact with each other to catalyze MgATP hydrolysis, intermolecular electron transfer and the reduction of dinitrogen gas to ammonia. The crystal structure of a nitrogenase Fe protein single site deletion variant reveals a distinctly new conformation of the Fe protein and indicates that upon binding of MgATP, the Fe protein undergoes a dramatic conformational change that is largely manifested in the rigid body reorientation of the homodimeric Fe protein subunits with respect to one another. The observed conformational state allows the rationalization of a model of structurally and chemically complementary interactions that occur upon initial complex formation with the MoFe protein component that are distinct from the protein-protein interactions that have been characterized previously for stabilized nitrogenase complexes. The crystallographic results, in combination with complementary UV-visible absorption, EPR and resonance Raman spectroscopic data, indicate that the [4Fe-4S] cluster of both the Fe protein deletion variant and the native Fe protein in the presence of MgATP can reversibly cycle between a regular cubane-type [4Fe-4S] cluster in the reduced state and cleaved form involving two [2Fe-2S] fragments in the oxidized state. Resonance Raman studies indicate that this novel cluster conversion is induced by glycerol and the crystallographic data suggest that glycerol is bound as a bridging bidentate ligand to both [2Fe-2S] clusters fragments in the oxidized state.

### The Structure of an Electron Transfer Complex Consisting of a Water-Soluble and Integral Membrane Protein from a Photosynthetic Bacterium

H. L. Axelrod,<sup>1</sup> E. C. Abresch,<sup>1</sup> M. Y. Okamura,<sup>1</sup> G. Feher,<sup>1</sup> A. P. Yeh,<sup>2</sup> and D. C. Rees<sup>2</sup>

<sup>1</sup>Department of Physics, University of California at San Diego, San Diego, La Jolla, CA 92093-0319, USA

<sup>2</sup>Division of Chemistry and Chemical Engineering, California Institute of Technology, Pasadena, CA 91125, USA

In the photosynthetic bacterium *Rhodobacter sphaeroides*, a water-soluble cytochrome  $c_2$  (cyt  $c_2$ ) is the electron donor to the photosynthetic reaction center (RC), a membrane-bound pigment-protein complex that is the site of the primary light-induced electron transfer. To determine the interactions important for docking and electron transfer within the transiently-bound complex of the two proteins, the RC and cyt  $c_2$  were co-crystallized and the structure determined by x-ray diffraction. Cyt  $c_2$  reduces the photo-oxidized RC donor ( $D^+$ ), a bacteriochlorophyll dimer, in the co-crystals in 0.9 microsecond, which is the same time as measured in solution. This provides strong evidence that the structure of the complex in the region of electron transfer is the same in the crystal and in solution. X-ray diffraction data were collected using synchrotron radiation to a resolution of 2.40 Å, and the coordinates were refined to an R-factor of 22% ( $R_{\text{free}} = 26\%$ ). The intermolecular interactions important for binding and electron transfer will be described. The binding interface can be divided into two domains: (i). A short-range interaction domain that includes groups exhibiting non-polar interactions, hydrogen bonding, and a cation- $\pi$  interaction. This domain is important for electron transfer and contributes to the strength and specificity of cyt  $c_2$  binding. (ii). A long-range, electrostatic interaction domain that contains solvated complementary charges on the RC and cyt  $c_2$ . This domain, in addition to contributing to the binding, may steer the unbound proteins toward the right conformation.

## **Instrumentation for Polarized XAS of Protein and Inorganic Single Crystals at SSRL**

M. J. Latimer and B. Hedman

Stanford Synchrotron Radiation Laboratory, SLAC, Stanford University, Stanford, CA 94309, USA

Polarized XAS of specifically oriented crystals offers a wealth of molecular and electronic structure information unavailable in un-oriented/isotropic systems. By alignment of molecular vectors to the e-vector of the x-ray beam, specific electronic transitions or EXAFS interactions can be emphasized or minimized. The angle-dependence of the polarized XAS signals allow direct electronic-molecular structure correlations and the precision of XAS distance determinations provide more detailed structural information for metallo-protein active sites than can be provided by crystallography alone.

Development of single crystal XAS instrumentation, which allows nearly simultaneous XAS and crystallographic data collection, has been carried out at SSRL BL9-3, the 16-pole 2T wiggler beam line 9 side station dedicated to general user biological XAS. The implementation includes a Huber kappa goniometer, Canberra 30-element Ge detector for XAS data collection, open-flow He and N<sub>2</sub> coolers, microscope for crystal alignment in the beam, and a MAR CCD or MAR 345 crystallography detector. The kappa goniometer allows a large accessible angle range with an open geometry affording access to detectors and open stream coolers. Applicable standard hardware on SSRL crystallography beam lines has been incorporated with crystallographic data collection controlled via the Blu-Ice software developed by the SSRL SMB macromolecular crystallography group. XAS data collection is handled through the SSRL standard XAS-Collect software. Initial diffraction and XAS data from single crystals using an open flow cryostat will be presented. The instrument will be available to general users after the SPEAR3 upgrade in early 2004.

## **X-ray Spectroscopy of Photosystem II Single Crystals**

J. Yano,<sup>1,2</sup> K. Sauer,<sup>1,2</sup> V. Yachandra,<sup>1</sup> J. Messinger,<sup>3</sup> J. Kern,<sup>4</sup> and A. Zouni<sup>4</sup>

<sup>1</sup>Physical Biosciences, Lawrence Berkeley National Laboratory, Berkeley, CA 94720, USA

<sup>2</sup>Department of Chemistry, University of California at Berkeley, Berkeley, CA 94720, USA

<sup>3</sup>Max-Planck-Institut für Strahlenchemie, D-45470 Mülheim an den Ruhr, Germany

<sup>4</sup>Max-Volmer Laboratorium der TU-Berlin, D-10623, Berlin, Germany

Photosystem II (PS II) is a membrane-bound protein complex found in green plants and cyanobacteria. During a light-driven process by PS II, single electron photooxidations of chlorophyll are coupled to the four- electron oxidation of water to evolve dioxygen. The catalytic site of this reaction is a protein-bound tetranuclear Mn complex. The crystal structure of PSII was recently reported by Zouni *et al.* (Nature 409, 739, 2001) and then by Shen *et al.* (PNAS 100, 98, 2003) with resolution of 3.6 Å. Our primary goal is to utilize X-ray spectroscopy of single crystals of PS II in conjunction with the low-resolution electron density of the Mn complex obtained from X-ray diffraction studies to derive the structure of the Mn complex at high resolution. We have obtained the Mn K-edge and EXAFS spectra of the single crystals of PS II with the e-vector of the X-rays oriented parallel to the three principal a, b and c axes of the crystal. The diffraction pattern for each crystal was collected *in situ* to determine the orientation of the crystal with respect to the X-ray electric-field vector. The X-ray spectra and diffraction data are collected at 10 K to minimize radiation damage. The dipole forbidden 1s-3d transition and the allowed main K- edge show distinct dichroism. The EXAFS spectra also show distinct dichroism in the Fourier peaks assigned to Mn-O/N vectors at ~1.8 - 2.0 Å and the Mn-Mn/Ca vectors at 2.7 and 3.3 Å. The S/N ratio of the spectra was improved by averaging data from many crystals.

## **Low-resolution Phasing by Se-MAD in the Presence of Radiation Damage**

C. Bakolitsa,<sup>1</sup> F. von Delft,<sup>2</sup> A. Gonzalez,<sup>3</sup> and R. C. Liddington<sup>1</sup>

<sup>1</sup>The Burnham Institute, La Jolla, CA 92037, USA

<sup>2</sup>The Scripps Research Institute, La Jolla, CA 92037, USA

<sup>3</sup>Stanford Synchrotron Radiation Laboratory, SLAC, Stanford University, Stanford, CA 94309, USA

With increasingly powerful synchrotron sources, radiation damage is emerging as a limiting factor in determining the success or failure of a Se-MAD experiment. Here, we present the phasing by two-wavelength Se-MAD of weakly diffracting protein crystals affected by significant radiation damage as indicated by a 0.5-0.7% increase in cell dimensions between initial and final frames collected. Data were collected at SSRL 9-1 and made use of some of SSRL's unique features. Internal non-isomorphism rendered global scaling of data invalid, and both Patterson and dual-space direct methods failed to locate the anomalous scatterers; these were eventually located by combining local scaling and direct methods.

## Small Angle X-ray Scattering

### ***In Situ* SAXS/WAXS Studies of the Precipitation, Crystallization and High Temperature Phase Transitions of Minerals**

S. Shaw,<sup>1</sup> L. G. Benning,<sup>2</sup> N. J. Terrill,<sup>3</sup> C. M. B. Henderson,<sup>4</sup> and J. A. Warner<sup>5</sup>

<sup>1</sup>Department of Earth Sciences, University of Oxford, Oxford, UK OX1 3PR

<sup>2</sup>Department of Earth Sciences, University of Leeds, Leeds, UK LS2 9JT

<sup>3</sup>Diamond Light Source Ltd, Rutherford Appleton Laboratory, UK OX11 0QX

<sup>4</sup>Department of Earth Sciences, University of Manchester, Manchester, UK M13 9PL

<sup>5</sup>Lawrence Berkeley National Laboratory, Berkeley, CA 94720, USA

Specialized sample cells used in conjunction with a high-flux synchrotron X-ray source allow the *in situ* characterization of mineralogical reactions using time-resolved Small Angle X-ray Scattering (SAXS). Cells have been developed to allow minerals to be analysed under a wide variety of chemical (pH, ionic strength) and physical (temperature) conditions with data collection on timescales down to 100's milliseconds. Three studies will be presented to show the range and diversity of the reaction processes that can be studied using time-resolved SAXS:

(1) High temperature (up to 1100°C) studies of the breakdown and recrystallization of cement minerals/phases e.g. tobermorite ( $\text{Ca}_5\text{Si}_6\text{O}_{16}(\text{OH})_2 \cdot 4\text{H}_2\text{O}$ ), hillebrandite ( $\text{Ca}_2\text{Si}_6\text{O}_3(\text{OH})_2$ ) and thaumasite ( $\text{Ca}_3\text{Si}(\text{CO}_3)(\text{SO}_4)(\text{OH})_6 \cdot 12\text{H}_2\text{O}$ ), have provided information on the mechanisms and kinetics of their dehydration processes and phases transitions which occur at high temperatures.

(2) The growth and crystallisation of environmentally important nanoparticles in aqueous solution (e.g. iron/aluminium oxyhydroxides) to characterise the crystallization of poorly-ordered particles to stable crystalline phases. Particle size and density information obtained from SAXS combined with crystallographic information from Wide Angle X-ray Scattering (WAXS) provide a detailed description of kinetics and mechanism of these reactions.

(3) Stopped-flow mixing apparatus used in conjunction with a high flux undulator insertion device allow high-speed, accurate and reproducible mixing of reactants along with high speed SAXS data collection (<1 sec). This has been used to characterise the nucleation and growth of nanoparticles from solution which occurs during base and oxidative hydrolysis reactions.

### **Counterion Behavior on the Surfaces of Condensed Biopolymers**

T. E. Angelini,<sup>1</sup> J. Butler,<sup>2</sup> H. Liang,<sup>2</sup> and G. C. L. Wong<sup>2</sup>

<sup>1</sup>Department of Physics, University of Illinois, Urbana, IL 61801, USA

<sup>2</sup>Department of Materials Science and Engineering, Urbana, IL 61801, USA

Electrostatics in aqueous media is commonly understood in terms of screened Coulomb interactions, where like-charged objects, such as polyelectrolytes, always repel. These intuitive expectations are based on mean field theories, such as the Poisson-Boltzmann formalism, which are routinely employed in colloid science and computational biology. Like-charge attractions, however, have been experimentally observed in a wide variety of systems. Intense theoretical scrutiny over the last 30 years has suggested that counterions play a central role, but no consensus exists for the precise mechanism. We have examined the organization of multivalent ions on actin filaments using synchrotron x-ray diffraction, and discovered a new collective mechanism in which the counterions self-organize into one-dimensional charge density waves parallel to the actin filaments and couple to their torsional distortions.

## **Probing the Counterion Atmosphere around DNA by Anomalous Small-Angle X-ray Scattering**

R. Das

Department of Physics, Stanford University, Stanford, CA 94305, USA

Counterion atmospheres condensed onto charged biopolymers strongly affect their physical properties and biological functions, but have been difficult to quantify experimentally. Here, monovalent and divalent counterion atmospheres around DNA double helices in solution are probed using small-angle x-ray scattering (SAXS) techniques. Modulation of the ion scattering factors by anomalous (resonant) x-ray scattering and by interchanging ion identities yields direct measurements of the scattering signal due to the spatial correlation of surrounding ions to the DNA. The quality of the data permit, for the first time, quantitative tests of extended counterion distributions calculated from atomic-scale models of biologically relevant molecules.

## **Closing the Folding Chamber of the Eukaryotic Chaperonin Requires the Transition State of ATP Hydrolysis**

A. S. Meyer,<sup>1</sup> J. R. Gillespie,<sup>1</sup> D. Walther,<sup>4</sup> I. S. Millett,<sup>2</sup> S. Doniach,<sup>3</sup> and J. Frydman<sup>1</sup>

<sup>1</sup>Department of Biological Sciences, Stanford University, Stanford, CA 94305, USA

<sup>2</sup>Department of Chemistry, Stanford University, Stanford, CA 94305, USA

<sup>3</sup>Department of Applied Physics, Stanford University, Stanford, CA 94305, USA

<sup>4</sup>Incyte Genomics, Inc., 3160 Porter Dr., Palo Alto, CA 94304, USA

The ATP-dependent chaperonin TRiC (TCP-1 ring complex) is proposed to mediate protein folding by opening and closing a "built-in" lid over its central cavity. Here we combine structural and biochemical analyses to elucidate the mechanism and function of lid closure. Nucleotide-free TRiC adopts an open conformation that binds unfolded substrates in its central cavity. We find that incubation with ATP indeed promotes lid formation, yielding a closed central cavity. Formation of this closed chamber is essential for substrate folding, as blocking lid closure does not impair ATP hydrolysis or substrate-binding but abolishes productive folding of the bound polypeptide. To define how the ATPase cycle drives lid closure, we employed nucleotide analogues that stabilize distinct pre- and post-hydrolysis states. ATP-analogues that mimic the pre-hydrolysis state do not generate the closed cavity and leave the bound substrate in an unstructured conformation. In contrast, lid closure is induced by a mimic of the trigonal-bipyramidal transition state of the hydrolysis reaction, thus confining the substrate in the central chamber. We conclude that closure of the "built-in" lid is triggered by the transition state of ATP hydrolysis and is essential for productive folding of substrate proteins.

## Surface Spectroscopy

### Investigations of the Structural Properties of Aqueous Geochemical Interfaces by the Use of X-ray Diffraction, Standing Waves and Spectroscopy

P. J. Eng,<sup>1,2</sup> T. P. Trainor,<sup>1</sup> G. A. Waychunas,<sup>3</sup> J. C. Catalano,<sup>4</sup> M. Newville,<sup>1</sup> and G. E. Brown, Jr.<sup>4</sup>

<sup>1</sup>Consortium for Advanced Radiation Sources, University of Chicago, Chicago, IL 60637, USA

<sup>2</sup>The James Franck Institute, University of Chicago, Chicago, IL 60637, USA

<sup>3</sup>Earth Sciences Division, Lawrence Berkeley National Laboratory, Berkeley, CA 94720, USA

<sup>4</sup>Department of Geological & Environmental Sciences, Stanford University, Stanford, CA 94305, USA

The interaction of liquid water with the surfaces of natural solids is one of the most fundamental chemical reactions occurring in nature. Its importance extends from geochemical processes such as mineral dissolution and sorption/desorption reactions -- which play major roles in the growth of crystals, chemical weathering, contamination of ground waters, environmental restoration, and the biogeochemical cycling of elements -- to the reaction of water with sulfate aerosol particles in the troposphere. I will present the results of our measurements of the structure of metal oxide surfaces under ambient or in-situ conditions (i.e., in the presence of bulk solution at ambient temperature). Utilizing x-rays from the Advanced Photon Source at GeoSoilEnviroCARS sector 13 we employ surface and interface sensitive x-ray methods in our studies, such as grazing incidence XAFS, crystal-truncation-rod x-ray scattering and x-ray standing wave. These probes are unrivaled in their ability to provide us with physical and chemical information about surfaces under natural conditions, and especially in the presence of bulk water.

### Investigations into the Surface Structure and Chemistry of ZnS Nanoparticles

B. Gilbert, F. Huang, H. Zhang, and J. F. Banfield

Department of Earth and Planetary Science, University of California at Berkeley, Berkeley, CA 94720, USA

The structure of nanoparticles, and surface structure in particular, can respond dynamically to surface environment changes at room temperature. Uncoated 3 nm ZnS nanoparticles undergo reversible structural change associated with methanol desorption and rewetting. Water addition causes a profound, non-reversible rearrangement to a more crystalline structure. Unexpected differences (relative to bulk ZnS) in the structural dynamics of ZnS nanoparticles capped with an organic ligand can be linked to the observation of surface reconstruction. Hence, solvent or ligand interactions can alter the structure, and potentially the reactivity of small nanoparticles at the interface with their environment. We combine vibrational and electronic spectroscopies with molecular dynamics modelling to investigate the structure of the nanoparticle surface, and nature of surface chemical interactions. We seek to determine how surface molecules, e.g. water, can drive substantial atomic reorganization, and to identify the reactive surface sites of the nanoparticles.

## Rapid and Heterogeneous Oxidation of Pyrite Surfaces by *Thiobacillus ferrooxidans* and *T. thiooxidans*

B. C. Bostick,<sup>1</sup> K. Lester,<sup>2</sup> C. Doyle,<sup>2</sup> T. Kendelewicz,<sup>2</sup> G. E. Brown, Jr.,<sup>2</sup> S. Fendorf,<sup>2</sup> and A. M. Spormann<sup>2</sup>

<sup>1</sup>Department of Earth Sciences, Dartmouth College, Hanover, NH 03755, USA

<sup>2</sup>Department of Geological & Environmental Sciences, Stanford University, Stanford, CA 94305, USA

Pyrite oxidation is the critical step in the formation of acid mine drainage, which acidifies the environment, and releases associated toxic metals. Certain microbes accelerate pyrite oxidation, and, as such, a more detailed understanding of their role in the production of acid mine drainage is needed. This study is one of the first to use x-ray photoelectron spectroscopy to probe bacterial pyrite oxidation, providing insights into the fundamental processes by which oxidation occurs. Freshly fractured pyrite surfaces were oxidized by *Thiobacillus ferrooxidans* over a range of solution pH and were compared with chemical oxidation of similar surfaces under the same conditions. Microbial oxidation resulted in the formation of distinct oxidized surface species distributed non-uniformly over the pyrite surface. In contrast, chemical oxidation proceeds through a similar mechanism but without the build-up of passivating oxidation products. Both mechanisms occurred at isolated regions of the surface that were not correlated with step edges or other topographical features. These results demonstrate that microbial oxidation occurs through a mechanism similar to chemical oxidation, but that the increased rate of biological oxidation results the formation of distinct products on the pyrite surface. This build-up of surface oxidation products may impact the long-term reactivity of pyrite surfaces.

## The Structure of Ni-based Refractory Metal Glasses

M. L. Tokarz and J. C. Bilello

Center for Nanomaterials Science, Department of Materials Science, University of Michigan, Ann Arbor, MI 48109-2136, USA

A series of Ni-based alloys of varying Nb/Sn concentrations was studied using the high resolution synchrotron powder diffraction beamline 2-1 and these results were correlated with a number of collary experiments using beamline 2-2, DSC, SEM and laboratory source x-rays. These alloys exhibited a glass transition temperature of  $\approx 600^\circ\text{C}$  for the entire composition range, where Ni concentration was nominally 60% and the Sn concentration ranged over 3, 5, 6.2 to 6.9%, with Nb the remainder. Specimens from all four concentrations appeared to be bulk glasses when studied with a standard lab x-ray source, but data taken on 2-1 indicated that there was a small, but persistent, nanocrystalline phase present for all samples with the exception of  $\text{Ni}_{60}\text{Nb}_{35}\text{Sn}_5$ . This nanocrystalline residue was subsequently confirmed by Laue transmission diffraction experiments on beamline 2-2, which provided global information, and by the more limited field of view provided high resolution SEM. Analysis of the Radial Distribution Functions, particularly at 16KeV, which allowed access to a wider range of k-space, indicated that these materials show definite structural order out to 4 (possibly 5) nearest neighbors, and these nearest neighbor distances do not agree with those predicted from a random packing model. This result is very different from what one would normally associate with a traditional glass forming material.

Appreciation is expressed to Professor W. L. Johnson and Dr. C-Y. Haein for preparing the alloy samples and to DoE and DARPA for support.

## **SSRL Reports**

### **Beam Line Development**

T. Rabedeau

### **SPEAR3 Status Report**

R. Hettel

Stanford Synchrotron Radiation Laboratory, SLAC, Stanford University, Stanford, CA 94309, USA

SSRL will enter a new era of synchrotron radiation experimentation with the completion of the SPEAR 3 upgrade project this Fall. The 3<sup>rd</sup> generation SPEAR 3 storage ring will produce beams having one to two orders of magnitude higher brightness and flux density than the old SPEAR 2 ring, accommodate several new high performance insertion devices and beam lines, and be capable of top-off operation by virtue of its improved at-energy injection system. The project completely replaces the SPEAR vacuum chamber, magnets, support rafts, RF, power supplies, cable plant and shielding tunnel floor in a 7-month shutdown period that began March 31 of this year. The status of the 4-year, 58 M\$ upgrade project will be reviewed.

### **SPEAR3 Commissioning Plans**

J. Safranek

Stanford Synchrotron Radiation Laboratory, SLAC, Stanford University, Stanford, CA 94309, USA

The SPEAR3 commissioning schedule and measurement plans will be presented. Achieving the design performance of SPEAR3 will require precise control of the electron beam. Diagnostics, measurement techniques and analysis that will be used to characterize and control the beam stability and optics will be discussed.

### **LCLS Update**

J. Galayda

### **SPPS Update**

J. Hastings

Stanford Synchrotron Radiation Laboratory  
30<sup>th</sup> Annual Users' Meeting



## ***WORKSHOP AGENDAS***



## Crystallography Beam Line Automation: Work Smarter Not Harder

---

Wednesday, October 8, 2003  
 SLAC Research Office Bldg. 48, Redwood Rm.  
 Organizer: Aina Cohen, SSRL

New technologies for high-throughput crystallography at synchrotrons will be described focusing on the benefits and concerns of general users. Speakers on each topic will be given a short list of issues to address during their presentations. These questions are based on user concerns such as system efficiency, information transport, remote accessibility, cross compatibility, and data retrieval. Following the speakers on each topic, there will be an open discussion about these issues. This feedback is extremely valuable for planning developments that best match the needs of our user community.

### Agenda

8:00–8:30 am	<i>Registration / Coffee and Muffins</i>
8:30–8:40 am	Welcome – Michael Soltis, SSRL Introduction – Aina Cohen, Timothy McPhillips, Frank Topper
8:40–10:10 am	Robotic Sample Mounting Systems 8:40 Thomas Earnest, ALS 9:05 Steve Ginell, APS SBC-CAT 9:30 Aina Cohen, SSRL 9:55 Discussion
10:10–10:30 am	<i>Break</i>
10:30–11:15 am	Automated Crystal Centering 10:30 James Holton, ALS 10:45 Timothy McPhillips, SSRL 11:00 Discussion
11:15 am–12:15 am	Data Backup 11:20 Ken Sharp, SSRL 11:40 James Holton, ALS 12:00 Discussion
12:15–1:00 pm	<i>Lunch</i>
1:00–2:10 pm	Crystal Screening Interfaces 1:00 Ana Gonzalez, SSRL 1:25 Nicholas Sauter, ALS 1:50 Discussion
2:10–2:20 pm	<i>Walk to Building 120</i>
2:20–4:30 pm	SSRL Hands-on Session (BL9-2) / Tour and Demonstration (BL11-1)  <i>A workshop and Users' Meeting pre-registration reception will be held in the SLAC Panofsky Auditorium Breezeway from 5:00–7:00 pm</i>

**Inelastic X-ray Scattering and Advanced Spectroscopy Facility for SPEAR3**

---

Wednesday, October 8, 2003  
SLAC Central Labs Bldg. 40, Orange Rm. 84  
Organizers: Uwe Bergmann and Anders Nilsson, SSRL

We are planning to build a dedicated facility for sub-eV inelastic x-ray scattering in the 5–15 keV range at SPEAR3. The techniques will include non-resonant and resonant X-ray Raman scattering, selective X-ray absorption and X-ray emission spectroscopy. In this workshop we will discuss scientific problems that can be addressed at this facility and the resulting beam line and x-ray optics parameters. The topics will include the study of low Z systems under ambient and extreme conditions and the study of 3d transition metal compounds.

**Agenda**

8:00–8:30 am	<i>Registration and Breakfast</i>
8:30–9:20 am	Welcome and Introduction Uwe Bergmann, SSRL
9:20–10:00 am	Applications of RIXS in Catalysis and Magnetism Frank Groot, Utrecht University
10:00–10:40 am	<i>Break</i>
10:40–11:20 am	Unsolved Problems in Biology – What can Advanced X-ray Spectroscopy Contribute James Penner-Hahn, University of Michigan
11:20 am–12:00 pm	New X-ray Techniques to Study the Mn Complex involved in Photosynthetic Water Oxidation Vittal Yachandra, LBNL
12:00–1:30 pm	<i>Lunch</i>
1:30–2:10 pm	New Perspective on Structure and Bonding in Water Using XAS and XRS Anders Nilsson, SSRL/Stockholm
2:10–2:50 pm	Needs for Advanced X-ray Microprobes to Study Crystal and Electronic Structures of Solids at High Pressures Choong-Shik Yoo, LLNL
2:50–3:20 pm	<i>Break</i>
3:20–4:00 pm	Petroleomics: from Chemical Composition to Commercial Reality – How XRS will Help Us Oliver Mullins, Schlumberger-Doll
4:00–4:40 pm	Probing Low Atomic Number Chemical Dynamics at the LCLS with Inelastic Scattering Kelly Gaffney, SSRL
4:40–6:00 pm	Workshop Summary All Participants
	<i>A workshop and Users' Meeting pre-registration reception will be held in the SLAC Panofsky Auditorium Breezeway from 5:00–7:00 pm</i>

## Synchrotron Techniques for Environmental Microbiology and Biogeochemistry

---

Wednesday, October 8, 2003

SLAC Panofsky Auditorium

Organizers: John Bargar, SSRL, Scott Fendorf, Stanford University and David Shuh, ALS

Environmental microbiology and geomicrobiology have emerged as vibrant scientific fields with cross-cutting research themes emphasizing the interplay and energy flow between microbial communities, inorganic and organic contaminants, (bio)minerals, groundwater, and other solutions. Synchrotron (SR)-based techniques are beginning to play important roles in these research areas because of the utility of SR methods for characterizing metal ion and organic molecule speciation under in-situ conditions in complex environmental materials. The purpose of this meeting is to bring together scientists from the environmental microbiology, biogeochemistry, and synchrotron communities to share ideas. Introductory talks regarding the core techniques will be mixed with scientific talks (not limited to SR-based techniques) to foster interactions between attendees. Topics to be discussed include mechanisms of metal binding by bacteria, microbially mediated redox cycling of metals in the environment and their application to bioremediation, biomineral structures, compositions, and formation mechanisms, and competitive sequestration of metals by inorganic sinks (oxide, sulfide surfaces, solids) and bacteria.

(Co-sponsored by SSRL, ALS and the DOE (Natural and Accelerated Bioremediation Research (NABIR) Program).

### Agenda

7:30–8:00 am	<i>Registration / Coffee and Muffins</i>
8:00–8:10 am	Welcome and Introductory Comments
8:10–8:50 am	Application of Absorption Fine Structure (XAFS) Spectroscopy in Biogeochemistry John Bargar, SSRL
8:50–9:30 am	Introduction to Micro-beam X-ray Fluorescence Imaging (SXRF) and XAFS Spectroscopy Matthew A. Marcus, ALS
9:30–10:10 am	Introduction to SR-based X-ray Scattering Techniques Samuel M. Webb, SSRL
10:10–10:25 am	<i>Break</i>
10:25–11:05 am	Introduction to SR-based Microdiffraction Techniques Nobumichi Tamura, ALS
11:05–11:50 am	Topic: Microbial Influences on Metals and Minerals Ken Neelson, USC
11:50 am–12:50 pm	<i>Lunch</i>
12:50–1:35 pm	Topic: Biogeochemical Processes in Surface and Subsurface Environments John Zachara, PNNL
1:35–2:20 pm	Topic: Submicron Scanning Transmission X-ray Microscopy (STXM) and Chemical Imaging of Humic Functional Groups in Humic and Biological Materials Satish Myneni, Princeton University
2:20–3:05 pm	The Role of Controlled Cultivation in Systems Biology and Biogeochemical Research Yuri Gorby, PNNL
3:05–3:15 pm	<i>Break</i>
3:15–4:00 pm	Mechanisms of Uranyl Sorption to Bacterial Surfaces of <i>Pseudomonas fluorescens</i> Rizlan Bencheikh-Latamani, Scripps Institute of Oceanography

## *workshop agendas*

4:00–4:45 pm	Mineral Biotransformations and Their Impacts on Contaminant Attenuation Scott Fendorf, Stanford University
4:45–5:30 pm	Molecular Microbial-Mineral Interactions Alfred Spormann, Stanford University
5:30 pm	Discussion

*A workshop and Users' Meeting pre-registration reception will be held in the SLAC Panofsky Auditorium Breezeway from 5:00–7:00 pm*

## Probing Mechanical Deformation and Failure via Synchrotron X-rays

---

Wednesday, October 8, 2003  
 SSRL Building 137, 2<sup>nd</sup> Floor Conference Rm. 226  
 Organizers: Apurva Mehta, SSRL and Nobumichi Tamura, ALS

The purpose of this workshop is to investigate mechanical properties of materials via synchrotrons. Salient issues in mechanical deformation and failures, such as crystal plasticity models, fatigue, crack propagation, etc., will be discussed. A brief survey of relevant synchrotron methods - parallel beam geometry for mesodiffraction, microdiffraction, and phase contrast imaging, and talks on actual experiments done at synchrotrons will also be included. At the end of the day, we plan to brainstorm future projects and developments.

### Agenda

#### Session 1

8:45–9:00 am Introduction  
 Apurva Mehta, SSRL

#### Session 2

(chair — William Nix)  
 9:00–9:45 am Introduction to Microdiffraction  
 Nobumichi Tamura, ALS

9:45–10:00 am *Break*

10:00–10:45 am Nanoindentation and Nanomechanics  
 Erica Lilleoden, LBNL

10:45–11:30 am An Introduction to Strain Gradient Plasticity: Theory and Experiment  
 James Stolken, LLNL

11:30–12:00 pm Microdiffraction Analysis of Strain Gradient Plasticity Theories  
 Monica Barney, LLNL

12:00–1:00 pm *Lunch*

#### Session 3

(chair — Brad Boyce)  
 1:00–1:45 pm Length Scale Effects on Mechanical Properties of Crystalline Materials  
 William Nix, Stanford University

1:45–2:30 pm Modeling Microstructural Deformation and Failure Processes at a Crack Tip  
 Don Shockey, SRI

2:30–3:15 pm Visualization of Cracks and Crack Propagation  
 Zofia Rek, SSRL

3:15–3:30 pm *Break*

#### Session 4

(chair — Nobumichi Tamura)  
 3:30–4:15 pm Spatially Resolved Residual Stress Measurements for Insight into Turbine Engine Fatigue Failures  
 Brad Boyce, Sandia National Laboratories

4:15–5:00 pm X-ray Microdiffraction on Diamond Shaped NiTi for Biomedical Applications  
 Valentina Imbeni, SRI

5:00 pm Discussion of Future Experiments

*A workshop and Users' Meeting pre-registration reception will be held in the SLAC Panofsky Auditorium Breezeway from 5:00–7:00 pm*

**Recent Advances in Synchrotron-Based Microscopy**

---

Tuesday, October 7 – Wednesday, October, 8, 2003

ALS Building 6 Rm. 2202

Organizers: Greg Denbeaux, University of Albany, Katharina Lüning, SSRL, Gary Mitchell, Dow Chemical, Piero Pianetta, SSRL and David Shuh, ALS

In recent years there have been many advances in scanning and full field x-ray imaging techniques such as improved spatial resolution, more stable instruments for spectromicroscopy and time resolved microscopy, phase contrast imaging techniques as well as high resolution tomographic capabilities in absorption and phase contrast. These techniques enable novel nanoscale research in various fields of materials science, environmental science and biology. This workshop, hosted jointly by the ALS and SSRL, is to discuss these new capabilities and to identify the scientific direction for soft- and hard x-ray microscopy at both light sources. Time will be given for open discussion with feedback on possible future directions from workshop attendees.

**Agenda****Tuesday, October 7, 2003**

1:25 pm	Opening Comments
	<b>Spectro-Microscopic Data Interpretation / New Instrumental Capabilities</b>
1:30–2:00 pm	Methods and Examples of Quantitative Chemical Mapping by Soft X-ray Spectromicroscopy Adam Hitchcock, McMaster University
2:00–2:30 pm	Cluster Analysis for Soft X-ray Spectromicroscopy Chris Jacobson, SUNY-Stony Brook
2:30–3:00 pm	Interferometrically Controlled Microscopes at 5.3.2 and 7-11 David Kilcoyne, ALS
3:00–3:30 pm	New Instrumental Capabilities at MES Beamline 11 Tyliszczak Tolek, ALS
3:30–3:45 pm	<i>Break</i>
	<b>Applications to Environmental Science</b>
3:45–4:10 pm	Comparative Scanning Transmission X-ray and Laser Scanning Microscopy of Microbial Biofilms John Lawrence, NHRC
4:10–4:35 pm	Applications of Scanning Transmission X-ray Spectromicroscopy (STXM) for the Characterization of Transition Element Precipitates in Environmental Science Klaus Pecher, PNNL
4:35–5:00 pm	TBD Alain Manceau, CNRS
5:00 pm	<i>Adjourn</i>

**Wednesday, October 8, 2003**

	<b>Applications to Soft Matter</b>
9:00–9:25 am	Spectroscopy of Organic Nanocrystals Using the ALS-STXMs Rainer Fink, University of Erlangen-Nurnberg
9:25–9:50 am	Linear Dichroism and Imaging of Polycrystalline Alkanes Stephen Urquhart, University of Saskatchewan

9:50–10:15 am	Probing Degradation Chemistry in the Cellular Membrane of Vascular Plants: Biogeochemical Applications of STXM George Cody, Geophysical Laboratory, Carnegie Institute of Washington
10:15–10:30 am	<i>Break</i>
10:30–10:55 am	Growth of Short Chain Alkanes and Polyethylene in Crystalline and Semi-crystalline Thin Films Ying Zou, NCSU
10:55–11:20 am	Quantitative Chemical Mapping of Polymer Reinforcement of Alginate Microcapsules by Soft X-ray Spectromicroscopy Torhu Araki, McMaster University
11:20–11:45 am	New Optical Designs for High Spatial and Spectral Resolution X-ray Microscope Chang Chang, Drexel University
11:45–12:10 pm	X-ray Tomography of Cells Carolyn Larabell, LBNL/University of California at San Francisco
12:10–1:30 pm	<i>Lunch</i>
	<b>Applications to Magnetic Materials and Hard X-ray Microscopy</b>
1:30–1:55 pm	High Resolution Magnetic Imaging with Full-field Soft X-ray Microscopy Peter Fischer, Max-Planck Institut
1:55–2:20 pm	Time and Spatially Resolved Microscopy on Magnetization Dynamics Using X-PEEM Sug-Bong Choe, ALS
2:20–2:45 pm	A Hard X-ray Imaging Facility at SPEAR3 Katharina Lüning, SSRL
2:45–3:00 pm	<i>Break</i>
3:00–3:25 pm	Imaging Biological Specimens with Hard X-rays: Advantages and Challenges Wenbing Yun, Xradia, Incorporated
3:25–3:50 pm	X-ray Fluorescence Microscopy and Microspectroscopy for Biological Applications Barry Lai, APS
3:50–4:20 pm	Zernike-type Phase Contrast X-ray Microscopy at 4 keV Photon Energy with 60 nm Spatial Resolution Ulrich Neuhaeusler, ESRF
4:20–4:45 pm	X-ray Microscopy – Filling the Gap between Light and Electron Microscopy in the Biological Sciences Eduardo Almeida, NASA
4:45 pm	<i>Adjourn</i>



Stanford Synchrotron Radiation Laboratory  
30<sup>th</sup> Annual Users' Meeting

---



***POSTER SESSION***

---



## Poster Index

Presenter	Poster No.	Title	Pg. No.
* Andrews, Scott D.	41	STXM Characterization of Spin Injection Samples	50
* Aubertine, Daniel B.	11	X-ray Scattering as a Probe of Interdiffusion in Si/SiGe Heterostructures	34
* Campbell, Mary L.	32	Comparison of $\Delta nifB$ and $\Delta nifH$ MoFe Proteins by X-ray Absorption Spectroscopy with Implications for P-cluster Biosynthesis	44
* Castro, Javier S.	21	Symmetry Break-downs in Aurivillius Phases	39
* Chui, Chi On	19	Synchrotron Radiation Photoemission Spectroscopy Analysis of High-k Gate Dielectric for Ge MOS Applications	38
* Cuk, Tanja	26	A Unified Understanding of Bosonic Renormalization Effects in High-Tc Superconductors	41
* Davies, Jason	33	Conformational Changes during Nucleotide Hydrolysis Cycle of P97 Probed by Small-Angle X-ray Scattering	45
Doukov, Tzanko I.	18	The CODH/ACS Family: Xenon in and the End of the Tunnel	37
* Downward, Lisa M.	31	Evidence for a Universal Relationship Between Magnetization and Changes in the Local Structure of La Manganites	44
* Doyle, Colin S.	35	Soft X-ray Spectroscopic Studies of the Reaction of Fractured Pyrite Surfaces with Cr(VI)-containing Aqueous Solutions	46
Frank, Patrick	4	Perophora Surprise: A New Structural Motif for Biological Iron	30
* Ginder-Vogel, Matthew	7	Biostimulation of Uranium Reduction in Sediment from the Oak Ridge Source Zone	32
* Jackson Rudd, Deanne	23	Investigating the Fe-Fe Scattering from the Binuclear Fe Site in Methane Monooxygenase Hydroxylase by X-ray Absorption Spectroscopy	40
Kavanagh, Kathryn L.	13	Mechanistic Insights from Substrate-bound Structures of <i>Pseudomonas fluorescens</i> Mannitol 2-dehydrogenase	35
Kendelewicz, Tom	34	Oxidation of Fractured Surfaces of FeS <sub>2</sub> (100) by Molecular Oxygen, Water Vapor, and Air	45
Kim, Chu-Young	36	From Structure to Pathogenesis: HLA-DQ2 and Celiac Disease	46
Kim, Hanseong	1	2.6 Å Resolution Crystal Structure of <i>E. coli</i> CTP Synthetase by Hg-MAD, Solvent-flattening and Map Averaging	29
Kwon, Ae-Ran	28	Structure and Reactivity of an Asymmetric Complex between HslV and I-domain Deleted HslU, a Prokaryotic Homolog of the Eukaryotic Proteasome	42
* Lacheen, Howard S.	14	<i>In Situ</i> Mo K-edge XAS Studies of Mo-ZSM-5 in CO <sub>2</sub> /CH <sub>4</sub> Mixtures	35
* Larochelle, Simon	30	X-ray diffraction on BaBiO <sub>3</sub>	43
* Lee, Dong-Ick T.	10	Synchrotron Radiation Photoemission Spectroscopy Study of Cs/O Co-adsorbed InP(100) Surface for Application in Negative Electron Affinity Devices	33

**poster session**

Presenter	Poster No.	Title	Pg No.
* Mang, Patrick K.	27	Spurious Magnetism in a High-Temperature Superconductor	42
Mathews, Irimpan I.	38	Structural Studies Reveal Thymidylate Synthase Complementing Protein as a Target for Multiple Diseases	48
* Meyer, Anne S.	2	Closing the Folding Chamber of the Eukaryotic Chaperonin Requires the Transition State of ATP Hydrolysis	29
* Murray, David S.	16	Structure of Glutamine Synthetase from <i>Bacillus subtilis</i> : Possible Insight into Regulation of Nitrogen Metabolism	36
* Neiss, Jim	24	Speciation Controls on the Reduction and Transport of Uranium	40
* Norman, Thaddeus J.	9	Local Structure of Dopant Ions [Mn(II), Cu(II), and Ag(I)] in ZnSe Nanoparticles	33
Piestrup, Melvin A	40	Inexpensive Compound Refractive Lenses for X-ray Imaging and Microspot Focusing	49
* Polizzotto, Matthew L.	3	Arsenic Speciation and Cycling within the Sediments of Lake Coeur d'Alene, Idaho	30
Ralston, Corie Y.	20	Performance and Characteristics of the Howard Hughes Medical Institute Beamlines at the Advanced Light Source	38
Rogers, Joe	5	The Environmental Science Grazing Incidence EXAFS Spectrometer at SSRL	31
* Sarangi, Ritimukta	29	X-ray Absorption Edge and EXAFS Studies of the Red Copper Site in Nitrosocyanin: Comparison to Plastocyanin	43
* Slowey, Aaron J.	17	Transport of Colloid-Associated Arsenic and Mercury: Column Experiments and Microscopic and Spectroscopic Analyses of Colloidal Material	37
Smolsky, Igor	22	Diffraction-Enhanced/Phase-Contrast Imaging Instrument at SSRL	39
Tatchyn, Roman	39	Conceptual Scheme for a High Peak Energy Inertial Confinement Fusion (ICF) X-ray Ignition Facility	49
Tsuruta, Hiro	37	New Small Angle Scattering/Diffraction Instrument on BL4-2 for Structural Molecular Biology	47
Wasinger, Erik C.	25	L-edge XAS Determined Differential Orbital Covalency of Non-Heme Iron Sites	41
Webb, Samuel M.	12	Structural Mechanism of Uranyl Sequestration into Biogenic Manganese Oxides	34
* Willey, Trevor M	8	Characterization of Carboxyl Functionalized SAMs Using NEXAFS	32
Winick, Herman	42	SESAME, a 3 <sup>rd</sup> Generation Synchrotron Light Source for the Middle East Region	51
Wong, Thiang Y.	6	Molecular Selectivity in the Anthrax Lethal Factor Catalytic Site: X-ray Crystal Studies	31
* Yoon, Tae-Hyun	15	Long-Period X-ray Standing Wave (XSW) Studies of Pb(II) and As(V) Ion Distributions at Organic Thin Film – Mineral Interfaces	36

\* graduate student submission

POSTER # 1

## 2.6 Å Resolution Crystal Structure of *E. coli* CTP Synthetase by Hg-MAD, Solvent-flattening and Map Averaging

J. A. Endrizzi,<sup>1</sup> H. Kim,<sup>1</sup> P. Anderson,<sup>2</sup> and E. P. Baldwin<sup>1</sup><sup>1</sup>Molecular and Cell Biology, University of California at Davis, Davis, CA 95616, USA<sup>2</sup>University of Minnesota, Duluth, MN 55804, USA

CTP synthetase generates CTP from UTP and glutamine in the last step of pyrimidine biosynthesis. We have solved the crystal structure of *E. coli* CTP synthetase using phases obtained from Hg-MAD data collected at SSRL beamline 9-2. The 3.0 angstrom MAD map (FOM=0.35) was markedly improved by solvent flattening, followed by phase extension to 2.6 angstrom resolution using two-fold averaging and flattening with DM (DMFOM=0.86). The present R-factor is 18.8% ( $R_{\text{free}}=26.9\%$ ). There are two CTPS molecules/asu and the biologically active 222 tetramer is generated by the crystallographic two-fold axis of the  $p2_12_12$  space group. The 222 origin is 3 angstrom from the crystallographic origin which created special problems for location of heavy atoms. The solvent flattening improvement was extremely sensitive to solvent percentage. Refinement was performed using the data set from the dispersion peak. Interestingly, the mercury sites are absent or poorly occupied in the initial and refined maps. This apparent photodissociation of mercury explains why the best phases were obtained using the inflection-remote differences only. CTPS belongs to the Cbi (CobQ) structural fold family, and is the first structure of a GATase domain-containing member. The two molecules in the asymmetric unit differ by a  $\sim 3$  degree hinge between the GATase and synthetase domains. This structure will provide a template for solving the structures of other CTP synthetases and potentially for structure-based design of anti-cancer and anti-parasitic therapeutics.

POSTER # 2

## Closing the Folding Chamber of the Eukaryotic Chaperonin Requires the Transition State of ATP Hydrolysis

A. S. Meyer,<sup>1</sup> J. R. Gillespie,<sup>1</sup> D. Walther,<sup>4</sup> I. S. Millett,<sup>2</sup> S. Doniach,<sup>3</sup> and J. Frydman<sup>1</sup><sup>1</sup>Department of Biological Sciences, Stanford University, Stanford, CA 94305, USA<sup>2</sup>Department of Chemistry, Stanford University, Stanford, CA 94305, USA<sup>3</sup>Department of Applied Physics, Stanford University, Stanford, CA 94305, USA<sup>4</sup>Incyte Genomics, Inc., 3160 Porter Dr., Palo Alto, CA 94304, USA

The ATP-dependent chaperonin TRiC (TCP-1 ring complex) is proposed to mediate protein folding by opening and closing a "built-in" lid over its central cavity. Here we combine structural and biochemical analyses to elucidate the mechanism and function of lid closure. Nucleotide-free TRiC adopts an open conformation that binds unfolded substrates in its central cavity. We find that incubation with ATP indeed promotes lid formation, yielding a closed central cavity. Formation of this closed chamber is essential for substrate folding, as blocking lid closure does not impair ATP hydrolysis or substrate-binding but abolishes productive folding of the bound polypeptide. To define how the ATPase cycle drives lid closure, we employed nucleotide analogues that stabilize distinct pre- and post-hydrolysis states. ATP-analogues that mimic the pre-hydrolysis state do not generate the closed cavity and leave the bound substrate in an unstructured conformation. In contrast, lid closure is induced by a mimic of the trigonal-bipyramidal transition state of the hydrolysis reaction, thus confining the substrate in the central chamber. We conclude that closure of the "built-in" lid is triggered by the transition state of ATP hydrolysis and is essential for productive folding of substrate proteins.

graduate student submission

POSTER # 3

### **Arsenic Speciation and Cycling within the Sediments of Lake Coeur d'Alene, Idaho**

M. L. Polizzotto,<sup>1</sup> S. E. Fendorf,<sup>1</sup> G. Toevs,<sup>2</sup> M. Morra,<sup>2</sup> and B. Bostick<sup>3</sup>

<sup>1</sup>Department of Geological & Environmental Sciences, Stanford University, Stanford, CA 94305, USA

<sup>2</sup>Soil and Land Resources Division, University of Idaho, Moscow, ID 83843, USA

<sup>3</sup>Department of Earth Sciences, Dartmouth College, Hanover, NH 03755, USA

Arsenic is a contaminant in the sediments of Lake Coeur d'Alene, Idaho, as a result of years of upstream mining. At the present time, there is debate over whether arsenic in the sediments is primarily associated with a detrital ferric-oxide phase or incorporated into an authigenic sulfide phase. Dissolved arsenic in sediment porewaters increases with depth near the surface to a maximum of 1000 mg/L at 5-10 cm; below this point the concentration decreases.

Sediment cores were obtained from three locations in Lake Coeur d'Alene where solid phase arsenic concentrations reach 400 mg/Kg at the sediment-water boundary. X-ray absorption spectroscopic (XAS) analyses indicate that solid phase arsenic, iron, and sulfur become more reduced with depth. Arsenic appears to be present as an adsorbed complex on surficial sediments but is incorporated into sulfide-bearing minerals with increasing depth. Based on XAS analysis, oxidized iron minerals are present at the surface, but siderite dominates deeper in the sediment, encompassing over 80% of the iron mineralogy in the bottommost sediments sampled. Additionally, reduced sulfur species (e.g. sulfides) also increase with depth.

On the basis of changes in solid speciation and porewater concentrations with depth, active diagenetic cycling of arsenic, iron, and sulfur drives their distribution in Lake Coeur d'Alene sediments. During ferric oxide reduction, arsenic is displaced from mineral surfaces and remains in solution until the genesis of sulfide minerals, which repartitions arsenic back into the solid phase. The spatial distribution of arsenic further reveals that the greatest potential for its release into porewater from the sediment environment occurs during changes in redox conditions; an influx of nutrients or competing ions could stimulate arsenic release into Lake Coeur d'Alene.

graduate student submission

POSTER # 4

### **Perophora Surprise: A New Structural Motif for Biological Iron**

P. Frank,<sup>1,2</sup> A. W. De Tomaso,<sup>2</sup> B. M. Hedman,<sup>1</sup> and K. O. Hodgson<sup>1,2</sup>

<sup>1</sup>Stanford Synchrotron Radiation Laboratory, SLAC, Stanford University, Stanford, CA 94309, USA

<sup>2</sup>Department of Chemistry, Stanford University, Stanford, CA 94305, USA

<sup>3</sup>Hopkins Marine Station, Stanford University, Pacific Grove, CA 93950, USA

In a surprise finding, blood cells from the colonial ascidian *Perophora annectens* were found to be rich in iron instead of the expected vanadium. Iron K-edge x-ray absorption spectra are similar in some respects to that of iron in magnetite, and show evidence of redox-activity. This implies metabolic participation. The EXAFS Fourier transform spectrum shows a strong iron-iron vector indicating a multinuclear site. Fits to EXAFS are most consistent with a unique flared Fe<sub>4</sub>O<sub>4</sub> cubane-like structure, new to biological iron.

POSTER # 5

**The Environmental Science Grazing Incidence EXAFS Spectrometer at SSRL**J. Rogers,<sup>1</sup> J. Bargar,<sup>1</sup> T. H. Yoon,<sup>2</sup> G. A. Waychunas,<sup>3</sup> and G. E. Brown, Jr.<sup>2</sup><sup>1</sup>Stanford Synchrotron Radiation Laboratory, SLAC, Stanford University, Stanford, CA 94309, USA<sup>2</sup>Department of Geological & Environmental Sciences, Stanford University, Stanford, CA 94305, USA<sup>3</sup>Earth Sciences, Lawrence Berkeley National Laboratory, Berkeley, CA 94720, USA

A new high-precision spectrometer system has been developed and tested for measuring grazing-incidence XAFS spectra, x-ray reflectivity, and standing wave intensity data from oriented single crystals, flat polycrystalline samples, and from adsorbed interfacial species. The instrument system was designed for routine, rapid installation and alignment in synchrotron experimental hutches utilized for XAFS measurements. Two major components comprise the system: (a) a strongback cradle on which flexor slits, sample positioners, and detectors are aligned, and (b) a multi-altitudinal detector support, which allows a 30-element Ge detector array to be positioned at vertical down-looking, horizontal side-looking, and intermediate observation angles. Motorized positioners provide seven axes of motion for samples, which provides accurate positioning of large sample cells and samples having uneven shapes. Precision slits located upstream of the sample provide aperture control to about 1 micron precision over a range of motion from zero to 1.5 mm, making possible measurements on mm-diameter samples and at very small incidence angles. An ion chamber detector located downstream of the sample is mounted on a bearing arc to rotate about the sample eucentric for specular reflectivity measurements. This detector can be replaced with an analyzer crystal and proportional counter to provide high-rate counting with high angular/energy resolution. Results from recent measurements demonstrate the utility and precision of this instrument for molecular environmental and interfacial sciences.

POSTER # 6

**Molecular Selectivity in the Anthrax Lethal Factor Catalytic Site: X-ray Crystal Studies**T. Y. Wong,<sup>1</sup> B. E. Turk,<sup>2</sup> R. Schwarzenbacher,<sup>1</sup> S. H. Leppla,<sup>3</sup> L. C. Cantley,<sup>2</sup> and R. C. Liddington<sup>1</sup><sup>1</sup>The Burnham Institute, La Jolla, CA 92037, USA<sup>2</sup>Beth Israel Deaconess Medical Center and Harvard Medical School, Boston, MA 02215, USA<sup>3</sup>National Institute of Allergy and Infectious Diseases, Bethesda, MD 20892, USA

Exposure to *Bacillus anthracis* spores, and subsequent rapid proliferation of vegetative cells in the mammalian host, leads to accumulative production of anthrax toxin in the bloodstream. Anthrax toxin is composed of the membrane pore-forming Protective Antigen (PA), and the translocated intracellular enzymes Lethal Factor (LF) and Edema Factor (EF). PA, combined with LF, gives rise to Lethal Toxin (LeTx), which is especially cytotoxic for the macrophage and the major cause of high fatality rates in inhalational Anthrax. LF, a zinc-dependent proteinase, intracellularly inactivates members of the Mitogen-Activated Protein Kinase Kinase (MKK) cell signaling protein family essential for normal cell growth. We are studying the molecular basis for LF substrate and inhibitor target affinities using highly purified LF, natural substrate-derived peptides, peptide library screening and x-ray crystallography. The aim is to probe and investigate in detail the active center of LF using LF protein crystals complexed to targets, obtaining three-dimensional information to design specific drugs. Currently, we have found the determining factors that explain how LF accommodates specific, high-affinity ligands, and identified the molecular environment that contributes to successful target recognition. In particular, a highly charged region along the 40 Å-long binding groove, combined with hydrophobic selection at the LF active center determines ligand binding conformation and affinity. At present, we have inhibitory compounds with  $K_i(\text{app})$  values in the range of ~1nM to 10microM. Further work look to provide insights into secondary target sites. This work lays the foundation for multiple approaches to neutralize LeTx, with possible applications to other proteases involved in pathogenic processes.

POSTER # 7

### **Biostimulation of Uranium Reduction in Sediment from the Oak Ridge Source Zone**

M. A. Ginder-Vogel,<sup>1</sup> J. Nyman,<sup>2</sup> W. Wu,<sup>2</sup> S. Fendorf,<sup>1</sup> and C. Criddle<sup>2</sup>

<sup>1</sup>Department of Geological & Environmental Sciences, Stanford University, Stanford, CA 94305, USA

<sup>2</sup>Department of Civil and Environmental Engineering, Stanford University, Stanford, CA 94305, USA

*In-situ* immobilization of heavy metals, such as uranium, through biological reduction is a promising remediation technique for contaminated subsurface sediments. Stimulation of uranium reduction at the field scale presents several challenges, including heterogeneous sediment mineralogy, a complex and evolving community of bacteria responsible for uranium reduction, and the presence of multiple electron donors and acceptors. These issues were investigated in a series of batch experiments using radionuclide contaminated sediment from Oak Ridge National Laboratory inoculated with a denitrifying bacterial community. The oxidation states of Fe and U were determined using X-ray absorption near edge structure (XANES) spectroscopy. Viable bacterial cells rapidly reduced U(VI) to U(IV), while no change in iron oxidation was observed. The presence of nitrate was shown to inhibit uranium reduction. This work demonstrates that in the absence of nitrate uranium is reduced under conditions similar to the Oak Ridge Field site.

graduate student submission

POSTER # 8

### **Characterization of Carboxyl Functionalized SAMs Using NEXAFS**

T. M. Willey,<sup>1,2</sup> A. L. Vance,<sup>2</sup> C. Bostedt,<sup>2</sup> R. W. Meulenberg,<sup>2</sup> A. J. Nelson,<sup>2</sup> L. J. Terminello,<sup>2</sup> and C. S. Fadley<sup>1,3</sup>

<sup>1</sup>University of California at Davis, Davis, CA 95616, USA

<sup>2</sup>Lawrence Livermore National Laboratory, Livermore, CA 94550, USA

<sup>3</sup>Lawrence Berkeley National Laboratory, Berkeley, CA 94720, USA

Self-Assembled Monolayers (SAMs) and other organo-thiol compounds on Au(111) have become increasingly important to achieve surface attachment of complex molecules. In order to produce useful films, one needs to understand the orientation, order, and substrate bonding of the molecules. Carboxyl and amino terminated SAMs are useful in functionalizing surfaces for surface attachment and immobilization of proteins, DNA, viruses, as well as using the charged state of the endgroups in switchable surfaces. We are also using such functionalization in surface attached interlocking molecules to attempt to induce and measure reorientation (switching) within interlocking molecules. However, functionalization often (adversely!) affects the synthesis, order, packing, and formation of these films. With proper synthetic protocol, many of these issues can be overcome.

Here, NEXAFS results probe the orientation of molecules while XPS is used to determine the attachment and chemical nature of the films. We present NEXAFS spectra to compare and contrast the ordering between SAMs from mercaptohexadecanoic acid ( $\text{HS}(\text{CH}_2)_{15}\text{COOH}$ ) a long-chain molecule with strong chain-chain interactions and thioctic acid ( $\text{S}_2(\text{CH}_2)_2\text{CH}(\text{CH}_2)_4\text{COOH}$ ) with a large base hindering strong interactions between its short chains. Stark conformational differences occur in these films of the endgroups with deprotonation ( $\text{COO}^-$  vs.  $\text{COOH}$ .) In addition, we will present initial results on ordering and attachment in amino-terminated alkanethiols on gold, and preliminary results using functional groups in switching interlocking molecules on surfaces.

graduate student submission

POSTER # 9

**Local Structure of Dopant Ions [Mn(II), Cu(II), and Ag(I)] in ZnSe Nanoparticles**T. Norman,<sup>1</sup> T. Wilson,<sup>1</sup> D. Magana,<sup>2</sup> D. Cao,<sup>3</sup> F. Bridges,<sup>1</sup> and J. Z. Zhang<sup>1</sup><sup>1</sup>University of California at Santa Cruz, Santa Cruz, CA 95064, USA<sup>2</sup>University of California at Santa Barbara, Santa Barbara, CA 93106, USA<sup>3</sup>Los Alamos National Laboratory, Los Alamos, NM 87545, USA

ZnSe nanoparticles were synthesized from molecular cluster precursors. Mn(II), Cu(II), or Ag(I) dopant ions were incorporated into the nanoparticles by mixing in the corresponding metal salt or cluster into the ZnSe nanoparticle reaction mixture. XAFS studies were conducted to determine local structure of the Zn and Se atoms as well as the local structure of the dopant ions. The XAFS data indicated there was a reduction in the Zn first neighbor Se coordination from the bulk value but a lack of a reduction in the Se first neighbor coordination. This suggests that the core of the nanoparticles resembles that of bulk ZnSe, and the surface of the particle has a higher concentration of metal atoms. However, the Zn had a full 4 coordinate coordination shell due to the presence of OH-/O<sub>2</sub>- or hexadecylamine (HDA) bound to the surface of the nanoparticle. The Mn(II) dopant possessed a greater reduction in first neighbor Se coordination and a higher OH-/O<sub>2</sub>- /HDA coordination. From this we propose that the surface Mn(II) possessed an octahedral geometry due to significant OH-/O<sub>2</sub>- coordination and the interior Mn(II) occupied the Zn tetrahedral site. In the case of Cu(II) doped ZnSe nanoparticles it was determined that the Cu(II) uniformly substituted for Zn(II) in the lattice. For the Ag(I) dopant the XAFS data suggested that the Ag(I) dopant occupied a variety of different environments in the ZnSe lattice. These structural properties can have a profound influence on the optical properties of the nanoparticles.

graduate student submission

POSTER # 10

**Synchrotron Radiation Photoemission Spectroscopy Study of Cs/O Co-adsorbed InP(100) Surface for Application in Negative Electron Affinity Devices**D.-I. T. Lee,<sup>1,2</sup> S. Sun,<sup>1,3</sup> Y. S. Sun,<sup>1,5</sup> P. Pianetta,<sup>1,4</sup> and W. E. Spicer<sup>1,4</sup><sup>1</sup>Stanford Synchrotron Radiation Laboratory, SLAC, Stanford University, Stanford, CA 94309, USA<sup>2</sup>Department of Materials Science and Engineering, Stanford University, Stanford, CA 94305, USA<sup>3</sup>Department of Physics, Stanford University, Stanford, CA 94305, USA<sup>4</sup>Department of Electrical Engineering, Stanford University, Stanford, CA 94305, USA<sup>5</sup>Department of Chemistry, Stanford University, Stanford, CA 94305, USA

The activation of III-V semiconductors by Cs/O co- adsorption is critical to the achievement of negative electron affinity for application in night vision goggles and electron beam lithography. InP(100) is one of the most important III-V semiconductors, having a band gap energy that corresponds to light emitters and receivers in the long wavelength region and, also having high-saturation-velocity electrons suitable for the active channel in high-power and high-speed devices. The chemistry and atomic geometry of the Cs/O co-adsorbed InP(100) surface, however, is far from clear. In this synchrotron radiation ultraviolet photoemission spectroscopy (SR-UPS) study, different oxygen species were observed, including both molecular and atomic oxygen species. By plotting the valence bands against different quantum yields (Q.E), the peak attributed to the molecular oxygen in the valence band increased as the Q.E increased. The molecular oxygen peak decreased with time, as Q.E did, to a great extent. These two phenomena show that the amount of molecular oxygen species is attributable to the Q.E of the Cs/O activated surface. The angle-resolved UPS has also been measured to investigate the orientation of the molecular oxygen on the surface. Since the beam was linearly polarized perpendicularly to the direction of the propagation, and the molecular oxygen peak was maximized with the polarization normal to the surface, the oxygen molecules must be placing themselves on their ends.

graduate student submission

POSTER # 11

### **X-ray Scattering as a Probe of Interdiffusion in Si/SiGe Heterostructures**

D. B. Aubertine, N. Ozguven, and P. C. McIntyre

Department of Materials Science and Engineering, Stanford University, Stanford, CA 94305, USA

X-ray scattering from compositionally modulated films has a long history as an ultra-high-sensitivity probe of interdiffusion and several groups have recently applied this strategy to Si/SiGe. We investigate numerical simulations that utilize a non-linear interdiffusion solver and dynamical x-ray diffraction calculations to predict the local composition evolution in low Ge concentration Si/SiGe superlattices and their diffraction patterns during annealing. Superlattice satellite peak decay rates are compared with experimentally measured values and simulated diffraction patterns are matched directly to data with good success. The simulations are used to show that diffraction from superlattices with initially square-wave concentration profiles produces valid results when applied to 004 superlattice satellite peaks even though it is only exactly applicable to sinusoidal composition modulations and to satellite peaks about 000 reflections. Finally, we show that proper interpretation of x-ray scattering data to extract Si/SiGe interdiffusivity values must account for the strong dependence of the interdiffusivity on Ge concentration.

graduate student submission

POSTER # 12

### **Structural Mechanism of Uranyl Sequestration into Biogenic Manganese Oxides**

S. M. Webb,<sup>1</sup> J. R. Bargar,<sup>1</sup> and B. M. Tebo<sup>2</sup>

<sup>1</sup>Stanford Synchrotron Radiation Laboratory, SLAC, Stanford University, Stanford, CA 94309, USA

<sup>2</sup>Scripps Institution of Oceanography, La Jolla, CA 92093, USA

Uranium is a toxic and radioactive contaminant in many environmental settings, such as groundwaters and sediments. In oxidizing environments, uranyl (U(VI)) is thermodynamically the most stable oxidation state. Sorption or incorporation of U(VI) into reactive mineral phases are processes of major importance because they retard its transport. Biogenic manganese oxides are an important source of reactive mineral surfaces in the environment and may be potentially enhanced in bioremediation cases to improve natural attenuation. Experiments were performed in which U(VI) at various concentrations was present during manganese oxide biogenesis. At all concentrations there was strong uptake of U onto the oxides. Synchrotron based EXAFS and WAXS studies were carried out to determine the manner in which uranyl is incorporated into the oxide and how this incorporation affects the resulting manganese oxide structure and mineralogy. The EXAFS experiments show that at low concentrations, uranyl does not appear to substitute into the lattice of the oxides, and is rather present as a strong surface complex. However, at high concentrations, the presence of U(VI) affects the stability and structure of the Mn oxide to form tunnel structures, similar to todorokite. EXAFS modeling shows that uranyl is present predominantly in the tunnels of the Mn oxide structure. Observations by WAXS corroborate these results. Presumably, this leads to more stable U(VI) sequestration that may be suitable for remediation uses. These observations, combined with the very high sorptive capacity of the Mn oxides, imply that bacteria can exert tremendous influence over U(VI) speciation in impacted waters via Mn oxide biomineralization.

POSTER # 13

### Mechanistic Insights from Substrate-bound Structures of *Pseudomonas fluorescens* Mannitol 2-dehydrogenase

K. L. Kavanagh,<sup>1</sup> M. Klimacek,<sup>2</sup> B. Nidetzky,<sup>2</sup> and D. K. Wilson<sup>1</sup><sup>1</sup>University of California at Davis, Davis, CA 95616, USA<sup>2</sup>Technical University of Graz, A-8010 Graz, Austria

*Pseudomonas fluorescens* mannitol 2-dehydrogenase (MDH) is a secondary alcohol dehydrogenase that catalyzes the NAD(P)H-dependent interconversion of D-mannitol and D-fructose. It belongs to a family of long-chain mannitol dehydrogenases that to date consists of 66 members. The structures of a binary complex with NADH and a ternary complex with NADH and mannitol were determined to 1.7 and 1.8 Angstroms respectively.

While most alcohol dehydrogenases utilize a metal ion or catalytic tyrosine, MDH employs a lysine general base. The catalytic lysine (Lys-295) donates hydrogen bonds to main-chain carbonyls on Val-229 and Asn-300, which orients its lone electron pair toward substrate. In return, the mannitol O2 accepts hydrogen bonds from Asn-300 and Asn-191 ensuring its proton is directed toward Lys-295. This emphasizes the important role of Asn-300, which is involved in positioning both Lys-295 and substrate for efficient catalysis.

A comparison of the two complexes indicates that Glu-292, which is adjacent to the active site, oscillates between buried, hydrophobic and solvent accessible environments. A solvent-lined channel is also observed in the ternary complex. This conformational difference along with the well-ordered water molecules suggests two mechanisms by which Glu-292 may be involved in proton transfer. In the first, Glu-292 acts as a gate opening a channel by which the proton can be shuttled to bulk solvent along a chain of water molecules. In the second mechanism Glu-292 carries the proton itself to bulk solvent. Experiments to elucidate the precise role of Glu-292 are planned.

POSTER # 14

### *In Situ* Mo K-edge XAS Studies of Mo-ZSM-5 in CO<sub>2</sub>/CH<sub>4</sub> Mixtures

H. S. Lacheen,<sup>1</sup> F. Requejo,<sup>2</sup> and E. Iglesia<sup>1</sup><sup>1</sup>Department of Chemical Engineering, University of California at Berkeley, Berkeley, CA 94720, USA<sup>2</sup>Departamento de Fisica. Facultad de Ciencias Exactas, UNLP, La Plata, Argentina

Mo/H-ZSM-5 was recently shown to achieve near- equilibrium conversions of CH<sub>4</sub> to C<sub>2+</sub> alkanes and aromatics. Synthesis gas generation occurs with CO<sub>2</sub> addition but leads to lower pyrolysis yields. Previous studies suggest oxidation of active Mo centers during CO<sub>2</sub> addition and thermodynamic constraints as a result of the concomitant formation of additional H<sub>2</sub> in CH<sub>4</sub>-CO<sub>2</sub> reactions lead to the decreased yields. Here, we explore the effect of CO<sub>2</sub> addition on the state of Mo with CO<sub>2</sub>/CH<sub>4</sub> cofeed at 950 K using *in situ* transient Mo K-edge XANES and mass spectrometric analysis of reaction rates; we use principal component analysis (PCA) to determine the number and identity of components, and k<sup>2</sup>-weighted EXAFS spectra fitted with theoretical standards to determine radial distances and coordination. PCA identified two components during CH<sub>4</sub> activation of Mo-ZSM-5, but only MgMo<sub>2</sub>O<sub>7</sub> was identified as a principal component. The Mo K-edge relative to Mo foil decreases from 4.90 eV to 0.21 eV upon addition of CH<sub>4</sub> and increases to 3.70 eV with CO<sub>2</sub>/CH<sub>4</sub> = 0.25. The pre-edge feature in the fresh catalyst arising from ditetrahedral (Mo<sub>2</sub>O<sub>5</sub>)<sup>2+</sup> centers completely disappears during carburization in CH<sub>4</sub> and reappears in CO<sub>2</sub>/CH<sub>4</sub> mixtures. EXAFS analysis indicates a Mo-O shell forms and the Mo-C coordination number decreases when carburized Mo/H-ZSM-5 is treated with CO<sub>2</sub>/CH<sub>4</sub>. XAFS results agree with results obtained in parallel by mass spectrometry of reaction products, which indicated that Mo reoxidized in CO<sub>2</sub>/CH<sub>4</sub> mixtures after fully reducing and carburizing in pure CH<sub>4</sub>, and the amount of O\* added is proportional to CO<sub>2</sub> concentration.

graduate student submission

POSTER # 15

### Long-Period X-ray Standing Wave (XSW) Studies of Pb(II) and As(V) Ion Distributions at Organic Thin Film – Mineral Interfaces

T. H. Yoon,<sup>1</sup> T. P. Trainor,<sup>2</sup> P. Eng,<sup>3</sup> J. Bargar,<sup>4</sup> and G. E. Brown, Jr.<sup>1,4</sup>

<sup>1</sup>Department of Geological & Environmental Sciences, Stanford University, Stanford, CA 94305, USA

<sup>2</sup>Department of Chemistry and Biochemistry, University of Alaska Fairbanks, Fairbanks, AK 99775, USA

<sup>3</sup>GSECARS, University of Chicago, Chicago, IL 60637, USA

<sup>4</sup>Stanford Synchrotron Radiation Laboratory, SLAC, Stanford University, Stanford, CA 94309, USA

The distributions of Pb(II) and As(V) ions in the interfacial region between organic thin films (e.g., PolyAcrylic Acid (PAA)) and single crystal  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> ((0001) and (1-102)) and  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>(0001) substrates were studied using synchrotron-based XSW techniques. The PAA/ $\alpha$ -Al<sub>2</sub>O<sub>3</sub> and PAA/ $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> interfaces are simple analogs of naturally occurring organic thin films on mineral surfaces, which are thought to play important roles in the sequestration of trace heavy metals in natural soils and aquatic systems. In this work, very thin (30 – 120nm), flat (rms roughness < 1.5nm) PAA films were prepared on  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> and  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> surfaces and exposed to Pb(II) or As(V) ions (<50 $\mu$ M), and their fluorescence yield (FY) XSW and reflectivity profiles were collected at APS (ID-13) and SSRL (11-2) beamlines. Highly resolved XSW FY profiles provide unique information on Pb(II) partitioning between the organic film and single crystal mineral surfaces. Compared to the  $\alpha$ -Al<sub>2</sub>O<sub>3</sub>(0001) surface, the  $\alpha$ -Al<sub>2</sub>O<sub>3</sub>(1-102) and  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>(0001) surfaces are more reactive to Pb(II) adsorption, and more adsorbed Pb(II) ions were observed on the (1-102) surface as pH increased.

graduate student submission

POSTER # 16

### Structure of Glutamine Synthetase from *Bacillus subtilis*: Possible Insight into Regulation of Nitrogen Metabolism

D. S. Murray,<sup>1</sup> L. V. Wray Jr.,<sup>2</sup> S. H. Fisher,<sup>2</sup> and R. G. Brennan<sup>1</sup>

<sup>1</sup>Department of Biochemistry and Molecular Biology, Oregon Health & Science University, Portland, OR 97239, USA

<sup>2</sup>Department of Microbiology, Boston University School of Medicine, Boston, MA 02215, USA

In *Bacillus subtilis*, the dodecameric ammonia ligase, glutamine synthetase (GS), catalyzes the reaction of ammonia and glutamate in the presence of ATP to produce glutamine and ADP. The ADP product is further processed into AMP and ATP by adenylate kinase. A second function of GS is to sense nitrogen levels via feedback inhibition primarily by glutamine and AMP. GS from *B. subtilis* is regulated principally at the transcriptional level by two similar MerR-like proteins, GlnR and TnrA. GlnR represses the transcription of *glnA* (GS), *urease*, and *TnrA* during excess nitrogen conditions. Conversely, TnrA transcriptionally represses GS during nitrogen poor conditions and activates the *nrg*, *ure*, and *nas* operons, which initiate nitrogen scavenging. However, during nitrogen excess conditions, feedback inhibited GS represses TnrA via a protein/protein interaction. To begin to understand the structural basis of nitrogen metabolism regulation in *B. subtilis*, we have crystallized GS in its apo form along with soaking apo crystals with glutamine and AMP. The protein crystallizes in space group P2<sub>1</sub> with unit cell dimensions of 133 Å x 144 Å x 174 Å and  $\beta = 91^\circ$ . Assuming 12 subunits per asymmetric unit (mw = 600 kDa), the Matthews coefficient is 2.8 Å<sup>3</sup> Da<sup>-1</sup> with a solvent content of 55.7%. Intensity data have been collected to 2.55 Å at SSRL (BL-9.1) for both apo ( $I/\sigma I = 3.1$ ,  $R_{\text{sym}} = 0.060$ ) and glutamine/AMP soaked ( $I/\sigma I = 2.3$ ,  $R_{\text{sym}} = 0.050$ ) crystals. Structure determination of both forms of GS by molecular replacement (employing EPMR) is underway utilizing a dodecamer of the *Mycobacterium tuberculosis* GS (38% identity) as a search model. These structures will begin to provide insight into the structural mechanisms by which TnrA and GS regulate each other.

graduate student submission

POSTER # 17

### Transport of Colloid-Associated Arsenic and Mercury: Column Experiments and Microscopic and Spectroscopic Analyses of Colloidal Material

A. J. Slowey,<sup>1</sup> S. B. Johnson,<sup>1</sup> M. Newville,<sup>3</sup> and G. E. Brown, Jr.<sup>1,2</sup>

<sup>1</sup>Department of Geological & Environmental Sciences, Stanford University, Stanford, CA 94305, USA

<sup>2</sup>Stanford Synchrotron Radiation Laboratory, SLAC, Stanford University, Stanford, CA 94309, USA

<sup>3</sup>GeoSoilEnviro Consortium for Advanced Radiation Sources, University of Chicago, Chicago, IL 60637, USA

Heavy metal contaminants in aqueous solutions can be transported as dissolved or particle-associated species. While transport of dissolved species has received more attention, under certain conditions, transport of heavy metals associated with colloids is significant. Important issues to address are (1) the physicochemical conditions under which colloidal particles are mobilized, (2) the nature of the colloidal particles and the way(s) in which heavy metals are associated with them, and (3) the extent of particle-associated heavy metal transport. Column experiments were performed to demonstrate particle-based transport of arsenic and mercury from a calcined ore obtained from the Sulphur Bank mercury mine, CA. Calcines were exposed to organic acids and electrolyte as a means of simulating environmental perturbations that commonly mobilize colloids. The arsenic and mercury species associated with the mobilized particles were characterized using Hg L<sub>III</sub>- and As K-edge x-ray absorption fine structure (XAFS) spectroscopy and scanning electron microscopy with energy dispersive spectroscopy (SEM-EDS). To obtain a useable range of As EXAFS (i.e., beyond  $k = 10.5 \text{ \AA}^{-1}$ ), interference of Hg L- $\alpha$  fluorescence with As K- $\alpha$  fluorescence measurements was minimized through enhanced energy resolution. Higher resolution was achieved by lengthening the shaping time of DXP digital electronics used to measure currents induced by fluorescent photons received by the 16-element Ge detector. High concentrations of Hg in the samples (~1,000 ppm) also caused sample self absorption and/or Ge detector deadtime, requiring correction of all As EXAFS spectra. Most of the mobilized arsenic and mercury was in particulate forms, comprising As(V)-Fe(oxy)hydroxide surface complexes or coprecipitates, As(V)-substituted jarosite ( $\text{KFe}_3(\text{SO}_4, \text{AsO}_4)_2(\text{OH})_6$ ), HgS (cinnabar and metacinnabar), and corderoite ( $\text{Hg}_3\text{S}_2\text{Cl}_2$ ).

graduate student presentation

POSTER # 18

### The CODH/ACS Family: Xenon in and the End of the Tunnel

T. I. Doukov,<sup>1</sup> J. Seravalli,<sup>2</sup> S. W. Ragsdale,<sup>2</sup> and C. L. Drennan<sup>1</sup>

<sup>1</sup>Department of Chemistry, Massachusetts Institute of Technology, Cambridge, MA 02139, USA

<sup>2</sup>Department of Biochemistry, Beadle Center, University of Nebraska-Lincoln, Lincoln, NE 68588, USA

The carbon monoxide dehydrogenase/acetyl-CoA synthase (CODH/ACS) family of enzymes catalyzes two unique reactions involving CO gas at a complex multimetal containing active sites. First activity, reversible CO<sub>2</sub> reduction, was assigned spectroscopically to the so called center C, containing Fe and Ni. The produced CO is utilized in the ACS active site some 70 Å away.

In our initial structural report we have calculated a possible tunnel connecting CODH and ACS active sites through the protein matrix. In this report we present our Xe pressurized crystal results confirming the tunnel. We observed 7 Xe sites in the tunnel and one separate site on the CODH surface. In addition, we observed a well ordered Xe site in the hydrophobic ACS active site. Xe is making VDW contact with two ACS metal ions.

POSTER # 19

### **Synchrotron Radiation Photoemission Spectroscopy Analysis of High-k Gate Dielectric for Ge MOS Applications**

C. O. Chui,<sup>1</sup> D.-I. Lee,<sup>2</sup> A. A. Singh,<sup>2</sup> P. Pianetta,<sup>2</sup> and K. C. Saraswat<sup>1</sup>

<sup>1</sup>Department of Electrical Engineering, Stanford University, Stanford, CA 94305, USA

<sup>2</sup>Stanford Synchrotron Radiation Laboratory, SLAC, Stanford University, Stanford, CA 94309, USA

The saturation of Si MOSFET drain current upon dimension shrinkage may limit the prospect of future scaling. The lower effective mass (and lower valley degeneracy) of Ge could alleviate the problem by providing a higher source injection velocity, which translates into higher drive current and smaller gate delay. In addition, the smaller optical bandgap for Ge broadens the absorption wavelength spectrum allowing opto-electronic integration to enhance CMOS functionality.

Nonetheless, unlike Si, the poor quality Ge native dielectrics for gate insulator and field isolation have hindered the realization of Ge MOS devices in the last four decades. Inspired by the recent successes of the high-k dielectric deposition technique on Si and the thermodynamically unstable nature of the common germanium native oxides, we have investigated the possibility of applying high-k dielectrics to Ge without a native oxide interlayer. We fabricated MOS capacitors on Ge with ZrO<sub>2</sub> gate dielectric and that has led to the demonstration of Ge MOSFETs with enhanced hole mobility.

Transmission electron microscopy was used to analyze the ZrO<sub>2</sub>/Ge interface cross-section, although the phase contrast between ZrO<sub>2</sub> and GeOx (if any) was not high enough for clear distinction. In this experiment, layer-by-layer wet etching of ZrO<sub>2</sub> was used to track the composition variation along vertical direction using synchrotron radiation photoemission spectroscopy. The measured spectra were fitted and the material stack depth profile was modeled accordingly.

graduate student submission

POSTER # 20

### **Performance and Characteristics of the Howard Hughes Medical Institute Beamlines at the Advanced Light Source**

C. Y. Ralston,<sup>1</sup> A. A. MacDowell,<sup>2</sup> R. S. Celestre,<sup>2</sup> H. A. Padmore,<sup>2</sup> M. Howells,<sup>2</sup> C. W. Cork,<sup>1</sup> J. Dickert,<sup>1</sup> D. Cambie,<sup>3</sup> J. Holton,<sup>1</sup> E. E. Domning,<sup>3</sup> R. M. Duarte,<sup>3</sup> N. Kelez,<sup>3</sup> D. W. Plate,<sup>3</sup> and T. N. Earnest<sup>1</sup>

<sup>1</sup>Physical Biosciences, Lawrence Berkeley National Laboratory, Berkeley, CA 94720, USA

<sup>2</sup>Advanced Light Source, Lawrence Berkeley National Laboratory, Berkeley, CA 94720, USA

<sup>3</sup>Engineering Division, Lawrence Berkeley National Laboratory, Berkeley, CA 94720, USA

Three new multiwavelength anomalous diffraction (MAD) beamlines have been built at the Advanced Light Source (ALS) off a superconducting-bending magnet source. Two of these recently commissioned beamlines, 8.2.1 and 8.2.2, are funded through the Howard Hughes Medical Institute, and are dedicated to protein crystallography. The x-ray optics include a parabolic premirror, a double crystal monochromator, and a final toroidal mirror. The achieved focused beam size is near 100 microns in diameter, an ideal match for the increasingly small crystals now used in protein crystallography. The characteristics and performance of these beamlines is evaluated.

POSTER # 21

**Symmetry Break-downs in Aurivillius Phases**

J. Castro-Carmona,<sup>1</sup> M. García-Guaderrama,<sup>2</sup> M. E. Fuentes-Montero,<sup>3</sup> A. Mehta,<sup>4</sup> H. Camacho-Montes,<sup>1</sup> Montero-Cabrera,<sup>2</sup> J. F. Fernandez-Lozano,<sup>5</sup> M. Villegas,<sup>5</sup> T. Jardiel,<sup>5</sup> and L. E. Fuentes-Cobas<sup>2</sup>

<sup>1</sup>Universidad Autónoma de Ciudad Juárez, 31109 Chihuahua, Chih., México

<sup>2</sup>Centro de Investigación en Materiales Avanzados, 31109 Chihuahua, Chih., México

<sup>3</sup>Universidad Autónoma de Chihuahua, 31109 Chihuahua, Chih., México

<sup>4</sup>Stanford Synchrotron Radiation Laboratory, SLAC, Stanford University, Stanford, CA 94309, USA

<sup>5</sup>Instituto de Ceramica y Vidrio, 28049 Madrid, Spain

So-called Aurivillius ceramics focus attention today, mainly due to their high-temperature ferroelectricity and their potentiality for magnetoelectric sensors. The crystalline structure of these materials is formed by perovskite octahedra, sandwiched between bismuth oxide layers. For temperatures above the Curie point,  $T_c$  (600°C – 800°C), Aurivillius crystals adopt centro-symmetrical tetragonal structures, with space group Fmmm. In the ferroelectric temperature domain ( $T < T_c$ ) tetragonal symmetry breaks down to orthorhombic or monoclinic, with lattice parameters slightly different from those corresponding to the high-temperature configuration. Determining the crystal symmetry group is difficult, but it is important, because symmetry is a decisive factor in the structure - polarization relationship. Recent specialized articles display interpretation errors, caused by insufficient resolution in the diffraction experiments. A wide range of Aurivillius phases has been obtained by cooperative work among research groups in Chihuahua and Madrid. Investigated compositions include phases with number of perovskite layers ranging from 3 to 7. Small octahedral cations are Ti, Fe and W. High resolution powder diffraction patterns of representative samples have been measured in SSRL, Station 2-1. The obtained diffraction spectra demonstrated non previously reported symmetry break-downs. Considered structures are refined by means of the Rietveld method. Present poster exhibits, for the first time, the results of this structural investigation. The physical significance of crystallographic findings is discussed.

graduate student submission

POSTER # 22

**Diffraction-Enhanced/Phase-Contrast Imaging Instrument at SSRL**

H. Tsuruta, I. Smolsky, and K. Ito

Stanford Synchrotron Radiation Laboratory, SLAC, Stanford University, Stanford, CA 94309, USA

Purpose of this technique is to visualize low electron density variations in crystalline and non-crystalline low electron density objects. Image contrast is formed as a result of absorption, refraction gradient and small angle scatter-rejection (extinction). The use of the synchrotron beam coherency and carefully designed diffractive x-ray optics allows preparation of quasi-plane x-ray wave and analysis of x-rays refracted or scattered at very small angles by an object. We obtain a highly parallel beam by an asymmetrically cut Si (113) crystal (asymmetry factor  $b \sim 11$ ), which also enlarges the beam size vertically for two-dimensional imaging, so that real time imaging studies could be performed. The transmitted, diffracted and refracted x-rays through the specimen are analyzed by a symmetrically cut Si (113) crystal in a (+,-) non-dispersive geometry. The system is optimized to work at high beam energies, typically 36 keV, in order to take full advantage of the refraction/phase-shift effects rather than absorption. A series of images, each typically 50 mm (h) by 16 mm (v) in size, are recorded on x-ray film or a CCD x-ray detector along the analyzer rocking curve, whose FWHM is typically less than 3.5 microrad, depending on specimen and wave length. We have successfully visualized small simulated calcification and fiber formation in the mammography phantom and calcium oxalate particles in porcine belly meat. We have also looked at amputated amphibian limbs at different regeneration stages as well as microscopic calcification in parts of sea urchin shell. Compared with conventional absorption radiography, this technique makes features in objects more visible and allows one to do analysis of electron density variations.

POSTER # 23

### **Investigating the Fe-Fe Scattering from the Binuclear Fe Site in Methane Monooxygenase Hydroxylase by X-ray Absorption Spectroscopy**

D. Jackson Rudd,<sup>1</sup> M. H. Sazinsky,<sup>2</sup> M. Merx,<sup>2</sup> S. J. Lippard,<sup>2</sup> B. Hedman,<sup>1,3</sup> and K. O. Hodgson<sup>1,3</sup>

<sup>1</sup>Department of Chemistry, Stanford University, Stanford, CA 94305, USA

<sup>2</sup>Department of Chemistry, Massachusetts Institute of Technology, Cambridge, MA 02139, USA

<sup>3</sup>Stanford Synchrotron Radiation Laboratory, SLAC, Stanford University, Stanford, CA 94309, USA

Methane monooxygenase (MMO) is the multicomponent enzyme responsible for conversion of methane to methanol in methanotrophic bacteria. In the presence of iron, the enzyme exists in a soluble form and is comprised of a hydroxylase (MMOH), a regulatory protein (MMOB), a reductase (MMOR) and a protein generated from an open reading frame of unknown function (MMOD). MMOH contains a binuclear non-heme iron active site, which has been investigated structurally by both protein crystallography and x-ray absorption spectroscopy (XAS). Despite several studies, there is still ambiguity about the Fe-Fe distance in the oxidized state. Here, we report studies on oxidized MMOH by XAS and, more specifically, by EXAFS analysis that in particular focuses on the ability to determine iron-iron backscattering contributions in the presence of lighter second-shell scatterers, and also the effect on the diiron site by the other components in the enzyme.

This work was performed at SSRL, which is funded by the Department of Energy, Basic Energy Sciences. The SSRL Structural Molecular Biology Program is supported by the National Institutes of Health, National Center for Research Resources, Biomedical Technology Program and by the Department of Energy Office of Biological and Environmental Research.

graduate student submission

POSTER # 24

### **Speciation Controls on the Reduction and Transport of Uranium**

J. Neiss,<sup>1</sup> P. S. Nico,<sup>2</sup> B. Stewart,<sup>1</sup> and S. Fendorf<sup>1</sup>

<sup>1</sup>Department of Geological & Environmental Sciences, Stanford University, Stanford, CA 94305, USA

<sup>2</sup>Department of Chemistry, California State University Stanislaus, Turlock, CA 95382, USA

Due to mining and nuclear-production activities, uranium is now an environmental contaminant of great concern, the hazard of which can be diminished through reduction of the oxidized species, uranyl, to reduced phases such as uraninite. Recent evidence, however, illustrates the importance of uranyl speciation on the extent of reduction. In the presence of calcium, a Ca-UO<sub>2</sub>-CO<sub>3</sub> complex is the dominant aqueous species, greatly limiting abiotic and biotic reduction of uranium. This species is, in fact, the most stable form of U(VI) in waters equilibrated with atmospheric carbon dioxide levels and calcium concentrations > 0.4 μM from pH 5 to 8. Here we investigate the role of calcium in two advective flow systems (one with μM Ca and one without Ca) as well as their respective solid-phase products using X-ray adsorption near edge structure (XANES) and x-ray absorption fine structure (EXAFS) spectroscopies. Appreciable uranium sequestration occurred in the absence of Ca, while in systems with Ca (μM levels), only minimal uranium was sequestered. XANES analysis confirms the complete reduction of uranyl to the precipitated mineral uraninite in systems absent of calcium; in contrast, when Ca was present in the aqueous phase, uranyl reduction did not transpire. Additionally, EXAFS spectroscopy indicates differences in hydrous ferric oxide transformations to magnetite between the two systems. We hence conclude that calcium in the aqueous phase has a profound effect on bacterial reduction, and hence mobility, of uranium within surface and subsurface environments.

graduate student submission

POSTER # 25

**L-edge XAS Determined Differential Orbital Covalency of Non-Heme Iron Sites**E. C. Wasinger,<sup>1</sup> F. M. F. de Groot,<sup>2</sup> B. Hedman,<sup>1,3</sup> K. O. Hodgson,<sup>1,3</sup> and E. I. Solomon<sup>1</sup><sup>1</sup>Department of Chemistry, Stanford University, Stanford, CA 94305, USA<sup>2</sup>Utrecht University, 3508 TC Utrecht, The Netherlands<sup>3</sup>Stanford Synchrotron Radiation Laboratory, SLAC, Stanford University, Stanford, CA 94309, USA

X-ray absorption spectroscopy has been utilized to obtain the L-edge multiplet spectra for a series of non-heme ferric and ferrous complexes. Using these data a methodology for determining the total covalency and the differential orbital covalency (DOC), i.e. differences in covalency in the different symmetry sets of the d orbitals, has been developed. The integrated L-edge intensity is proportional to the number of one-electron transition pathways to the unoccupied molecular orbitals as well as to the covalency of the iron site. The covalency reduces the total L-edge intensity and redistributes the remaining intensity, producing shake-up satellites. Furthermore, differential orbital covalency leads to differences in intensity for the different symmetry sets of orbitals, and thus further modifies the experimental spectra. The ligand field multiplet model commonly used to simulate L-edge spectra does not adequately reproduce the spectral features, especially the charge transfer satellites. The inclusion of charge transfer states with differences in covalency gives excellent fits to the data and experimental estimates of the different contributions of charge transfer shake-up pathways to the  $t_{2g}$  and  $e_g$  symmetry orbitals. The resulting experimentally determined DOC is compared to values calculated from density functional theory and used to understand chemical trends in high- and low-spin ferrous and ferric complexes with different covalent environments. The utility of this method toward problems in bioinorganic chemistry is discussed.

Professor Roland Meier is thanked for providing the [Fe(ida)<sub>2</sub>] compound used in this study. This work was supported by grants from the National Institutes of Health: GM40392 to E.I.S. and RR-01209 to K.O.H. SSRL operations and research are funded by the U.S. Department of Energy (Office of Basic Energy Science). The Structural Molecular Biology program at SSRL is funded by the National Institutes of Health, National Center for Research Resources, Biomedical Technology Program, and the Department of Energy, Office of Biological and Environmental Research.

POSTER # 26

**A Unified Understanding of Bosonic Renormalization Effects in High-Tc Superconductors**T. Cuk,<sup>1,2</sup> F. Baumberger,<sup>1</sup> N. Ingle,<sup>1</sup> X. J. Zhou,<sup>1,3</sup> H. Eisaki,<sup>1,4</sup> N. Kaneko,<sup>1</sup> Z. Hussain,<sup>3</sup> and Z.-X. Shen<sup>1,2,6</sup><sup>1</sup>Department of Applied Physics, Stanford University, Stanford, CA 94305, USA<sup>2</sup>Stanford Synchrotron Radiation Laboratory, SLAC, Stanford University, Stanford, CA 94309, USA<sup>3</sup>Advanced Light Source, Lawrence Berkeley National Laboratory, Berkeley, CA 94720, USA<sup>4</sup>Nanoelectronics Research Institute, Tsukuba, Ibaraki 305-8568, Japan<sup>5</sup>CREST, Applied Physics, Japan<sup>6</sup>Physics Institute, University of Zürich, CH-80547 Zürich, Switzerland

Important revelations in physics sometimes start from small signals. The subtle wiggles seen in the electron tunneling spectra of conventional superconductors are a classical example. The explanation of this phenomenon, renormalization due to bosonic coupling, as the finger print of the electron-phonon interaction, cemented the consensus on the phonon pairing mechanism. A parallel is occurring in the field of high-temperature superconductivity where the recent observation of an analogous effect in photoelectron spectra has resulted in intense debate. Here, by analyzing extensive momentum and temperature dependence data, we uncover a signature of strong coupling of the 40 meV oxygen bond buckling phonon to electronic states where d-wave pairing is the strongest. This provides the missing link that allows a unified understanding.

graduate student submission

POSTER # 27

### Spurious Magnetism in a High-Temperature Superconductor

P. K. Mang,<sup>1</sup> S. Larochele,<sup>2</sup> A. Mehta,<sup>3</sup> and M. Greven<sup>1,3</sup>

<sup>1</sup>Department of Applied Physics, Stanford University, Stanford, CA 94305, USA

<sup>2</sup>Department of Physics, Stanford University, Stanford, CA 94305, USA

<sup>3</sup>Stanford Synchrotron Radiation Laboratory, SLAC, Stanford University, Stanford, CA 94309, USA

We present structural characterization and magnetic neutron scattering work on a secondary epitaxial phase formed inside the electron-doped superconductor Nd<sub>2-x</sub>Ce<sub>x</sub>CuO<sub>4</sub> (NCCO) [1]. The phase is identified as cubic (Nd,Ce)<sub>2</sub>O<sub>3</sub> and results from the decomposition of 0.01-0.1% of the parent phase when subjected to a high-temperature oxygen reduction procedure necessary for the creation of a superconducting phase. Because the lattice constant of the secondary phase is approximately 2sqrt(2) times the in-plane lattice constant of NCCO, scattering peaks from the secondary phase appear at fractional positions that are rational with respect to the NCCO lattice in the (H,K,0) plane. In particular, the (2, 0, 0) structural peak of the secondary phase coincides with an antiferromagnetic Bragg peak of NCCO at (1/2, 1/2, 0). These peaks have mistakenly been identified by others [2] as resulting from a superstructure associated with superconductivity. Recent work [3] has also mistakenly associated paramagnetism of the secondary phase Nd ions with the emergence of antiferromagnetic order in the superconducting state of NCCO in a magnetic field.

[1] P. K. Mang, S. Larochele, M. Greven, cond-mat/0308607 (to appear in Nature).

[2] K. Kurahashi *et al.*, J. Phys. Soc. Jpn. 71, 910 (2002).

[3] H. J. Kang *et al.*, Nature 423, 522 (2003).

graduate student submission

POSTER # 28

### Structure and Reactivity of an Asymmetric Complex between HsIV and I-domain Deleted HsIU, a Prokaryotic Homolog of the Eukaryotic Proteasome

A. R. Kwon,<sup>1</sup> B. M. Kessler,<sup>2</sup> H. S. Overkleeft,<sup>2</sup> and D. B. McKay<sup>1</sup>

<sup>1</sup>Department of Structural Biology, Stanford University School of Medicine, Stanford, CA 94305, USA

<sup>2</sup>Department of Pathology, Harvard Medical School, Boston, MA 02115, USA

HsIU is a prokaryotic homolog of the eukaryotic proteasome. HsIU is a AAA protein of the Clp/Hsp100 family. HsIV is a "double donut" dodecameric protease whose protomer is homologous to proteolytic subunits of proteasomes. Binding of HsIU allosterically enhances the proteolytic activity of HsIV. The HsIU protomer has three domains (amino-terminal, carboxy-terminal, and intermediate); the amino- and carboxy-terminal domains form a hexameric "ring", while the intermediate domains extend in a tentacle-like manner from the ring. The intermediate domains of HsIU have been deleted, an asymmetric HsIU( $\Delta$ )<sub>6</sub>HsIV<sub>12</sub> complex has been crystallized, and the structure has been solved to 2.5 Å resolution, revealing an assembly in which a HsIU( $\Delta$ ) hexamer binds one end of the HsIV dodecamer. The conformation of the protomers of the HsIU( $\Delta$ )-complexed HsIV hexamer is similar to that of the HsIU-complexed protomers in the symmetric, wild-type HsIU complex; the protomer conformation of the uncomplexed HsIV hexamer is similar to that found in the structure of HsIV alone. Reaction in the crystals with a vinyl sulfone inhibitor reveals that the HsIU( $\Delta$ )-complexed HsIV hexamer is active, while the uncomplexed HsIV hexamer is inactive. These results confirm that HsIV can be activated by binding of a hexameric HsIU( $\Delta$ )<sub>6</sub> ring lacking the I domains, that activation is effected through a conformational change in HsIV rather than through alteration of the size of the entry channel into the protease catalytic cavity, and that the two HsIV<sub>6</sub> rings in the protease dodecamer are activated independently rather than cooperatively.

POSTER # 29

**X-ray Absorption Edge and EXAFS Studies of the Red Copper Site in Nitrosocyanin: Comparison to Plastocyanin**R. Sarangi,<sup>1</sup> L. Basumallick,<sup>1</sup> B. Elmore,<sup>2</sup> A. Hooper,<sup>2</sup> K. O. Hodgson,<sup>3</sup> B. Hedman,<sup>3</sup> and E. I. Solomon<sup>1</sup><sup>1</sup>Department of Chemistry, Stanford University, Stanford, CA 94305, USA<sup>2</sup>Department of Biochemistry, Molecular Biology and Biophysics, University of Minnesota, St. Paul, MN 55108, USA<sup>3</sup>Stanford Synchrotron Radiation Laboratory, SLAC, Stanford University, Stanford, CA 94309, USA

Nitrosocyanin (NC) is a soluble red copper protein, with a mononuclear Cu site, from the autotrophic bacterium *Nitrosomonas europae*. The NC monomer has some sequence homology to blue-copper proteins and contains one Cu-S (Cys) and two Cu-N (His) bonds in parallel to blue copper (BC) sites. The spectroscopy characteristics, however, are drastically different from that of the well-understood BC proteins. NC is brilliant red in color with an intense absorption band at 390 nm unlike the ~600 nm absorption band of BC proteins. Its redox potential is +85 mV, which is much lower than the +184 to +680 mV range for BC proteins. This study is aimed at elucidating the active site structure of NC using EXAFS, and at the understanding of structure-function correlation through a detailed edge analysis of the Cu K- and S K-edge structures.

The crystal structures of NC in both its oxidized (1.65 Å) and reduced (2.3 Å) forms are known. Results from EXAFS analysis of data for both the oxidized and reduced forms corroborate the crystallographically determined structures. The XAS Cu K-edge 1s@4p shakedown transition energy and intensity for the reduced form are consistent with a 3-coordinate Cu<sup>1+</sup> site. A quantitative comparison to plastocyanin, a classic BC protein, has been made to understand the ligand field strength and the  $Z_{\text{eff}}$  of the Cu<sup>2+</sup> form. Finally, S K-edge pre-edge analysis sheds light on the extent of Cu-S(cys) overlap and its correlation to protein function.

This work was performed at SSRL, which is funded by the Department of Energy, Basic Energy Sciences. The SSRL Structural Molecular Biology Program is supported by the National Institutes of Health, National Center for Research Resources, Biomedical Technology Program, and by the Department of Energy, Office of Biological and Environmental Research.

graduate student submission

POSTER # 30

**X-ray Diffraction on BaBiO<sub>3</sub>**S. Larochele<sup>1</sup> and M. Greven<sup>2,3</sup><sup>1</sup>Department of Physics, Stanford University, Stanford, CA 94305, USA<sup>2</sup>Department of Applied Physics, Stanford University, Stanford, CA 94305, USA<sup>3</sup>Stanford Synchrotron Radiation Laboratory, SLAC, Stanford University, Stanford, CA 94309, USA

BaBiO<sub>3</sub> is a semiconducting material with a highly distorted perovskite structure with two bismuth sites. Its transport properties are thought to be due to charge ordering of the bismuth ions (Bi<sup>3+</sup> and Bi<sup>5+</sup>). We have used resonant x-ray powder diffraction to measure the site-specific anomalous factors  $f'(E)$  of the two bismuth sites around the Bi L(III) edge in order to establish the spectroscopic differences between the two sites. Furthermore, using high resolution powder diffraction, we have established that the structure is monoclinic, albeit with rather significant anisotropic strain broadening.

graduate student submission

POSTER # 31

### Evidence for a Universal Relationship between Magnetization and Changes in the Local Structure of La Manganites

L. M. Downward,<sup>1</sup> F. Bridges,<sup>1</sup> S. Bushart,<sup>1</sup> J. Neumeier,<sup>2</sup> and L. Zhou<sup>3</sup>

<sup>1</sup>Physics Department, University of California at Santa Cruz, Santa Cruz, CA 95064, USA

<sup>2</sup>Physics Department, Montana State University, Bozeman, MT 59717, USA

<sup>3</sup>Stanford Synchrotron Radiation Laboratory, SLAC, Stanford University, Stanford, CA 94309, USA

We present XAFS data as a function of applied magnetic field (up to 11T) and temperature to investigate changes in the local structure of CMR manganites with varying Ca concentrations,  $x$  ( $\text{La}_{1-x}\text{Ca}_x\text{MnO}_3$ ). We have modeled the disorder using a single parameter, the width ( $\sigma$ ) of the Mn-O pair distribution function. For temperatures near the ferromagnetic transition temperature,  $T_c$ , the application of a magnetic field makes the sample become more ordered. In our earlier work, we showed that the change in the Mn-O distortions associated with polarons ( $\sigma^2$ ), is approximately logarithmically related to the magnetization,  $M - \ln[\sigma^2] = A M/M_0 + B$ . However in those measurements the magnetization was varied by changing the temperature. In the new measurements, at a fixed temperature the magnetization is changed by applying a magnetic field. The data fall along the same line in the plot of  $\ln[\sigma^2] = A M/M_0 + B$ . This suggests that a more universal relationship exists between the magnetization and the local distortions, which likely depends on the number of magnetized neighboring sites. A new model for the development of sample magnetization below  $T_c$  is also presented. In this model the sample distortion increases slowly with magnetization at first, as an undistorted and distorted site pair up and become magnetized; then faster once no undistorted hole sites remain.

graduate student submission

POSTER # 32

### Comparison of $\Delta nifB$ and $\Delta nifH$ MoFe Proteins by X-ray Absorption Spectroscopy with Implications for P-cluster Biosynthesis

M. L. Campbell,<sup>1</sup> Y. Hu,<sup>2</sup> F. Naderi,<sup>2</sup> M. W. Ribbe,<sup>2</sup> B. Hedman,<sup>3</sup> and K. O. Hodgson<sup>1,3</sup>

<sup>1</sup>Department of Chemistry, Stanford University, Stanford, CA 94305, USA

<sup>2</sup>Department of Molecular Biology and Biochemistry, University of California at Irvine, Irvine, CA 92697-3900, USA

<sup>3</sup>Stanford Synchrotron Radiation Laboratory, SLAC, Stanford University, Stanford, CA 94309, USA

Two *Azotobacter vinelandii* nitrogenase MoFe protein mutants, expressed by the deletion of either the *nifH* or *nifB* gene, have been studied. Mutants of these types are iron-molybdenum cofactor deficient yet presumably contain the [8Fe-7S] P-cluster.  $\Delta nifB$  mutants can be directly activated *in vitro* by the addition of isolated FeMo-co, whereas  $\Delta nifH$  mutants cannot. Crystallographic data support the presence of a "normal" P-cluster, similar to that observed in wild-type MoFe protein, in the  $\Delta nifB$  protein. EPR studies suggest that the cluster in the  $\Delta nifH$  protein could be a P-cluster precursor. We will report the details of the electronic and structural differences between these two mutants as determined by Fe K-edge x-ray absorption and extended fine structure. The results of these studies support the presence of a "normal" P-cluster in the  $\Delta nifB$ - protein and identify a novel Fe-S cluster, akin to a [4Fe-4S] cluster, in the  $\Delta nifH$  protein that potentially represents a biosynthetic precursor to the P-cluster.

This work was performed at the SSRL, which is funded by the DOE Office of Basic Energy Sciences. The SSRL Structural Molecular Biology Program is supported by the NIH National Center for Research Resources, Biomedical Technology Program and by the DOE Office of Biological and Environmental Research.

graduate student submission

POSTER # 33

**Conformational Changes during Nucleotide Hydrolysis Cycle of P97 Probed by Small-Angle X-ray Scattering**J. Davies,<sup>1</sup> H. Tsuruta,<sup>2</sup> and W. Weis<sup>1</sup><sup>1</sup>Department of Structural Biology, Stanford University School of Medicine, Stanford, CA 94305, USA<sup>2</sup>Stanford Synchrotron Radiation Laboratory, SLAC, Stanford University, Stanford, CA 94309, USA

Valosin-containing protein (VCP)/p97 is an AAA ATPase that has been implicated in a variety of cellular processes, including homotypic fusion of membranes and ubiquitin-proteasome mediated protein degradation. VCP/p97 is a homohexamer whose protomers consist of an N-terminal (N) domain responsible for binding to effector proteins, followed by two AAA ATPase domains. The various roles of VCP/p97 are facilitated through binding of the N-domain to one of a collection of adaptor proteins, each adaptor being responsible for a specific cellular task. It is likely that VCP/p97 accomplishes its varied functions by converting the energy of ATP hydrolysis into conformational changes that allow mechanical work to be performed on the bound effector protein. It is thus important to understand how the individual domains move during the ATPase cycle.

To this end, small-angle x-ray scattering (SAXS) studies have been undertaken to monitor changes between the four states in hydrolysis: no nucleotide, ATP-bound, ADP-Pi-bound, and ADP-bound. It was observed that the major change in conformation, as measured by changes in the radius of gyration, arises upon binding of nucleotide to the enzyme. Preliminary modeling of the SAXS data indicates that the major changes are due to the N-domains opening out into solution, with some accompanying movement of the middle portion of the hexameric ring. By elucidating the kinetics and conformational changes produced by nucleotide hydrolysis, it is hoped that the underlying mechanism of action for VCP/p97 might be better understood.

graduate student submission

POSTER # 34

**Oxidation of Fractured Surfaces of FeS<sub>2</sub>(100) by Molecular Oxygen, Water Vapor, and Air**T. Kendelewicz,<sup>1</sup> C. S. Doyle,<sup>1</sup> B. C. Bostick,<sup>3</sup> and G. E. Brown, Jr.<sup>1,2</sup><sup>1</sup>Department of Geological & Environmental Sciences, Stanford University, Stanford, CA 94305-2115, USA<sup>2</sup>Stanford Synchrotron Radiation Laboratory, SLAC, Stanford University, Stanford, CA 94309, USA<sup>3</sup>Department of Earth Sciences, Dartmouth College, Hanover, NH 03755, USA

Synchrotron-based photoelectron spectroscopy was used to study the oxidation of UHV-fractured (100) surfaces of pyrite by molecular oxygen, water vapor, and air. Molecular oxygen reacts appreciably with pyrite surface only for high doses of ~107 L, while reaction with water vapor does not take place even at doses as high as 1010 L. For high O<sub>2</sub> partial pressures the extent of reaction with oxygen is comparable to equivalent air exposures. We show that the presence of water is not strictly necessary for oxidation of pyrite (100) to take place in our experiments. Intermediate oxidation products, quantified by spectral decomposition as a function of dose, are discussed.

POSTER # 35

**Soft X-ray Spectroscopic Studies of the Reaction of Fractured Pyrite Surfaces with Cr(VI)-containing Aqueous Solutions**

C. S. Doyle,<sup>1</sup> T. Kendelewicz,<sup>1</sup> B. C. Bostick,<sup>1,3</sup> and G. E. Brown, Jr.<sup>1,2</sup>

<sup>1</sup>Department of Geological & Environmental Sciences, Stanford University, Stanford, CA 94305-2115, USA

<sup>2</sup>Stanford Synchrotron Radiation Laboratory, SLAC, Stanford University, Stanford, CA 94309, USA

<sup>3</sup>Department of Earth Sciences, Dartmouth College, Hanover, NH 03755, USA

We have used synchrotron-based soft x-ray core-level photoemission and adsorption spectroscopies to study the reaction of aqueous sodium chromate solutions with freshly fractured pyrite surfaces. The study has two complementary goals: (1) to investigate the oxidation of pyrite surfaces and identify reaction intermediates, and (2) to assess pyrite as a potential material for the reductive removal and immobilization of hexavalent chromium from aqueous solution. Pyrite surfaces were reacted with 50  $\mu\text{M}$  sodium chromate solution at pH 7 for reaction times between one minute and thirty-seven hours. Additional experiments were performed at pH 2 and pH 4 with 50  $\mu\text{M}$  sodium chromate solutions, and at pH 7 with 5 mM solutions. At chromate concentrations of 50  $\mu\text{M}$ , all chromate present on the pyrite surface was in the form of Cr(III), while at 5 mM, both Cr(III) and Cr(VI) were present at the pyrite surface. Minor quantities of oxidized sulfur species (sulfate, sulfite, and zero-valent sulfur) were identified as reaction products on the pyrite surface. The amount of oxidized sulfur species observed on the surface was greater when pyrite was reacted with 5 mM Cr(VI) solutions because the rate of chromium deposition exceeded the rate of dissolution of pyrite oxidation products, effectively trapping Cr(VI) and oxidized sulfur species in an overlayer of iron(III)-containing Cr(III)-hydroxide. This work shows that pyrite, an extremely cheap and readily available waste material, may be suitable for the removal of hexavalent chromium from acidic to circumneutral waste streams. The reduced chromium ultimately forms a coating on the pyrite surface, which passivates the pyrite surface towards further oxidation.

graduate student submission

POSTER # 36

**From Structure to Pathogenesis: HLA-DQ2 and Celiac Disease**

C.-Y. Kim,<sup>1</sup> H. Quarste,<sup>4</sup> E. Bergseng,<sup>4</sup> C. Khosla,<sup>1,2,3</sup> and L. M. Sollid<sup>4</sup>

<sup>1</sup>Department of Chemical Engineering, Stanford University, Stanford, CA 94305, USA

<sup>2</sup>Department of Chemistry, Stanford University, Stanford, CA 94305, USA

<sup>3</sup>Department of Biochemistry, Stanford University, Stanford, CA 94305, USA

<sup>4</sup>Institute of Immunology, University of Oslo, N-0316 Oslo, Norway

Celiac Disease is a gluten-induced autoimmune disorder of the small intestine associated with HLA-DQ2. The structure of HLA-DQ2 complexed with an immunodominant epitope from gluten has been determined to atomic resolution by x-ray crystallography, and provides insight into why this disease is DQ2-associated. Furthermore, the binding of this Pro-rich peptide demonstrates a new type of sequence-specific binding of peptides to class II MHC molecules.

POSTER # 37

**New Small Angle Scattering/Diffraction Instrument on BL4-2 for Structural Molecular Biology**

H. Tsuruta, I. Smolsky, and K. Ito

Stanford Synchrotron Radiation Laboratory, SLAC, Stanford University, Stanford, CA 94309, USA

During the SPEAR3 upgrade, we are building a new small-angle scattering/diffraction instrument for structure biology studies on BL4-2, which is now dedicated 100% for this class of studies. The new instrument has been designed to take full advantage of the high brilliance SPEAR3 beam. The anticipated flux in the typical sample area (0.5mmx1.5mm) is  $10^{12}$  ph/s in FY2003 with the Si(111) monochromator and  $10^{13}$  when we complete the BL4 optics upgrade. The flux level will be higher at least by one order of magnitude in both cases when the multilayer monochromator is used. The new instrument also incorporates many of existing and new features. Two most notable features are: 1) built-in Bonse-Hart geometry ultra-small angle scattering set up, based on our recent USAXS development; 2) automated sample-to-detector distance selection (0.5-2.5m). The new instrument will cover characteristic lengths ranging from a few Angstroms to a few micrometers.

The new USAXS geometry employs the 5-bounce Si(111) channel-cut analyzer crystal along with the avalanche photodiode, both carried over from the existing instrument. The analyzer crystal will be mounted inside the most downstream section of the vacuum flight path. It will be moved out of way by a sliding mechanism for regular SAXS/D data collection. Beam collimation will be made by a 4- or 6-bounce Si(111) channel-cut crystal located at an upstream location. This crystal is also retractable for high-flux SAXS/D studies, but can remain in place to clean up tailing edge of the vertical beam profile for regular SAXS/D experiments. We expect the level of USAXS performance comparable to the existing instrument, achieving the small angle resolution of  $\sim 1.3$  micrometers.

The new instrument has a segmented vacuum flight path, made up of five separate sections. All but the most downstream section has a motorized sliding mechanism underneath. The sample stage and the upstream vacuum flange share the same optical rail, which sits on a motorized sliding mechanism for moving these components together automatically in the beam direction. Our goal is to provide quick change of sample-to-detector distance within 10 minutes, including the fine adjustment of sample position and slit opening, without staff support.

POSTER # 38

**Structural Studies Reveal Thymidylate Synthase Complementing Protein as a Target for Multiple Diseases**

I. I. Mathews,<sup>1</sup> A. M. Deacon,<sup>1,2</sup> J. M. Canaves,<sup>2,3</sup> D. McMullan,<sup>2,4</sup> S. A. Lesley,<sup>2,4</sup> S. Agarwalla,<sup>5</sup> and P. Kuhn<sup>2,6</sup>

<sup>1</sup>Stanford Synchrotron Radiation Laboratory, SLAC, Stanford University, Stanford, CA 94309, USA

<sup>2</sup>The Joint Center for Structural Genomics, Stanford University, Stanford, CA 94309, USA

<sup>3</sup>The San Diego Supercomputer Center, 9500 Gilman Drive, La Jolla, CA 92093, USA

<sup>4</sup>The Genomics Institute of Novartis Foundation, 10675 John Jay Hopkins Dr., San Diego, CA 92121, USA

<sup>5</sup>Department of Biochemistry and Biophysics, University of California at San Francisco, San Francisco, CA 94143, USA

<sup>6</sup>The Scripps Research Institute, 10550 N. Torrey Pines Rd, La Jolla, CA 92037, USA

Like thymidylate synthase (TS) in eukaryotes, the thymidylate synthase complementing proteins (TSCP) are essential for cell survival of many prokaryotes in the absence of external sources of thymidylate. TSCP's employ a flavin-dependent reaction for the thymidylate synthesis [1,2] and the details of the mechanism of this novel family of enzymes are unknown. Here, we report the detailed structural and functional analysis of a TSCP enzyme from *Thermotoga maritima* [2]. Multiple complexes with and without FAD, substrate and substrate analogs have mapped out many of the principal binding determinants of this novel enzyme class. The structures presented here provide a basis for rationalizing the TSCP catalysis and reveal the possibility of the design of an inhibitor. Since many pathogenic bacteria depend on TSCP/thy1 for survival, an inhibitor for these enzymes would be a highly effective antibacterial drug. Furthermore, the absence of structural or sequence homology between TSCP enzymes and classical thymidylate synthase offers the possibility of developing very specific TSCP inhibitors with low cross-reactivity with the TS enzymes and, hence, reduced toxic effects. We have also identified a new helix-loop-strand FAD-binding motif characteristic of the enzymes in the TSCP family. The presence of a hydrophobic core with residues conserved among the TSCP family suggests a common overall fold. Therefore, TSCP enzymes provide a unique alternative for the development of antimicrobials capable of simultaneously targeting a wide range of pathogenic organisms. TSCP inhibitors also have important implications for Homeland Security and the development of therapeutic approaches to combat potential bioterrorism. The fact that most of the bioagents containing TSCP have different mechanisms of infection (anthrax, botulism, epidemic typhus, borreliosis, spotted fever, gas gangrene, etc) complicates prevention and treatment.

1. Myllykallio, H., Lipowski, G., Leduc, D., *et al.*, (2002) *Science*, 297, 105-107.
2. Mathews, I. I., Deacon, A. M., Canaves, J. M., *et al.*, (2003) *Structure*, 11, 677-690.

POSTER # 39

**Conceptual Scheme for a High Peak Energy Inertial Confinement Fusion (ICF) X-ray Ignition Facility**R. Tatchyn<sup>1</sup> and S. Shlyaptsev<sup>2</sup><sup>1</sup>Stanford Synchrotron Radiation Laboratory, SLAC, Stanford University, Stanford, CA 94309, USA<sup>2</sup>Lawrence Livermore National Laboratory, Livermore, CA 94550, USA

In recent work systematic simulations of ignition threshold processes in solid Inertial Confinement Fusion (ICF) targets have indicated that efficient coupling of ignition energy to a typical compressed target may be realizable using soft x-ray photons in the 0.6- 0.2 nm range. At these wavelengths virtually all the radiation energy would be absorbed in, say, a solid compressed D-T target of radius ~25 microns and a compression factor of ~600. The total energy requirement, of the order of 100-1000 Joules, which should be efficiently delivered into the target volume, has led us to consider whether or not the associated phase space requirements could be met with an appropriately designed synchrotron radiation source. One noteworthy aspect of our investigation is that in the past few decades' push toward "4th Generation" sources, the "10-fold or more" increase in the total energy within a single photon pulse has been the one phase space parameter that has heretofore received little or no attention. In this work we present basic design concepts for an ICF X-ray igniter facility based on moderate-energy (1-2 GeV) Energy Recovery Linacs (ERLs) using photocathode RF guns, ultra-long wigglers, and special X-ray mirrors. The facility, which could attain per-pulse energies of up to several hundred Joules, is shown to be of the same order of scale as the DESY TESLA FEL project and associated with similar areas of research and development. If constructed, the igniter facility would exceed the per-pulse energy of any existing or proposed synchrotron radiation source by 3-4 orders of magnitude.

POSTER # 40

**Inexpensive Compound Refractive Lenses for X-ray Imaging and Microspot Focusing**M. A. Piestrup,<sup>1</sup> Y. I. Dudchik,<sup>2</sup> N. N. Kolchevsky,<sup>2</sup> F. F. Komarov,<sup>2</sup> J. T. Cremer,<sup>1</sup> C. Gary,<sup>1</sup> and R. H. Pantell<sup>3</sup><sup>1</sup>Adelphi Technology, Inc., Palo Alto, CA 94306, USA<sup>2</sup>Institute of Applied Physics Problems, Kurchatova 7, 220064, Minsk, Belarus<sup>3</sup>Department of Electrical Engineering, Stanford University, Stanford California, 94305, USA

We have fabricated and tested short focal-length compound refractive lenses (CRLs) composed of micro-bubbles embedded in epoxy. The interface between the bubbles formed 90 to 350 spherical bi-concave microlenses reducing the overall focal length inversely by the number of lenses. When compared with CRLs manufactured using other methods, the micro-bubble lenses have shorter focal lengths with higher transmissions and gains for moderate energy X-rays (e.g. 5 – 30 keV). In addition, lens surface quality is extremely good resulting in high resolution imaging when the CRL is used for magnification. We used beamline 2-3 at the Stanford Synchrotron Radiation Laboratory (SSRL) and beamline DND-CAT at the Advanced Photon Source (APS) to measure focal lengths between 100-250 mm and absorption apertures between 90 to 200 microns. Transmission profiles were measured giving, for example, a peak transmission of 46 % for a 25-cm focal length CRL at 20 keV. The focal-spot sizes were also measured yielding, for example, a vertical spot size of 1.4 microns resulting from an 18-fold demagnification of the 25-micron DND-CAT source size. The micro-bubble technique opens a new opportunity for designing lenses in the 5-30 keV range with focal lengths as low as 30 mm. These lenses compare favorably with Fresnel Zone Plates and cost much less. These inexpensive lenses are available for sale at Adelphi Technology, Inc.

POSTER # 41

### **STXM Characterization of Spin Injection Samples**

S. D. Andrews,<sup>1</sup> Y. Acremann,<sup>2</sup> J. Strachan,<sup>1</sup> V. Chembrolu,<sup>1</sup> G. Wang,<sup>1</sup> Y. Zhao,<sup>4</sup> T. Tyliczszak,<sup>3</sup> B. Clemens,<sup>1</sup> and J. Stöhr<sup>2</sup>

<sup>1</sup>Department of Materials Science and Engineering, Stanford University, Stanford, CA 94305, USA

<sup>2</sup>Stanford Synchrotron Radiation Laboratory, SLAC, Stanford University, Stanford, CA 94309, USA

<sup>3</sup>Advanced Light Source, Lawrence Berkeley National Laboratory, Berkeley, CA 94720, USA

<sup>4</sup>Department of Materials Science and Engineering, Cornell University, Ithaca, NY 14850, USA

Recent experiments show that a spin polarized current injected into a ferromagnet can influence the magnetization. But so far, the dynamic response of the magnetization to a spin current pulse as well as the switching behavior on the nanometer scale is unknown. The high spatial resolution and the ability to obtain chemical and magnetic information of buried layers make the scanning transmission x-ray microscope (STXM) the ideal tool to investigate spinelectronic devices.

Our samples consist of magnetic pillars embedded in an insulating film. The entire structure is on a SiN membrane to allow x-ray transmission. We report first STXM measurements showing that the instrument will allow us to observe magnetization reversal by spin injection. Furthermore, micro-spectroscopy on a scale of 50 nm is an important diagnostic tool for the sample fabrication process.

graduate student poster

POSTER #42

**SESAME, a 3<sup>rd</sup> Generation Synchrotron Light Source for the Middle East Region**R. Boyce,<sup>1</sup> D. Ernst,<sup>1</sup> M. Molloy,<sup>2</sup> H. Morales,<sup>1</sup> T. Rabedeau,<sup>1</sup> B. Scott,<sup>1</sup> M. Widmeyer,<sup>1</sup> and H. Winick<sup>1</sup><sup>1</sup>Stanford Synchrotron Radiation Laboratory, SLAC, Stanford University, Stanford, CA 94309<sup>2</sup>DOE Site Office, SLAC, Menlo Park, CA 94025

Developed under the auspices of UNESCO, SESAME (**S**ynchrotron-light for **E**xperimental **S**cience and **A**pplications in the **M**iddle **E**ast; [www.sesame.org.jo](http://www.sesame.org.jo)) is being established as a major international research centre in the Middle East/Mediterranean region. It will have as its centrepiece a 2.5 GeV 3<sup>rd</sup> Generation synchrotron light source with 13 straight sections for insertion devices and an emittance of 25 nmrad [1,2]. The project emerged from a German gift of the 0.8 GeV BESSY I facility, evolving from an initial plan for a 1 GeV ring [3]. Capital funds for the ring, beam lines, and essential user-support laboratories are being sought from the EU, Japan, the USA and other sources. In addition, gifts of ring and beam line equipment are being solicited from existing synchrotron radiation facilities. It is planned to send to SESAME equipment from SPEAR2 that will not be needed for SPEAR3. This includes wiggler magnets, power supplies, vacuum components and beam line equipment as these become available.

As of July 2003, Members of SESAME are Bahrain, Egypt, Iran, Israel, Jordan, Pakistan, Palestinian Authority, Turkey and the United Arab Emirates, with a total population of over 305 millions. Members provide the annual operations budget and more are expected to join. SESAME is located in Allan, Jordan, 30 km North-West of Amman. Jordan is providing a site and funds for construction of the building, which began in July 2003. Plans for the initial beam lines and scientific program include MAD Protein Crystallography, SAXS and WAXS for polymers and proteins, Powder Diffraction for material science, UV/VUV/SXR Photoelectron Spectroscopy and Photoabsorption Spectroscopy, IR Spectroscopy, and XAFS.

An accelerator group is being formed from among 19 Middle East scientists and engineers who have completed 2 years of training at European synchrotron radiation laboratories. Five scientific workshops and schools and a Users' meeting (reports on the web site) have been held. Several hundred regional users are expected at the start of operation in 2008, growing to about one thousand or more after a few years.

*References*

- [1] D. EINFELD, R.H. SARRAF, M. ATTAL, K. TAVAKOLI, H. HASHEMI, H. HASSANZADEGAN, E. AL-DMOUR, B. KALANTARI, A. ALADWAN, H. TARAWNEH, S. VARNASERY: SESAME, A 2.5 GeV Synchrotron Light Source for the Middle East Region. Presented at PAC2003; Available on-line at [http://warrior.lbl.gov:7778/pacfiles/papers/TUESDAY/PM\\_ORAL/TOPA006/TOPA006.PDF](http://warrior.lbl.gov:7778/pacfiles/papers/TUESDAY/PM_ORAL/TOPA006/TOPA006.PDF)
- [2] D. EINFELD, H. SCHOPPER, Z. SAYERS, S.HASNAIN, H. WINICK: SESAME, A 3<sup>rd</sup> Generation Synchrotron Light Source for the Middle East Region; To be published by AIP as Proc. of SRI2003.
- [3] G.-A. VOSS, T. RABEDEAU, S. RAITHER, H. SCHOPPER, E. WEIHRETER, H. WINICK: SESAME - An extended spectral range synchrotron radiation facility in the Middle East based on an upgrade of BESSY I, Nucl. Instr. & Meth. A 467-468 (2001) 55-58.



Stanford Linear Accelerator Center  
Stanford Synchrotron Radiation Laboratory  
2575 Sand Hill Road  
Menlo Park, California 94025  
Tel:650-926-4000

<http://www-ssrl.slac.stanford.edu>

