

Soft X-ray microscopy and spectroscopy at the molecular environmental science beamline at the Advanced Light Source

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Abstract

We present examples of the application of synchrotron-based spectroscopies and microscopies to environmentally relevant samples. The experiments were performed at the molecular environmental science beamline (11.0.2) at the Advanced Light Source, Lawrence Berkeley National Laboratory. Examples range from the study of water monolayers on Pt(1 1 1) single crystal surfaces using X-ray emission spectroscopy and the examination of alkali halide solution/water vapor interfaces using ambient pressure photoemission spectroscopy, to the investigation of actinides, river water biofilms, Al-containing colloids and mineral–bacteria suspensions using scanning transmission X-ray spectromicroscopy. The results of our experiments show that spectroscopy and microscopy in the soft X-ray energy range are excellent tools for the investigation of environmentally relevant samples under realistic conditions, i.e., with water or water vapor present at ambient temperature.

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1. Introduction

Molecular environmental science (MES) is an expanding field that studies, on a molecular level, chemical and biological processes occurring at environmentally relevant surfaces and interfaces. Fundamental understanding of these processes is necessary to solve important problems in environmental science related to the speciation, distribution, reactivity, transformations, mobility, and potential bioavailability of pollutants and contam-

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inants in the environment. Synchrotron-based spectroscopies enable the study of, e.g., aqueous solute complexes, amorphous and crystalline materials, solid/liquid, and liquid/vapor interfaces, as well as the interaction of heavy metals with biofilms and plants [1]. With the growth of the MES community, the demand for dedicated beamlines and endstations for conducting MES research has been increasing over the last decade. The MES beamline at the Advanced Light Source (ALS), Lawrence Berkeley National Laboratory, was designed with the goal of providing access for the MES community to different kinds of synchrotron-based techniques in the soft X-ray energy range (75–2150 eV), in particular scanning transmission X-ray microscopy (STXM), ambient pressure photoemission spectroscopy (PES), near-edge X-ray absorption fine structure (NEXAFS) spectroscopy, and X-ray emission spectroscopy (XES). The energy range of the MES beamline coincides with the K-edges of the most abundant elements on Earth's surface (C, N, O, Na, Mg, Al, and Si) and the L-edges of all the important transition metals (Ti, V, Cr, Mn, Fe, Co, Ni, Cu, and Zn) as well as the L-edges of P, S, Cl, K, and Ca. For the heavier elements there are other absorption edges, which can be accessed at the MES beamline. The three permanent endstations at the beamline allow investigations of samples under different environments, from UHV surface-science-type studies of well-defined surfaces, to spectromicroscopy studies of complex samples, including mixtures of inorganic solids, natural organic matter, and microorganisms under wet conditions.

One of the areas of special importance for MES is the interaction of water with surfaces, starting from the adsorption of monomers, to the formation of monolayers and eventually the presence of bulk water and its reaction with the surface [1–3]. Water plays a very important role in the distribution of contaminants and pollutants that originate from, e.g., agriculture, mining, industrial production, and radioactive waste disposal sites. Most of the chemical reactions that are important for environmental processes take place at the solid–liquid interface between water or aqueous solutions and metals, minerals, soils, and rocks. The water solubility of contaminants and pollutants also greatly influences their mobility. An important task for the MES beamline is therefore the investigation of environmentally relevant samples under “wet conditions”, which is also essential for the study of biological samples that can be kept fully hydrated if they are mounted in an aqueous solution. STXM [4] is ideally suited to investigate small particles, membranes, or even cells and whole bacteria immersed in aqueous solution. Since photons have an attenuation length in liquid water on the order of several hundred nanometers at energies below and above the oxygen K-edge, imaging samples in bulk solution is possible, if the thickness of the sample is in the sub-micrometer range. STXM also allows one to obtain NEXAFS spectra with a spatial resolution of 30 nm [5], so that the chemical state and the morphology of the sample can be directly correlated. In addition to STXM, XES [6] as a photon-in-photon-out technique is well suited for studies of bulk aqueous solution samples in an atom-specific way. Ambient pressure PES [7] on the other hand, is an excellent tool for the investigation of the solution/vapor interface, where aqueous solution samples

can be studied in thermodynamic equilibrium with their vapor. It is also a powerful method for studying the initial stages of water adsorption on surfaces under ambient conditions, e.g., determining the number of monolayers of water on the surface as a function of relative humidity. The combination of PES with XES and NEXAFS provides information about the electronic and geometric structures of water layers on solid surfaces, in particular the chemical bonding of water molecules to the substrate and the hydrogen bonding network within the water layer [8].

Another area of special importance to MES is the investigation of radioactive materials, in particular actinides, to understand the sequestration of these materials in complex natural environments. STXM offers the opportunity to investigate radioactive samples in an efficient and safe way, since the amount of material that is needed in the experiments is on the order of only 10 fg.

In the following section, we discuss the setup and properties of the beamline and the dedicated endstations. In the main part of the manuscript, we present examples for the application of synchrotron-based soft X-ray spectroscopy and microscopy to the study of environmental materials and processes. The examples cover a wide range of MES research, among them fundamental studies of well-defined adsorbate layers on single crystalline surfaces under ultra-high vacuum conditions, alkali halide solutions in the presence of water vapor, uranium oxide particles, as well as biofilms, bacterial-mineral samples, and aluminum flocs in aqueous solutions.

2. Description of experimental facilities

2.1. Beamline

The design and the properties of the ALS-MES beamline 11.0.2 will be explained in detail in a forthcoming paper. Here, we describe the main design features of the beamline in brief. A schematic overview of the main beamline components is shown in Fig. 1. The MES beamline uses photons from an elliptically polarizing undulator with a 5 cm period [9]. With the Advanced Light Source operating at an energy of 1.9 GeV it provides photons in the energy range from 75 to 2150 eV. The photons from the undulator are focused vertically by a sagittal cylinder mirror onto the pre-mirror of a SX700 style plane-grating monochromator [9–11]. There is no entrance slit in front of the monochromator. The monochromator is equipped with two gratings (150 and 1200 lines/mm). The whole monochromator tank is moved when the gratings are changed. The light from the monochromator is focused by either of two toroidal focus mirrors that generate a stigmatic focus at the exit slits of either the STXM or the spectroscopy branch line. The branch line is selected by the position of the spectroscopy mirror either in or out of the beam path. The exit slits are illumination sources for the zone plate of the STXM, or a pair of Kirkpatrick–Baez (KB) mirrors in the spectroscopy branch line. The KB mirrors (horizontal and vertical) are able to focus the spot in the experimental chambers down to $7\ \mu\text{m} \times 10\ \mu\text{m}$.

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