Pacific Northwest NATIONAL LABORATORY

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APPLICATIONS

Areas in which RSSL capabilities may be applied include:

Energy: Understanding the properties and behavior of advanced fuels, such as migration and seg-segregation of fission and activation products in spent fuel; investigating orrosion and degradation of plant and pipe-line components; and examin-ation and diagnostic testing of materials.

National and Homeland Security: Attribution; forensics; interactionof radionuclides with urban surfaces; and analysis of materials for weapons production, maintenance and disassembly.

Environmental: Interactions of actinides and other radionuclides with mineral and engineered barrier surfaces; analysis of waste forms; and speciation of high-level-waste solids components.

Biological: Interactions of actinides and other radionuclides with biological materials, such as bone and tissues.

U.S. DEPARTMENT OF ENERGY

PACIFIC NORTHWEST NATIONAL LABORATORY

PNNL's Radiological Surface Science Laboratory

A Unique Facility for Surface and Interface Analysis of Radiological Samples

The Physical Electronics 560 Surface Analysis System, one of the Radiological Surface Science Laboratory's three surface analysis systems, may be used for AES, XPS and SIMS experiments on radioactive samples.

The Radiological Surface Science Laboratory (RSSL) is a unique facility that provides an array of surface analysis instruments for the examination of radioactive samples. Surface and interface analysis tools have been used extensively and with great success over the past 50 years in corrosion, geochemistry, catalysis, electronics, tribology, and many other areas. The RSSL now combines these powerful research tools with the ability to examine radiological samples, creating new opportunities for basic through applied research. The RSSL is located in PNNL's Radiochemical Processing Laboratory (http://rpl.pnl.gov/), giving direct access to radiological labs and hot cells, where highly active and dispersible samples may be received, tested, and prepared for surface analysis.

RSSL SURFACE ANALYSIS CAPABILITIES

Auger Electron Spectroscopy (AES) provides elemental composition of the top 5 - 20 atomic layers of a sample. Spatial resolution is currently as high as 2 um; 0.1 um will soon be available. Analysis modes include: spot analysis; larger area average (up to 0.5 sq cm); line scan; and elemental mapping.

X-ray Photoelectron Spectroscopy (XPS) provides elemental composition and also oxidation state information of the top 5 - 20 atomic layers. The highest spatial resolution is currently 150 um - 250 um. Two different anodes are available: magnesium (1253.6 eV x-rays); and aluminum (1486.7 eV x-rays).

Secondary Ion Mass Spectrometry (SIMS) uses a rastered beam of mono-energetic ions (argon, krypton, xenon, or other) to create and eject ions from the surface. These ions are mass analyzed with a quadrupole mass spectrometer, giving an isotopic composition of the surface. This isextremely useful in the analysis of actinides in a sample, particularly when combined with the elemental and oxidation state information from XPS.

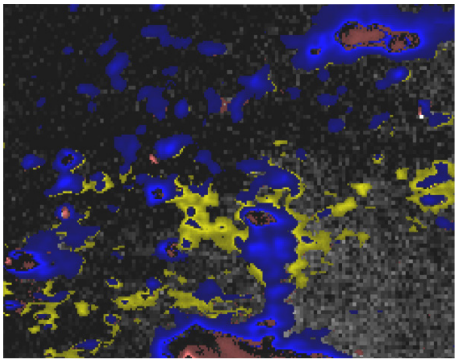
This closeup shows the tip of the cylindrical mirror analyzer (CMA) inside the Physical Electronics 560 Surface Analysis System. The CMA is used to collect AES and XPS data. Up to twelve samples can be staged inside the system for analysis.

Depth profiles, or plots of how the concentrations of different elements vary with depth, can be collected using any of these techniques, allowing investigation of buried interfaces. SIMS inherently creates a depth profile as the probe ions remove material from the surface; AES and XPS depth profiles may be collected by sputtering (bombarding with ions) between measurements.

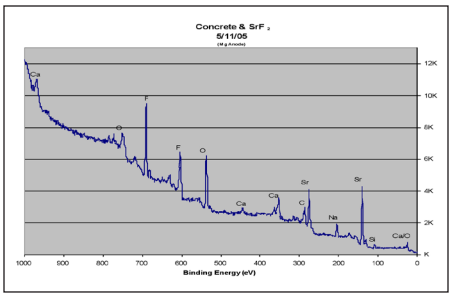
SYSTEMS FOR EXAMINATION OF RADIOACTIVE AND NON-RADIOACTIVE SAMPLES

Physical Electronics 660 Scanning Auger Microprobe – provides scanning AES, SIMS, and secondary-electron and absorbed-current imaging systems. The capabilities of this system are very similar to the 590 system, but the AES and imaging systems can resolve much smaller features, of nominally 100 nm. The system has an in situ fracture unit and a separate sputtering chamber for high-activity samples. A radiological containment box over the sample introduction port—plus a sample introduction airlock—allows high throughput of radiological samples.

Physical Electronics 590 – provides scanning AES and SIMS. Secondary-electron and absorbed-current imaging systems allow imaging of surfaces at the same spatial resolution in the surface plane as the scanning AES (2 um).



An elemental map produced using Auger Electron Spectroscopy shows strontium deposited on a concrete sample. Strontium is colored blue, calcium is white, silicon is red, and sodium is yellow. The association of strontium and silicon suggests that strontium is bound more strongly to the silicate minerals in the concrete than to the other concrete components.



An x-ray photoelectron spectrum shows the peaks that arise from the various elements in the strontium/concrete sample. Such data can be used to determine oxidation states and, in some cases, identify compounds of the elements observed. This, combined with the elemental maps, can be used to determine where the strontium binds to the concrete and can guide development of efficient decontamination technologies.

All instruments are focused on one point. This allows visual inspection, selection of a region of interest, sample analysis with either the AES or SIMS, and follow-up visual inspection, with no major sample movement or re-alignment. Samples can be heated before or during analysis. A radiological containment box over the sample introduction port, plus a sample introduction airlock, allow high through-put of radiological samples without breaking vacuum.

Kratos AXIS 165 – provides scanning AES, XPS, and SIMS, in addition to secondary-electron and absorbed-current imaging. Uses an aluminum anode for XPS, either as a flood source or monochromated, with spot size as small as 30 um. AES and electron imaging modes have a spatial resolution of < 1000 Å. A radiological containment box over the sample introduction port—plus a sample introduction airlock—allow high through-put of radiological samples.

Physical Electronics 560 – provides scanning AES, XPS, and SIMS, in addition to secondary-electron and absorbed-current imaging. The AES and imaging instruments have a spatial resolution of approximately 50 um. As with the Physical Electronics 590, all instruments in this system are focused on the same point inside the chamber, allowing analysis by all methods with minimal sample movement. Samples can be heated before or during analysis, and a radiological containment box over the sample introduction port—plus a sample introduction airlock—allow high throughput of radiological samples.

With the 560 system, various reaction chambers can be used, including one that allows reaction of solid samples with gases, an electrochemical reaction cell, and a dipping chamber that allows immersion of a solid sample in a liquid.

These ancillary chambers are sealed to the 560 system, allowing a sample to be cleaned and examined in the main vacuum chamber, transferred to one of the small chambers for reaction under controlled conditions, then returned to the main chamber for examination to determine the effects on the surface. The transfer chambers are backfilled with an inert gas to avoid contamination/reaction of the sample with onstituents in air.

All AES, XPS and SIMS systems are controlled with standard PC computer systems, allowing rapid data acquisition andease of file manipulation. Secure (Limited Area) laboratories and offices are available for classified data collection and analysis.

RESEARCH AREAS

Current areas of investigation with these systems include the speciation of uranium and other elements in weapons components and spent fuel, the interaction of potential contaminants (strontium, cesium, technetium, actinides) with urban and environmental surfaces (oxides and minerals, metals, polymers, etc.), analysis of tritium production related materials, speciation of metals in high-level waste simulants, and distribution of heavy metals in biological tissues.

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