NUCLEAR MATERIALS CHARACTERIZATION

Six critical roles your R&D team will want to know about.

Q&A with four experts at CAMECA:

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Anne-Sophie Robbes is product manager for EPMA instruments, SXFIVE-TACTIS and SKAPHIA product lines. She regularly gives advanced application training and presents at international conferences.



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Robert Ulfig is senior project manager, APT. He brings broad expertise in materials science, semiconductor manufacturing and technology to product development worldwide.

CAMECA is a global leader in elemental and isotopic microanalysis solutions for government, academic and industry labs. A business unit of AMETEK and four-time R&D 100 Awards recipient, CAMECA provides transformational characterization instruments for nuclear and other materials used in a broad array of applications. SKAPHIA is one of the company's new-generation instruments, a shielded Electron Probe that supports post-irradiation materials analysis and research. ACTINIS is the world's unique shielded magnetic sector SIMS, performing high-precision elemental and isotopic analyses of radioactive samples in a safe environment.

Ask any nuclear professional: Radiation is everywhere. While radioactive materials are often misunderstood, today's characterization techniques allow for superior detection and analysis and play a vital role in taking the mystery out of these materials. Nuclear scientists and engineers now have access to analytical instruments that enable the characterization of irradiated fuels. As a result, they can study its microstructure and composition to predict its behavior and develop strategies to prevent the release of radiation in accident scenarios.

Others need to perform high-precision elemental and isotopic analyses of radioactive samples in a safe environment. Advanced instruments are now available for that. Whatever the need, understanding these six critical roles will assist in identifying a complete solution to your nuclear materials characterization challenges.

1. Ensuring longevity of nuclear materials

Understanding the diffusion and migration mechanisms of fission products in nuclear fuel and structural materials is key to ensuring their longevity. For nuclear materials researchers, determining the behavior



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of noble gases produced by fission is of particular importance.

For example, low-solubility fission products such as xenon and krypton can agglomerate into bubbles that induce mechanical stress in the fuel pellets, or be released from the pellets, increasing pressure within the cladding. This second outcome can create serious safety issues. Also, helium migration and trapping may lead to void swelling, hardening or creep fracture, and negatively affect the microstructural and mechanical properties of structural materials.

Due to its outstanding depth-profiling and ion-imaging capabilities, dynamic Secondary Ion Mass Spectrometry (SIMS) is routinely used in the study of irradiation effects to better understand the migration behavior of fission products in nuclear materials. SIMS provides elemental as well as isotopic information ranging from low mass (H) to high-mass species (Pu and beyond). This technique is also known for its high sensitivity (low detection limits) for several elements of interest, including light elements and low-yield fission products.

RADIATION IS EVERYWHERE. AND WHILE RADIOACTIVE MATERIALS ARE OFTEN MISUNDERSTOOD, TODAY'S CHARACTERIZATION TECHNIQUES PLAY A VITAL ROLE IN TAKING THE MYSTERY OUT OF THESE MATERIALS.

2. Characterizing nuclear fuel for improved safety

Nuclear scientists rely on both computational simulations and laboratory experiments to study and reduce the risks of environmental emissions of radioactive products in the event of a nuclear accident.

Nuclearized analytical tools are used to characterize highly radioactive samples in a safe environment. The experimental data obtained can be integrated in sequences to better predict events in case of an accident. Such scenarios include loss of cooling accidents and the failure of structural components. ACTINIS is a shielded SIMS instrument that allows researchers to perform high-precision elemental and isotopic analyses of radioactive samples. The study of nuclear reactions occurring during in-core irradiation is made possible by performing isotopic ratio measurements. The physical and chemical behavior of fission products can also be investigated thanks to SIMS excellent lateral and depth-resolution capabilities.



The SKAPHIA shielded EPMA can analyze almost all elements of the periodic table, revealing compositional information for both major and trace elements of a radioactive sample. The information is obtained from sub-micron areas with excellent precision and accuracy.

The CAMECA shielded Electron Probe Microanalyzer (shielded EPMA) tool, SKAPHIA, provides compositional information about radioactive materials. It enables the study of the microstructure and chemical composition of nuclear fuel under accidental conditions. The unparalleled precision and accuracy obtained from sub-micron areas allow for detailed observations of interactions between the nuclear fuel and cladding material. EPMA also is used to investigate the impact of the oxidizing or reducing atmospheres emitted during incidental conditions on fission products speciation.

3. Verifying peaceful use of nuclear materials

Radioactive particle analysis has been largely applied to air samples collected from nuclear handling facilities when searching for undeclared nuclear activities.

Modern safeguard labs are equipped for timely analyses, allowing for detection of often scarce and small (micron to submicron) uranium-bearing particles within an immense background of environmental particles. This enables accurate and precise measurements of the U isotope composition of individual particles.

For µm-sized uranium particle analyses, SIMS is the only technique that can both localize particles of interest and determine the uranium isotopic composition of single particles. The combination of ion mapping and sample-stage movement enables fast particle screening over a large sample area (a few cm's in diameter). The screening measurements provide the exact location of U particles and rapid indication of the enrichment level (235U content).

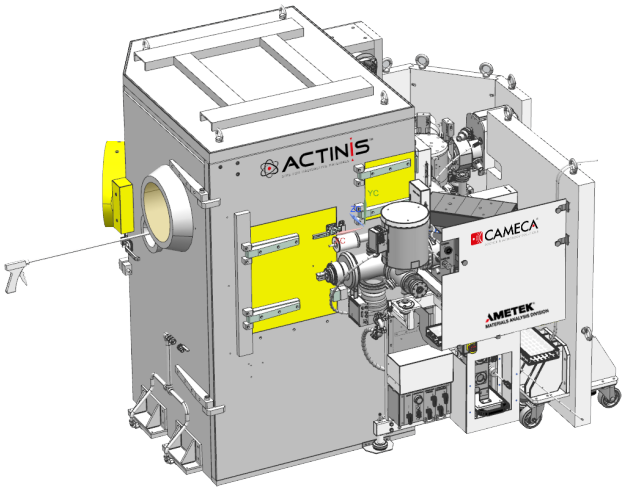
The U isotope signature can then be determined from individual particles as small as a few picograms (10–12 grams). Large-geometry SIMS instruments, such as CAMECA's IMS 1300-HR³, are equipped with automated particle-screening software, and provide the best sensitivity, precision and throughput for these applications.

4. Optimizing nuclear waste management and evaluation

Understanding the long-term behavior of glasses used to confine nuclear waste is critical to their safe geological disposal. Research of glass-alteration mechanisms requires a focus on the formation of altered layers that develop in contact with an aqueous solution.

This requires elemental in-depth characterization as well as the use of isotopic tracers, a powerful method for studying the exchanges between the solution and glass. Offering excellent depth-profiling capabilities for low-concentration elements and high-precision isotopic ratio analysis, dynamic SIMS and Atom Probe Tomography (APT) are a powerful team for analyzing nuclear glasses and investigating glass-alteration mechanisms.

Shielded EPMA is a tool of choice for characterizing lanthanide phase formation and elements redistribution in such complex fuels. Other studies focus on the long-lived minor actinides (MA) that result from irradiation of oxide fuels in light water reactors because they increase the radiotoxicity of nuclear waste. To facilitate disposal, scientists have investigated various processes to recycle the MA.



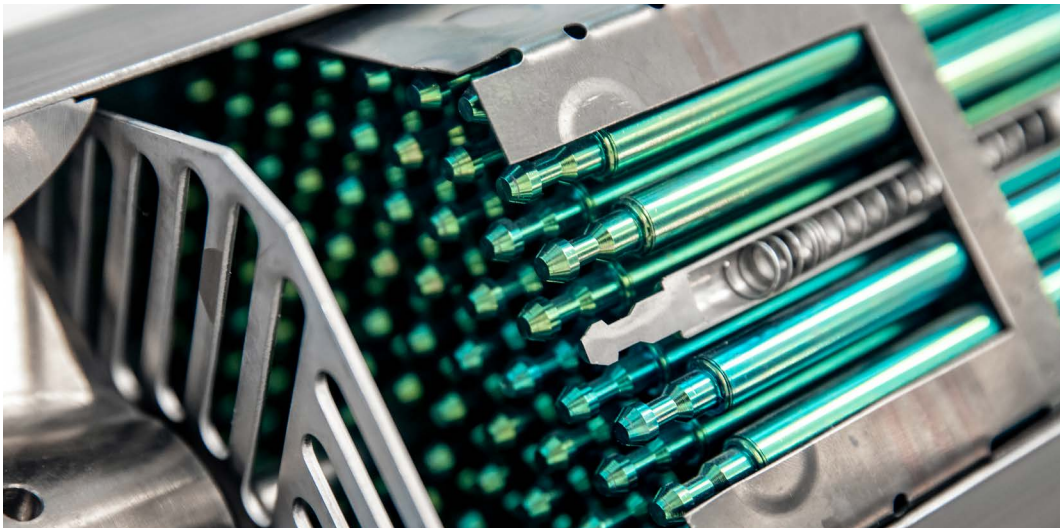
Derived from the IMS 7f-GEO SIMS, ACTINIS delivers high-sensitivity depth-profiling, isotope ratio measurements, as well as element and isotope mapping of highly radioactive materials in a safe environment.

One consists of separating the MA from the spent fuel and incorporating the MA in novel U-Pu-Zr metallic fuels prior to irradiation under a fast-neutron spectrum. EPMA offers excellent spectral resolution (ffi10eV), which is essential for differentiating complex overlapping spectral peaks.

5. Controlling corrosion and stress corrosion cracking (SCC)

SCC is a form of corrosion, or tiny blemishes, that can cause huge problems. They can appear unexpectedly in metals used in steam generators and structural components of nuclear and conventional power plants. In severe cases, major components must be removed and replaced, costing months of downtime and hundreds of millions of dollars.

Although electron microscopy has been used to study SCC for decades, only with the relatively recent use of NanoSIMS and Local Electrode Atom Probe (LEAP) have researchers been able to study the surprisingly complex chemistry at the crack growth front, with nanoscale spatial resolution and trace element analysis in 3D. Not only does oxygen penetrate the crack causing expansion and



more stress at the growth front, but key anti-oxidation agents such as aluminum and chromium show strong diffusion away from the growth front, which exacerbates the problem.

Alloy composition and water chemistry have been shown to strongly influence crack growth. SIMS and APT have given researchers the tools necessary to improve the alloy as well as to control lead and other trace elements in the coolant that can be detrimental to these structural materials.

THE NUCLEAR SCIENCE R&D COMMUNITY HAS LONG BEEN PURSUING CLEAN AND INEXPENSIVE ENERGY FROM FUSION, BUT TECHNICAL CHALLENGES REMAIN.

6. Addressing fusion reactor challenges

The nuclear science R&D community has long been pursuing clean and inexpensive energy from fusion, but technical challenges remain. They must be resolved, particularly as the most ambitious international project on this front, International Thermonuclear Experimental Reactor (ITER), will be operational in a few years.

One of the most demanding materials problems is the longevity of plasma-facing materials, especially in the plasma-diverting region of the vacuum vessel. Here materials must hold up against plasma temperatures 10 times hotter than the sun's core (150M° C) while withstanding high-energy hydrogen and helium implantation (up to 14MeV). Researchers have studied tungsten alloys with complex heat treatments, as well as radiation-resistant nanocrystalline and high-entropy alloys with this application in mind.

Understanding the 3D nanoscale mixing caused by high-temperature ion bombardment is key to designing alloys that won't erode. The erosion prevention is not only critical from a structural point of view, heavy-metal ions also have a dramatic cooling effect on plasmas. Minimizing this loss is a key criterion for the success of ITER and the other approximately dozen fusion-energy projects in progress around the world.

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