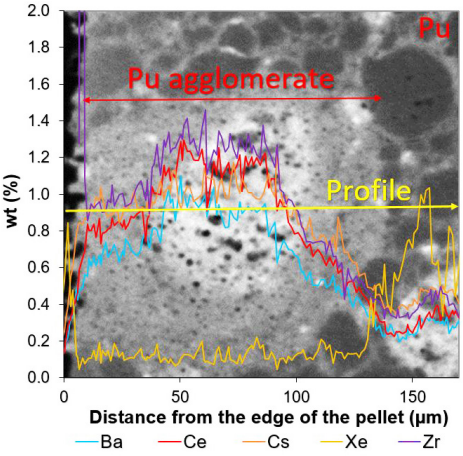
CAMECA
SCIENCE & METROLOGY SOLUTIONS

AMETEK
MATERIALS ANALYSIS DIVISION

Empowering nuclear research with a CAMECA EPMA at CEA Cadarache

A SUCCESS STORY



Ba, Ce, Cs, Xe, and Zr quantitative profiles across a Pu agglomerate (MOX fuel).



In a shielded cell at CEA Cadarache's LECA-STAR labs, Karine Hanifi checks CAMECA EPMA data.

The Challenge

France currently draws more than 70% of its electricity from nuclear power — a greater percentage than any other country. So it focuses strongly on nuclear plant safety, maintaining an excellent record in that regard. Karine Hanifi works as an analytical scientist at the CEA Cadarache energy research center in southeastern France. Her efforts help safeguard that record.

In her Microanalysis Laboratory within the facility's LECA-STAR complex, Hanifi assists in a wide variety of national and international research programs, conducting analyses to help improve the safety and performance of solid radioactive fuels and materials used in a range of reactor types.

Analyzing samples from irradiated nuclear fuel rods presents some unique challenges. The complex includes 15 hot cells: rooms shielded to protect workers from radiation. And high levels of background radiation can interfere with precise measurement.

Despite these difficulties, LECA-STAR scientists are able to apply a variety of analytical techniques, including scanning electron microscopy (SEM), secondary ion mass spectrometry (SIMS), X-ray diffraction (XRD), transmission electron microscopy (TEM), and optical microscopy.

Plus one more key technology that's Karine Hanifi's specialty: EPMA.

The Instrument

An electron probe microanalyzer (EPMA) bombards a solid specimen with a focused electron beam, then analyzes the characteristic X-rays each element in the sample emits, determining its location and concentration.

The CEA Cadarache lab uses an SX100-R EPMA from CAMECA. Its design combines powerful wavelength-dispersive spectrometers (WDS) with secondary electron/backscatter electron (SE/BSE) detectors plus an optical microscope in a single high-performance microprobe.

The instrument's measurements are specially shielded against the background X-rays, gamma rays, and beta particles that

CAMECA EPMA Advantages

- Shielded electron probe microanalyzer (EPMA) for radioactive samples
- High-precision trace analysis for almost all periodic table elements
- Four powerful wavelength-dispersive spectrometers (WDS)
- Full hot cell construction & shielding
- High spatial resolution
- High sensitivity
- Low detection limits
- Nuclear fuel studies
- Post-irradiation examination
- Severe nuclear accident simulation & analysis
- Evolution of fission products
- Impact on microstructure & chemical composition

radioactive samples emit, which would otherwise prevent precise microanalysis. So even on irradiated fuel pellets or cladding, this advanced EPMA analyzer can map the distribution of elements at or near the sample surface, while also accurately measuring each element's concentration within excellent detection limits. (The EPMA's shielding and hot cell also ensure optimum safety for operators.)

"Our samples are precious," says Hanifi. "So it's important that the EPMA technique is nondestructive. We can do our analysis, and then analyze the same area with a technique like SIMS for additional information. The CAMECA EPMA gives us both quantitative and mapping data for major and minor elements. You can also see fission products *under* the surface. For instance, you can probe a bubble of xenon gas that's 1 micron down in the sample."

Installed at CEA Cadarache in 2009, the SX100-R is the second shielded CAMECA EPMA employed there since 1984. In fact, CAMECA is the foremost supplier of shielded EPMA instruments for hot lab facilities worldwide. (Today, the company's latest solution is the advanced SKAPHIA EPMA.)

The Work

For one study, Hanifi worked with fuel pellets from two different scenarios wherein her colleagues had used very high temperatures (> 2300° C) under an oxidizing and reducing atmosphere to simulate the conditions of severe nuclear accidents.

"During irradiation, in the lifetime of the fuel rod, the microstructure develops cracks, and fission products are created," she says. "But during an accident, how would it develop differently? We must apprehend the evolution of fuel in a given accident condition."

Specifically, she applied EPMA to evaluate the impact of the oxidation/reduction potential of the high-temperature atmosphere generated during these tests in the facility's VERDON simulator on the behavior of the fuel and its fission products.

The work was meticulous. A sample could contain 30 elements; mapping a single 1 mm square — quantifying all its major and minor elements of interest at sub-micron scale — took days. And after this rigorous EPMA analysis, the sample might undergo testing by other techniques to derive further measurements.

Trace amounts of barium (Ba), cesium (Cs), xenon (Xe), and cerium (Ce) were analyzed. With detection limits down to 300 ppm, the SX100-R was able to supply precise mapping and quantitative data. (Again, a nonshielded instrument would be unable to achieve these measurements on radioactive samples.)

The Results

The study produced several significant findings. Broadly: atmospheric and temperature effects in the reactor during the simulated accidents had specific impacts on the microstructure of the fuel. These included interdiffusion of some elements between fuel and cladding; some cracks in the fuel's molten zones; and creation of certain metallic precipitates that could fragilize the material.

"This permitted us to gain a better comprehension of the reaction mechanism in the reactor — as simulated in our VERDON test device — and the evolution of microstructures in the fuel," Hanifi reports. "So we can predict these accidents' consequences in terms of contamination."

The Future

In late 2020, CEA Cadarache installed a CAMECA ACTINIS Shielded Ion Microprobe — a new SIMS instrument to provide the very latest capabilities where its results can complement EPMA analysis.

"Like our shielded CAMECA EPMA, it's a good investment," says Karine Hanifi. "Because the research is never finished!"



Hanifi's colleague Nicolas Tarisien at work. Radioactive samples must be handled in a glovebox using manipulators.

About the Labs

Located near Aix-en-Provence in southeastern France, CEA Cadarache — a facility of the French Commission of Atomic and Alternative Energies — is Europe's largest center for energy research and development. The Microanalysis Laboratory in its "hot lab" LECA facility is devoted to characterization of post-irradiation nuclear fuels using EPMA, SEM, SIMS, XRD, and TEM analyses, as well as other techniques. The adjoining STAR facility also studies nuclear plant accident scenarios, utilizing its unique VERDON testing device.

About CAMECA

CAMECA is a world-leading supplier of microanalytical and metrology instrumentation for research and process control. Our instruments measure elemental and isotopic composition in materials down to atomic resolution. Advanced CAMECA technologies include electron probe microanalysis (EPMA), secondary ion mass spectrometry (SIMS), atom probe tomography (APT), and low-energy electron-induced X-ray emission spectrometry (LEXES). We address challenging characterization needs in diverse markets, including nuclear, materials sciences, life sciences, geology, cosmochemistry, environmental, and semiconductor research.

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