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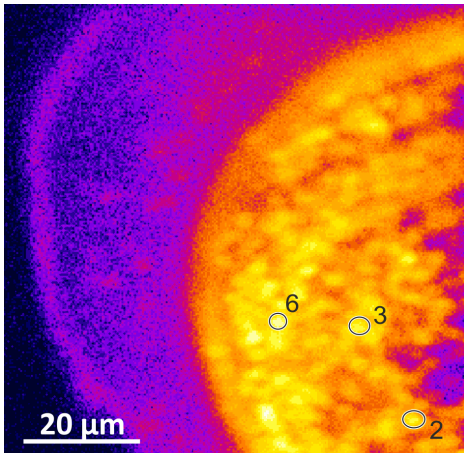
# Digging deep into geochronology with a CAMECA LG-SIMS at the Swedish Museum of Natural History

A SUCCESS STORY

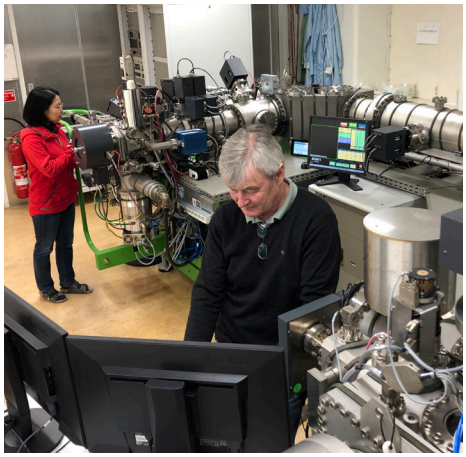
## CAMECA LG-SIMS Advantages

- Ultra-high-sensitivity ion microprobe / large-geometry secondary ion mass spectrometer (LG-SIMS)
- In-situ microscale isotopic & elemental analyses
- High sensitivity at high mass resolution
- High precision for isotopic ratios (down to tenth-permil level)
- Multicollection capability for high throughput
- Wide versatility (spot analysis, ion imaging, depth profiling, & more)
- Geochronology
- Geochemistry
- Geobiology
- Cosmochemistry
- Materials research & trace element analysis

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*Dating zircon samples with isolated lead (Pb) anomalies like these presents a real geochronological challenge — until you apply insight plus LG-SIMS.*



*At NordSIMS, research engineer Dr. Heejin Jeon and lab head Prof. Martin Whitehouse prep their CAMECA ion microprobe for another run.*

## The Challenge

The history of our planet is written in the rocks. One key way that earth scientists read that history: measuring the radioactive decay of uranium (U) and thorium (Th) into lead (Pb) within zircon crystals. When zircon first solidifies, its crystal structure rejects lead but incorporates uranium. Over known periods of time, this uranium decays into different lead isotopes via well-characterized pathways. Using this evolution as a *geochronometer*, researchers measure the ratios of lead to uranium and of different lead isotopes to each other — and can determine when a given zircon crystal formed.

Unfortunately, radioactive decay can damage the crystal, allowing lead to escape, or high temperatures may diffuse it away from its uranium source. So it had been assumed that a sample showing this *unsupported lead* would, in a sense, "break the geochronometer," making accurate age determination impossible.

Then pioneering research by Prof. Martin Whitehouse, in the Department of Geosciences at the Swedish Museum of Natural History in Stockholm, dug a little deeper. His team revealed the history of a complicated rock formation by combining systematic, precise U-Th-Pb analysis with a powerful research tool.

## The Instrument

The museum's NordSIMS lab houses a CAMECA IMS 1280 magnetic sector large-geometry secondary ion mass spectrometer (LG-SIMS).

The instrument's high-precision quantification of elemental and isotopic concentrations is unmatched for studies such as zircon U-Th-Pb. Its sensitivity comes from its large-radius magnetic sector, which provides optimal transmission at high mass resolution. Its lateral resolution — down to 1-2 microns — also allows in-situ analysis of individual grains, or even subgrain domains. Features on this scale can't be resolved with bulk or large-spot analytical methods such as laser ablation ICP-MS.

Since the LG-SIMS' installation in 1997, enhancements under CAMECA's flexible upgrade scheme have included new state-of-the-art electronics, low-noise Faraday cup detectors, and an RF-plasma oxygen ion source — all features found in their latest SIMS model, the IMS 1300-HR$^3$.

The spectrometer's exceptional capabilities and versatility make the lab a prestigious — and incredibly productive — place for multiple research communities.

"We always have more users than running time," says Prof. Whitehouse. "We're a worldwide operation: currently we have projects with people in Scandinavia, Australia, the U.S., Brazil. Geochronology and cosmochemistry dominate, followed by geobiology, ecology, and nuclear safeguards applications. Over 24 years, that instrument has generated data for 670 papers so far! A lot of those projects would be impossible without it; of course, my own research has evolved around the LG-SIMS."

## The Work

In a key case study, Prof. Whitehouse and his coworkers first reviewed the literature for anomalous U-Th-Pb data — such as spikes in lead signals. "From the spot analysis point of view, spikiness is terribly bad news," he says, "because you suddenly lose your age accuracy."

They then used the ion microprobe to pursue these anomalies via detailed analyses of zircon in two rock samples from southern India — including spot analysis of individual grains for U-Th-Pb aging; rare earth element measurements; and scanning ion imaging (SII).

During the SII work, they experienced one of science's most exciting combinations: serendipity and insight. Seeking the best possible images, they had left the LG-SIMS running overnight.

Recalls Prof. Whitehouse: "In the morning, yes, we had a fantastic image — but then we started looking back through the night. Instead of each scan just getting brighter and brighter in the same place, they were fading away in some places, getting brighter in others."

They realized the changes were coming from unsupported patches of lead that varied in the depth direction. As the top layer was sputtered away, the patches appeared and disappeared in the scans. "We saw things coming and going on that scale, and realized: these things must be tens of nanometers in size, not much bigger. That was a real revelation!"

The instrument's depth resolution is around 10 nm — about three orders of magnitude better than its already excellent lateral resolution. By stacking successive SII scans, the scientists constructed lead depth profiles with nm-scale resolution in a new technique they termed "scanning ion tomography," or SIT.

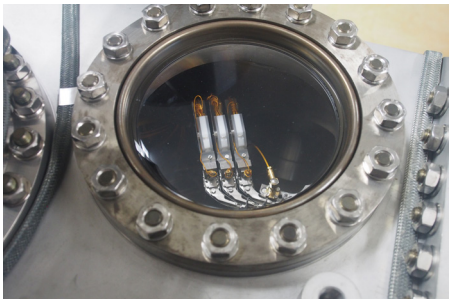
## The Results

By combining SIT data with calculations of the ratio of isotopes $^{207}$Pb to $^{206}$Pb (which form at different rates), Prof. Whitehouse and his colleagues concluded that the tiny lead patches resulted from radiation damage during uranium decay, followed by annealing during metamorphism.

The unique capabilities of the IMS 1280 had enabled them to "repair the geochronometer." They were able to reliably determine that the Indian zircons were formed around 1,850 million years ago (Ma), with subsequent ultra-high-temperature metamorphisms taking place at 570 and 520-510 Ma.

The study has wide implications. "It adds to our overall understanding of limitations in the methodology that can bias your results. So it's a powerful tool for understanding what can happen to the most widely used geochronometer we have," says Prof. Whitehouse.

"In the past, researchers had seen the abnormalities — spikiness — in their spot analyses, but didn't have the tools to dig any deeper. Could it be unsupported lead? That was just an idea. Then the imaging gave us the first indication that it was actually a *good* idea."



*Heart of the NordSIMS lab: its advanced CAMECA IMS 1280 magnetic sector large-geometry secondary ion mass spectrometer (LG-SIMS).*

## About the Lab

*The NordSIMS laboratory is located in the Department of Geosciences at the Swedish Museum of Natural History in Stockholm. The lab is part of the Swedish Research Council-funded NordSIMS-Vegacenter microanalytical and microimaging infrastructure. NordSIMS' CAMECA ion microprobe is used for a broad range of topics, including geochronology and cosmochemistry, geobiology, ecology, and nuclear safeguards. For more information, see https://www.nrm.se/en/forskningochsamlingar/geovetenskap/nordsim.904.html*

## 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 secondary ion mass spectrometry (SIMS), atom probe tomography (APT), and electron probe microanalysis (EPMA). We address challenging characterization needs in diverse markets, from geology, life sciences, materials sciences, and cosmochemistry to environmental, nuclear, and semiconductor research.*

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