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SIMS Lab Potsdam: Secondary Ion Mass Spectrometry Lab Potsdam

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Abstract: Secondary Ion Mass Spectrometry (SIMS) is among the most powerful laboratory tools available to the analytical geochemist. Its strength lies in SIMS' ability to produce high precision trace element and isotope ratio data on polish sample mounts on total analysis masses as small as 100 picograms. The Helmholtz-Centre Potsdam GFZ German Research Centre for Geosciences operates a fully equipped, large geometry SIMS instrument, which is supported by a comprehensive spectrum of peripheral instrumentation. This facility operates as an open user facility which supports the needs of the global geochemical community.

1 Introduction

A secondary ion mass spectrometer (SIMS) uses a finely focused ion beam to probe a selected sample domain on the polished surface of a solid material. In the case of Potsdam's large geometry instrument, the typical diameters for such probe beams are in the range of 2 to 30 µm. A small percentage of the material eroded from the polished surface of the sample is ionized, and these ions are accelerated into a mass spectrometer where they are separated according to their mass-over-charge ratio. An important characteristic of SIMS is its high sensitivity compared to other microbeam sampling techniques: the ability to count individual ions results in detection limits in the 10's of ng/g range for many elements.

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Also the fact that ions derived from the sample are separated by their mass-over-charge ratio allows high precision isotopic analyses to be performed on test portion masses that can be as small as 150 picograms. By observing how a signal varies as a function of time, such instrumentation can record depth profiles with a depth resolution reaching into the low nanometer range. Finally, operating either in microscope or in scanning ion imaging mode, the CAMECA 1280-HR instrument can determine elemental and isotopic maps at a $\sim$ 2 µm spatial resolution.

2 The Potsdam SIMS User Facility

The Helmholtz-Centre Potsdam GFZ German Research Centre for Geosciences operates a large geometry CAMECA 1280-HR SIMS instrument (Figure 1), which entered service in November 2013. Importantly, the Potsdam SIMS laboratory is supported by a comprehensive spectrum of peripheral instrumentation, including a full range of microanalytical equipment. The SIMS laboratory is an open user facility that supports the analytical needs of the global geochemical community. Scientists from Europe and beyond are invited to discuss collaboration opportunities with laboratory members. Currently this facility is staffed by 3.5 full-time positions. The CAMECA 1280-HR SIMS is housed in a unique, purpose-designed laboratory environment. In order to achieve the best possible instrument stability the main electronics chasses are supplied with a floor-level air cooling system, which also expels heat from the laboratory environment by an extraction hood directly above the chasses. A second air handling system provides air flow from broad areas at floor level, allowing the entire air within the laboratory to be exchanged every 3 minutes. A further system extracts cold air from beneath a liquid nitrogen Dewar. These design features are able to maintain a stable temperature that remains within a 0.3°C window at all times of operation. The 1280-HR instrument itself is positioned on a low magnetic susceptibility steel frame that is filled with fiber-reinforced concrete. This 5-ton platform is positioned on four oak blocks, thereby mechanically decoupling the platform from the surrounding laboratory flooring. The entire system is located in a magnetically quiet region of the institute.

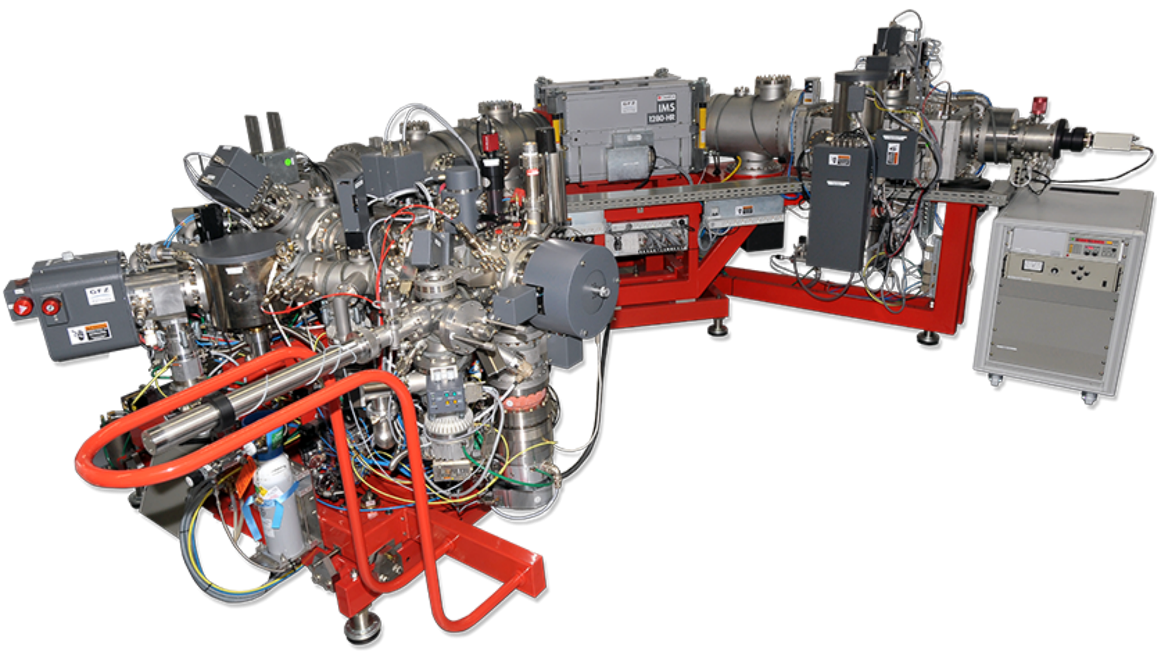

Figure 1: The Cameca 1280-HR instrument. The total length of the vacuum system is 8 meters. The vacuum pressure is between e-6 and e-7 Pa throughout most of the system.

2.1 Instrumentation and Upgrades

- CAMECA 1280-HR with dual primary ion sources and 5-trolly multi-collection system
- Long life liquid nitrogen Dewar with cold trap for improved vacuum in the sample chamber
- Monochromatic LED illumination for improved sample viewing
- All metal oxygen lines for improved primary ion source performance
- Resistive anode image digitizing system

2.2 Within-Laboratory Peripheral Instrumentation and Upgrades

- ZYGO New View 7100 white light optical profilometer (Figure 2)
- Nikon Eclipse X-Y-Z motorized optical microscope
- Vacuum oven for storage of cleaned oxygen ion source
- Argon sputter-coater for producing high-purity sample gold coats
- Cold cathode colour imaging system
- Flatbed scanner for advanced sample imaging
- Data transfer and back-up system

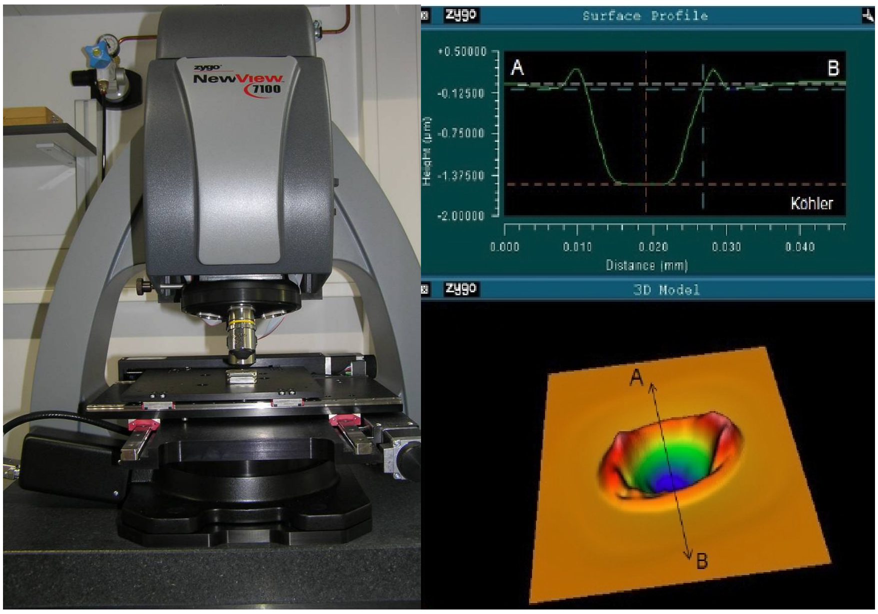

Figure 2: The New View 7100 optical profilometer (left). Example of a data set from the profilometer which can determine the geometry of the SIMS carters with a resolution of 500 nm in X and Y and 1 nm in the Z dimension. This allows us to determine crater volume and test portion mass of our various analytical methods (right).

2.3 Instrumentation available on Campus

- Fully equipped sample preparation facility
- Field emission scanning electron microscope
- Field emission and LaB6 electron microprobes
- Dual-Beam FIB instrument
- Machine shop, including design capabilities for small parts
- Broad range of geochemical instrumentation for characterizing the bulk composition of calibration materials

3 Applications

The primary research focus of the Potsdam SIMS laboratory is high precision isotope ratio determinations on geomaterials. Although the CAMECA 1280-HR can address topics related to nearly all multi-isotopic elements, our work has mainly focused on boron, carbon, oxygen and sulfur. We have also conducted numerous projects devoted to determining the ages of samples based on the U-Th-Pb radioactive decay system as applied to the mineral zircon. Some examples of our research include:

- $\delta^{11}B$ characterization of the source of gold-bearing fluids
- $\delta^{13}C$ analyses of African diamonds to quantify the isotopic heterogeneity of the Earth's mantle
- $\delta^{15}N$ investigation of synthetic diamonds in order to understand the early outgassing history of the Earth
- $\delta^{18}O$ study of microfossils in order to establish rates of change during ancient global environmental catastrophes
- $\delta^{34}S$ of iron sulfides that formed in 3.2 billion year old soils, from this we established the presence of non-marine biological activity at this early epoch
- U-Pb dating of zircons associated with the Vredevort structure in South Africa, the world's largest known meteorite impact crater.

Thanks to the stability of our lab environment, the Potsdam instrument is currently a world leader in terms of analytical precision as applied to isotope ratio determinations at the picogram sampling scale. We have demonstrated analytical repeatability as low as ± 0.09‰ (1sd) on oxygen isotope determinations in both silicate glasses and in zircon.

The depth profiling capability of the instrument has been extensively used both for determining rates of diffusion of key elements in silicate mineral phases as well as for performing trace element quantifications via ion-implant calibration materials. The CAMECA 1280-HR's highly flexible imaging system, capable of working in both scanning ion and ion microscope modes, has been used to investigate complex chemical distributions at 2 μm imaging resolution.

4 The Helmholtz SIMS Network

In cooperation with colleagues at the Helmholtz-Centre for Environmental Research in Leipzig and the Helmholtz Zentrum Dresden-Rossendorf we are developing a network of SIMS facilities which will be able to provide access to essentially all classes of SIMS instrumentation. With NanoSIMS and time-of-flight tools in Leipzig, an ion accelerator-based Super-SIMS instrument in Dresden-Rossendorf and the CAMECA 1280-HR in Potsdam we will effectively cover the complete range of ion-based sampling mass spectrometry in terms of spatial resolution, limit of detection and analytical precision. Within the network we support the analytical needs of the various partners; mutual support in terms of instrument development, technical assistance for maintenance of the instruments and cooperation in the area of methodological technique validation are also important aspects of this structure. Thanks to the highly complementary nature of the various SIMS instruments, this network within the Helmholtz Association provides a globally unique analytical platform for the characterization of Earth, environmental and engineered materials.

Web Address
www.gfz-potsdam.de/SIMS/

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