New capabilities for small-scale and high-precision SIMS analyses

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Secondary ion mass spectrometry (SIMS) has wide application for *in situ* geochronology, trace element and isotope analysis. Despite the strengths of SIMS techniques, their usefulness has been limited for many problems by the relatively coarse scale of analysis (~10-30 µm), and poor precision for element abundance ratios (~1-10 %, relative). Three recent innovations in SIMS instrumentation have improved capabilities in both regards, creating opportunities for new kinds of applications: (1) The NanoSIMS provides a reduced primary beam size (typically 100-300 nm; as small as 10's of nm). A conventional SIMS instrument equipped with a Gallium source can achieve similar resolutions, though at the cost of significantly reduced sensitivity. (2) The NanoSIMS is also the first ion microprobe capable of simultaneous detection of elements differing greatly in mass (up to a factor of 22; e.g., ^{12}C and ^{238}U). This allows for multi-collection of most element ratios, much as isotope ratio multi-collection is done on lower-dispersion mass spectrometers. (3) The ims-7f Geo provides a 'pseudo multi-collection' capability. This is intended as a poor-man's instrument for high-precision isotope ratio analysis, but also permits element ratio measurements at greater than normal speed and precision.

We will illustrate the limits and uses of these new capabilities, focusing on applications to problems in earth science, including petrology, fossilized and recent bio-materials, and geochronology. External errors for major and trace element ratios measured by multi-collection and 'psuedo multi-collection' are demonstrably controlled by counting statistics across a wide range in concentration and down to ~ ±2-3 per mil precision. This capability exceeds those of e-beam methods in several respects and enables a variety of new applications to paleoclimatology, geothermometry and 'geospeedometry'. For example, we will demonstrate how NanoSIMS images can be used to characterize diffusion gradients of minor elements between adjacent phases in metamorphic rocks, with spatial resolution an order of magnitude smaller than the e-probe and sensitivity sufficient to measure concentrations below the detection limits of ATEM. Finally, we will discuss limits of U-Pb geochronology at µm and smaller scales through processing of NanoSIMS ion images.

Non-equilibrium stable isotope partitioning in igneous processes?

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Studies of the stable isotope compositions of igneous rocks commonly assume that crystallization occurs under conditions of melt/solid equilibrium and that disequilibrium fractionations, when observed, reflect mixing processes. However, igneous phenocrysts commonly exhibit oscillatory, sector, and more complex zoning of major and trace elements that could be produced by diffusion-limited transport near the crystal/melt interface and/or by partitioning into structurally anomalous surface sites. And, melt-phase inter-diffusion is known to lead to gradients in stable isotope compositions. We look for evidence that stable isotope compositions of phenocrysts could reflect such disequilibrium during growth.

We made 255 ion probe (IMS-1280) measurements of δ^{18}O in 35 magmatic and mantle olivines and pyroxenes from 5 hand samples of diverse igneous environments and compared δ^{18}O to zonation in other elements (especially P; analyzed by EMPA and NanoSIMS). 29 of the 35 studied grains are homogeneous in δ^{18}O within the ±0.2 ‰ analytical precision. This population includes grains with oscillatory µm-scale P banding. Their lack of δ^{18}O variations suggest that whatever factors lead to this common mode of trace element zonation have no detectable effect on melt/crystal partitioning of oxygen isotopes. Large (2‰) oxygen isotope variations are observed in one olivine glomerocryst from Mauna Kea, Hawaii. This glomerocryst contains skeletal and equant P-rich domains, and these P-rich domains are systematically low in δ^{18}O compared to adjacent, later-grown, P-poor olivine. This trend appears to contrast with previous measurements showing decreasing δ^{18}O with decreasing Mg# in Mauna Kea olivines that have been attributed to AFC processes. The unusual oxygen isotope zonation pattern we observe might reflect a kinetic fractionation during nucleation and growth of the cores of some olivine phenocrysts (e.g., if diffusion-limited growth 'feeds' the crystal surface with components that are lower in δ^{18}O than equilibrium with bulk co-existing melt.) Alternatively, these data might reflect addition of a low δ^{18}O, P-rich component to some Hawaiian magmas just before nucleation of olivine. We are testing the first of these hypotheses by measuring δ^{18}O distributions in synthetic olivines grown at a range of rates and exhibiting diverse patterns of P enrichment.