X-ray microspectroscopic analyses of mineral-fluid and melt-fluid interactions at extreme temperatures and pressures

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Fluid-solid and fluid-melt interactions up to 850 °C and 500 MPa in a hydrothermal diamond anvil cell were monitored using X-ray microspectroscopy. The high spatial resolution of the X-ray microprobe permits separate *in situ* analyses of different phases at a given temperature and pressure. Spectra were collected continuously for periods of up to 12 hours on beam lines 13ID-C or 201D-B at the Advanced Photon Source, Argonne National Laboratory, Chicago. The local structure or partial composition of the aqueous fluid or melt is determined using X-ray absorption fine structure (XAFS) and X-ray fluorescence (XRF) spectra, respectively. Quantitative elemental analysis of the aqueous fluid is achieved by using an internal spike. Analytical sensitivity is enhanced by reducing the path length of the X-rays in diamond to 80 micrometers by laser-milling a sample chamber and grooves in the culet face of one of the diamond anvils. The potential of this technique will be demonstrated using examples involving zircon-fluid and melt-fluid interaction.

Submicrometer-scale minor and trace element mapping in comet dust

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Comets are primitive bodies that likely preserve the fundamental building blocks of the solar system. Dust from comets is collected in the Earth's stratosphere by NASA aircraft and direct samples of a comet will be returned to Earth by the Stardust mission. Microanalysis of comet dust is extremely challenging owing to the small size of the collected particles (10-20 μm dia.) and the even smaller size of their constituent mineral grains and other components (from ~0.5 μm down to few nanometers).

We use a coordinated approach to study the mineralogy and chemistry of thin sections (70 nm thick) of these particles using 1) a field emission, scanning and transmission electron microscope (FE-STEM) equipped with an energy-dispersive X-ray (EDX) spectrometer for quantitative spectrum imaging and diffraction analysis, and 2) a new synchrotron-based X-ray microprobe (XRM) at the Advanced Photon Source (Beamline 2-ID-D, Argonne National Lab). With the STEM, the incident probe is 1-4 nm and a quantitative EDX spectrum is collected at each pixel of the scanned image. We achieve <1% counting statistics for major elements and practical detection limits of ~0.1% for minor elements in parts of the spectrum without major element overlap. The XRM has ~200 nm spatial resolution and minimum detection limits in the ~40-100 ppm range for Cr through Zn given typical analysis conditions (200 nm spot, 1 s dwell).

The XRM mapping results reveal distinct minor element enrichments of Mn, Cr, Cu and Zn in different phases in the comet dust thin sections, with Cu and Zn co-located with Fe and S. Coordinated STEM analysis of the same thin section shows that Mn and Cr are hosted by enstatite ($MgSiO_3$) grains and confirms the presence of Zn in some but not all of the Fe-sulfide grains in the section By using the combined approach, the X-ray mapping results are correlated to the petrography of the particle and allows us to discriminate primordial trace element contents established during the formation of the grains from those acquired more recently during the particles residence in the stratosphere.