

Nanogeology of Metamorphic Magnetite using Three-Dimensional Atom Probe and Focused Ion Beam Secondary Ion Mass Spectroscopy

K. R. Kuhlman¹, P. G. Conrad¹, R. L. Martens², T. F. Kelly², D. Dunn³, N. D. Evans⁴, and M. K. Miller⁴

¹California Institute of Technology, Jet Propulsion Laboratory, Pasadena, CA 91109-8099, USA, kkuhlman@jpl.nasa.gov, ²Imago Scientific Instruments Corp., Madison, WI 53719 USA, ³University of Virginia, Charlottesville, VA 22903, ⁴Microscopy and Microanalytical Sciences Group, Metals and Ceramics Division, Oak Ridge National Laboratory, Oak Ridge, TN 37831-6376, USA

Abstract

Three-dimensional atom probe (3DAP) microanalysis and focused ion beam secondary ion mass spectroscopy (FIB-SIMS) are highly attractive techniques for the nanoanalysis of geological materials despite the difficulties inherent in analyzing semiconducting and insulating materials. Both techniques have been proposed for development into *in-situ* instruments for planetary science. We present a brief description of each technique and the results from the nanoanalysis of a metamorphic magnetite.

The local electrode atom probe (LEAP) and Mini-LEAP are new instruments for performing three-dimensional microscopy with near atomic spatial resolution and high mass resolution. The data obtained allow the three-dimensional reconstruction of the sample's elemental composition with sub-nanometer resolution. Compositional changes, phase differences and defects become measurable at the near-atomic scale. Focused ion beam secondary ion mass spectroscopy (FIB-SIMS) is a relatively new analytical technique resulting in elemental maps as a function of depth with resolution approaching 20 nm. Compositional changes and phase differences are measurable on small scales with FIB-SIMS, and difficulties due to differential sputtering in conventional SIMS analyses are overcome by sampling at specific depths rather than continuously profiling the sample.

Field ion specimens have been successfully fabricated from samples of metamorphic magnetite crystals. These magnetite crystals contain nanometer-scale, disk-shaped lamellae with elevated concentrations of manganese and aluminum. These lamellae make this magnetite particularly attractive for investigating the capabilities of techniques for the nanoanalysis of geological materials. Field ion microscope (FIM) images of these magnetite crystals were obtained in which the observed size and morphology of the precipitates agree with previous results. A preliminary study of this magnetite with the FIB-SIMS demonstrates the potential of the instrument. The enhanced aluminum in the lamellae is clearly seen during elemental mapping. The FIB-SIMS also shows promise for sectioning and analyzing geobiological samples.

Research at the California Institute of Technology and University of Virginia was sponsored by the Cross Enterprise Technology Development Program, U.S. National Aeronautics and Space Administration and by the Director's Research and Development Fund, Jet Propulsion Laboratory. Research at the Oak Ridge National Laboratory SHaRE User Facility was sponsored by the Division of Materials Sciences and Engineering, U.S. Department of Energy, under contract DE-AC05-00OR22725 with UT-Battelle, LLC, and through the SHaRE Program under contract DE-AC05-76OR00033 with Oak Ridge Associated Universities.