

OXYGEN ISOTOPIC MICROANALYSIS OF MINERALS IN THIN SECTION USING THE ION MICROPROBE. Richard L. Hervig, Center for Solid State Science, Peter Williams, Robert Thomas, Department of Chemistry, Arizona State University, Tempe, AZ 85287, and Ian M. Steele, Department of Geophysical Sciences, University of Chicago, Chicago, IL 60637.

INTRODUCTION: The measurement of oxygen isotopes on a micro-scale on minerals in thin section is a desirable goal in the study of: 1) provenance of isolated minerals in polymict meteorites; 2) zoning profiles either to document growth zoning or to access effects of possible diffusion modification of primary compositions; 3) terrestrial weathering and diagenesis. In cosmochemistry, the coupling of conventional measurements of mineral separates and the growing number of microanalyses of individual interplanetary dust particles with the study of minerals in thin section from particular meteorites holds promise for understanding processes in the solar nebula. We have been working on a microanalysis technique for oxygen isotopic determination in insulating minerals for 5 years and present a report on progress made to date. Our immediate goal is to evaluate the instrumental effects on our precision and accuracy and then to obtain data on selected minerals within meteorites.

ANALYTICAL: We use a Cameca IMS 3f ion microscope equipped with a home made Cs⁺ ion source and a high energy electron gun. Primary Cs⁺ ions are focussed to a spot 30-50 μm in diameter and negative singly charged oxygen secondary ions are detected. The problem of sample charging is alleviated with the electron gun which delivers electrons to the sample at energies in excess of 3 keV. Negative secondary ions are accelerated into the mass spectrometer, where an energy filter is used to exclude those ions emitted with other than 350 \pm 20 eV initial kinetic energy. The reasons for using such high energy ions (most ion probe studies concentrate on ions ejected with 0-100 eV kinetic energy) are: 1) variations in degree of charge compensation do not grossly change the measured isotopic ratio; 2) hydroxide cluster ions with energies of 350 eV are unstable, effectively eliminating interference of ¹⁶OH on ¹⁷O and allowing measurement of the 3 oxygen isotopes at low mass resolving power. This procedure is in contrast to collecting low energy secondary ions and analyzing at high mass resolving power.

RESULTS ON STANDARDS: We have studied several mineral types characterized by bulk techniques, including plagioclase, quartz, magnetite, and olivines with a range of Mg/(Mg+Fe). Instrumental mass fractionation during sputtering of high energy ions is extreme (up to 40 per mil per mass unit compared to more typical values of 20 per mil for low energy ions) and can be related to the atomic number of the target. Apparently, the dominant control on the measured ratio relates to transfer of energy during atom-atom collisions, with the result that heavy targets (e.g., magnetite) show small, and light targets (e.g., quartz) large, sample dependent mass fractionation effects. Preliminary results show a linear trend between mass fractionation and atomic weight of the sample. This effect appears to be too small to generate observable fractionation factors for olivines between Fo₈₀ and Fo₉₀. Internal precision is equal to or \sim 0.2 per mil greater than predicted by counting

statistics (~1.3 per mil for 18/16 in a 45 minute analysis), but crater to crater comparisons on standard grains can be as large as 6 per mil over a single analysis session. During the last 15 months, we have measured the 18/16 ratios of quartz and feldspar in a single terrestrial thin section, finding the relative values reproducible to better than 1 per mil. Our recent first attempt measurements for olivine standards are illustrated in the figure and illustrate the variation among different points in one sample and the apparent lack of recognizable fractionation (18/16) within this small compositional range.

PROPOSED STUDY OF UNKNOWN PHASES: To date, we have studied only quartz and feldspars in terrestrial rocks. However, we now have natural and synthetic olivine standards which range from 1.0 to 0.80 with respect to $Mg/(Mg+Fe)$, and which have had oxygen isotopic determination by bulk techniques. We plan to continue to measure 18/17/16 ratios in these isolated olivine standards mounted in thin section to determine our precision and accuracy in known samples simulating typical unknowns. These measurements will place limits on interpretation of similar data obtained from olivines in thin sections of meteorites, in particular carbonaceous and unequilibrated ordinary chondrites.

Oxygen isotopic measurements for separated fragments of forsterite from Allende (1) have shown enrichment in ^{16}O relative to bulk Allende. These grains are relatively common in C3 and unequilibrated ordinary chondrites and are prime candidates for in-situ oxygen measurements because of the large grain size, observed ^{16}O enrichment in some grains, and their occurrence in different classes of meteorites. We anticipate being able to recognize zoning profiles to test oxygen migration or changes in oxygen composition during growth and to correlate any variation with composition which shows regular patterns within these grains (2, 3).

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ACKNOWLEDGEMENTS: Research supported by NASA NAG 9-47 (IMS) and NAGW-2272 (IMS).

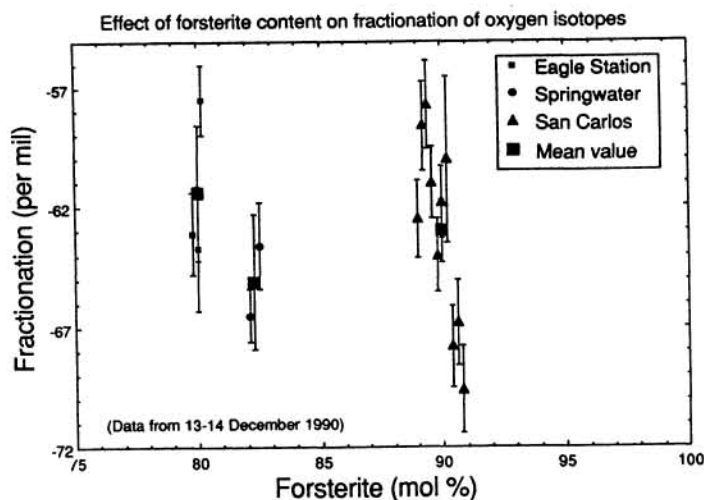


Figure: Initial ion probe measurements for standard olivines showing the apparent lack of recognizable fractionation changes between ^{18}O and ^{16}O as a function of mol% forsterite. Individual points represent different analysis areas and the large symbol is the mean for each sample. Error bars indicate 1σ errors and individual points are displaced from true composition for clarity.