

FIRST LOCAL ELECTRODE ATOM PROBE ANALYSIS OF MAGNETITE (Fe₃O₄)

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We have successfully fabricated atom probe samples of a metamorphic magnetite and performed an analysis of one of these samples using a local electrode atom probe (LEAP) [1]. This particular magnetite, previously designated LP204-1, was extracted from a polymetamorphosed, granulite-facies marble and contains grain scale heterogeneity in its oxygen isotope ratios [2]. Crystals of LP204-1 contain a high number density of nanometer-scale, disk-shaped Al-Mn-Fe-spinel precipitates making this magnetite particularly attractive for demonstrating the capabilities of the LEAP with regard to geological materials [3]. Field ion microscope images of these magnetite crystals show precipitate size and morphology that agrees with previous results [4].

A sample of LP204-1 was analyzed in the LEAP, resulting in a cylindrical analyzed volume approx. 26 nm in diameter and 21 nm high. The mass spectrum contained nearly 106,000 atoms, 97.1% of which were identified. Peaks for singly, doubly and triply ionized species were fully resolved. The analysis volume appeared to be purely magnetite, i.e. no precipitates were observed. If it is assumed that 77% of the ions in the peak at 16 are O₂⁺⁺ rather than O⁺, the stoichiometry measured for this sample using electron probe microanalysis [2] is achieved. The high fraction of O₂⁺⁺ can be explained by lack of a peak for O⁺⁺ and significant peaks for FeO_x indicating a relatively low field strength, which in turn favors molecular ions. This work is an encouraging beginning for analysis of geological materials in atom probes.

Acknowledgements

The research described in this publication was carried out at JPL, a division of the California Institute of Technology, under a contract with the U.S. National Aeronautics and Space Administration. Research at the SHaRE User Center was sponsored by the Division of Materials Sciences and Engineering, U. S. Department of Energy, under Contract DE-AC05-00OR22725 with UT-Battelle, LLC.

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