American Mineralogist, Volume 92, pages 1031–1037, 2007

Investigation of synthetic $Mg_{1.3}V_{1.7}O_4$ spinel with MgO inclusions: Case study of a spinel with an apparently occupied interstitial site

Hinako Uchida,[1,*] Kevin Righter,[2] Barbara Lavina,[3,4] Matthew M. Nowell,[5] Stuart I. Wright,[5] Robert T. Downs,[1] and Hexiong Yang[1]

[1]Department of Geosciences, University of Arizona, Tucson, Arizona 85721-0077, U.S.A.
[2]Mailcode KT, NASA Johnson Space Center, 2101 NASA Parkway, Houston, Texas 77058, U.S.A.
[3]High Pressure Science and Engineering Center, University of Nevada Las Vegas, Las Vegas, Nevada 89154-4002, U.S.A.
[4]GSECARS, University of Chicago, 5734 S. Ellis Avenue, Chicago, Illinois 60637, U.S.A.
[5]EDAX-TSL, Draper, Utah 84020, U.S.A.

Abstract

A magnesium vanadate spinel crystal, ideally MgV_2O_4, synthesized at 1 bar, 1200 °C and equilibrated under FMQ + 1.3 log f_{O_2} condition, was investigated using single-crystal X-ray diffraction, electron microprobe, and electron backscatter diffraction (EBSD). The initial X-ray structure refinements gave tetrahedral and octahedral site occupancies of $^{T}(Mg_{0.966}\square_{0.034})$ and $^{M}(V^{3+}_{0.711}V^{4+}_{0.109}Mg_{0.180})$, respectively, along with the presence of 0.053 apfu Mg at an interstitial octahedral site (16*c*). Back-scattered electron (BSE) images and electron microprobe analyses revealed the existence of an Mg-rich phase in the spinel matrix, which was too small (≤3 μm) for an accurate chemical determination. The EBSD analysis combined with X-ray energy dispersive spectroscopy (XEDS) suggested that the Mg-rich inclusions are periclase oriented coherently with the spinel matrix. The final structure refinements were optimized by subtracting the X-ray intensity contributions (~9%) of periclase reflections, which eliminated the interstitial Mg, yielding a structural formula for spinel $^{T}Mg^{M}(V^{3+}_{1.368}V^{4+}_{0.316}Mg_{0.316})O_4$. This study provides insight into possible origins of refined interstitial cations reported in the literature for spinel, and points to the difficulty of using only X-ray diffraction data to distinguish a spinel with interstitial cations from one with coherently oriented MgO inclusions.

Keywords: Spinel, crystal chemistry, XRD, inclusion, periclase, electron backscatter diffraction