American Mineralogist, Volume 102, pages 867–875, 2017

Polarized FTIR spectroscopic examination on hydroxylation in the minerals of the wolframite group, $(Fe,Mn,Mg)[W,(Nb,Ta)][O,(OH)]_4$

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Abstract

Polarized FTIR spectroscopic measurements of 11 natural wolframite single crystals from different occurrences revealed the common presence of structurally bound OH groups in their crystal lattice, with potential influence on the properties of thisse geologically and technologically important group of compounds. Despite differences in the appearance of the OH absorption pattern, dependent among other on the end-member ratio, two types of "intrinsic" OH defects could be discerned from detailed studies of the pleochroic behavior of the absorption bands both at 80 K and room temperature. The accompanying chemical analyses by the electron microprobe helped to clearly identify the substitution trend $W^{6+} + O^{2-} \leftrightarrow (Nb,Ta)^{5+} + OH^-$ as the prevailing hydrogen incorporation mechanism into wolframite. The assignment of the observed IR absorption phenomena to hydrous defects was confirmed by the results of deuteration experiments and the negligible contribution of included impurities to the FTIR spectra in the OH absorption region. The results obtained in this study of natural wolframite crystals can be used to detect and analyze hydrous defects in synthetic technologically important tungstates.

Keywords: Wolframite, FTIR spectroscopy, OH defects, nominally anhydrous minerals, deuteration, flux synthesis