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Intergrown niobian rutile phases with Sc- and W-rich ferrocolumbite: An electron-microprobe and Rietveld study

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ABSTRACT

A pegmatite from Diethensdorf, Saxonian Granulite Massif, Germany, contains fine-grained aggregates of at least three different varieties of niobian rutile and a (W, Sc)-rich ferrocolumbite. In addition, rutile 1 occurs as larger grains. Electron microprobe analyses of niobian rutile gave compositions close to the general formula $(Fe,Mn)_x(Nb,Ta)_{2x}Ti_{3-3x}O_6$, with Nb and Fe contents decreasing in the order rutile 1 → rutile 2 → rutile 3 and Ti increasing accordingly. The substitution of (Ti,Sn) by Fe + (Nb,Ta) is 35–32 at% in rutile 1, 28–26 at% in rutile 2, and 24–19 at% in rutile 3. The intergrown ferrocolumbite has Ta/(Nb + Ta) ratios of <0.10 and Mn/(Fe + Mn) ratios <0.25, and shows unusually high contents of Sc_2O_3 and WO_3 (up to 4.0 and 8.8 wt%, respectively). Powder X-ray diffraction (XRD) analysis with Guinier and Bragg-Brentano methods identified at least three rutile phases (herein termed A, B, and C) with cell volumes decreasing with the Nb (+ Ta, W) contents. Nb/Ti ratios of rutiles estimated from Rietveld refinements roughly conform to the results of electron microprobe analysis. The cation distribution in the ferrocolumbite was refined on the basis of a two-scatterer model at sites 8*d* and 4*c* in space group *Pbcn*, leading to ordering of the heavy atoms on site 8*d*. Textural evidence suggests that the fine-grained intergrowths of ferrocolumbite + rutile 2 + rutile 3 (+ rutile 1) were formed by exsolution from a precursor phase that most probably was not rutile 1.