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High-pressure synthesis of skiagite-majorite garnet and investigation of its crystal structure

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Abstract

Skiagite-rich garnet was synthesized as single crystals at 9.5 GPa and 1100 °C using a multi-anvil apparatus. The crystal structure [cubic, space group $Ia\bar{3}d$, a = 11.7511(2) Å, V = 1622.69(5) Å^3, D_{calc} = 4.4931 g/cm^3] was investigated using single-crystal synchrotron X-ray diffraction. Synchrotron Mössbauer source spectroscopy revealed that Fe^{2+} and Fe^{3+} predominantly occupy dodecahedral (X) and octahedral (Y) sites, respectively, as expected for the garnet structure, and confirmed independently using nuclear forward scattering. Single-crystal X-ray diffraction suggests the structural formula of the skiagite-rich garnet to be $Fe_3^{2+}(Fe_{0.234(2)}^{2+}Fe_{1.532(1)}^{3+}Si_{0.234(2)}^{4+})(SiO_4)_3$, in agreement with electron microprobe chemical analysis. The formula is consistent with X-ray absorption near-edge structure spectra. The occurrence of Si and Fe^{2+} in the octahedral Y-site indicates the synthesized garnet to be a solid solution of end-member skiagite with ~23 mol% of the Fe-majorite end-member $Fe_3^{2+}(Fe^{2+}Si^{4+})(SiO_4)_3$.

Keywords: Skiagite, majorite, garnets, single-crystal X-ray diffraction, Mössbauer spectroscopy, nuclear forward scattering, XANES