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In-situ study of ferric iron distribution in synthetic spinels by electron microprobe analysis

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The iron oxidation state in mantle minerals is a key value in oxygen fugacity calculation and the most widely used analytical approach for Fe3+/ΣFe determination is Mössbauer spectroscopy, which is a bulk method and there is a lack of information on Fe3+/ΣFe zonation in individual mineral grains and Fe3+/ΣFe in inclusions. Here we present application of the flank method using the electron microprobe by analysing the FeLα and FeLβ X-ray emission spectra to a suite of 20 synthetic $MgAl2O_2$-$Cr2O_3$-$Fe2O_3$(FeO) spinels. Materials were done with 5 – 25 FeO wt.%, and 2-70 $Cr2O_3$ wt.% and Fe3+/ΣFe = 0.10 to 0.80, where Fe3+/ΣFe was determined independently using Mössbauer spectroscopy on the same grains used for the flank method measurements.
Synthesis of the samples produced using a pyrolysis method of organic salt compositions in $MgAl2O_2$-$Cr2O_3$-$Fe2O_3$(FeO) system with following heating in corundum crucibles at 1300 ° C for 5 -10 hours under controlled oxygen fugacity. All synthetic materials were investigated by X-ray and Mössbauer spectroscopy to examine a phase and iron oxidation state features. In terms of chemical composition and Fe3+/ΣFe resulting synthetic material covers a whole range of spinels derived in mantle peridotites and pyroxenites. These synthetic products were used as a standard sample to investigate co-variations of ratios of intensities measured on the flanks of FeLα and Lβ peaks and Fe3+/ΣFe, FeO content and Cr#. The obtained correlations can be used to perform in-situ studies of ferric iron distribution in natural mantle spinels. The presented approach will allow investigating the difference in mantle spinel Fe3+/ΣFe at a microscale from core to rim in individual grain, inclusion, melting pocket and in intergrows with other mantle mineral assemblage.
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