

Iron oxidation state determination in garnets by EPMA and XANES

H.E. HÖFER¹, G.P. BREY¹, G.M. YAXLEY²

AND A.J. BERRY³

¹Institute of Geosciences, Universität Frankfurt, Germany;
hoefer@em.uni-frankfurt.de

²Research School of Earth Sciences, The Australian National University

³Dept. of Earth Science and Engineering, Imperial College London, United Kingdom

Techniques for the determination of iron oxidation state with micron spatial resolution have gained new input due to recent advances with the electron microprobe flank method [1] and synchrotron-based K-edge XANES spectroscopy [2]. We are giving here first results on a comparative study to establish both techniques for synthetic and natural Cr-rich garnets.

Although arising either from X-ray absorption or emission processes, both techniques are based on core electron transitions involving 3d electron states. As a consequence, both methods are strongly dependent on the Fe coordination geometry. For this reason, we focus on synthetic garnets where the two Fe species are either in dodecahedral or octahedral coordination, respectively.

The calibration of the EPMA flank method is performed with 19 different synthetic garnets with variable Mg, Fe, and Ca compositions. After correcting the data for self-absorption [Höfer and Brey, in prep.], the error in the Fe³⁺ determination is ~0.4 wt.-%. In natural samples with very low Fe contents of 5-10 wt.-%, the uncertainty in Fe³⁺/ΣFe determination is estimated to 0.04 (1s).

The presence of Mn and Cr in the garnets does not affect the flank method calibration as found for Mn- and Cr-rich synthetic garnets. The samples investigated by EPMA will be used for XANES Fe K-edge measurements together with Mössbauer spectroscopy to develop complementary techniques for microscale application.

References

- [1] Höfer H.E., Brey G.P. and Hibberson W.O. (2004) *Lithos* **73**, 551.
- [2] Yaxley G.M., Berry A.J., O'Neill H.St.C., and Woodland A.B. (2005) *GCA* **69**, Suppl., A828.