In situ $Fe^{3+}/\Sigma Fe$ measurements in Ti-rich mantle-derived amphiboles: results and limitations as evidenced by EMP and SIMS.

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Mantle kaersutite are known to have variable $Fe^{3+}/\Sigma Fe$. Usually, Fe_2O_3 and FeO were calculated assuming stoichiometric OH contents, which does not apply for Ti-rich oxy-amphiboles. Direct in situ measurements are thus required. The samples studied were kaersutite megacrysts from lava flows, scoria cones, dikes and maars, with variable H_2O (0.17 to 1.74 wt%). We measured $Fe^{3+}/\Sigma Fe$ by electron probe microanalysis (EMP) based on the "self absorption induced Fe $L\alpha$ peak shift" method *(Fialin et al., 2001, Am Mineral, 86, p456-465*; 2004, *Am Mineral, 89, 654-662)*. $Fe^{3+}/\Sigma Fe$ (%) values range from 36 (± 6) to 89 (± 6). Compared to bulk wet chemistry, EMP $Fe^{3+}/\Sigma Fe$ is significantly overestimated when H^+ > 0.5 apfu. The difference cannot be simply explained by megacryst heterogeneities, and may result from an electron beam-induced dehydrogenation and a subsequent oxidation of Fe^{2+} to Fe^{3+} according to the known relation: $Fe^{2+} + OH^- = Fe^{3+} + O^{2-} + 1/2 \ H_2$. We measured H-losses by secondary ion mass spectrometry (SIMS) after EMP irradiation at different beam currents on two amphiboles with 1.1 and 1.7 wt% H_2O respectively. H-losses occur under 240 and 100 nA in both amphiboles, but no dehydrogenation is observed under lower beam currents for the 1.1 wt% H_2O amphibole. However, H-loss still occurs from 240 nA down to at least 50 nA in the amphibole with the greatest H_2O contents. Thus, the current 240 nA EMP protocol is applicable for amphiboles with H^+ lower than ~ 0.5 apfu., but a reduced electron beam current density should be used for

amphiboles with higher H_2O contents.