American Mineralogist, Volume 99, pages 139–148, 2014

The effect of XPS background removing method on the appraisal of Ti and Fe: The case of phlogopites and brookite

Maria R. Guascito[1], Ernesto Mesto[2,*], Cosimino Malitesta[1], Rosaria A. Picca[1] and Fernando Scordari[2]

[1]Dipartimento di Scienze e Tecnologie Biologiche e Ambientali, Università del Salento, via Monteroni, 73100 Lecce, Italy
[2]Dipartimento di Scienze della Terra e Geoambientali, Università degli Studi di Bari "Aldo Moro," via E. Orabona 4, 70125 Bari, Italy

Abstract

The determination of the oxidation state and structural role of transition metals in minerals is a crucial challenge. XPS has proven to have a great potential in probing the site distribution and chemical states of Fe and Ti transition elements, provided that the right method to process the spectra is used. XPS spectra of these elements have the 2*p* core level region usually rich of features but the choice of the method for background removing can seriously affect the results of the quantitative analysis. Single crystals of brookite (TiO_2) and natural micas (phlogopites) are investigated to examine the effect of background subtraction on Ti2*p* and Fe2*p* signals. The backgrounds used are: the "Linear" background; the traditional "Shirley" background; three different Tougaard-like backgrounds; and the more recent "shape parameter, κ" method. In the case of the studied natural micas, the Fe chemical state proportion (Fe^{2+}/Fe_{tot}) obtained with the corrected spectra varies by 10%. It is shown that TiO_2 oxides are not suitable as standard for octahedral Ti^{4+} signal in the studied micas. The "shape parameter, κ" method proves to provide supplementary information useful for a full interpretation of XPS signals.

Keywords: XPS, background subtraction, Fe,Ti-oxidation state, mica, brookite