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Structural and spectroscopic characterization of a natural Zn-rich spinel approaching end-member gahnite

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A natural Zn-rich spinel crystal coming from the Jemaa, Kaduna State, Nigeria, was analyzed by electron microprobe. The gahnite component ($ZnAl_2O_4$) of this sample is $\sim$94%, the remaining being dominated by a hercynite component ($FeAl_2O_4$). To our knowledge, a gahnite component of 94% is the highest value among the mineral samples studied in literature. For this reason a full characterization of this crystal was made by using single-crystal X-ray diffraction, optical absorption and Raman spectroscopies.

The sample studied is an octahedral, 3 mm large single crystal with a pale blue-grey colour. The values of its main structural parameters are: unit cell parameter of 8.0850(3) Å, T-O bond distance of 1.9485(6) Å and M-O bond distance of 1.9137(3) Å. The empirical structural formula is $^{T}(Zn_{0.94}\ Fe^{2+}_{0.04}Al_{0.02})^{M}(Al_{1.97}Fe^{2+}_{0.02}Fe^{3+}_{0.01})O_4$ shows a very low degree of inversion (0.02), with Zn and Al almost fully ordered in the tetrahedrally- and octahedrally-coordinated T and M sites, respectively.

Raman spectra were collected over the range 100-800 cm^{-1} Raman shift using two different laser wavelengths, 632.8 nm and 473.1 nm. A strong fluorescence was observed, but in spite of this, two Raman modes of the five Raman-active modes of the spinel phase were recorded. They are the E_g mode at 419 cm^{-1} due to Zn in ZnO_4 tetrahedra and the T_{2g} mode at 662 cm^{-1} due to Al in AlO_6 octahedra. What observed is in excellent agreement with the literature.

The optical absorption spectra were collected in the UV/VIS-NIR range 5000-30000 cm^{-1}. They show a dominant absorption band at ca. 5200 cm^{-1} that has been assigned to a spin-allowed $d-d$ transition in Fe^{2+} located at the tetrahedrally coordinated T site.