

ENERGY-DISPERSIVE X-RAY FLUORESCENCE ANALYSIS OF MONO- AND POLYCRYSTALS OF SELENIDE SPINELS BY FUNDAMENTAL PARAMETER METHOD

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The analysis of mono- and polycrystals of selenide spinels of general formula $M_xN_yCr_zSe_4$ (where M^{+2} and N^{+3} are cations of metals, e.g. Zn^{+2} , V^{+3} , Ga^{+3}) by energy-dispersive X-ray fluorescence spectrometry (EDXRF) is presented. The monocrystals of spinels (sizes from ca. 1 to 5 mm) were analyzed without any treatment. The polycrystals were powdered and pressed into pellets of 10 mm in diameter with liquid binder (polyvinylpyrrolidone/methylcellulose solution). The tungsten pinhole collimator was placed in front of the X-ray tube to reduce the size of the analyzed area. The monocrystals were analyzed using the pinhole collimators of 200 or 400 μm holes (280, 581 μm focal spot sizes) depending on the size of the analyzed crystal, whereas the polycrystalline spinels pressed into pellets were analyzed using 1000 μm collimator (1469 μm focal spot size).

The quantitative analysis was performed using the fundamental parameters method with and without reference samples. The agreement between the results of standardless analysis and the results obtained using reference samples was quite satisfactory and the average relative difference between both quantification methods was ca. 3%. The synthesized monocrystals have well-formed flat faces. Nevertheless, various positions of a monocrystal can strongly influence EDXRF analysis. Thus, the dependence of quantitative analysis results on monocrystal position was studied. The accuracy of the EDXRF results was also verified using inductively coupled plasma optical emission spectrometry (ICP-OES).