Type of presentation: Poster

IT-5-P-5988 Distributions of cations and inversion parameter in nonstoichiometric magnesium aluminate spinel characterized by electron energy-loss spectroscopy

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The effect of composition and heat treatments on the distribution of cations and on the inversion parameter in magnesium aluminate spinel was studied using Electron Energy Loss Spectroscopy (EELS). Powders of MgO•nAl2O3 (0.95<n<1.07) with nano-sized grains were synthesized by solution combustion and heat treated using Spark Plasma Sintering (SPS) and Pressure-less Sintering (PS) methods. EEL spectra were collected at varying distances perpendicular to grain boundaries from which the Mg to Al cation ratio and the inversion parameter, which is the fraction of tetrahedral sites occupied by Al cations, were calculated. The Mg to Al cation ratio was calculated from their core loss K-edges. To estimate the fraction of tetrahedral sites occupied by Al cations, the inversion parameter was calculated from the Al L-edge using the methodology suggested by Bruley et al., [1] which is based on the integral ratio between L3 to L2 peaks.
We report that cations which segregate to the grain boundaries are the excess component relative to the stoichiometric composition of the spinel (Fig. 1). Heat treatment does not change the type of segregate cation but does affect the degree of order of spinel. We find that spinel powders with nano-sized grains subjected to SPS treatment results in higher order compared to PS, namely lower inversion parameter. Finally, we discuss the experimental requirements for measuring reliable EEL spectra from these materials which are sensitive to damage from the electron beam.

[1] J. Bruley, M.-W. Tseng and D. B. Williams "Spectrum-Line Profile Analysis of a Magnesium Aluminate Spinel Sapphire Interface," Microsc. Microanal. Microstruct, pp. 1-18, 1995.

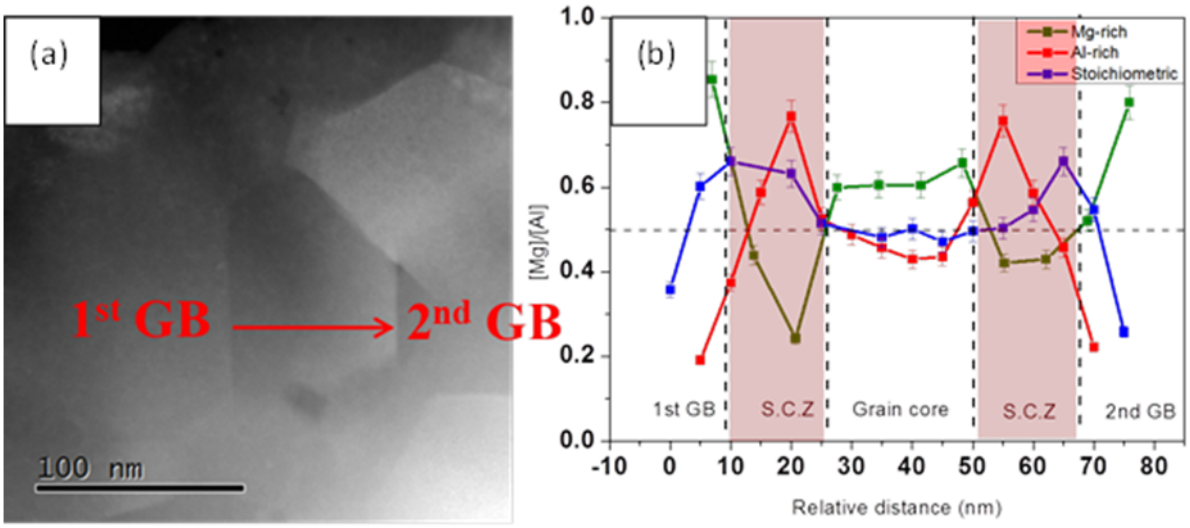

Fig. 1: (a) Schematic representation of the collection of EEL spectra perpendicular to grain boundaries. (b) Mg to Al cation ratio as a function of relative position perpendicular to the grain powders of spinel samples heat treated by SPS. (SCZ represents space charge zone)