

Akihiko Nakatsuka<sup>3</sup>, Maki Okube<sup>4</sup>, Satoshi Sasaki<sup>5</sup>, Eiji Ito<sup>5</sup>

<sup>1</sup>Tohoku University, School of Engineering, Institute for Materials Research, Tohoku University, 2-1-1 Katahira, Aoba-ku, Sendai, Miyagi, 980-8577, Japan, <sup>2</sup>Graduate School of Science and Technology, Kumamoto University, Kumamoto, 860-8555, Japan, <sup>3</sup>Department of Advanced Material and Engineering, Faculty of Engineering, Yamaguchi University, Ube, 755-8611, Japan, <sup>4</sup>Materials and structure laboratory, Tokyo Institute of Technology, Yokohama, 226-8503, Japan, <sup>5</sup>Institute for study of the Earth Interior, Okayama University, Tottori, 682-0193, Japan, E-mail: ssakaikumadai@gmail.com

Single crystals of  $MgAl_{2-x}B_xO_4$  ( $x = 0.11$  and  $0.13$ ) spinel were synthesized under 11 GPa and 1873-1273 K using a large volume split sphere-type multi-anvil apparatus. These crystals were ascertained to satisfy the systematic absence of space group  $Fd\bar{3}m$  from precession photographs. Crystal structure refinements were carried to determine the site occupancy of cations and Debye-Waller factors in spinel structure. The R factors for  $x = 0.11$  and  $x = 0.13$  were 2.97 % and 2.52 %, respectively. Based on the site preference refinements and observed bond length of each site, the chemical formula of the B-bearing spinels are  $(Mg_{0.62}, Al_{0.38})[Mg_{0.60}, Al_{1.29}, B_{0.11}]O_4$  and  $(Mg_{0.63}, Al_{0.37})[Mg_{0.63}, Al_{1.24}, B_{0.13}]O_4$ , respectively. All  $B^{3+}$  occupy the octahedral site in the synthetic B-bearing spinels. These results are very important; the small B ion does not occupy the small tetrahedral site, but it occupies the large octahedral site. When we compare the  $MgAl_2O_4$  spinel end-member, disorderness which can also be detected by Debye-Waller factors is largely increased with boron contents. The large octahedral site in normal  $MgAl_2O_4$  spinel is proffered by smaller Al ion than larger Mg ion. Moreover, the smallest B ion gives priority to the octahedral site most in the Mg-Al-B system. The peculiar site preference depends greatly on the host  $MgAl_2O_4$  spinel structure with high symmetry. Such a feature is not observed in olivine structure of the Mg-Al-B system.

Keywords: spinel, high pressure, powder and single crystal diffraction

### P11.16.118

*Acta Cryst.* (2008). A64, C543

#### Magnetic and structural investigations of nanocrystalline nickel ferrite $NiFe_2O_4$

Alia Adam<sup>1</sup>, Z Ali<sup>2</sup>, E Abdeltwab<sup>3</sup>, Y Abbas<sup>4</sup>

<sup>1</sup>Al-Azhar university, Faculty of Science, Girls Branch, Physics, Al-Azhar Univ. Faculty of Sci. Yossef Abbas Street, Naser city, Cairo, Cairo, 11201, Egypt, <sup>2</sup>Al-Azhar university, Faculty of Science, Girls Branch, Al-Azhar Univ. Faculty of Sci. Yossef Abbas Street, Naser city, Cairo, Cairo, 11201, Egypt, <sup>3</sup>Al-Azhar university, Faculty of Science, Girls Branch, Al-Azhar Univ. Faculty of Sci. Yossef Abbas Street, Naser city, Cairo, Cairo, 11201, Egypt, <sup>4</sup>Suez Canal Univ. Faculty of Science, Physics Dep. Ismailia, Egypt., E-mail: alia\_adam@hotmail.com

Nickel ferrite is an important high frequency magnetic material due to its ultrahigh resistivity. Nanoparticles of  $NiFe_2O_4$  ferrite have been synthesized by the chemical co-precipitation of the amorphous metal hydroxides (under controlled pH values). The prepared powders have been sintered at 800°C. Room temperature X-ray measurements of the prepared samples of  $NiFe_2O_4$  reveal the production of single cubic phase with average particle size of about 30 nm. The lattice parameters, the oxygen position and the cation distribution have been determined by using Rietveld analysis. The results of Mossbauer Effect spectroscopy (ME) and the Vibrating Sample Magnetometer (VSM) indicate the correlation between the magnetic properties and the nanocrystallinity of the investigated nickel ferrite.

Keywords: nanoparticles, ferrites, magnetization

### P11.16.119

*Acta Cryst.* (2008). A64, C543

#### X-ray structural study of intercalation compound $M_xTiS_2$ ( $M=Ni, Co$ )

Takuro Kawasaki, Ken-ichi Ohshima

University of Tsukuba, Institute of Materials Science, Tennodai 1-1-1, Tsukuba, Ibaraki, 305-8573, Japan, E-mail: kawasaki@jb.bk.tsukuba.ac.jp

Transition-metal intercalated titanium disulfides  $M_xTiS_2$  have various structures and characteristic physical properties depending on the kind or composition of intercalated atoms M. It is reported that long-range ordered structure, short-range ordered structure and staging structure of intercalated atoms appear in  $M_xTiS_2$ . However, there are few detailed structural studies of  $Ni_xTiS_2$  and  $Co_xTiS_2$  until now. Single and polycrystalline crystals of  $Ni_xTiS_2$  and  $Co_xTiS_2$  were grown in evacuated silica tubes by chemical vapor transport method using iodine as a transport gas. M compositions x were determined by electron probe micro analysis (EPMA). Both superlattice reflections and diffuse scattering, depending on x, were observed by electron diffraction and single-crystal X-ray diffraction methods. X-ray intensity data were collected at room temperature with the use of an automatic four-circle diffractometer. Crystal structure refinements and electron density distribution analysis by maximum entropy method of  $Ni_xTiS_2$  and  $Co_xTiS_2$  were performed. Intercalated atoms occupy octahedral sites and tetrahedral sites in the van der Waals gap. Lattice parameter of the compounds was decreasing with increasing the intercalated M atoms. The covalent electron exists between Ti, M and S atoms. Further, order-disorder transition phenomena of intercalated atoms were observed by high temperature *in-situ* powder X-ray diffraction and differential scanning calorimetry (DSC) measurements.

Keywords: intercalation compounds, sulfides, crystal structures

### P11.16.120

*Acta Cryst.* (2008). A64, C543

#### A synchrotron powder diffraction study for luminescent materials

Namsoo Shin<sup>1</sup>, Docheon Ahn<sup>1</sup>, Kee-sun Sohn<sup>2</sup>

<sup>1</sup>Pohang Accelerator Laboratory, POSTECH, Pohang, Kyungbuk, 790-784, Korea (S), <sup>2</sup>Sunchon National University, Suncheon, 540-742, Korea (S), E-mail: sns@postech.ac.kr

The crystal structure of some luminescent materials were solved *ab initio* from synchrotron powder data without preliminary knowledge of the chemical formula. The time resolved luminescence and concentration quenching behavior for the phosphors were also examined. Each deconvoluted Gaussian peak in the emission spectra was assigned to its corresponding crystallographic site with the assistance of density functional theory (DFT) calculations. The thermal stability and the color purity were also discussed from the crystallographic analysis.

Keywords: luminescence, *ab initio* structure determination, synchrotron