Neutron Diffraction Study of Powders Prepared by Self-propagating High Temperature Synthesis

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ABSTRACT

Non-stoichiometric ceramics of $Ni_xZnO_{1-x}Fe_2O_4$ were prepared by self-propagating high temperature synthesis reaction with various processing conditions and their stoichiometric numbers were determined by neutron diffraction. The neutron diffraction patterns were measured at room temperature using monochromatic neutrons with a wave length of 0.18339 nm from a Ge(331) monochromator at a 90 degree take off angle. The Rietveld refinement of each pattern converged to good agreement ($\chi 2$=1.88-2.24). The neutron diffraction analysis revealed the final stoichiometries of the ferrites were $Ni_{0.38}Zn_{0.62}Fe_2O_4$ and $Ni_{0.33}Zn_{0.67}Fe_2O_4$, respectively. This supports that final stoichiometric number of the self-propagating high temperature synthesis product can be controlled by the processing parameters during the combustion reaction.

INTRODUCTION

Non-stoichiometric compound such as Ni-Zn ferrites are attractive for micro-device applications owing to their high resistivity, low eddy current losses, high curie temperature and chemical stability. Although there have been many attempts to obtain various ferrites through SHS products, little information is available on the effects of initial high oxygen pressure on the non-stoichiometry and formation mechanism of the final product. Hence, the objective of present work is to determine non-stoichiometric number of Ni-Zn ferrite prepared through SHS at high oxygen pressure by neutron diffraction.

EXPERIMENTAL METHOD

Reagent grade of iron, iron oxide, nickel oxide and zinc oxide powders were used as starting materials. The mixture of these powders with suitable molar composition was

throughly mixed and ground for 24 hours followed by drying for 15 hours in vacuum at 150℃. The chemical analysis of the SHS products was carried out by neutron diffraction. The powder neutron diffraction pattern from 5° to 155° was measured at room temperature using a high resolution powder diffractometer (HRPD) at Korea Atomic Energy Research Institute. Monochromatic neutrons with a wavelength of 0.18339 nm was obtained from a Ge(331) monochromator with a 90° take-off angle.

RESULTS AND DISCUSSION

The SHS reaction occurred well at the oxygen pressure range of 0.5 MP and 5 Mpa. The combustion temperatures were 943 °C at 0.5 MPa and 1120 °C at 5.0 MPa, respectively. From the typical neutron pattern of the ferrites prepared at the oxygen pressures of 0.5 and 5.0 MPa, respectively, spinel peaks of ferrites were clearly observed in the neutron diffraction pattern of the combustion products. The neutron diffraction analysis results obtained by FULLPROF showed that the Rietveld refinement of each pattern converged to good agreement ($\chi 2$=1.88~2.24). In the AF sample, the Rietveld refinement of a structural model consisting of $Ni_xZn_{1-x}Fe_2O_4$ converged to $\chi 2$=2.24. From the Rietveld refinement, the lattice parameters of the $Ni_{0.38}Zn_{0.62}Fe_2O_4$ phase formed at 0.5 MPa of oxygen pressure and the $Ni_{0.33}Zn_{0.67}Fe_2O_4$ phase formed at 5.0 MPa of oxygen pressure were 0.84125 nm and 0.84129 nm, respectively.

SUMMARY

The chemical analysis of Ni-Zn ferrite powders prepared by self-propagating high temperature reaction at various oxygen pressure were studied and the following conclusions were drown : The combustion propagating rate was the extreme value of 7.8 mm/sec at 5.0 MPa and remained almost constant in the error range with further increase in the oxygen pressure up to 5.0 MPa. The combustion temperature increased with oxygen pressure, however, higher compacting pressure of the reactant powders resulted in the lower combustion temperature. The combustion reaction did not occur above the uniaxial compacting pressure of 15 MPa even the oxygen pressure was at 5.0 MPa. The Rietveld refinement of neutron diffraction data for $Ni_xZn_{1-x}Fe_2O_4$ converged to $\chi 2$=2.24, which suowed the lattice parameters of the $Ni_{0.38}Zn_{0.62}Fe_2O_4$ and $Ni_{0.33}Zn_{0.67}Fe_2O_4$ were 0.84125 nm and 0.84129 nm, respectively.

ACKNOWLEDGEMENT

The authors wish to acknowledge the Korea Ministry of Science and Technology(MOST) for their support of neutron diffraction study.