共沈法とフラックス法による$(Cu,Fe)Fe_2O_4$微粒子の作製と特性評価

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Characterization of $(Cu,Fe)Fe_2O_4$ nanoparticles obtained via coprecipitation and flux methods

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Introduction

Magnetic nanoparticles such as magnetite (Fe_3O_4) and transition metal doped ferrites in the form of MFe_2O_4 (M= Zn, Mn, Fe, Co, Ni etc.) are promising materials with intrinsic magnetism whose stoichiometry can be manipulated to tailor their properties for specific applications. Most spinel ferrites usually crystallize into a cubic lattice structure. Copper ferrite, $CuFe_2O_4$, may exist in cubic and tetragonal symmetries. The structural phase transition occurs as a result of the Jahn-Teller effect and is typically accompanied by a Jahn-Teller distortion in the structure. [1-2] This lattice distortion along with its effect on the realization of ferrimagnetism as a consequence of the Cu^{2+} cationic distribution is of fundamental importance. We aim to investigate the control of lattice distortion of Cu doped spinel ferrite particles. In this paper, we report the fabrication and characterization of Cu doped spinel ferrite nanoparticles obtained by co-precipitation and the flux treatment method.

Experimental procedures

The samples were prepared with the addition of Cu^{2+} of different molar ratios to $FeFe_2O_4$ ($Cu_xFe_{(1-x)}Fe_2O_4$ (x=0, 3, 13, 50%)). Aqueous solutions containing Cu^{2+}, Fe^{2+}, and Fe^{3+} were mixed with NaOH aqueous solution to form a precipitate. The precipitate was then mixed with KBr flux, and annealed at 850℃ for 3 hours. The obtained particles were then rinsed with water a few times to remove the flux. Finally, the particles were reduced by in hydrogen gas at 380-530℃ for 3 hours. X-ray Diffraction (XRD) and a vibrating sample magnetometer (VSM) were used to characterize the materials.

Results

Figure 1 (a) shows the XRD pattern obtained from the sample with x=3% before reduction with H_2 gas. The confirmed hematite corundum structure is due to oxidation during high annealing temperature. After reduction, a single spinel phase could be confirmed (Fig.1 (b)). Fig. 2 shows diffraction patterns after reduction for samples with increasing Cu content (x=0, 3, 13, and 50%). Single spinel phase was obtained for sample x=0, 3, 13%. A slight peak attributable to Cu is observed when the Cu content is increased to x=50%. The crystallite sizes calculated from Scherrer's equation were in the range of 30 – 50 nm. The saturation magnetization of the sample with x=3% is 37.4 emu/g and coercivity was 171 Oe. By using this fabrication method, we were able to dope up to 13% of Cu and obtain a single phase spinel structure. All samples were cubic spinel and we were not able to find any Jahn-Teller distortion. In this case, further control in the annealing condition is necessary.

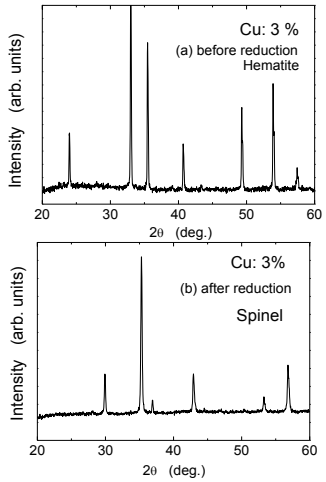

Fig.1. XRD patterns of sample x=3% (a) before and (b) after reduction with H_2 gas.

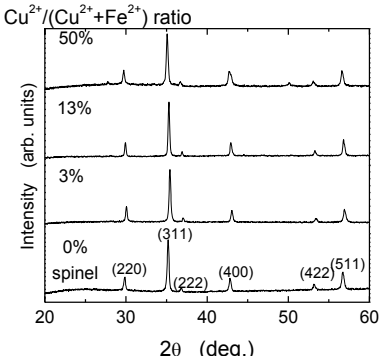

Fig.1. XRD patterns of samples x=0, 3, 13 and 50% after H_2 gas reduction.

References

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