Sol-gel synthesis and dilute magnetism of nano MgO doped with Fe

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Ferromagnetic semiconductor and insulator are expected as spintronics materials. Oxide insulators such as YAG and Al\textsubscript{2}O\textsubscript{3} show ferromagnetic behavior by doping dilute Fe\textsuperscript{3+}. MgO with large band gap (7.8 eV) is used for spintronics devices as thin insulator layer between ferromagnetic layers. We are interested in magnetism of dilute \textsuperscript{57}Fe\textsuperscript{3+} doped MgO, and tried to synthesize dilute Fe doped MgO powders by a sol-gel method. It is found that nano-powders of Mg ferrite are precipitated at high temperatures, and that the size of MgO powders can be easily controlled by annealing temperatures.

Fe doped MgO powders were synthesized by a sol-gel method. The nominal composition was prepared by mixing appreciate amounts of 0.5 M Mg solution and 0.01 M \textsuperscript{57}Fe solution. After citrate acid and ethylene glycol were added, the solution was heated at around 90 °C to get the gel, which was decomposed by heating at 250 °C for 2 hours, and annealing at 550 °C for 30 min. The crushed powders were further annealed at various temperatures for 3.5 hours. Crystal structures and magnetic properties are analyzed by XRD and vibrational sample magnetometer (VSM). Chemical states of iron are characterized by Mossbauer spectroscopy.

XRD patterns of sol-gel synthesized powder showed a cubic structure of MgO. The lattice parameters decreased a little and the crystalline size of MgO increased with increasing the annealing temperature up to 1000 °C as shown in Fig. 1. The crystalline size was not so strongly affected by different atmosphere (in vacuum condition of 10\textsuperscript{-5} torr). After heating at 1000 °C, the VSM showed strong ferromagnetism and XRD peaks of ferromagnetic MgFe\textsubscript{2}O\textsubscript{4} were clearly detected although VSM showed weak ferromagnetic behavior and no XRD peaks of MgFe\textsubscript{2}O\textsubscript{4} for samples heated up to 800 °C. The saturation magnetization was 0.4 \mu B/Fe, and the coercivity was 85 Oe. In the \textsuperscript{57}Fe Mossbauer spectra (as shown in fig.2), two kinds of doublets due to high spin Fe\textsuperscript{3+} were observed for samples heated until 800 °C. The full width half maximum (\Gamma) of doublets became small (0.85, 0.64, and 0.49 mm/s) with the increasing annealing temperatures (550, 600, and 800 °C). It is considered that the doublet D2 with small isomer shift (\delta) and large quadrupole splitting (\Delta) is due to paramagnetic Fe\textsuperscript{3+} in distorted lattice or at the grain boundary. Doublet D1 is considered to be due to Fe\textsuperscript{2+} substituted at Mg site in MgO or a part of super paramagnetic MgFe\textsubscript{2}O\textsubscript{4}. In the spectrum of sample heated at 1000 °C, two kinds of sextets (M1 and M2) were observed. The hyperfine fields of 41.3 T and 45.1 T are due to Fe\textsuperscript{3+} occupied at tetrahedral and octahedral sites in MgFe\textsubscript{2}O\textsubscript{4}. Two sextets are further decomposed into four sextets, which correspond to a part of inside core and another part of outside layer, respectively. The particle size for nano powder of MgO can be controlled by post-annealing at high temperatures.

![Figure 1. Temperature dependence on crystalline size of 1% Fe doped MgO, heated at various temperatures in air atmosphere.](image)

![Figure 2. Mossbauer spectra of 1% Fe doped MgO, annealed at 550, 600, 800, and 1000 °C for 3.5 hours in air atmosphere.](image)