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Magnetic properties of Sr-ferrites synthesized in molten (NaCl + KCl) flux

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Abstract

The Sr-ferrite powders, $SrFe_{12}O_{19}$, were synthesized by the molten salt method using (NaCl + KCl) mixture. Particle morphology was homogeneous and hexagonal platelet like. Both particle size and thickness increased as the reaction temperature and time increased. The sintering density of Sr-ferrite magnet prepared with powders by the molten salt method showed the maximum value at the sintering temperature of 1200 °C. The magnetic properties of the Sr-ferrite magnet were investigated with various sintering temperatures. The maximum values of remanent magnetization (σ_r, 45 emu/g) and coercivity field ($_jH_c$, 298 kA/m) occurred at the sintering temperatures of 1150–1200 °C. The Sr-ferrite magnet by a molten salt method showed higher remanent magnetization and coercivity field than those of the Sr-ferrite magnet prepared with the same starting materials by a conventional ceramic process.

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Keywords: Sr-ferrite; Molten salt method; Hexagonal ferrite; Magnetic properties

1. Introduction

Hexagonal barium and strontium ferrites have been intensively investigated during the last few decades due to their considerable importance to the electronic material industry. These hexagonal ferrites are characterized with high magnetocrystalline anisotropy, moderate hard magnetic properties and high chemical stability, compared with other magnetic materials [1–3]. The common processing methods of hexagonal ferrites are conventional ceramic process of solid-state reaction [4], co-precipitation/hydrothermal synthesis [5,6], sol–gel process [7], spray pyrolysis [8], and molten salt method [9,10], etc.

The conventional ceramic process, which includes mixing the raw materials, calcinations, milling, pressing, and sintering at 1200–1350 °C, has been often used in industrial manufacturing. However, this production process has difficulty to form the hexagonal platelets of homogeneous composition and uniform particle distribution. The chemical co-precipitation and sol–gel method were used to prepare the hexagonal ferrite for high-density magnetic media and microwave devices [11,12]. In addition to $SrCO_3$, $SrSO_4$, $BaCO_3$, and $BaSO_4$ as starting materials, the molten salt method utilizes various salts such as NaCl, KCl, Na_2SO_4, and Na_2CO_3, etc. The molten salt flux promotes the reaction of the compound, then resulting in forming the $SrFe_{12}O_{19}$ and $BaFe_{12}O_{19}$ at much lower reaction temperature (800–1000 °C) than the conventional ceramic process (1200–1350 °C) [13]. The molten salt method does not require any further pulverization that is common in the conventional ceramic process. In addition, the control of powder morphology is found to be much easier using this technique [14].

In this study the hexagonal platelet powders of Sr-ferrite were prepared by using the molten salts of NaCl and KCl and its characterizations were studied, with emphasis on the variation of platelet size as reaction temperature and reaction time. Also, its magnetic properties were investigated as sintering temperature and compared with the ferrite prepared by the conventional ceramic process.

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2. Experimental

The hexagonal strontium ferrites of nominal composition $SrO \cdot 5.7Fe_2O_3$ were synthesized starting from ball-milling mixtures of $SrCO_3$ (99.9%, Aldrich), $5.7Fe_2O_3$ (99.9%), 1.5(NaCl + KCl) and additives of $0.08Al_2O_3$, 0.10CaO, and $0.12SiO_2$ (the front number indicates molar ratio) in ethanol for 16 h. After drying at 60 °C for 6 h, the powder mixture was heated at the temperatures of 800–1200 °C for 2 h in a lid-covered alumina crucible with a heating rate of 5 °C/min in air. Then, after cooling to room temperature in furnace the oxide–molten salt mixture was washed with distilled water several times until the Cl^- free was confirmed by the $AgNO_3$ examination.

The preparation of Sr-ferrite powders with the conventional ceramic process was nearly identical to that of molten salt method, except that the salt of (NaCl + KCl) was not used. The starting powders of $SrCO_3$ (99.9%, Aldrich), $5.7Fe_2O_3$ (99.9%) and additives of $0.08Al_2O_3$, 0.10CaO, and $0.12SiO_2$ (molar ratio) were mixed using YSZ balls in ethanol for several hours. After drying, the powder mixture was calcined by heating at 1200 °C for 6 h in air and ground in ethanol using YSZ balls for 16 h.

In order to make the sintered magnet, the Sr-ferrite powders were wet mixed in acetone medium with addition of 4% polyvinyl alcohol (PVA) binder solution by using a ball mill. After drying the pellets of 16 mm × 12 mm × 4.4 mm were prepared by pressing at 600 MPa in a 10 kOe magnetic field applied along the pressing direction. Then, the pellets were sintered in a resistance heated furnace for 3 h at each specified level of sintering temperature from 1050 to 1250 °C. The magnetic properties such as remanent magnetization (σ_r) and coercivity field ($_jH_c$) of the sintered magnets were measured using a vibrating sample magnetometer (VSM, Lake Shore 735, USA) with an applied field of up to 796 kA/m (10 kOe). The crystal structure of samples was examined by using a X-ray diffractometer (Zeifert 3000 diffractometer) with CuK_α radiation. The microstructure was investigated using scanning electron microscopes (SEM, JSM-5800 and Hitachi S-1400). The sintered density of samples was measured by using the Archimedes water immersion technique.

3. Results and discussion

Fig. 1 shows the SEM micrographs of Sr-ferrite powders synthesized at 800 °C with different reaction times. The particles demonstrate uniform hexagonal platelet shape with the particle size of 1–2 μm. The size and thickness of platelet particles increases as the reaction time increases.

SEM micrographs of Sr-ferrite powders synthesized at different reaction temperatures for 2 h are also shown in Fig. 2. The size of platelet particles slightly increases by 1200 °C as the reaction temperature increases, while the aspect ratio (diameter to thickness) significantly decreases, then close to almost 1.0 at 1200 °C.

Fig. 3 shows the XRD pattern of the Sr-ferrite powders synthesized at 800 °C with different reaction times. XRD peaks of Fe_2O_3 were detected for the samples heated for 10 min and 1 h. However, its intensities decreased as the heating time increased, then completely disappeared after 2 h heating, indicating that the solid-state reaction was completed.

Table 1 shows the apparent sintered densities of the sintered Sr-ferrites which were measured by the Archimedes method. The Sr-ferrite powders were synthesized at 800 °C for 2 h in molten salt. The sintered density increased up to the highest value by 4.82 g/cm^3 at 1200 °C beyond

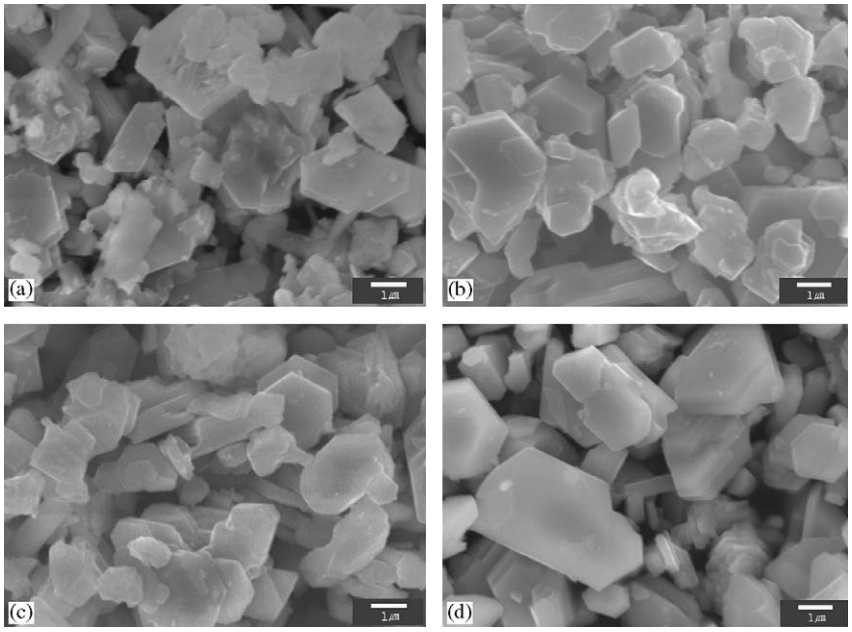

Fig. 1. SEM micrographs of Sr-ferrite powders synthesized at 800 °C for different reaction times: (a) 10 min; (b) 1 h; (c) 2 h; and (d) 6 h.