



Electromagnetic and microwave absorption properties of Fe–Sr_{0.8}La_{0.2}Fe_{11.8}Co_{0.2}O₁₉ shell-core composites

Zeyang Zhang^a, Xiangxuan Liu^{a,*}, Xuanjun Wang^a, Youpeng Wu^b, Yuan Liu^a

^a No. 503 Faculty, Xi'an Research Institute of High Technology, Xi'an 710025, China

^b Beijing Research Institute of High Technology, Beijing 100085, China

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ABSTRACT

Shell-core Fe–Sr_{0.8}La_{0.2}Fe_{11.8}Co_{0.2}O₁₉ composites are prepared by chemical vapor deposition (CVD) for use as microwave absorbing materials. Scanning electron microscopy and X-ray diffraction analyses show that the CVD method yields Sr_{0.8}La_{0.2}Fe_{11.8}Co_{0.2}O₁₉ powders with a uniform coating of Fe. Compared with Sr_{0.8}La_{0.2}Fe_{11.8}Co_{0.2}O₁₉, Fe–Sr_{0.8}La_{0.2}Fe_{11.8}Co_{0.2}O₁₉ composites have higher electrical conductivity, permittivity, and dielectric loss, which gradually increase with increasing Fe content. When Sr_{0.8}La_{0.2}Fe_{11.8}Co_{0.2}O₁₉/Fe=7:3, a reflection loss (RL) exceeding –10 dB is obtained in the frequency range of 10–14 GHz at a coating thickness of 2.0 mm. A minimum RL of –30 dB was found at 8.0 GHz, corresponding to a matching thickness of 2.8 mm.

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1. Introduction

The increase in electromagnetic pollution due to the rapid development of gigahertz-scale electronic systems and telecommunications has resulted in intense and growing interest in electromagnetic absorber technology [1–5]. An absorbent is the core of an absorbing material. Many studies have been carried out to investigate various aspects of microwave absorption materials with high magnetic and dielectric loss [6–8]. M-type strontium ferrite has a hexagonal molecular structure (SrFe₁₂O₁₉) and is a promising material for permanent magnets, advanced recorders, and microwave absorbers because of its fairly large magneto-crystalline anisotropy, high Curie temperature, relatively large magnetization, excellent chemical stability, and corrosion resistivity. However, its magnetic and electric properties must be adjusted to satisfy the requirements of different applications. In an attempt to improve the microwave absorption properties of SrFe₁₂O₁₉, La–Co substituted M-type strontium hexaferrites were prepared in our previous study. Sr_{0.8}La_{0.2}Fe_{11.8}Co_{0.2}O₁₉ was found to exhibit better microwave absorption compared with SrFe₁₂O₁₉. However, poor dielectric loss was also found to be an intrinsic disadvantage of Sr_{0.8}La_{0.2}Fe_{11.8}Co_{0.2}O₁₉ for microwave absorption.

Carbonyl iron (CI) powders, which features high saturation magnetization and relative permeability at radar wave frequencies is a superior material that is widely accepted as microwave absorbers [9–14]. The high-frequency dielectric properties of CI

powders have been found to be tunable by adjusting the iron content in the samples [15].

In this article, shell-core-structured Fe–Sr_{0.8}La_{0.2}Fe_{11.8}Co_{0.2}O₁₉ composites are prepared by chemical vapor deposition (CVD), and the influence of the iron shell on the crystal structure, morphology, complex permittivity and complex permeability, and microwave absorption properties of the Sr_{0.8}La_{0.2}Fe_{11.8}Co_{0.2}O₁₉ particles is investigated.

2. Experimental

2.1. Synthesis of Sr_{0.8}La_{0.2}Fe_{11.8}Co_{0.2}O₁₉

Sr_{0.8}La_{0.2}Fe_{11.8}Co_{0.2}O₁₉ hexagonal ferrites were prepared by a sol–gel–auto combustion method. Based on Sr_{0.8}La_{0.2}Fe_{11.8}Co_{0.2}O₁₉, stoichiometric amounts of strontium nitrate, cobalt nitrate, lanthanum nitrate, and iron nitrate were dissolved in de-ionized water, and an appropriate amount (1:1 mol ratio of citric acid to NO₃) of citric acid was added to complex the metal ions completely. The pH value of the sol was adjusted to 6.0 using ammonia solution (25 wt%). The solution was evaporated at 80 °C until a viscous gel formed. The gel was air-dried at 120 °C, ignited in air, and burned into dendritic powders. Finally, the precursor powder was sintered at 1200 °C for 3 h.

2.2. Synthesis of Fe–Sr_{0.8}La_{0.2}Fe_{11.8}Co_{0.2}O₁₉ shell-core composite

Synthesis was performed in a CVD setup designed by our group. A typical preparation process for Fe–Sr_{0.8}La_{0.2}Fe_{11.8}Co_{0.2}O₁₉

* Corresponding author. Tel.: +86 15902957276; fax: +86 209 83348220.
E-mail address: xiangxuan_study@yahoo.com (X. Liu).

composites is as follows: First, 1 g of $\text{Sr}_{0.8}\text{La}_{0.2}\text{Fe}_{11.8}\text{Co}_{0.2}\text{O}_{19}$ powders and 5 mL of iron pentacarbonyl [$\text{Fe}(\text{CO})_5$] were added into an airproof reactor and evaporator, respectively. $\text{Fe}(\text{CO})_5$ was evaporated at 65 °C, while $\text{Sr}_{0.8}\text{La}_{0.2}\text{Fe}_{11.8}\text{Co}_{0.2}\text{O}_{19}$ powders were heated and maintained at 250 °C. $\text{Fe}(\text{CO})_5$ vapor was bowed into the reactor by argon gas at a rate of 150 mL/min for 3, 9 and 18 min. To obtain a uniform coating, the reactor was vigorously and continuously stirring mechanically. Shell-core $\text{Fe}-\text{Sr}_{0.8}\text{La}_{0.2}\text{Fe}_{11.8}\text{Co}_{0.2}\text{O}_{19}$ composites were obtained after slow-cooling to room temperature, and the whole experiment was protected by argon. The weight content of iron in the composites was determined by comparing the weights of specimens before and after the experiment. The mass ratios of $\text{Sr}_{0.8}\text{La}_{0.2}\text{Fe}_{11.8}\text{Co}_{0.2}\text{O}_{19}$ to Fe were approximately 9:1, 7:3 and 4:6.

2.3. The preparation of composites coatings

The $\text{Fe}-\text{Sr}_{0.8}\text{La}_{0.2}\text{Fe}_{11.8}\text{Co}_{0.2}\text{O}_{19}$ powders were dispersed into the epoxide resin via adding solvent and a high energy ultrasonic treatment for 30 min. Afterwards, hardener was added into the mixtures, followed by stirring at 1000 rpm for 10 min. Finally, the composite materials are fabricated on an aluminum substrate with a standard size (180 mm × 180 mm × 3 mm).

2.4. Measurement of properties

The phase structure of the powder was identified by X-ray diffraction (XRD; D/max-IIB, Japan). A VEGA II XMU INCA scanning electron microscope (SEM) was employed for morphological analysis. EM parameters (i.e., complex permeability and permittivity) were measured using a vector network analyzer (HP-8720ES) in the frequency range of 2–18 GHz. The samples used for EM parameter measurements were prepared by dispersing powders into paraffin wax at a mass fraction of 60%, and then pressing the mixtures into a compact toroidal shape with outer and inner diameters of 7.0 and 3.0 mm, respectively. The reflection loss of the prepared absorbers versus the frequency is studied using an HP 8510B vector network analyzer and standard horn antennas in an anechoic chamber.

3. Results and discussion

3.1. SEM analysis

An SEM image of the $\text{Sr}_{0.8}\text{La}_{0.2}\text{Fe}_{11.8}\text{Co}_{0.2}\text{O}_{19}$ particles shows irregularly shaped platelet-like crystals with smooth surfaces, as shown in Fig. 1(a). The particles stack on top of each other due to magnetic attraction between particles and are uniformly coated with iron, as illustrated in Fig. 1(b)–(d). The $\text{Fe}-\text{Sr}_{0.8}\text{La}_{0.2}\text{Fe}_{11.8}\text{Co}_{0.2}\text{O}_{19}$ micro-structured composites have a full shell-core structure. When a small amount of $\text{Fe}(\text{CO})_5$ vapor was added, CI particles with average diameters of approximately 0.02–0.1 μm were found in some of the $\text{Sr}_{0.8}\text{La}_{0.2}\text{Fe}_{11.8}\text{Co}_{0.2}\text{O}_{19}$ particles [Fig. 1(b)]. $\text{Sr}_{0.8}\text{La}_{0.2}\text{Fe}_{11.8}\text{Co}_{0.2}\text{O}_{19}$ particles coated with CI showed coarse surfaces, as shown in Fig. 1(c). An increase in the CI content of the $\text{Fe}-\text{Sr}_{0.8}\text{La}_{0.2}\text{Fe}_{11.8}\text{Co}_{0.2}\text{O}_{19}$ composites changed the shape of the particles to coralloid, and their arris disappeared due to increasing thickness of the carbonyl shells.

Fig. 2 (a) and (b) are the surface and section SEM observation, which show the microstructure of the coating filled with 60% $\text{Fe}-\text{Sr}_{0.8}\text{La}_{0.2}\text{Fe}_{11.8}\text{Co}_{0.2}\text{O}_{19}$ composites. It was observed that the fillers are well dispersed in the EP matrix and no significant porosity was noticed.

3.2. Crystal structures

Fig. 3 shows the X-ray diffraction (XRD) patterns of $\text{Sr}_{0.8}\text{La}_{0.2}\text{Fe}_{11.8}\text{Co}_{0.2}\text{O}_{19}$, CI, and $\text{Fe}-\text{Sr}_{0.8}\text{La}_{0.2}\text{Fe}_{11.8}\text{Co}_{0.2}\text{O}_{19}$ composites. Fig. 3(b) shows peaks at 19.34°, 23.22°, 30.93°, 31.13°, 32.61°, 34.2°, 36.05°, 37.80°, 41.06°, 57.46°, and 64.3°, which are respectively attributed to the (102), (006), (110), (008), (107), (201), (203), (205), (206), (2011), and (220) phases of the $\text{Sr}_{0.8}\text{La}_{0.2}\text{Fe}_{11.8}\text{Co}_{0.2}\text{O}_{19}$ particles. These peaks can be indexed to the pure M-type hexa ferrite structure, in agreement with JCPDS Card no. 720739. No impurity peaks were detected, indicating the high purity of the powders. The shell-core-structured $\text{Fe}-\text{Sr}_{0.8}\text{La}_{0.2}\text{Fe}_{11.8}\text{Co}_{0.2}\text{O}_{19}$ composites [Fig. 3(c)] had similar diffraction peaks to $\text{Sr}_{0.8}\text{La}_{0.2}\text{Fe}_{11.8}\text{Co}_{0.2}\text{O}_{19}$ and CI, but the intensity of the peaks decreased. In addition, a peak around $2\theta=44.6^\circ$ was observed, indicating that the shell layer was composed of iron.

3.3. Complex permittivity and permeability

Fig. 4 shows the complex permittivity and permeability of pure $\text{Sr}_{0.8}\text{La}_{0.2}\text{Fe}_{11.8}\text{Co}_{0.2}\text{O}_{19}$ and $\text{Fe}-\text{Sr}_{0.8}\text{La}_{0.2}\text{Fe}_{11.8}\text{Co}_{0.2}\text{O}_{19}$ composites. Fig. 4(a) and (b) respectively show the real (ϵ') and imaginary (ϵ'') parts of the permittivity of the specimens plotted as a function of frequency. ϵ' and ϵ'' represent the energy storage ability and loss ability, respectively. The ϵ' and ϵ'' values of the specimens remained constant with slight fluctuations over the entire frequency range and increased with increasing weight ratio of Fe. The ϵ' and ϵ'' of $\text{Sr}_{0.8}\text{La}_{0.2}\text{Fe}_{11.8}\text{Co}_{0.2}\text{O}_{19}$ were almost constant with nearly no difference throughout the frequency range studied ($\epsilon'=6$, $\epsilon''=0.5$), indicating poor dielectric loss. When the mass fraction of Fe was increased by 60%, the ϵ' values increased from 6 to 12 while the ϵ'' values increased from 0.5 to 2.3. Dielectric loss commonly results from electron polarization, iron polarization, and electric dipolar polarization [16]. The experiments showed that the Fe shell, which forms a surface layer, greatly increases the conductivity of the samples and contributes to the enhancement of permittivity. Because of the metallic properties of the shell, electrons can travel freely and accumulate on the $\text{Fe}-\text{Sr}_{0.8}\text{La}_{0.2}\text{Fe}_{11.8}\text{Co}_{0.2}\text{O}_{19}$ interface, forming a structure similar to a boundary-layer capacitor and generating interfacial electric dipolar polarization. According to previous research [17], electron and iron polarization only occur at frequencies higher than the infrared range. Thus, the enhanced dielectric loss found in our samples could be attributed to electric dipolar polarization. Optimizing the weight ratio of Fe would likely cause the composites to attain the permittivity necessary in microwave absorbing materials.

The real (μ') and imaginary (μ'') parts of the permeability are shown in Fig. 5(a) and (b), respectively. An intersecting point at 8.5 GHz was observed on the curve of μ' . Higher μ' values were found for $\text{Fe}-\text{Sr}_{0.8}\text{La}_{0.2}\text{Fe}_{11.8}\text{Co}_{0.2}\text{O}_{19}$ composites with higher Fe contents prior to this intersecting point. Inverse changes were detected after the intersecting point. Meanwhile, the μ'' of $\text{Fe}-\text{Sr}_{0.8}\text{La}_{0.2}\text{Fe}_{11.8}\text{Co}_{0.2}\text{O}_{19}$ was uniformly smaller than that of pure $\text{Sr}_{0.8}\text{La}_{0.2}\text{Fe}_{11.8}\text{Co}_{0.2}\text{O}_{19}$ over the range of 2–18 GHz, and decreased with increasing Fe content. Broad peaks at 3–7 GHz were present in the $\text{Fe}-\text{Sr}_{0.8}\text{La}_{0.2}\text{Fe}_{11.8}\text{Co}_{0.2}\text{O}_{19}$ composites. Generally, for ferrite magnetic materials, the microwave magnetic loss originates mainly from domain wall resonance and natural ferromagnetic resonance [18]. Domain wall resonance is usually exhibited in the low-frequency region (< 2 GHz). However, resonance due to the spin rotational component occurs at high-frequency regions. Thus, the resonance peak observed may be attributed to natural resonance.

3.4. Microwave absorption properties

The normalized input impedance, Z_{in} , of a metal-backed microwave absorption layer is derived from the following

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