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Selective microwave absorption in Nd^{3+} substituted barium ferrite composites

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ABSTRACT

Microwave (MW) frequency based wireless communications and electronic devices became prospective due to several ramifications. To meet this need, a series of neodymium ions (Nd^{3+}) substituted barium ferrite composites with composition $(20)BaO:(80\text{-}x)Fe_2O_3:(x)Nd_2O_3$ ($0 \le x \le 3$ mol%) was prepared at 1100 °C using solid-state reaction method. We evaluated the effect of various Nd^{3+} ions contents on the surface morphology, structure, and magnetic properties of the as-synthesized barium ferrite composites. Meanwhile, microwave reflection loss, complex permittivity, and permeability ware determined using the transmission/reflection line method in the X-band (8–12 GHz). SEM image of the composites showed that the surface morphology consisted of rough and porous microstructures. XRD patterns of the un-doped composites revealed the existence of $BaFe_{12}O_{19}$ (hexagonal) and $Fe_{21.333}O_{32}$ (tetragonal) crystalline phases. Furthermore, a new hexagonal crystalline phase of $Ba_6Nd_2Fe_4O_{15}$ with the crystallite sizes between 15 and 67 nm was observed due to Nd^{3+} ions substitution in the composite. The saturation magnetization of the composite containing 2 mol% of Nd^{3+} did not exhibit any significant alteration compared to the one devoid of Nd^{3+}. The complex relative permittivity and permeability of the achieved composites enriched in $Ba_6Nd_2Fe_4O_{15}$ and $BaFe_2O_4$ phases disclosed significant MW frequency dependence. The composites also displayed selective MW absorption in the X-band which could be useful for diverse applications.

Keywords: neodymium doping, barium ferrite composites, magnetic properties, porosity, MW reflection loss

1. Introduction

In recent times, wireless communications and electronic devices based on the microwave (MW) frequency in the range of 8-12 GHz (X-band) received focused attention, wherein dedicated efforts have been made to achieve the emission of such frequencies from a transmitter and radar. Materials required for such purposes must be capable in attenuating and dissipating the excess amount of electromagnetic energy in the form of thermal energy via magnetic and dielectric loss mechanisms [1]. Thus, novel magnetic materials (ferrites based) appropriate for absorbing the electromagnetic energy (MW) have continually been explored [2–10]. In this regard, barium hexaferrite (BHF) owing to its extremely high uniaxial magnetic anisotropy appeared an emergent MW absorbing material [3–5,11,12]. Thus, it became essential to optimize the MW absorption capacity of these ferrites in the X-band (8–12 GHz).

It was demonstrated that the natural resonance-frequencies, saturation magnetization, permeability, permittivity and magnetic field anisotropy of such ferrites could be altered by incorporating divalent or trivalent ions into the crystalline structures [13–15]. Consequently, the effects of electromagnetic interferences in these ferrites could be improved. Meanwhile, outstanding relaxation attributes of rare earth ions (REIs) as dopants inside the BHF have also been exploited to alter its magnetic and MW absorption characteristics [3,13,16–19]. Besides, BHF doped with REIs revealed weak coercive field (H_c) and low saturation magnetization (M_s)

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which could be useful for the enhancement of MW absorption capacity [6,13,20,21]. Inspired by these innovations, we took an attempt to diminish the values of H_c and M_s of the prepared barium ferrite composites (BFCs), thereby enhancing the selective absorption of MW frequencies.

This communication reports the production and characterizations of new BFCs (hereafter referred as NdBFCs) wherein the Fe^{3+} ions in pure BHF were replaced by Nd^{3+} ions (at various concentration). Trivalent Nd^{3+} due to their compatible hexagonal lattice structure and excellent relaxational attributes were selected as doping agents (so-called substituent ions). Two types of composites (undoped and doped with Nd^{3+} ions) were synthesized via tailored solid–state reaction method. As-prepared composites were characterized using different analytical tools to determine the influence of Nd^{3+} ions on the microstructures, surface morphologies, magnetic characteristics, and MW reflection loss in the X–band. Achieved $Ba_6Nd_2Fe_4O_{15}$ and $BaFe_2O_4$ phases of the proposed BFCs were shown to be beneficial for sundry applications.

2. Materials and methods

Few BFCs (one without and three with Nd^{3+} ions doping) with the composition of $(20)BaO{:}(80\text{-}x)Fe_2O_3{:}(x)Nd_2O_3$, where $0 \leq x \leq 3$ mol% were prepared using modified solid-state reaction strategy. High purity powders (analytical grade) of $BaCO_3$ (Merck, 99%), Nd_2O_3 (Sigma Aldrich, 99%) and Fe_2O_3 (Sigma Aldrich, 99%) were used as preparatory materials to synthesize such BFCs. Furthermore, the $BaCO_3$ powder was calcined at 300 °C for an hour to remove the existed carbon element. Later, BaO powder was mixed gradually with Fe_2O_3 via annealing and sintering processes. Afterward, the Nd_2O_3 powder was added to the mixture and thoroughly mixed using a magnetic stirrer at the revolution speed of 700 rpm (rotation per minute) for two hours. Polyvinyl alcohol (PVA) was mixed and stirred continually until a sol was achieved. After that, the composite powder was compacted to produce pellets with the respective thickness and diameter of 1 mm and 1 cm [13]. Besides, some pellets of rectangular shape with a dimension of (2.5 cm × 1.5 cm × 0.2 cm) were obtained for additional characterizations. These pellets were further annealed at 800 °C for one hour and sintered at 1100 °C for five hours [22] before being cooled down naturally to room temperature. The achieved pellets were named as NdBF0, NdBF1, NdBF2, and NdBF3 based on their corresponding Nd^{3+} ions content of 0, 1, 2, and 3 mol%. Finally, the prepared composites were characterized at room temperature using different analytical techniques to determine the feasibility of achieving their selective MW absorption effectiveness in the X-band.

A scanning electron microscope (SEM, Hitachi SU 3500) equipped with energy dispersive X-ray (EDX, Horiba) was utilized to determine the surface morphologies, microstructures and elemental compositions of the synthesized NdBFCs [13,21,23]. The presence of crystalline phases in the studied NdBFCs was verified using X-ray diffraction measurement (XRD, SmartLab 3 kW) equipped with Cu–Kα radiation of wavelength (λ) 0.1541874 nm. A vibrating sample magnetometer (VSM, Oxford 1.2H) was employed to examine the magnetic behavior of the studied composites. The scattering (*S*) parameters of the proposed composites were measured using a vector network analyzer (VNA, Advantest R3770) operated in the frequency range of 8–12 GHz. Additionally, the transmission/reflection line method was exploited to determine the MW reflection loss (R_L), the complex relative permittivity, and permeability.

3. Results and Discussion

3.1. Surface morphology and elemental traces

Fig. 1 displays the SEM images of all synthesized composites, wherein the surface morphology consisted of porous microstructures with roughness. Overall, the composites surface morphology and the distribution of particles size were influenced due to the Nd^{3+} ions substitution into the crystalline lattices. At higher Nd^{3+} concentrations, the microstructure manifested two phases including the hexagonal grains of barium ferrite (dark color) and Nd-rich (bright color) regions. The microstructure of NdBF2 was more porous than the other samples. The mean grain size BFCs was shrunk from 2.5 to 1 µm with the increase in the Nd^{3+} ions content from 0 to 3 mol% accompanied by porosity enhancement. This enrichment in the porosity led to an improvement in the magnetic characteristics and MW-reflection loss [21]. Fig. 2 depicts the EDX spectra of all BFCs, confirming the appropriate elemental traces of O, Fe, Ba, and Nd wherein the inclusion of Nd in the composite caused an adjustment in the level of other elements (Table in the inset). Sample NdBF2 revealed the minimum traces of Nd (0.1 At.%), and NdBF3 exhibited the highest level of Nd (0.3 At.%).

Fig. 1. SEM micrographs of the as-synthesized NdBFCs.

Fig. 2. EDX spectrum of the as-synthesized NdBFCs

3.2. Crystalline structure and sizes

Fig. 3 illustrates the XRD patterns of the synthesized composites which comprised of several sharp peaks characteristics of different crystalline lattices. For the sample without Nd^{3+} substitution (NdBF0), all the observed peaks were assigned to the dominant hexagonal crystalline structure of $BaFe_{12}O_{19}$ which matched with the ICDD number 00-027-1029 with crystallographic parameters of $a = b =$ 0.5892 nm, $c =$ 2.3198 nm, $\alpha = \beta = 90°$ and $\gamma = 120°$. However, the peak appeared at 28.36° was allocated to the tetragonal crystal structure of $Fe_{21.333}O_{32}$ which was tallied with the ICDD numbers 01-076-1470. Furthermore, substitution of Fe^{3+} by Nd^{3+} ions (at 1 mol% of Nd_2O_3) in the barium ferrite lattice could produce a significant change in the in the crystal structure as verified by the XRD pattern of NdBF1. The emergence of two new peaks in NdBF1 corresponding to a primary phase of $Ba_6Nd_2Fe_4O_{15}$ and a secondary phase of $BaFe_{18}O_{27}$ were clearly disclosed. The occurrence of sharp XRD peaks were allotted to the hexagonal lattice of $Ba_6Nd_2Fe_4O_{15}$ (matched with the ICDD number 01-079-2141) with $a = b =$ 1.1815 nm, $c =$ 0.7078 nm, $\alpha = \beta = 90°$ and $\gamma = 120°$. Moreover, the peak centered at 32.17° was endorsed to the hexagonal crystalline lattice of $BaFe_{18}O_{27}$ (matched with the ICDD number 01-075-0406). The inclusion of 2 mol% of Nd_2O_3 (sample NdBF2) into the BFCs could transform the crystalline phase from hexagonal $BaFe_{18}O_{27}$ to orthorhombic $BaFe_2O_4$ structure (matched with the ICDD number 01-077-2337). Meanwhile, further addition of Nd_2O_3 up to 3 mol% in the BFCs (sample NdBF3) led to an alteration in the XRD peak position of $BaFe_2O_4$ without significant variations in the phase and structure. It was affirmed that the replacement of Fe^{3+} by Nd^{3+} ions in the studied BFCs could generate a new primary hexagonal crystalline phase of $Ba_6Nd_2Fe_4O_{15}$. Widening of the diffraction peaks for all as-synthesized NdBFCs is credited to the nano-crystalline size and lattice strain [24,25]. The crystallite sizes of the studied $Ba_6Nd_2Fe_4O_{15}$ (calculated using the Debye-Scherrer's equation) were ranged from 15 to 67 nm.

Fig. 3. XRD patterns of all as-synthesized NdBFCs

3.3. Magnetic Properties

Fig. 4 depicts the magnetization (M) concerning the applied field (H) of all the studied barium ferrites without and with Nd^{3+} ions substitution. The composite without containing Nd^{3+} ions (NdBF0) revealed that the saturation magnetization (M_s), remanence magnetization (M_r), and coercivity field (H_c) were 16.36 emu/g, 11.82 emu/g, and 0.24 T, respectively. The overall magnetic behavior of the composite (NdBF1), except for the M_s did not significantly change due to addition 1 mol% of Nd_2O_3. The Inclusion 2 mol% of Nd_2O_3 into the composite (NdBF2) did not affect the value of M_s. However, incorporation 3 mol% of Nd_2O_3 into the composite (NdBF3) could lead to an increase in the value of M_s comparable to NdBF1, which was ascribed to the enhanced porosity of the barium ferrite surface. It was argued that the occurrence of such excessive porosity could isolate the magnetic domain walls, leading to natural polarization of the unpaired magnetic spins in the presence of an external applied magnetic field. The magnetic hysteresis loop of NdBF0 coincided with NdBF2. Similarly, the magnetic hysteresis loop of NdBF1 was overlapped with NdBF3. Thus, it was established that such pair ferrites (NdBF0 and NdBF2 as well as NdBF1 and NdBF3) had almost identical magnetic properties. Disclosure of such narrow hysteresis loops (loop-gap area) indeed confirmed the soft magnetic character of the proposed BFCs. On top, the studied composites failed to display the complete saturation magnetization because of the presence of antimagnetic materials such as $BaFe_2O_4$ and $Fe_{21.333}O_{32}$. The underlying development of the non-magnetic phase because of a sintering procedure at high temperature is likewise in charge of altering M_s [25,26].

Fig. 4. Hysteresis loops of the studied NdBFCs

3.4. Complex relative permitivity and permeability

Fig. 5a shows the frequency-dependent complex relative permittivity (ε_r) for all composites in the X-band. The real (ε') and imaginary (ε'') components of the permittivity of the prepared composites aroused from the intrinsic electric dipole polarization were strongly depended on the MW frequencies [27]. Actually, the real part of the permittivity signified the energy stored in the material from an external electric field [28]. The real part of the permittivity revealed three prominent resonance peaks around 8.7, 10.0, and 11.8 GHz, indicating the storage of energy in the composites from the electric field. Nevertheless, the lack of stored energy could be observed at other MW frequencies. Meanwhile, the imaginary part of the permittivity signified the dielectric loss factor, denoting the energy dissipation capacity of the material. The appearance of the resonance peaks in the ε' spectrum was trailed by the disappearance of the resonance peaks in the ε'' spectra [13]. It was affirmed that the proposed composites presented the best energy storage performance around 11.6 GHz, wherein the absorbed energy was consumed for the magnetic domain wall motion.

Fig. 5b illustrates the frequency dependent complex relative permeability for all composites in the X-band. The real part of the permeability (μ') exhibited three significant resonance peaks around 8.5, 9.3, and 11.0 GHz, demonstrating the magnetic energy storage capacity of the composites. Alternatively, the imaginary part of the permeability (μ'') revealed the magnetic loss factor around 8.2, 9.0, and 10.5 GHz, which was allocated to the natural resonance frequency f_r [13]. Besides, the magnetic loss factor was increased at the resonance frequency. It was observed that the substitution of Fe^{3+} in the BFCs by Nd^{3+} at varied concentrations caused the frequency shift. This disclosure was attributed to the emergence of different phase and porosity in the composites. The imaginary part of the permeability plays an important role in the microwave absorbing property of hexa-ferrites [29] that related to the relaxation of domain wall resonance [30].

Fig. 5. Frequency-dependent complex relative (a) permittivity and (b) permeability for all NdBFCs

3.5. Microwave reflection loss

Fig. 6 represents the variation in the reflection loss (R_L) of all prepared BFCs as a function of applied MW frequency (f). The NdBF0 and NdBF3 composites revealed a similar trend in the R_L pattern which comprised of two weak absorption bands with values above -10 dB.

Meanwhile, the NdBF1 specimen exhibited R_L value below -10 dB at frequencies range of 9.14 to 10.18 GHz. Besides, NdBF2 composite disclosed two prominent absorption bands centered around 9.94 and 11.64 GHz with R_L values of -22.94 and -23.82 dB, respectively. All the observed spectral bands showed a significant shift with varying contents of Nd^{3+} ions with the bandwidth of less than 1 dB. In short, the microwave absorption capacity of the proposed BFCs was improved due to Nd^{3+} ions substitution assisted enhanced porosity in the structure that in turn could confine the MW. Consequently, the Nd^{3+} ions substitution into the BFs could change the magnetocrystalline anisotropy direction of the composites, leading to an alteration of the natural resonance frequency. This alteration could finally cause a shift of the MW reflection loss to high-frequency zone as revealed by NdBF2. Interestingly enough, the doped composites enriched in $Ba_6Nd_2Fe_4O_{15}$ and $BaFe_2O_4$ phases could absorb microwaves selectively in the X-band.

Fig. 6. Frequency-dependent MW reflection loss of all the proposed NdBFCs

4. Conclusion

Synthesis and room temperature characterization of some new hexagonal barium ferrite composites with Nd^{3+} ions substitution was reported. An appropriate amount of chemical constituents were gradually mixed to prepare these composites following solid-state reaction route. The proposed BFCs revealed the excellent capacity of MW absorption in the X-band. EDX spectra revealed the right elemental compositions of the prepared BFCs and presence of Nd in the structures. The surface morphology, structure, magnetic properties and MW absorption ability of the prepared BFCs were strongly influenced because of the inclusion of Nd^{3+} ions into the crystalline lattice. The XRD pattern disclosed the formation of new crystalline phases of $Ba_6Nd_2Fe_4O_{15}$ and $BaFe_2O_4$ due to the incorporation of Nd^{3+} ions into the BFCs structure. SEM images of the studied BFCs exhibited highly rough and porous surface morphology. At 2 mol% of Nd^{3+} ions, the saturation magnetization of the BFCs displayed insignificant alteration. The complex relative permittivity and permeability of the studied BFCs was found to depend appreciably on the MW frequencies. Furthermore, the achieved low reflection loss at 9.94, and 11.64 GHz frequencies with selective MW absorption shown by the proposed BFCs may be advantageous for diverse device applications in the X-band.

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