Synthesis and electromagnetic characteristics of MnFe$_2$O$_4$/TiO$_2$ composite material

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Abstract
The development of absorbing materials with a wide frequency band is still challenging. Here we successfully fabricated the MnFe$_2$O$_4$/TiO$_2$ composite powder through the micro-emulsion method. We characterized the phase structures, morphologies, particle size, and chemical compositions of MnFe$_2$O$_4$/TiO$_2$ composite powders, and measured the microwave absorption capacity as well. The MnFe$_2$O$_4$/TiO$_2$ composite powders present a coating structure. The microwave absorption test revealed that the maximum microwave loss reaches $-38.6$ dB at a thickness of 3.05 mm and the corresponding bandwidth is about 4 GHz in the ranges from 7.8 to 11.4 GHz. The excellent performance is attributed to the high surface area endowed by the coating structure. This research provided an alternative route for the preparation of high bandwidth electromagnetic wave absorbing materials.

1. Introduction

With the widespread use of communication devices, which provide convenient services in daily life, the electromagnetic environment is suffering from rapid deterioration [1, 2]. Thus, it is urgent to develop environmental-friendly, high-performance electromagnetic wave-absorbing material. To date, numerous research has been devoted to exploiting a type of microwave-absorption materials with high saturation magnetization, wide effective frequency range, strong absorption, low density, and high resistivity in the GHz range [1, 3–6]. Relevant research has confirmed that conventional absorbing materials such as ferrite materials, metallic magnetic materials, conductive polymers, and carbon-based electromagnetic wave shielding materials can overcome the Snoek limit and extend the magnetic resonance frequency to GHz [2, 7–15]. Kong et al. make a summary of characteristics, which include density, absorption frequency, broad bandwidth, absorption properties, mechanical performance, and cost et al., of different lightweight and broadband wave-absorbing materials. They conclude that ferrite materials, such as MnFe$_2$O$_4$, are the kind of absorbing material with optimal overall properties [16].

Unfortunately, the abovementioned absorbing materials with a single material constructed have limited properties, including ferrite materials, and are difficult to meet the increasing requirements of impedance matching and electromagnetic absorption [1, 17]. Numerous studies have shown that introducing reinforcing dielectric materials into Fe-based alloy particles or forming dielectric coating layer nanocomposites can improve the electromagnetic wave dissipation capacity [18–25]. The primary reason is that the above methods can help to regulate the multiple dielectric relaxations, complex permittivity, attenuation capacity, and impedance matching. In general, TiO$_2$, a typical semiconductor, has high resistivity and low complex permittivity. It is used to weaken the eddy current effect and optimize the impedance matching degree. Liu et al. reported that the minimum reflection loss value of TiN@TiO$_2$ nanocomposites achieves $-35.9$ dB at 10.4 GHz. The excellent
properties are attributed to the optimized impedance matching and the introduction of plenty of heterogeneous interfaces [26]. Green et al demonstrated that Mg/H treated TiO2 nanoparticles present extraordinary dielectric and magnetic properties as well as excellent microwave absorbing performance [27]. Therefore, to broaden the narrow absorbing frequency band of ferrite and improve its absorbing performance, here we select TiO2 to form a dielectric coating layer and achieve the above goals with the help of the synergistic effect [19, 26, 28, 29].

In this context, attempts have focused on improving the electromagnetic wave-absorbing property by forming the MnFe2O4/TiO2 nanocomposite powders with coating structure through the micro-emulsion method. The crystalline structure, microstructure, microwave absorption properties, and the effect of TiO2 on the surface of MnFe2O4 are discussed.

2. Experimental

In the present work, MnSO4·H2O, FeSO4·7H2O, FeCl3·6H2O, FeCl2, NaOH, Ti(OCH2CH3)4, H2O2, C6H12, NH3·H2O, CH3COCH3, C19H42BrN, C6H14O, Ti(SO4)2, C33H60O10, C4H13NO and H2SO4 were used as raw materials, and all reagents are of analytical grade.

2.1. Preparation of manganese ferrite powder

The preparation process for manganese ferrite powder consists of three stages, and the detailed procedures are described below.

2.1.1. Preparation of microemulsions

The initial composition of microemulsion is deionized water (30 wt%), CTAB (30 wt%) and n-Hexanol (40 wt%), and subsequently three different microemulsions were obtained by adding different substances. Microemulsion I: MnSO4·1H2O and FeSO4·7H2O were dissolved in deionized water, and the mixed solution with Mn/Fe molar ratio of 1/2. Then, adding a vitriol solution to the mixed solution is set to adjust the PH value to 2.5. Finally, the resulting solution was mixed with the original microemulsion.

Microemulsion II: A certain amount of (TMAH)-(CH3)4NOH was dissolved into deionized water to obtain a solution, and then mixed with the original microemulsion.

Microemulsion III: Appropriate amount of H2O2 was added to the primordial microemulsion.

2.1.2. Precipitation reaction

The microemulsion I and II were mixed, followed by stirring and finally standing at least 20 min to obtain precipitates. To get the optimal pH value (11.8), the extra microemulsion II was added to the mixed solution. After an hour of precipitation reaction, the microemulsion III was added to oxidate the precipitates. The precipitation reaction was carried out at about 50 °C.

2.1.3. Washing and separation of product

A certain amount of deionized water and alcohol was added to the precipitate reaction product, and then obtained the precipitates through centrifugation. In order to ensure the purity of the product, the product was washed repeatedly. The precipitates were dried at 70 °C for 6 h and followed by calcining at 1200 °C for 6 h.

2.2. Preparation of composite

The solution was prepared by mixing 15 ml cyclohexane, 3.6 ml NPE (nonylphenol polyoxyethylene ether), and 3.6 ml hexanol and stirring for 20 min. And then 200 μl MnFe2O4 nanoparticles were added to the solution and stirred for 30 min. After that, 120 μl NH4OH and 120 μl PDDA solution were added to the mixed solution and stirred for 20 min. Afterward, Ti(SO4)2 was added to the above solution and kept stirring for 24 h. For accelerating the precipitation reaction, acetone was added to break the equilibrium of the mixed solution. Finally, separating and washing the precipitates with deionized water and alcohol, and then drying the precipitates at 60 °C for 6 h to obtain the composite powders.

2.3. Measurements

To investigate the crystalline structure of the composite powder, the x-ray diffraction (XRD) experiments were performed using a Rigaku Ultima IV diffractometer with a Cu-Kα radiation source. The scan range (2θ) was set to 10°—90°, and the angle resolution was set to 0.02° with a 0.5 s integration time to ensure an accurate measurement. The microstructures of materials were examined by scanning electron microscopy (SEM, Hitachi S-4800) equipped with an energy dispersive spectrometer (EDS). The specimen, which presents toroidal shape with a height of 2 mm, for microwave absorption performance test were prepared by mixing 80 wt%
MnFe₂O₄/TiO₂ composite powder with paraffin. The external diameter and inner diameter of the toroidal shape specimen were 7 mm and 3.04 mm, respectively. The relative permittivity and permeability values of the specimen were measured ranging from 2 GHz to 18 GHz using a network analyzer Agilent HP-8722ES.

3. Results and discussion

3.1. Crystalline structure analysis

Figure 1 (a) shows the XRD patterns of the MnFe₂O₄ powder, TiO₂ powder and MnFe₂O₄/TiO₂ composite powder. According to the JCPDS file No.73–1964, the MnFe₂O₄ powder with face-centered cubic (FCC) crystal structure belongs to manganese ferrite. Of special noting is that the diffraction peaks of MnFe₂O₄ shift back to the lower angle, as shown in Figure 1 (b). The above phenomenon means TiO₂ reacts with MnFe₂O₄ and MnFe₂O₄/TiO₂ composite powders were formed as well. In addition, except for the phase corresponding to MnFe₂O₄/TiO₂ appears, no other phases were observed. Compared with the XRD pattern of MnFe₂O₄, most diffraction peaks of the composite powders show well agreement with the XRD pattern of pure manganese crystal ferrite. Based on this, it can be concluded that the synthesized MnFe₂O₄/TiO₂ composite powders are pure and have good crystallinity.

3.2. Morphology

The morphology of the precursor powders and as-prepared powder are shown in figure 2. Of special noting is that the evenly distributed MnFe₂O₄ powder presents a lamellar structure. In addition, these laminated material presents a smooth and flat surface. Compared with MnFe₂O₄ powder, the morphology of MnFe₂O₄/TiO₂ composite powders is remarkably different. Numerous spherical particles with an average size of about 80 nm are coated on the lamellar structure. The surface morphology of the as-prepared powders is inherited from the TiO₂ powder, which presents rough and granulated characteristics. By comparing the precursor powders with as-prepared powders, therefore we can be regarded that the MnFe₂O₄ powders are successfully wrapped with nanosized TiO₂ powder.

3.3. Electromagnetic performance

To investigate the electromagnetic wave absorption performance, we obtained the complex permittivity and complex permeability, which were shown in figures 3 and 4, respectively. The real permittivity and imaginary permittivity of MnFe₂O₄ powders and MnFe₂O₄/TiO₂ composite powders are shown in figures 3(a) and (b), respectively. As the frequency increases, both the real permittivity and imaginary permittivity of the two samples present significantly different evolutional trends. Except for fluctuation appearing at the frequency ranging from 14 to 16 GHz, the real permittivity of MnFe₂O₄ powder is almost unchanged in other frequency ranges. The evolutional trend for the imaginary permittivity of MnFe₂O₄ powder is consistent with that for real permittivity. Compared with MnFe₂O₄ powder, real permittivity and imaginary permittivity of MnFe₂O₄/TiO₂ composite powders have obvious fluctuation peaks, especially at high-frequency bands. The fluctuations are ascribed to displacement current lag at the interface. The polarization reaction occurs at the interfaces due to the charge accumulation between the lamellar structure and spherical particles, therefore resulting in polarization relaxation and dielectric loss. A similar phenomenon has been reported in previous research. More importantly,
the real permittivity value of MnFe₂O₄/TiO₂ composite powders is higher than that of the MnFe₂O₄ powder. The higher real permittivity means that MnFe₂O₄/TiO₂ composite powders have a higher energy density when the electromagnetic wave transmits inside the materials, which helps the transmitting. The higher real permittivity of MnFe₂O₄/TiO₂ composite powders is resulting from the polarization of the space charge on the interface. The lamellar structure of MnFe₂O₄ enlarged the contact area between MnFe₂O₄ and TiO₂ powders, which supplies more space for the polarization of the space charge on the interface [29, 30], thus presenting the outstanding interfacial polarization effect. In addition, under the influence of an external electromagnetic field, TiO₂ regarded as the dielectric loss material can form a strong local inner electric field [26, 31], which is also beneficial to improving the real permittivity. Considering these reasons, it can be also concluded that the
dielectric loss of MnFe$_2$O$_4$/TiO$_2$ composite powder is higher than MnFe$_2$O$_4$ powder. Thus, the imaginary permittivity of MnFe$_2$O$_4$/TiO$_2$ composite powders is higher than that of MnFe$_2$O$_4$ powder. Real permeability and imaginary permeability of MnFe$_2$O$_4$ powder and MnFe$_2$O$_4$/TiO$_2$ composite powders are shown in figures 4(a) and (b), respectively. The values of real permeability and imaginary permeability of the MnFe$_2$O$_4$ exhibit similar variation, while that of MnFe$_2$O$_4$/TiO$_2$ composite powders present obviously different trends. It is noteworthy that the values of real permeability and imaginary permeability of MnFe$_2$O$_4$/TiO$_2$ composite powder present distinguishable absorption peaks in frequencies ranging from 8 GHz to 12 GHz. In general, there are two different resonance mechanisms (natural resonance and domain wall resonance) to explain the resonance absorption peaks of polycrystalline ferrites in an AC field [31]. The resonance absorption peak that appeared in high-frequency bands is attributed to natural resonance, while that appeared in low-frequency bands is attributed to domain wall resonance. At the same mass, the absolute MnFe$_2$O$_4$ content in MnFe$_2$O$_4$ powder is higher than in MnFe$_2$O$_4$/TiO$_2$ composite powder. Besides, TiO$_2$ is one of the dielectric materials. So, the imaginary permeability values of MnFe$_2$O$_4$ powder are larger than the MnFe$_2$O$_4$/TiO$_2$ composite powder at the frequency of less than 7 GHz or more than 14 GHz. In general, excellent matching characteristics depend on special boundary conditions. Absorbing materials with high conductivity and permeability can wrap the medium, remarkably improving the incident amount of an electromagnetic wave. The distinguish absorption peaks appeared at frequencies ranging from 8 GHz to 12 GHz, implying that the MnFe$_2$O$_4$ and TiO$_2$ have excellent interface matching. In figure 4(b), we can also find that there’s a distinguish peak of the imaginary permeability of MnFe$_2$O$_4$/TiO$_2$ composite powder, and this result can be explained by the magnetic dissipation. According to a study, the magnetic dissipation of ferrite contains hysteresis loss, eddy current loss, residual loss, ferromagnetic resonance loss, and intragranular domain wall loss [32]. TiO$_2$ with a band gap energy of 3.2 eV is often used as a semiconductor, and it leads to the enhancement of eddy loss, which can make a contribution to the higher imaginary permeability of MnFe$_2$O$_4$/TiO$_2$ composite powder. According to the above results, the MnFe$_2$O$_4$ powder is completely wrapped with the TiO$_2$ powder. The MnFe$_2$O$_4$/TiO$_2$ composite powder presents excellent dielectric properties and magnetic properties, and therefore the microwave absorbing properties show the maximum value in figure 4.

3.4. The reflection loss
In order to investigate the electromagnetic wave absorbing properties of the MnFe$_2$O$_4$/TiO$_2$ composite powder, it needs to supply the reflection loss (RL) of MnFe$_2$O$_4$ powder and MnFe$_2$O$_4$/TiO$_2$ composite powder. The reflection loss values of absorbers with different thicknesses can be calculated from the following equation [1, 2]:

$$RL = 20 \times \log_{10} \left| \frac{Z_{in} - Z_0}{Z_{in} + Z_0} \right|$$

where $Z_0$ is the impedance of free space, and $Z_{in}$ is the normalized input impedance of free space and materials interface. The value of $Z_{in}$ is acquired by [1, 2]

$$Z_{in} = Z_0 (\mu_r / \varepsilon_r)^{1/2} \tanh \left[ j(2\pi fd/c)(\mu_r \varepsilon_r)^{1/2} \right]$$

where $\varepsilon_r$ and $\mu_r$ are the complex permittivity and the complex permeability of absorbers, respectively. $C$ is the velocity of electromagnetic waves in free space, $f$ is the frequency of the microwave, and $d$ is the matching thickness.
According to equations (1) and (2), the reflection loss of the MnFe$_2$O$_4$ powder and MnFe$_2$O$_4$/TiO$_2$ composite powder with a thickness of 3.05 mm is shown in figure 5(a). The MnFe$_2$O$_4$/TiO$_2$ composite powder possesses a larger reflection loss value at the frequency of 10 GHz, which implies that the MnFe$_2$O$_4$/TiO$_2$ composite powder has excellent microwave absorption properties. However, the reflection loss of the MnFe$_2$O$_4$ powder remains a lower value over the whole frequency range, namely MnFe$_2$O$_4$ powder has inferior microwave absorption properties. Figure 5(b) shows the reflection loss of MnFe$_2$O$_4$/TiO$_2$ composite powder with different thicknesses. As the thickness increases, the maximum value increases first and then decreases. The maximum value of reflection loss reaches $-38.6$ dB at the optimum parameter (frequency is 10.4 GHz and thickness is 3.05 mm). More importantly, there is only one reflection loss peak for the MnFe$_2$O$_4$/TiO$_2$ composite powder with a thickness of 3.05 mm. The value of the reflection loss peak is higher than that of the other ferrite reported in several literatures, i.e., La-substituted strontium ferrite (9–9.5 GHz) [33], Ba-ferrite/PVDF composites (12.4 GHz) [34], ferrite- polymer composites (12 GHz) [35]. Given the above discussion, it has been determined that the electromagnetic absorption of the MnFe$_2$O$_4$/TiO$_2$ composite powder is mainly ascribed to dielectric loss. As known to all, intrinsically dielectric properties, structure, magnetic loss, and eddy loss affect the dielectric loss. As the particle size decreases, the proportion of surface atoms increases remarkably. The superficial atoms have high activity, and they can be polarized and magnetized in an external electromagnetic field [36–40]. It can also consume electromagnetic energy by converting it into heat. In addition, the micro-emulsion method introduces several defects as well as improves the dispersity of MnFe$_2$O$_4$. Therefore, it may be contributing to the higher electromagnetic reflection loss in MnFe$_2$O$_4$/TiO$_2$ composite powder.

4. Conclusions

In summary, both MnFe$_2$O$_4$ powder and MnFe$_2$O$_4$/TiO$_2$ composite powders were prepared by the microemulsion method. The MnFe$_2$O$_4$/TiO$_2$ composite powders with coating structure increase the specific area and lead to an excellent property. The maximum microwave loss reaches $-38.6$ dB at the thickness of 3.05 mm, and the corresponding bandwidth is about 4 GHz in the ranges from 7.8 to 11.4 GHz. The results indicate that MnFe$_2$O$_4$/TiO$_2$ composite powders with the coating structure have the potential to be utilized for fabricating electromagnetic wave absorbers.

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Data availability statement

The data generated and/or analysed during the current study are not publicly available for legal/ethical reasons but are available from the corresponding author on reasonable request.
Declaration of interests

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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