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The microwave absorbing properties of ZnO/Fe₃O₄/paraffin composites in low frequency band

To cite this article: Pengfei Yin *et al* 2018 *Mater. Res. Express* **5** 026109

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The microwave absorbing properties of ZnO/Fe₃O₄/paraffin composites in low frequency bandRECEIVED
2 January 2018REVISED
5 February 2018ACCEPTED FOR PUBLICATION
9 February 2018PUBLISHED
23 February 2018Pengfei Yin¹ , Yu Deng², Limin Zhang³, Juan Huang¹, Huayao Li¹, Youhongyu Li¹, Yali Qi¹ and Yu Tao¹¹ College of Science, Sichuan Agricultural University, Ya'an 625014, People's Republic of China² College of Water Conservancy and Hydropower Engineering, Sichuan Agricultural University, Ya'an 625014, People's Republic of China³ Key Laboratory of Space Applied Physics and Chemistry (Ministry of Education), School of Science, Northwestern Polytechnical University, Xi'an 710072, People's Republic of ChinaE-mail: yudeng21@sicau.edu.cn**Keywords:** zinc oxide, ferroferric oxide, microwave absorbing properties, reflection loss, low frequency band**Abstract**

ZnO/Fe₃O₄/paraffin composites with good microwave absorption performance in low frequency band were prepared by physical blending technology. The morphology, phase structures, frequency-dependent electromagnetic and microwave absorbing properties of the composites were investigated. The results showed that the addition content of ZnO can adjust the microwave absorbing properties i.e. the position, intensity, and absorption bandwidth of composites, and the synergetic consequence of dielectric loss and magnetic loss is the main microwave absorption mechanism of the composites. The bandwidths with RL below -10 dB over different frequency ranges were obtained in the low frequency range of 0.5 ~ 3 GHz at a thickness of 5 mm, e.g. 0.93 GHz from 1.59 to 2.52 GHz and 0.85 GHz from 1.26 to 2.11 GHz corresponding to the mass ratios of ZnO and Fe₃O₄ are 1:2 and 1:4, respectively. Thus, such absorbers can be applied as effective microwave absorbers in low frequency range of 0.5 ~ 3 GHz.

1. Introduction

With the rapid development of modern radar and electronic technology, microwave absorbing materials within the range of gigahertz have attracted more and more attention in military and civil fields [1–4]. Up till now, the detection frequency of many spaceborne radars has been extended from 1.8 GHz to 1.2 GHz, and the working frequency band of phased-array radars used today is in S-band (1.55 ~ 3.4 GHz), which poses a huge challenge to the research of stealth materials. For the absorbing frequency bands of existing absorbent materials are mainly focus on 2 ~ 18 GHz, the stealth effect of which is much worse to the low frequency microwave, especially in the frequency range of 1 ~ 2 GHz. Hence, the design and development of composites with strong absorption ability in low frequency band is strongly demanded [5, 6].

To address these issues, multifarious microwave absorbing materials, which can attenuate microwave by converting them into heat or dissipating them via interference, have been extensively investigated, including conductive macromolecules [7, 8], magnetic metals [9, 10], dielectric materials [11, 12], and ferrites. Among all kinds of absorbing materials, magnetic ferrites have been widely used to absorb microwave due to their high magnetic loss [13]. H Zhao *et al* [14] synthesized NiFe₂O₄ microwave absorbing materials by the polyacrylamide gel method, the composite with 65 wt% ferrite content showed a minimum reflection loss of -13 dB at 11.5 GHz with a -10 dB bandwidth over the extended frequency range of 10.3 ~ 13 GHz for an absorber thickness of 2 mm. C Hou *et al* [15] prepared multi-wall carbon nanotube/Fe₃O₄ hybrids by a chemical synthesis-hydrothermal treatment method, and the microwave absorbing properties in the frequency range of X-band were investigated as well. The minimum reflection loss of -18.22 dB at 12.05 GHz was achieved for the magnetism of hybrids was enhanced by increasing the content of Fe₃O₄. D Min *et al* [16] prepared flake carbonyl iron/Fe₃O₄ composites with enhanced microwave absorbing properties by a direct and flexible surface oxidation technique, the results showed that lower permittivity as well as modest permeability was obtained by

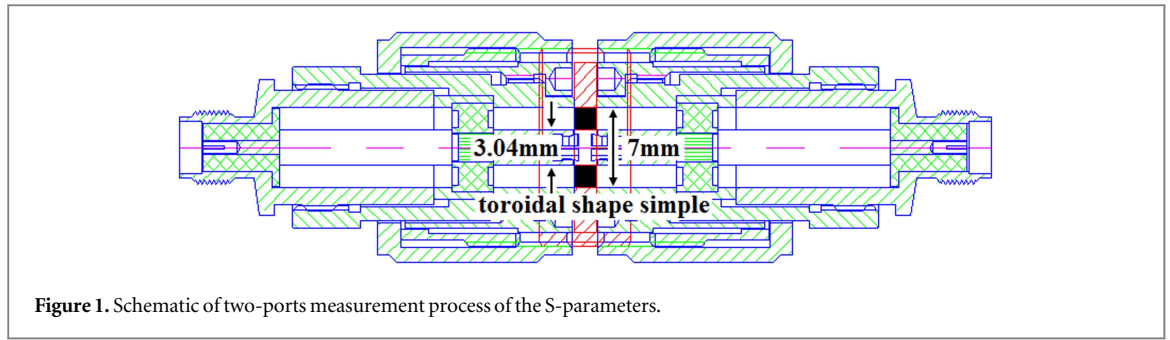


Figure 1. Schematic of two-ports measurement process of the S-parameters.

the FCI/Fe₃O₄ composites, such absorbers can act as effective and wide broadband microwave absorbers in the GHz range. P Sardarian *et al* [17] evaluated the synergistic effect of magnetic/dielectric composites system i.e. Fe₃O₄/BaTiO₃@MWCNT on the mechanism of enhanced microwave absorbing properties, the composites with more magnetic percentage showed better absorption in low frequency band. Due to the complex permittivity and permeability of pure Fe₃O₄ particles are relatively higher, which results in a poor impedance matching condition. This situation causes that the absorption bandwidth of pure Fe₃O₄ particles is too narrow, thus the microwave absorption efficiency of which is unsatisfactory over a wide frequency range. Therefore, combining Fe₃O₄ particles with dielectric materials has been promised to be an effective route to improve their microwave absorbing properties.

In our present work, ZnO was employed as a dielectric absorbent composite with Fe₃O₄ for its excellent dielectric properties. The ZnO/Fe₃O₄/paraffin composites have been prepared via physical blending technology on account of convenience and high efficiency of this method. In addition, the microstructure, phase identification and microwave absorption performances of the as prepared composites in the low frequency range of 0.5 ~ 3 GHz were investigated in detail.

2. Experimental details

The Fe₃O₄ and ZnO micropowder used in this investigation were purchased from Tianjin ZhiYuan Chemical Reagent Co. Ltd, China, and Tianjin Fuchen Chemical Reagent Factory, China, respectively. The purity of the two micropowders are both analytical pure, and the former is a powder of amorphous structure. Paraffin was used as matrix to adhere Fe₃O₄ and ZnO particles, and the physical blending technology was employed to prepare ZnO/Fe₃O₄/paraffin composites with different blending ratios of magnetic and dielectric powder at the room temperature. The mass ratios of ZnO and Fe₃O₄ micropowder are 4:1, 2:1, 1:2, and 1:4 respectively, while the mass ratio of ZnO/Fe₃O₄ mixed powder and Paraffin is fixed on 1:1, for the physical performance of composites becomes too poor to shaping as the powder is overloading.

The phase components and crystal microstructures were characterized by a Rigaku SmartLab x-ray diffractometer with Cu-K_α (λ = 0.154 18 nm) radiation at a voltage of 35 kV and a current of 200 mA at a scan step of 0.04°, and the microstructure morphology of the composites was investigated and analyzed by the Scanning Electron Microscope (SEM, XL30, Philips).

The electromagnetic parameters of composites were measured at 0.5 ~ 3 GHz by using a TIANDA TD3618C Vector Network Analyzer, and the sweep rate is set to 1601 points. The produced materials were pressed into a toroidal shape with 7 mm outer diameter, 3.04 mm inner diameter and 2 ~ 5 mm thicknesses. Then, the coaxial transmission and reflection method was used for measurement of the S-parameters, i.e. S₁₁ and S₂₁, the exact process of two-ports measurement was showed in figure 1. Finally, the relative complex permittivity and permeability of composites were calculated based on the theory of Nicolson and Ross [18].

The microwave reflection loss of ZnO/Fe₃O₄/paraffin composites was calculated according to the above electromagnetic parameters and transmission-line theory [19], in which the input impedance Z_{in} is given by:

$$Z_{in} = Z_0 \sqrt{\frac{\mu_r}{\epsilon_r}} \tanh \left(j \frac{2\pi f d}{c} \sqrt{\mu_r \epsilon_r} \right) \quad (1)$$

Where c is the velocity of light, f the frequency, d the thickness of the materials, ϵ_r the complex permittivity, and μ_r the complex permeability. Hence, the reflection loss of normal incident microwave at the absorber surface can be express as:

$$RL = 20 \log \left| \frac{Z_{in} - Z_0}{Z_{in} + Z_0} \right| \quad (2)$$

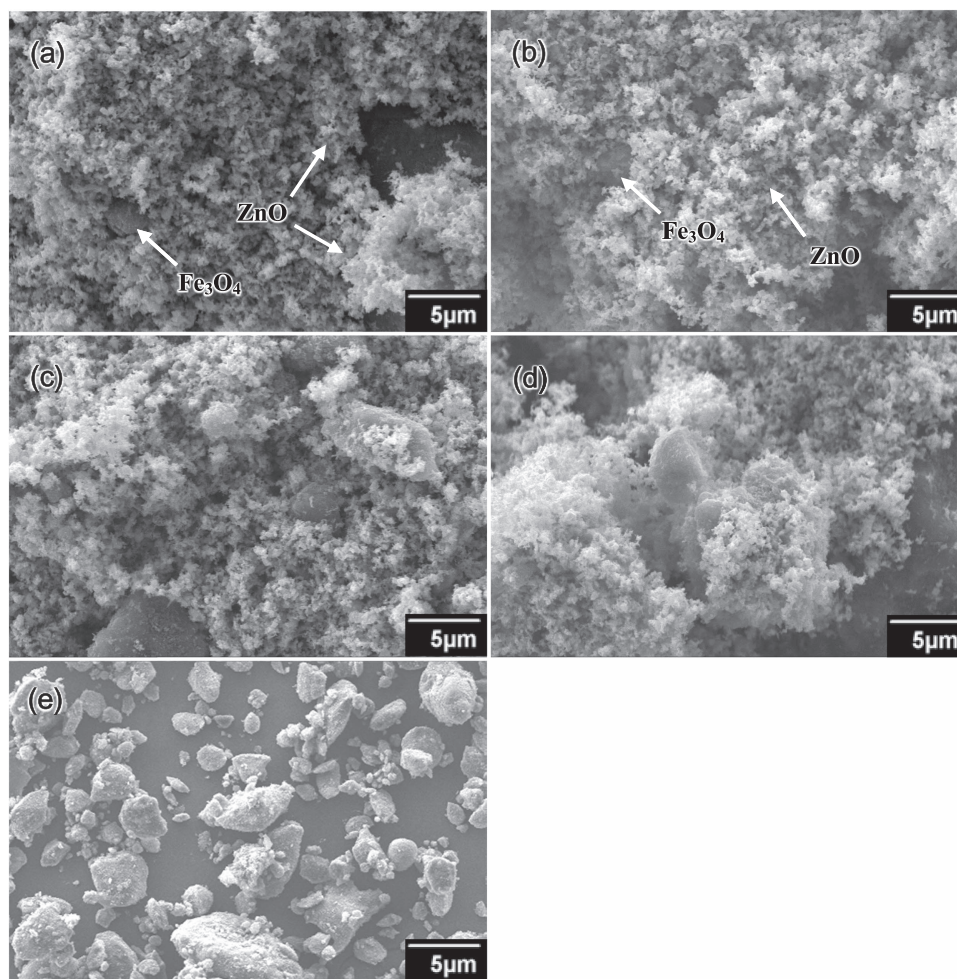


Figure 2. SEM images of ZnO/Fe₃O₄ composites with mass ratio of (a) 4:1, (b) 2:1, (c) 1:2, (d) 1:4 and (e) pure Fe₃O₄.

Here, the Z_0 is the impedance of air. Furthermore, the whole calculation procedure of electromagnetic parameters and reflection loss was programmed with Matlab language.

3. Results and discussion

3.1. Morphology and phase identification of ZnO/Fe₃O₄ composites

The morphologies of ZnO/Fe₃O₄ composites and pure Fe₃O₄ are shown in figure 2, we can see that there are only Fe₃O₄ particles dispersed independently in the field of view when the Fe₃O₄ micropowder was not composited with ZnO micropowder. Whereas, it indicates that fewer Fe₃O₄ particles can be observed in the figures 2(a) and (b), for the mass percentage of ZnO is relatively high, a large number of ZnO fine particles agglomerate with each other to form the porous structures. The Fe₃O₄ particles dot in the agglomerations of ZnO with the increasing content of Fe₃O₄, but which were not enveloped well by the ZnO agglomerations. This kind of situation is improved as the mass percentage of Fe₃O₄ be added up to 80%, most of Fe₃O₄ particles are enveloped by the agglomerations of ZnO fine particles. It is well known that modest complex permittivity and its suitable combination with the complex permeability is one of the most important factors for improving the microwave absorption efficiency of absorbers. Fe₃O₄ with ZnO enveloped can modify its electromagnetic parameters, which is helpful to improve the microwave absorbing properties of composites.

The crystal structures of ZnO/Fe₃O₄ mixed powders were analyzed with the x-ray diffractometer. The diffraction patterns of the as obtained samples were shown in figure 3. The characteristic diffraction peaks at $2\theta = 31.770^\circ, 34.422^\circ, 36.253^\circ, 47.539^\circ, 56.603^\circ, 62.864^\circ$, and 67.963° are in good agreement with the (100), (002), (101), (102), (110), (103) and (112) planes of ZnO reported in the standard card (JCPDS card No. 36-1451). The sharp strong peaks of ZnO indicate that the component of ZnO is polycrystalline, x-ray diffraction peaks of the (100), (002), and (101) planes are characteristic of ZnO with a wurtzite structure [20], and wurtzite is the most stable phase of ZnO under standard conditions [21]. It can be also observed that the intensity of ZnO

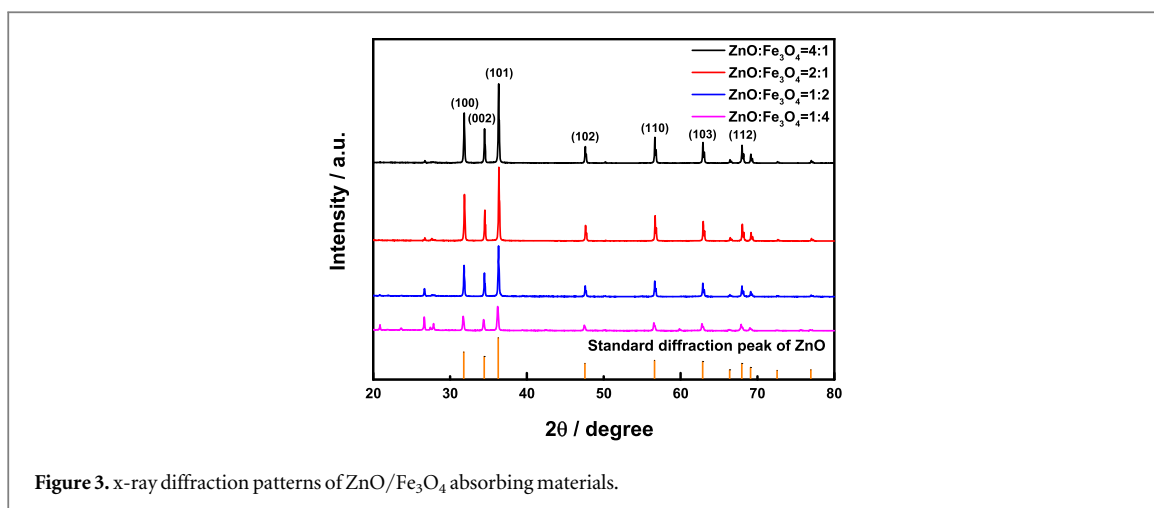


Figure 3. x-ray diffraction patterns of ZnO/Fe₃O₄ absorbing materials.

diffraction peaks decrease with the increase of Fe₃O₄ content. While, the diffraction peaks of Fe₃O₄ were not observed as its content increases, suggesting that the Fe₃O₄ micropowder is amorphous structure indeed.

3.2. Electromagnetic properties of ZnO/Fe₃O₄/paraffin composites

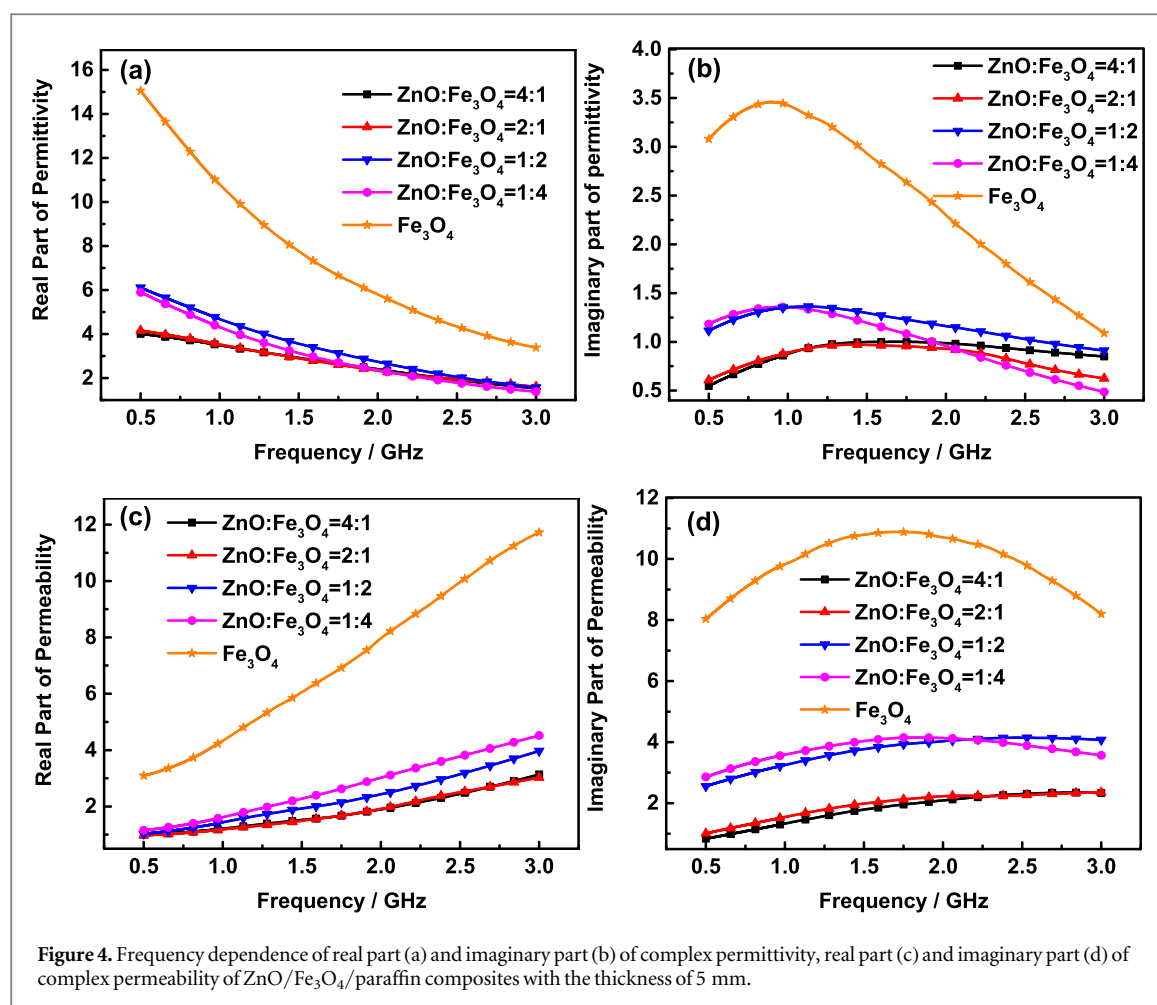
the complex permittivity ($\varepsilon = \varepsilon' - j\varepsilon''$) and permeability ($\mu = \mu' - j\mu''$) of microwave absorbing materials play an important role in determining the reflection and transmission of microwave, and the dielectric and magnetic loss are reflected by the complex permittivity and permeability in microwave absorption process as well [22]. To investigate the microwave absorbing properties of ZnO/Fe₃O₄/paraffin composites, the electromagnetic parameters were measured at the room temperature, and are shown in figure 4. As shown in figures 4(a) and (b), it is clear that the values of real part of permittivity decrease, and the values of imaginary part increase firstly then decrease with the increase of frequency, which exhibited obviously frequency dependence dielectric properties. In general, the permittivity of composites in GHz frequency range strongly depends on the interfacial polarization and inherent dipole polarization, the generation of displacement current significantly lags behind the build-up potential as the frequency increased. Thus, both real and imaginary parts of permittivity show the frequency dependence response [23]. Besides, we can find that the real part of permittivity changes little as the content of Fe₃O₄ is lower, while it increases significantly when the Fe₃O₄ content is relatively higher, especially in the low frequency domain, and the value is largest at a mass ratio of 1:2. In addition, it is interesting to note that when the content of Fe₃O₄ is lower, the imaginary part of permittivity in high frequency domain decreases with the decrease of ZnO content. Moreover, it increases in the whole measurement range as the content of Fe₃O₄ increases continuously, while the imaginary part of permittivity decreases sharply in the high frequency domain when the content of ZnO reduces further to a lower ratio. Hence, it can be concluded that the content of ZnO is mainly influence the imaginary part of permittivity in the high frequency domain, but the content of Fe₃O₄ contributes to the whole frequency range. According to the relationship between the imaginary part of permittivity and the resistivity [24]:

$$\varepsilon'' = \frac{1}{\rho\omega\varepsilon_0} \quad (3)$$

Where, ω is the angular frequency, ρ the resistivity and ε_0 the dielectric constant of free space. The resistivity of composite with the mass ratio of 1:4 is minimum at 0.5 GHz and maximum at 3 GHz among all the samples, and the doping of ZnO can adjust the resistivity of composites under different frequency ranges.

As seen in figures 4(c) and (d), the real part of permeability changes little when the content of Fe₃O₄ is lower as well, while it increases observably as the content of Fe₃O₄ increased further. This is primarily because the M_s of composites increases signally with the high doping of Fe₃O₄, and the real part of permeability is in direct proportion to M_s , i.e. $\mu' \propto 4\pi M_s$ [25]. Besides, the values of real part of permeability under different mass ratios are all increasing as the frequency increased. The change laws of imaginary part of permeability are mainly the same as the former, and the difference is that there is a peak value of 4.15 at 1.8 GHz when the mass content of Fe₃O₄ is 80%, which showed that the excessive content of Fe₃O₄ is helpless to enhance the imaginary part of permeability, but leads to a reduction of the imaginary part of permeability in the high frequency domain. It thought that the resonance peak in imaginary part of permeability spectra is attributed to natural resonance and domain wall resonance [14].

Furthermore, it does note that the complex permittivity and permeability of ZnO/Fe₃O₄/paraffin composites decrease substantially compared to Fe₃O₄/paraffin composite without the addition of ZnO. The



permittivity and permeability of composites can be adjusted by doping the ZnO micropowder, for the complex permittivity and permeability of ZnO is far lower than the Fe₃O₄. Thus, the complex permittivity and permeability of composites would reduce when the ZnO micropowder was added, which makes the value of permittivity be closer to the value of permeability further, it means that the μ'/ϵ' ratio of ZnO/Fe₃O₄/paraffin composites is closer to the free space than the composites without ZnO added. Based on the impedance matching theory, the incident microwave can access into the composites to be depleted much easier, which is very helpful to modify the absorption properties, e.g. position of absorption peak and absorption bandwidth etc of the composites.

The dielectric loss tangent and magnetic loss tangent of the composites as a function of frequency can be calculated based on the measured complex permittivity and permeability, which are shown in figures 5(a) and (b). We can see that the dielectric loss of ZnO/paraffin composite increases with the increase of frequency, which presents the linear relationship approximately. When the mass ratio of ZnO and Fe₃O₄ is 4:1, which means that a little quantity of Fe₃O₄ added would be helpful to enhance the dielectric loss. While, the dielectric loss decreases in high frequency domain as the content of ZnO decreased further for ZnO mainly influence the imaginary part of permittivity in the high frequency domain. Nonetheless, the dielectric loss still increases when the content of Fe₃O₄ is relatively higher, because the dipole polarization can enhance owing to the electron transfer process between Fe²⁺ and Fe³⁺ ions in Fe₃O₄ particles [26]. Therefore, there is a peak value of 0.41 at 2.04 GHz in dielectric loss tangent as the content of ZnO further reduces to a lower ratio. Additionally, the magnetic loss value is almost increasing as the content of Fe₃O₄ increased for its magnetic loss mechanism, and which of ZnO/paraffin composite is lower relatively, particularly in the low frequency domain. However, the excessive content of Fe₃O₄ leads to a reduction of magnetic loss. The magnetic loss is in direct proportion to the content of Fe₃O₄ in the low frequency domain, and it decreases with the increase of frequency, especially when the content of Fe₃O₄ is high enough, this is mainly due to the magnetic loss properties of Fe₃O₄ clearly.

It is interesting to note that the dielectric loss and magnetic loss can both reach the high values when the mass ratio of ZnO and Fe₃O₄ in composites is in the suitable range, this is owing to that the interface polarization increases for the charges transfer along the boundaries between ZnO and Fe₃O₄ particles. Moreover, it is obvious

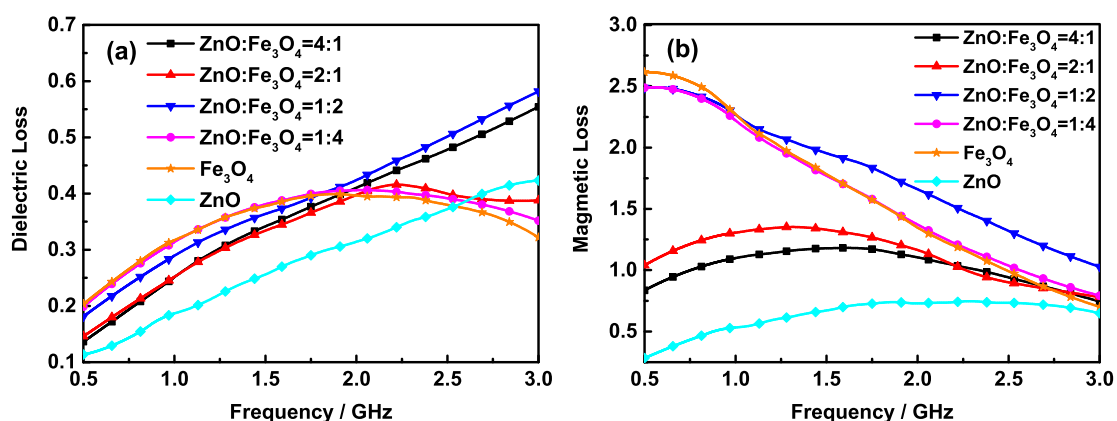


Figure 5. Frequency dependence of dielectric loss tangent (a) and magnetic loss tangent (b) of the ZnO/Fe₃O₄/paraffin composites with the thickness of 5 mm.

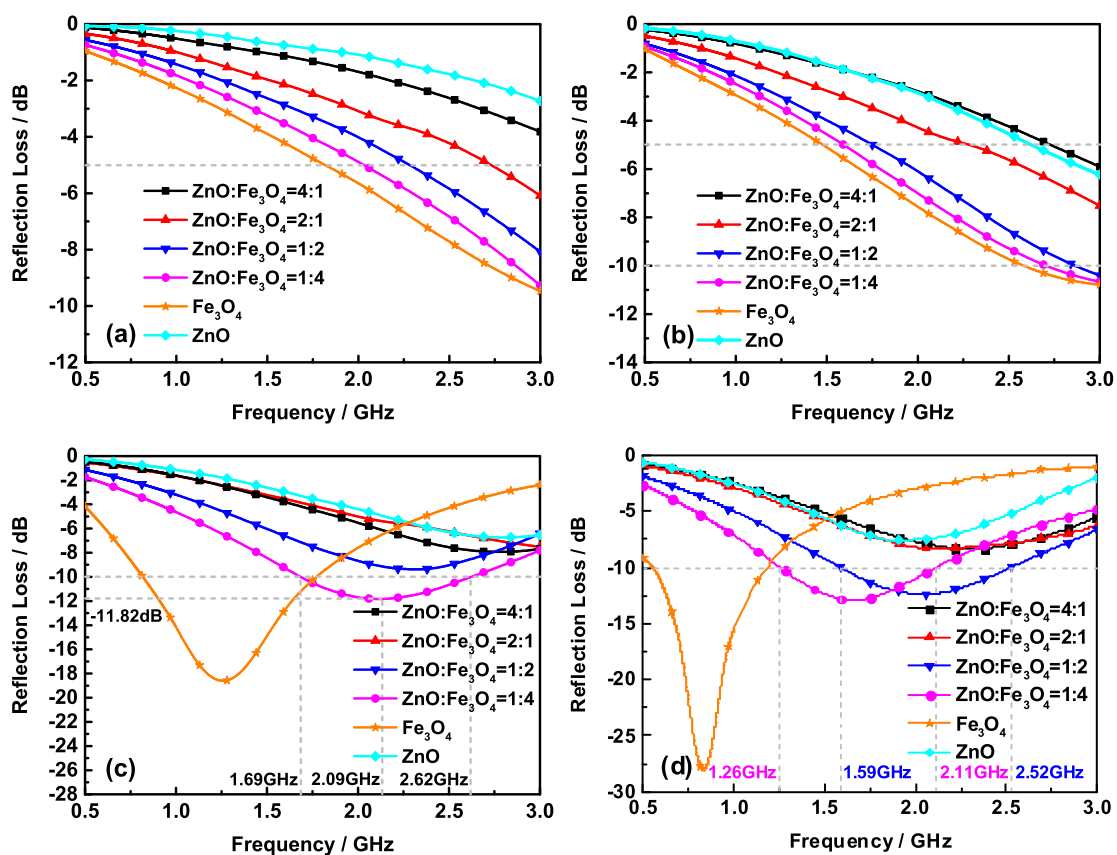


Figure 6. Frequency dependence of RL curves at different thicknesses for (a) 2 mm, (b) 3 mm, (c) 4 mm, (d) 5 mm.

that the magnetic loss is a little larger than dielectric loss in the measurement range, which indicates that the dielectric loss and magnetic loss are all contributes to the microwave absorption in the composites.

3.3. Microwave absorbing properties of ZnO/Fe₃O₄/paraffin composites

In order to characterize the microwave absorbing properties, microwave reflection losses (denoted RL henceforth) of the ZnO/Fe₃O₄/paraffin composites were calculated at different absorber thicknesses according to the electromagnetic parameters and transmission-line theory, which are shown in figure 6. It suggests that there is none of absorption peak can be observed between 0.5 GHz and 3 GHz in figures 6(a) and (b), i.e. the thicknesses of composites are 2 mm and 3 mm respectively. The reflectivity of microwave decreases with the increase of frequency, and the curve of RL is lower with the increase of Fe₃O₄ content as well. Furthermore, it is clear that the absorption bandwidths with RL below -5 dB of composites at the thickness of 2 mm are 0 GHz,

0.26 GHz (2.74 ~ 3 GHz), 0.71 GHz (2.29 ~ 3 GHz) and 0.96 GHz (2.04 ~ 3 GHz), corresponding to the mass ratios of 4:1, 2:1, 1:2 and 1:4 respectively. However, the RL bandwidths below -5 dB add up to 0.27 GHz (2.73 ~ 3 GHz), 0.71 GHz (2.29 ~ 3 GHz), 1.24 GHz (1.76 ~ 3 GHz) and 1.4 GHz (1.6 ~ 3 GHz) respectively when the thickness of composites is 3 mm, particularly the RL bandwidths below -10 dB start to appear, this is because that the composites have more effective electromagnetic absorption in the frequency range as the thickness added.

As shown in figure 6(c), we can see that the minimum RL peaks appeared in the low frequency range of 0.5 ~ 3 GHz when the thickness of composites is 4 mm. Although, there exists a stronger absorption peak in the Fe₃O₄/paraffin composite, the frequency position of minimum RL peak is too low. As we all know that the minimum RL value and its peak frequency strongly depend on the absorber thickness and the RL peak moves to a lower frequency with the increase of absorber thickness. This phenomenon can be explained by the $\lambda/4$ resonance effect and the resonance frequency f is expressed as:

$$f = \frac{c}{4d\sqrt{\mu_r\epsilon_r}} \quad (4)$$

Here, c is the velocity of light in free space, d the thickness of absorber, μ_r the complex permeability, ϵ_r the complex permittivity. Therefore, it is reasonable that the minimum RL peak shifts to lower frequency due to the increase of thickness [27]. Hence, it is evident that the microwave absorbing properties of Fe₃O₄/paraffin composite in a wide frequency range might not be improved only by increasing the thickness, but the addition of ZnO micropowder can be helpful to modify this behavior precisely. For instance, the minimum loss is -11.82 dB at 2.09 GHz with a -10 dB bandwidth over the extended frequency range of 1.69 ~ 2.62 GHz for the mass ratio of ZnO and Fe₃O₄ is 1:4, above 90% microwave energy can be absorbed as the RL is below -10 dB.

The electromagnetic absorption of ZnO/Fe₃O₄/paraffin composites is more effective in the low frequency range of 0.5 ~ 3 GHz when the thickness adds to 5 mm, and which are shown in figure 6(d). The minimum RL peaks appeared in the all samples with different mass ratios of ZnO and Fe₃O₄, and the microwave absorbing properties of composites with the mass ratio of 1:2 and 1:4 are better. Besides, the microwave reflection loss of Fe₃O₄/paraffin and ZnO/paraffin were also measured here, which further presents the impact of ZnO on the microwave absorbing properties of ZnO/Fe₃O₄/paraffin composites clearly. We can find that the minimum RL of Fe₃O₄/paraffin and ZnO/paraffin are -28.04 dB at 0.83 GHz and -7.60 dB at 1.90 GHz, respectively. Unfortunately, the microwave absorbing properties of Fe₃O₄/paraffin is unsatisfactory at a wide frequency range of 1 ~ 3 GHz, and the microwave absorption intensity of ZnO/paraffin is also undesirable. Usually, the minimum RL of composites used for absorbing microwave should below -10 dB, which means that above 90% microwave energy can be absorbed theoretically. The addition of ZnO micropowder can adjust the position, intensity, and absorption bandwidth of absorption peak. The minimum RL with the mass ratio of 1:2 is -12.42 dB at 2.05 GHz with a -10 dB bandwidth over the extended frequency range of 0.93 GHz (1.59 ~ 2.52 GHz), meanwhile the minimum RL is -12.92 dB at 1.66 GHz with a -10 dB bandwidth over the extended frequency range of 0.85 GHz (1.26 ~ 2.11 GHz) when the mass ratio of composites is 1:4. As mentioned above, the main microwave absorption mechanism of ZnO micropowder is dipole polarization and which of Fe₃O₄ micropowder are domain wall resonance and natural resonance. Consequently, the microwave absorption mechanism of ZnO/Fe₃O₄/paraffin composites will have the complex mechanism of the two components and the interface polarization between different particles, hence the final microwave absorption effect is a synergetic result of dielectric loss and magnetic loss of the composites [28]. Besides, the surface enveloped structure of Fe₃O₄ particles with the fine ZnO agglomerations is also contributed for enhancing the impedance matching [29]. Generally speaking, it suggests that we can get the superior absorbing property with -10 dB bandwidth over different frequency ranges in the low frequency range of 0.5 ~ 3 GHz by controlling the mass ratio of ZnO and Fe₃O₄ in composites. Most importantly, the preparation process used here are simple, without complex treatment and reaction. Thus, such absorbers can be applied as effective microwave absorbers in the low frequency range of 0.5 ~ 3 GHz.

4. Conclusions

In this paper, ZnO/Fe₃O₄/paraffin composites were prepared by physical blending technology, and the microwave absorbing properties of composites in the low frequency range of 0.5 ~ 3 GHz were investigated as well. The content of ZnO micropowder added can adjust the microwave absorbing properties of composites, i.e. the position, intensity, and absorption bandwidth. The surface enveloped structure and synergetic consequence of dielectric loss and magnetic loss are all contribute to the microwave absorption of ZnO/Fe₃O₄/paraffin composites. The bandwidths with RL below -10 dB over different frequency ranges in the low frequency range of 0.5 ~ 3 GHz were obtained, e.g. 0.93 GHz from 1.59 to 2.52 GHz and 0.85 GHz from 1.26 to 2.11 GHz with

the mass ratios of ZnO and Fe₃O₄ are 1:2 and 1:4 respectively, at a thickness of 5 mm. This study presents an easy and effective approach to prepare microwave absorbing materials with good performance in low frequency band, and indicates that ZnO/Fe₃O₄/paraffin composites are promising candidates as microwave absorbing materials in the frequency range of 0.5 ~ 3 GHz.

Acknowledgments

The authors are grateful to the National Natural Science Foundation of China (No. 51704242), and the Research Interest Training Program of Sichuan Agricultural University (No. 04055798).

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