

Synthesis and enhanced microwave-absorbing properties of SnO₂/ **α-Fe₂O₃@RGO composites**

Ziyao Shen1 · Honglong Xing1 · Yangting Zhu1 · Xiaoli Ji1 · Zhenfeng Liu1 · Lei Wang1

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Abstract $SnO₂/\alpha$ -Fe₂O₃@RGO composites were prepared through a solvothermal method. The phase structure, morphology, and electromagnetic (EM)-wave-absorbing properties of the as-prepared samples were characterized by X-ray diffraction, Fourier transform infrared spectroscopy, transmission electron microscopy, X-ray photoelectron spectroscopy, and vector network analyzer. Results indicated that the remarkable EM wave absorption properties of the samples resulted from the interaction between lamella-structured reduced graphene oxide and semiconductor-heterostructured $SnO₂/\alpha$ -Fe₂O₃. The optical reflection loss calculated from the measured complex permittivity and permeability was −44.33 dB at 12.64 GHz. Meanwhile, the Fe^{3+}/Sn^{4+} molar ratio in the composites was 6:1, and the coating thickness was 2 mm. The bandwidth below −10 dB was 4.4 GHz (10.80–15.20 GHz). Therefore, $SnO₂/\alpha$ -Fe₂O₃@RGO could serve as a high-performance EM-wave-absorbing material.

1 Introduction

Electromagnetic (EM) wave pollution has become increasingly serious due to the rapid growth of modern science technologies, and several investigators have focused their attention on EM wave absorption materials that possess a thin coating, low weight, wide frequency range, and strong absorption [[1,](#page-7-1) [2\]](#page-7-2). Composite absorbing materials are expected to become a new trend in the coming years

 \boxtimes Honglong Xing austxhl@163.com because the practical application of traditional microwaveabsorbing materials is limited by their weak absorption capability and narrow absorption band. Graphene oxide (GO), a new type of carbon material, possesses a high dielectric constant and abundant oxygen-containing groups on its surface [[3\]](#page-7-0). In addition, GO is characterized by a well-developed surface area, high mechanical strength, and low weight. Given these characteristics, GO is widely used in chemical sensors [[4–](#page-8-0)[6\]](#page-8-1), fuel cells [[7,](#page-8-2) [8\]](#page-8-3), catalysts $[9-11]$ $[9-11]$, and EM-wave-absorbing materials $[12-14]$ $[12-14]$. Chemically reduced GO (RGO) and its composites have recently attracted the attention of researchers because of their excellent EM wave absorption properties [[15,](#page-8-8) [16](#page-8-9)]. However, when RGO acts alone as an absorber, its high conductivity would worsen the impendence match and results in poor microwave absorption. The use of a composite with other materials to make the composite present multiple losses and to enhance the impendence match is an effective means to improve the microwave absorption performance. α -Fe₂O₃ is the most stable iron oxide and an n-semiconductor; it has a band gap of 2.1 eV $[17]$ $[17]$. As a promising functional material, α -Fe₂O₃ is always composited with RGO to synthesize the EM absorber. $SnO₂$, which is an n-type semiconductor with a wide band gap of 3.6 eV and an excellent conductive material, has attracted considerable attention because of its diverse applications $[18–21]$ $[18–21]$ $[18–21]$. The addition of SnO₂ and α -Fe₂O₃, not only regulates the conductivity of RGO, but also improves impendence match and provides the composite a remarkable EM absorption performance. $SnO₂$ and α -Fe₂O₃ have the properties of an n-type semiconductor; they can constitute semiconductor nanoheterostructures, and the effective complementarities help enhance the polarization process and promote EM wave absorbance [[22,](#page-8-13) [23\]](#page-8-14).

In this study, $SnO₂/\alpha$ -Fe₂O₃@RGO composites were successfully synthesized through a solvothermal method.

 1 School of Chemical Engineering, Anhui University of Science and Technology, Huainan 232001, China

The effects of the RGO flake and the ratio of Fe^{3+}/Sn^{4+} (2:1, 6:1, and 10:1) on microwave absorption properties were discussed. The morphology, structure, and the electromagnetic parameters of the composites were also systematically analyzed. The EM absorption measured within 2–18 GHz indicated a maximum refection loss of −44.33 dB at 12.64 GHz for the Fe^{3+}/Sn^{4+} ratio of 6:1 and coating thickness of 2 mm. The absorption band below −10 dB was 4.40 GHz (10.80–15.20 GHz).

2 Experimental

SnO₂/α-Fe₂O₃@RGO

2.1 Materials

The natural graphite flake (25 mesh, purity of 99.9%), ammonia (purity of 25%), potassium permanganate $(KMnO₄)$, sodium nitrate $(NaNO₃)$, five hydrated stannic chlorides ($SnCl₄·5H₂O$), ferric chloride hexahydrate $(FeCl₃·6H₂O)$, and other chemical reagents in this work were of analytical grade and not subjected to further purification.

2.2 Synthesis of SnO₂/α-Fe₂O₃@RGO composites

Graphene oxide (GO) flakes were synthesized from graphite powder through a modified Hummers' method [\[24](#page-8-15)]. SnO₂/ α -Fe₂O₃@RGO composites were prepared through a solvothermal method. Following a typical procedure, 50 mg of GO was dispersed uniformly in 70 mL of a solution that contained 30 mL of H_2O and 40 mL of ethanol and subjected to ultrasonic dispersion for 1 h. Ferric chloride hexahydrate (FeCl₃·6H₂O) and five hydrated stannic chlorides ($SnCl₄·5H₂O$) were added to the solution at different molar ratios of 2:1 (S1), 6:1 (S2), and 10:1 (S3). The solution was then transferred into a 100-mL Teflon-lined stainless steel autoclave and kept at 180° C for 12 h in an electric oven. The autoclave was cooled down to room temperature. Afterward, the obtained product was dried in a vacuum drying oven at 60° C for 12 h. The SnO₂/ α -Fe₂O₃ ω RGO composites were then obtained. The preparation process is illustrated in Fig. [1](#page-1-0).

2.3 Characterization

X-ray diffraction (XRD) using a Cu-Kα source (*λ*=1.5481 Å) (XRD, Beijing Purkinje General Instrument Co., Ltd.) was conducted to identify the crystal structure of the as-prepared samples. A Fourier transform infrared spectroscope (FTIR, 170SX) was used to record the FTIR spectra of the samples over the range of 400–4000 cm−1. The morphologies of the samples were observed with a transmission electron microscope (TEM, FEI Tecnai 12) with an acceleration voltage of 200 kV. The surface composition was obtained through X-ray photoelectron spectroscopy (XPS, ESCALAB210). The complex permittivity (ε_r) and complex permeability (μ_r) of the samples (uniformly mixing 60 wt.% $SnO₂/\alpha$ -Fe₂O₃@RGO composites with paraffin and pressing the mixture into a toroid mold with an outer diameter of 7.0 mm and an inner diameter of 3.04 mm) were measured in a frequency range of 2–18 GHz by using a vector network analyzer (AV3629D) at room temperature.

3 Results and discussion

Figure [2a](#page-2-0) presents the XRD pattern of the $SnO₂/α-Fe₂O₃@$ RGO composites (S1, S2, and S3). The RGO diffraction peaks cannot be observed in Fig. [2](#page-2-0)a because the ordered structure of the natural graphite flake was damaged in the GO preparation process, and reassembly of GO was prevented by the development of SnO₂ and α -Fe₂O₃ on the RGO surface in the solvothermal process. The relative intensities of the diffraction peaks at 2*θ*=24.14°, 33.14°, 35.61°, 40.84°, 49.43°, 54.02°, 57.57°, 62.40°, 63.97, 71.85°, 75.37° are assigned to the reflections from the (012), (104), (110), (113), (024), (116), (018), (214), (300), (1010), and (220) planes of α -Fe₂O₃ (JCPDS card No. 33-0664), respectively. For $SnO₂$, three peaks at 26.60° , 33.85°, and 51.76° were observed and could be indexed as characteristic (110), (101), and (211) reflections, respectively, of the $SnO₂$ crystal (JCPDS card No. 41-1445). These results suggest the existence of $SnO₂$ and α -Fe₂O₃ [\[25](#page-8-16), [26\]](#page-8-17). No other impure phases were detected. Notably, no obvious diffraction peaks for RGO can be observed in $SnO₂/\alpha$ -Fe₂O₃@RGO composites, which might be the relative low diffraction intensity of RGO in the composite of

Fig. 2 XRD pattern of S1, S2, and S3 and FTIR spectra of GO and S1

the SnO₂/ α -Fe₂O₃@RGO composites compared with SnO₂ and α -Fe₂O₃ nanoparticles [[27\]](#page-8-18).

The FTIR spectra of GO and SnO_2/α -Fe₂O₃@RGO com-posites (S1) are shown in Fig. [2b](#page-2-0). The peak at 3420 cm⁻¹ is assigned to the O–H deformation vibration in graphene and intercalated water in the samples [[28\]](#page-8-19). The peak at 1705 cm^{-1} is attributed to the C=O stretching of carbonyl and carboxyl groups at the edges of the GO networks. The peaks at 1380 and 1057 cm⁻¹ are due to –CH₃ deformation vibrations and C–O stretching vibration, respectively [[29,](#page-8-20) [30](#page-8-21)]. Comparison of the FTIR spectrum of GO with that of S1 revealed that the peaks at 1705 and 1057 cm^{-1} weakened in S1, indicating that GO was reduced in the preparation of SnO₂/α-Fe₂O₃@RGO composites. The peak at 1560 cm−1 is associated with the formation of –COO after RGO was coated with $SnO₂$ and α -Fe₂O₃. The broad peak at 548 cm−1 corresponds to Fe–O and Sn–O vibration.

To further determine the chemical composition of the SnO₂/ α -Fe₂O_{[3](#page-3-0)}@RGO composites, Fig. 3 shows the XPS spectra of S1. Figure [3](#page-3-0)a presents the full-scan XPS spectra of S1 and indicates that the elements on the surface of S1 were Fe, C, O, and Sn. The C1s spectra (Fig. [3](#page-3-0)b) revealed four types of carbon bonds at 284.7 eV (C–C), 285.6 eV $(C=0)$, 286.7 eV $(C=0)$, and 288.8 eV $(O-C=0)$ [\[31](#page-8-22)]. The intensities of C–O, C=O, and O–C=O were much weaker than that of C–C, which suggests a reduction in GO. Figure [3](#page-3-0)c provides the O1s XPS spectra. The banding energy peaks at 530.3, 530.5, 530.9, 531.6, and 532.8 eV repre-sent Fe–O, Sn–O, C=O, O–H, and C–O, respectively [\[27](#page-8-18)]. Thenergy peaks at 711.4 and 725.4 eV in the Fe 2p Xe banding PS spectra (Fig. [3](#page-3-0)d) agreed well with those in Fe $2p_{3/2}$ and Fe $2p_{1/2}$. The binding energy peaks at approximately 716.3 and 732.7 eV that accompanied Fe $2p_{3/2}$ and Fe 2p_{1/2} are characteristics of γ-Fe₂O₃. The two peaks at 486.8 and 459.2 eV are assigned to Sn $3d_{5/2}$ and Sn $3d_{3/2}$, respectively [[32\]](#page-8-23).

The morphology of composites is characterized by SEM and TEM, Fig. [4](#page-4-0) shows the results. From Fig. [4a](#page-4-0), it's obvious that the GO flake was fabricated successfully. Figure [4](#page-4-0)b shows that the nanoparticles were well loaded on the surface of GO flake. A thin-layered structure RGO with some wrinkles can be observed in the Fig. [4](#page-4-0)c, d. And it also can be seen from the images that there are two sizes of particles well scattered on the RGO, only a small fraction dispersed in the solution. HRTEM image of composites from the surface of RGO (inset of Fig. [4d](#page-4-0)) show two different clear lattice fringes. The crystal lattice spacing of the bigger particles is 0.27 nm (Fig. [4](#page-4-0)e) can be assigned to the (104) plane of α-Fe₂O₃ particles, while the lattice of the smaller particles is 0.35 nm (Fig. [4f](#page-4-0)), corresponding to the (110) plane of $SnO₂$ particles. The results are consistent with the XRD pattern. Therefore, we can identify the nanoparticles with range of $5-10$ nm in the Fig. [4d](#page-4-0) are $SnO₂$ nanoparticles, and α -Fe₂O₃ have a lager particles size around 25 nm.

Transmission coaxial line theory states that complex permittivity ($\varepsilon_r = \varepsilon' - j\varepsilon''$), complex permeability ($\mu_r = \mu'$ − *jμ″*), coating thickness (*d*), and frequency (*f*) are the main factors that affect the absorption performance of samples. We studied the EM parameters of the sample-paraffin mixture at the frequency of 2–18 GHz at room temperature to obtain additional EM absorption properties of the samples. Figure [5a](#page-5-0) shows that that the real part of the relative permittivity (ε') of the three samples exhibited a downward trend. The *ε*′ of S2 was larger than those of the other two samples in the entire testing frequency range. Figure [5](#page-5-0)b indicates that the imaginary part of the relative permittivity (*ε″*) of S3 exhibited a distinct decrease, whereas the *ε″* of S2 and S3 almost remained unchanged from 2 to 14 GHz and then exhibited some fluctuation at increased frequency. In the beginning of 6 GHz, the *ε″* values of S2 exceeded those of S3 and became the largest among the three samples. The increased value of *ε*′ might be due to

Fig. 3 XPS spectra of S1: **a** wide span, **b** C1s spectrum, **c** O1s spectrum, **d** Fe2p spectrum, and **e** Sn3d spectrum

the enhancement of interfacial polarization [[1\]](#page-7-1). The multiinterface between the RGO layer and $SnO₂/\alpha-Fe₂O₃$ and the proper Fe^{3+}/Sn^{4+} molar ratio promoted the polarization process. Free electron theory posits that $ε'' = σ/(2πfε₀)$ [\[33](#page-8-24)], where σ is the electrical conductivity; thus, the ε ^{*"*} values of the composites at 2–18 GHz represent the electrical conductivities. The higher *ε″* value of S3 from 2 to 6 GHz indicate high conductivity, and the same applies to S2 from 6 to 18 GHz. Therefore, an enhanced dielectric loss could be achieved by changing the Fe^{3+}/Sn^{4+} molar ratio.

Figure [5c](#page-5-0) shows that the real part of the permeability (μ') of S1 decreased with slight undulation and exhibited the same tendency as S2 and S3. Figure [5d](#page-5-0) shows that the imaginary part of the permeability (μ'') of the three samples had two resonance peaks at a low frequency, whereas the peaks were not distinct with increased frequency. Under the coaction of RGO and SnO_2/α -Fe₂O₃ nanoparticles, the values of μ 'and μ ^{*"*} remained stable at 0.98–1.20 and −0.017–0.21, respectively. With the change in the $Fe³⁺/Sn⁴⁺$ molar ratio, the magnetic loss property of the

composites improved and was thus more competent in dissipating magnetic energy [\[34](#page-8-25)].

Figure [5e](#page-5-0), f show the dielectric loss tangent (tan δ_e) and magnetic loss tangent (tanδ_μ) of SnO₂/α-Fe₂O₃@RGO-paraffin composites. The flaws of the nanoparticles (vacancy, hanging key, etc.) were easy to be polarized and subsequently generated a dielectric loss in the process of new polarization under the influence of EM wave. Figure [5e](#page-5-0) shows that the tan δ_{ε} values of the three samples changed to above 0.18, which indicates that the dielectric loss existed in the entire frequency range and proved the obvious dielectric loss capability of RGO, $SnO₂$, and α-Fe₂O₃. The tan $\delta_{\rm s}$ of S2 was higher from 2 to 10.4 GHz. When the frequency increased, the tan δ_{ε} of S3 became the highest. The tan δ_{μ} curves of the three samples in Fig. [5f](#page-5-0) fluctuate within a narrow range at approximately 0. All of the samples had higher tan δ_{ε} values than tan δ_{μ} in 2–18 GHz, which reveals that the dielectric loss mainly contributed to EM absorption.

The microwave absorption properties of absorbers have a close connection with impedance match. The synergistic effect of dielectric and magnetic losses makes composites satisfy material impedance matching, thereby promoting the microwave-absorbing performance. Dipole polarization in α -Fe₂O₃ and α -Fe₂O₃ at the nanoscale could enhance the polarization capability [[35\]](#page-8-26). In the ternary composites, electronic and ionic polarizations caused by electron transform among Fe^{3+} , Sn^{4+} , and RGO also occur.

The multi-interfaces among $SnO₂/\alpha-Fe₂O₃$, SnO₂/ α -Fe₂O₃@RGO, and α -Fe₂O₃@RGO could induce more interfacial polarization and corresponding relaxations, which led to enhanced microwave attenuation ability [\[36](#page-8-27)]. Specially, as their similar properties of n-type semiconductor, $SnO₂$ and α -Fe₂O₃ will constitute semiconductor

Fig. 5 Real and imaginary parts of permittivity (a ε' and b ε''), permeability (c μ' and d μ''), and loss tangent: e dielectric loss tangent; f magnetic loss tangent of S1, S2, and S3

nanoheterostructures in the composites, and the interfacial polarization could happens in the heterogeneous system [\[37,](#page-8-28) [38](#page-8-29)]. Therefore, the desirable performance in microwave absorbing could be ascribed to the unique interfacial interaction of the binary components [[22](#page-8-13)].

We used the Debye dipolar relaxation model [\[39,](#page-8-30) [40\]](#page-8-31) (Cole–Cole model) to explain further the mechanism of dielectric loss. Relative complex permittivity ε_r can be described as follows [\[41\]](#page-8-32):

$$
\varepsilon_r = \varepsilon_\infty + \frac{\varepsilon_s - \varepsilon_\infty}{1 + j2\pi f\tau},\tag{1}
$$

where *f* is the frequency; τ is the polarization time; and ε_s and ε_{∞} are the static dielectric constant and the optical dielectric constant at the infinite frequency, respectively. Equation ([1\)](#page-5-1) shows that

$$
\varepsilon' = \varepsilon_{\infty} + \frac{\varepsilon_s - \varepsilon_{\infty}}{1 + (2\pi f)^2 \tau^2},\tag{2}
$$

$$
\varepsilon'' = \frac{2\pi f \tau \left(\varepsilon_s - \varepsilon_\infty\right)}{1 + \left(2\pi f\right)^2 \tau^2}.\tag{3}
$$

The above equations show that the relationship between ε' and ε'' could be deduced as

$$
\left(\varepsilon'-\varepsilon_{\infty}\right)^{2}+\left(\varepsilon''\right)^{2}=\left(\varepsilon_{s}-\varepsilon_{\infty}\right).
$$
\n(4)

Therefore, from graph *ε″* according to *ε′*, we can generate a semicircle that is usually defined as a Cole–Cole semicircle, and each semicircle corresponds to a type of Debye relaxation process [\[42\]](#page-8-33). Figure [6a](#page-6-0)–c display the Cole–Cole vibration curves of the as-prepared samples. Two semicircles of the curves are shown in Fig. [6a](#page-6-0), c. Hence, S1 and S3 had two types of Debye relaxation at the least. Figure [6b](#page-6-0) shows that the number of semicircles for S3 is three. The increase in the Debye process implied that the S2 composite had more enhanced dielectric properties.

Microwave absorption properties were investigated by mixing 60 wt% of the samples with paraffin. From the obtained EM parameters, the reflection loss (RL) of S1, S2, and S3 was calculated according to transmission theory by using the following equations [[43,](#page-8-34) [44](#page-8-35)].

$$
Z_{in} = \sqrt{\frac{\mu_r}{\varepsilon_r}} \tanh\left[j\left(\frac{2\pi df}{c}\right)\sqrt{\mu_r \varepsilon_r}\right],\tag{5}
$$

$$
RL(dB) = 20lg \left| \frac{Z_{in} - Z_0}{Z_{in} + Z_0} \right|,
$$
\n(6)

where Z_{in} is the input impedance of the absorber, Z_0 is the impedance of free space, μ_r is complex permeability, ε_r is complex permittivity, *f* is the frequency of microwaves, *d* is the thickness of the absorber, and *c* is the velocity of EM waves in free space. The absorber achieved 90% microwave absorption when the RL values of the sample reached −10 dB [[45\]](#page-8-36).

The RL of the three samples with different thicknesses is shown in Fig. [7.](#page-7-3) The RL curves of the samples presented different variation trends with different Fe^{3+}/Sn^{4+}

molar ratios. For S1, the minimum RL values shifted to a low frequency when the thickness was increased. The optimal RL of S1 was −12.14 dB with a thickness of 4.5 mm at 6.72 GHz, and the bandwidth below −10 dB was 1.12 GHz (6.16–7.28 GHz). Figure [7b](#page-7-3) shows that the minimum RL of S2 reached −44.33 dB with a thickness of 2 mm at 12.64 GHz, and the bandwidth below −10 dB was 4.40 GHz (10.80–15.20 GHz). When the Fe^{3+}/Sn^{4+} ratio increased to 10:1, the minimum RL was −33.42 dB with a thickness of 3.5 mm at 7.76 GHz, and the bandwidth below −10 dB was 2.72 GHz (6.56–9.28 GHz). S2 possessed better EM-absorbing properties than the other two samples. The promotion of EM absorption performance can be explained by the following facts. Firstly, the addition of $SnO₂/\alpha$ -Fe₂O₃ to RGO led to interfacial and electric polarization. Secondly, the residual defects and groups in RGO with lamellar structure can act as polarized centers, which are favorable for the EM wave attenuation [[46](#page-8-37)]. With an increase in frequency, the minimum value of the $SnO₂/\alpha$ -Fe₂O₃@RGO composites corresponding to different matching thicknesses moved to a reduced frequency mainly because the increasing thickness of the absorber multiplied the wavelength of the EM waves that could interfere with one another; the corresponding frequency decreased. The result could be explained by the following equations [[47\]](#page-8-38).

$$
d = n\lambda_m / 4(n = 1, 3, 5, 7, 9......),
$$
\n(7)

$$
\lambda_m = \frac{c}{f\sqrt{|\varepsilon_r||\mu_r|}},\tag{8}
$$

where ε_r and μ_r are the complex relative permittivity and permeability of the absorber, respectively. The aforementioned equations indicate that the matching thick-ness increased with decreasing frequency. Figure [7](#page-7-3) also reveals that all of the three $SnO₂/\alpha$ -Fe₂O₃@RGO composite samples could complete absorption over the range of

Fig. 6 Typical Cole–Cole semicircles for SnO₂@α-Fe₂O₃@RGO (S1, S2, and S3) composites

Fig. 7 Frequency dependence of the RL values of the SnO₂@α-Fe₂O₃@RGO composites (S1, S2, and S3)

4–18 GHz with 90% absorptivity and a wide bandwidth. The good absorbing performance of the $SnO₂/\alpha-Fe₂O₃@$ RGO composites could be explained by the excellent interfacial and dipole polarization. When the Fe^{3+}/Sn^{4+} molar ratio reached 6:1, the composites exhibited superior performance. The enhanced microwave absorption properties of S2 may be attributed to the proper ratio positively affecting the properties of $SnO₂/\alpha$ -Fe₂O₃ nanoparticles and meeting the demands of impedance matching and attenuation.

4 Conclusions

A series of $SnO₂/\alpha$ -Fe₂O₃@RGO composites with different $Fe³⁺/Sn⁴⁺$ molar ratios was successfully prepared through a solvent–thermal method. In the ternary composite, SnO₂/ α -Fe₂O₃ nanoparticles were uniformly distributed on the surface of lamellar RGO, which led to interfacial and dipole polarization. The heterostructure between $SnO₂$ and α -Fe₂O₃ improved the EM wave attenuation of the absorber by promoting the polarization process. All three samples achieved effective absorption in the measuring frequency scale. The EM wave absorption of the as-prepared composites could be optimized by setting the Fe^{3+}/Sn^{4+} molar ratio to 6:1. The minimum RL was −44.33 dB at 12.64 GHz with a thickness of 2 mm, and the bandwidth below -10 dB was 4.40 GHz (10.80–15.20 GHz). SnO_2/α -Fe₂O₃@ RGO composites are therefore promising potential absorbers with high performance.

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