

Molten salt electrochemical synthesis of Fe@CNTs for broadband microwave absorption

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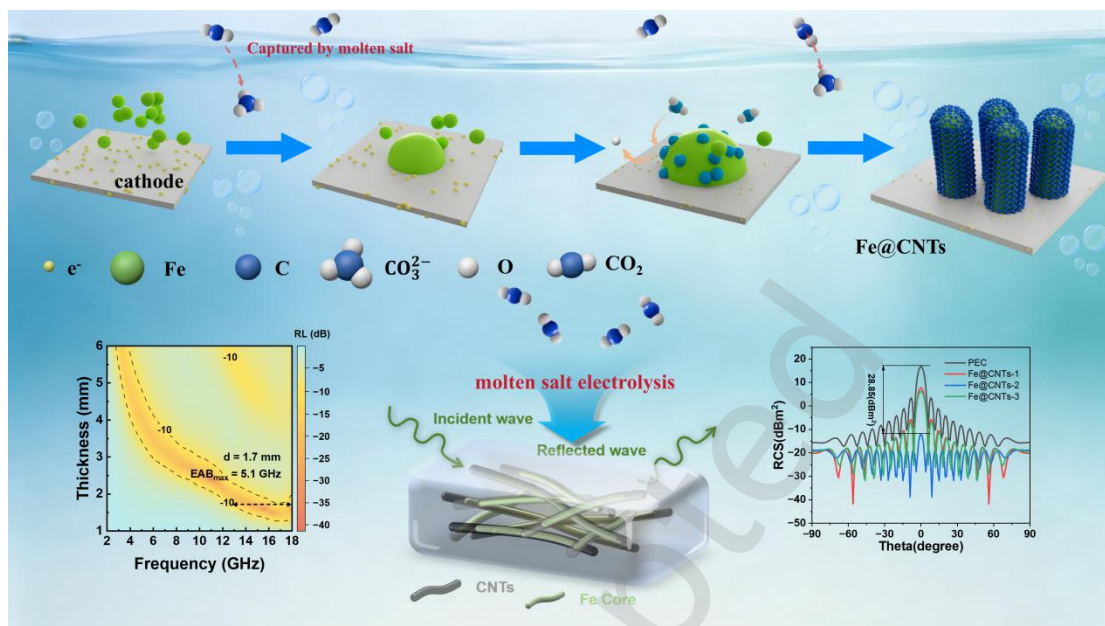
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Graphical Abstract

Fe@CNTs composite absorbers are synthesized in situ via molten salt electrochemical reduction using CO₂ as the carbon source, achieving an effective absorption bandwidth of 5.1 GHz at only 1.7 mm through the synergy of dielectric and magnetic losses.

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ABSTRACT: The rapid development of communication technologies has intensified electromagnetic pollution, creating an urgent demand for high-performance microwave absorbers. Herein, Fe@CNTs composites were synthesized via molten salt electrochemical reduction using CO₂ as the carbon source in a Na₂CO₃–K₂CO₃–CaCO₃ electrolyte. The microstructure, electromagnetic parameters, and microwave absorption performance were systematically investigated. The as-prepared sample exhibits an effective absorption bandwidth of 5.1 GHz at a thin thickness of 1.7 mm, surpassing most reported CNTs-based absorbers. Furthermore, a trapezoidal pyramid metastructure was designed to further broaden the bandwidth; the optimized metastructure achieves full-band absorption covering 2–18 GHz at a total thickness of 16.4 mm, representing a three-fold bandwidth enhancement over the planar configuration. This work provides a green, scalable synthesis route and an effective structural engineering strategy for ultra-broadband microwave absorption.

KEYWORDS: carbon nanotubes; iron-encapsulated; microwave absorbing materials; molten salt; electrolysis

1 Introduction

Mobile communications have now entered the fifth-generation (5G) era, making wireless connectivity an indispensable part of daily life. Nevertheless, the concomitant issues of electromagnetic pollution and signal interference have been intensifying year by year.[1–5] Electromagnetic wave-absorbing materials capable of attenuating unwanted electromagnetic waves are therefore in urgent demand. Such materials also hold critical strategic significance in national defense: aircraft, naval vessels, and other weapon platforms all rely on high-performance absorbing materials to reduce their radar cross-section (RCS)[6–10]. Ideally, a qualified absorbing material should be characterized by a thin profile, light weight, broad absorption bandwidth, and strong electromagnetic attenuation capacity[11–16]. Among conventional material candidates, ferrites and metallic powders deliver considerable magnetic loss, yet they suffer from high density, narrow operating bandwidth, and susceptibility to corrosion. These inherent drawbacks make them difficult to meet the performance requirements set by next-generation electromagnetic protection and stealth technologies. Carbon nanotubes (CNTs) offer a very different profile: a one-dimensional tubular geometry, an ultrahigh specific surface area, and an excellent conductive network, with an electromagnetic response that can be adjusted by defect engineering and heteroatom doping. For these reasons, CNTs have been widely studied as lightweight broadband absorbers.[17–22]

CNTs alone, however, absorb almost exclusively through conduction loss. The resulting high conductivity mismatches the free-space impedance, and a large share of the incident wave is reflected at the surface before it can be dissipated inside, which restricts the effective bandwidth. Introducing magnetic metal nanoparticles such as Fe, Co, or Ni into CNTs has thus become a standard remedy, since their magnetic loss complements the dielectric loss.[23–28] These particles additionally combine high saturation magnetization, natural ferromagnetic resonance loss, and low price. Gu et al.[29] pyrolyzed ZIF-67 together with melamine to prepare core-shell Co-C@CNT and embedded the filler into a self-made S-PDI matrix; the composite delivered a thermal conductivity of 0.84 W/(m·K) and an RL_{min} of –53.8 dB simultaneously. Even so, Fe-containing CNT composites are still mostly produced from high-purity carbon sources and metal precursors, and the cost of these raw materials runs counter to the green, low-carbon development philosophy. With carbon neutrality now a global goal, the capture and utilization of CO₂ has moved to the foreground of materials science and electrochemistry. CO₂ is a cheap, renewable, and practically unlimited carbon feedstock, so converting it electrochemically into functional carbon materials carries both environmental and economic significance. Molten salt electrolysis provides a direct means to this end: the melt absorbs CO₂, and the resulting carbonate ions (CO₃^{2–}) act as the actual reactant that turns into carbon nanomaterials at the cathode, closing the loop from CO₂ to Fe@CNTs absorbing materials. High productivity and simple equipment further make this route suitable for scale-up. Yuan et al.[30] have demonstrated a closely related process, in which multi-walled and single-walled CNTs were cathodically polarized in

molten $\text{CaCl}_2\text{-NaCl}$ and thereby unzipped into oxygen-free graphene stacks with a very low defect density (D/G Raman ratio down to 0.1). Varying the melt temperature between 800 and 1000 °C enabled precise control over the interlayer spacing, particle size, and stacking order of the graphitic flakes, and the flakes served well as lithium-ion battery anodes.

Herein, we prepared Fe@CNTs composite absorbers in situ on the cathode by molten salt electrochemical reduction; a $\text{Na}_2\text{CO}_3\text{-K}_2\text{CO}_3\text{-CaCO}_3$ ternary melt served as the electrolyte, and the CO_2 captured by the melt provided the carbon. The microstructure, electromagnetic parameters, and microwave absorption performance of the resulting products were investigated systematically, and the absorption mechanism of Fe@CNTs is discussed in detail.

2 Experimental

2.1 Reagents

Sodium carbonate (Na_2CO_3 , AR, $\geq 99.8\%$), potassium carbonate (K_2CO_3 , AR, $\geq 99.0\%$), and calcium carbonate (CaCO_3 , AR, $\geq 99.0\%$) came from Sinopharm Chemical Reagent Co., Ltd. (Shanghai, China); ferrous oxide (FeO , AR, $\geq 99\%$) from Yuanfeng Chemical Reagent Co., Ltd. (Guangdong, China); and carbon dioxide (CO_2 , 99.99%) from Xilian Special Gas Technology Co., Ltd. (Shaanxi, China). All reagents were used directly as received.

2.2 Preparation of Fe@CNTs

Na_2CO_3 (236 g) and K_2CO_3 (214 g) were thoroughly mixed and placed in a corundum crucible. The mixture was maintained at 400 °C for 24 h to remove residual moisture, after which the temperature was elevated to 800 °C to achieve complete melting of the salts. Pellets prepared from a mixture of CaCO_3 (49 g) and FeO (1 g) were added to the melt in batches, with a continuous flow of carbon dioxide maintained at 200 sccm throughout the process. Once the pellets had fully dissolved, two electrodes were immersed in the molten salt, and electrolysis was conducted at a cell voltage of 2.2 V, 2.4 V, or 2.6 V for 2 h. Upon completion of electrolysis, the cathode was withdrawn from the furnace. The deposit collected from the cathode was washed sequentially with 1 mol·L⁻¹ hydrochloric acid and deionized water, separated via filtration, and dried at 60 °C for 24 h to yield the final Fe@CNTs product.

2.3 Characterization and testing

Field emission scanning electron microscopy (FE-SEM, Hitachi SU8010, Japan) and high-resolution transmission electron microscopy (HRTEM, JEOL JEM-2100F, Japan) were employed to observe the morphology and microstructure of the products. The crystal phases were identified on a Bruker D8 Advance X-ray diffractometer (XRD); surface chemical states were probed on a Thermo Fisher K-Alpha X-ray photoelectron spectrometer (XPS); and the carbon structure was examined on a Renishaw inVia Raman spectrometer. A Ceyear 3674G vector network analyzer (VNA) was used to measure the electromagnetic parameters over 2-18 GHz via the coaxial-line method. The electrical conductivity of the samples was measured by the four-probe method using an ST2643 four-probe tester (Suzhou Jingge Electronic Co., Ltd., China).

The electrochemical reduction behavior of the Fe species was examined by cyclic voltammetry (CV) in a three-electrode cell at 800 °C, using an equimolar $\text{Na}_2\text{CO}_3\text{-K}_2\text{CO}_3$ melt and a scan rate of 50 mV/s. The working electrode was a homemade powder microelectrode, namely, a Mo cavity of 2 mm diameter with a 1 mm hole that was filled with FeO powder. The reference was an $\text{Ag/Ag}_2\text{SO}_4$ electrode[31] built by immersing a silver wire of 1.0 mm diameter into an internal filling of $\text{Na}_2\text{CO}_3\text{-K}_2\text{CO}_3\text{-Ag}_2\text{SO}_4$ (1.31, 1.19, and 0.078 g, respectively) enclosed within an alumina tube of 0.76 cm outer diameter and 0.55 cm inner diameter, while a Ni-Fe-Cu alloy acted as the counter electrode.

The Fe@CNTs products electrolyzed at 2.2, 2.4, and 2.6 V were mixed with paraffin at a filler loading of 20 wt% (Fe@CNTs:paraffin = 1:4 by mass), giving three composites referred to as Fe@CNTs- 1, Fe@CNTs- 2, and Fe@CNTs- 3, respectively. For measurement, each mixture was warmed to 80 °C until the paraffin melted, agitated thoroughly for uniformity, and molded into a toroidal specimen of 7.00 mm outer diameter and 3.04 mm inner diameter. The electromagnetic parameters of the specimens were collected with the VNA from 2 to 18 GHz, and the reflection loss (RL) at different thicknesses was then evaluated on the basis of transmission line theory with the following equations[32–35]:

$$Z_{in} = Z_0 \sqrt{\mu_r/\epsilon_r} \tanh [j(2\pi f d \sqrt{\mu_r \epsilon_r})/c] \quad \#(1)$$

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

$$M_\eta = \frac{2\eta'}{|\eta|^2 + 1} \quad \#(3)$$

$$\alpha = \frac{\sqrt{2\pi f}}{c} \sqrt{(\mu''\epsilon'' - \mu'\epsilon') + \sqrt{(\mu''\epsilon'' - \mu'\epsilon')^2 + (\mu'\epsilon'' + \mu''\epsilon')^2}} \quad \#(4)$$

$$\varepsilon_r'(\omega) = \varepsilon_\infty + (\varepsilon_s - \varepsilon_\infty) \frac{1}{1 + \omega^2 \tau^2} \quad \#(5)$$

$$\varepsilon_r''(\omega) = \varepsilon_p'' + \varepsilon_c'' = (\varepsilon_s - \varepsilon_\infty) \frac{\omega \tau}{1 + \omega^2 \tau^2} + \frac{\sigma}{\omega \varepsilon_0} \quad \#(6)$$

$$C_0 = \frac{\mu''}{(\mu')^2 \cdot f} \quad \#(7)$$

2.4 Metastructure design and simulation

A trapezoidal pyramid metastructure was further designed in CST Microwave Studio with the aim of extending the absorption band. From top to bottom, the model comprises a periodic array of trapezoidal pyramids (Component 1, Fe@CNTs-paraffin composite), a substrate slab made of the same composite (Component 2), and a perfect electric conductor (PEC) back plate. After optimization, the bottom edge a is 4.4 mm, the top edge b is 4.6 mm, the pyramid height h_1 is 4.7 mm, and the substrate height h_2 is 11.7 mm, which sums to a total thickness of 16.4 mm. The unit cell has a period of 9.0 mm along both x and y .

In the simulation, the x and y boundaries were set as unit cell boundaries to reproduce an infinite periodic array, and the z boundaries as open (add space). A Floquet port provided the plane-wave excitation. The frequency-domain solver computed the S -parameters from 2 to 18 GHz, and the magnitude of S_{11} was taken as the RL. Electric field distributions at 2, 10, and 18 GHz were exported for visualizing the absorption process. The radar cross-section (RCS) of a 200 mm $\times$ 200 mm plate coated with the optimized absorber was additionally simulated, using a bare PEC plate of the same size as the reference.

3 Results and discussion

The formation of Fe@CNTs inside the melt is schematically illustrated in Fig. 1 a. The Na_2CO_3 - K_2CO_3 - CaCO_3 ternary salt functions simultaneously as the electrolyte and as the reaction medium. At 800 °C, FeO dissolved in the melt accepts electrons at the carbon paper cathode and is reduced to Fe nanoparticles ($\text{Fe}^{2+} + 2e^- \rightarrow \text{Fe}$). Concurrently, CO_2 dissolved in the melt. The carbonate anions is cathodically reduced to elemental carbon ($\text{CO}_3^{2-} + 4e^- \rightarrow \text{C} + 3\text{O}^{2-}$). The nascent carbon deposits on the Fe nanoparticles and develops into nanotubes, and the Fe nanoparticles in turn catalyze the nucleation and growth of these CNTs. This process parallels the well-known vapor-liquid-solid mechanism, with the difference that it proceeds in a molten salt medium. Of the O^{2-} ions released at the cathode, one part is oxidized to O_2 at the anode, while the rest recombines with CO_2 and thereby regenerates the carbonate feedstock.

The electrochemical behavior of the Fe species was examined by CV (Fig. 1 b, c). During the first cycle (Fig. 1 b), a strong cathodic peak emerges at about -1.42 V (vs. $\text{Ag}/\text{Ag}_2\text{SO}_4$), corresponding to the two-electron reduction of Fe^{2+} to metallic Fe ($\text{Fe}^{2+} + 2e^- \rightarrow \text{Fe}$); on the reverse scan, the anodic peaks at -1.05 V and -0.78 V reflect the successive oxidation of Fe to Fe^{2+} and subsequently to Fe^{3+} . The third cycle (Fig. 1 c) shows essentially the same profile with slightly increased peak currents, implying a highly reversible and stable Fe^{2+}/Fe redox couple in this melt. Redox peaks that remain well defined over repeated cycling attest to the continuous production of Fe nanoparticles at the cathode, which is a precondition for the uninterrupted growth of CNTs over the 2 h electrolysis.

Fig. 1 d-f show the micromorphology characterization of Fe@CNTs-2 samples. The SEM and TEM images in Fig. 1 d reveal a large number of entangled carbon nanotubes several micrometers in length. Fe nanoparticles of 10-30 nm in size are uniformly encapsulated inside graphitic tubes with diameters of 20-50 nm, and the frequent contacts among tubes supply conductive pathways for electron transport. In the TEM image (inset of Fig. 1 d), particles of dark contrast can be clearly distinguished within the hollow tubular channels, evidencing the core-shell Fe@CNTs architecture. The HRTEM image (Fig. 1 e) shows two sets of lattice fringes with spacings of 0.33 and 0.20 nm; these are indexed to the (002) planes of graphitic carbon and the (110) planes of body-centered cubic (bcc) Fe, respectively, corroborating that crystalline Fe nanoparticles are enclosed within the graphitic nanotubes.

Elemental mapping by EDS (Fig. 1 f) shows a homogeneous distribution of C and Fe over the sampled area, and the Fe signals are concentrated exactly at the positions occupied by the CNTs, once more confirming the confinement of Fe nanoparticles within the nanotube framework. Given this relatively high Fe loading, an appreciable magnetic loss contribution can be expected.

XPS was used to analyze the surface composition and chemical states of sample Fe@CNTs-2. Deconvolution of the C 1s spectrum (Fig. 1 g) yields four components at 283.4, 284.6, 286.2, and 288.0 eV, which are assigned to Fe-C, C-C (sp^2), C-O, and C=O species, respectively. The Fe-C signal is especially noteworthy because it proves the existence of direct Fe-C chemical bonds at the Fe-CNT interface, and such interfacial bonded sites can act as polarization centers that intensify the interfacial polarization loss. Such heterogeneous interfaces can generate a built-in electric field and greatly boost the dielectric response performance, which further corroborates the critical role of the Fe-C bonding interfaces constructed in this work. [36,37] The Fe 2p spectrum (Fig. 1 h) contains a spin-orbit doublet at 710.6 eV (Fe 2p_{3/2}) and 724.5 eV (Fe 2p_{1/2}) attributable to

oxidized Fe-O species, together with an Fe⁰ 2p_{3/2} peak at 706.8 eV that attests to metallic iron. In the O 1s spectrum (Fig. 1 i), the peaks at 531.2 eV (O-Fe) and 532.8 eV (O-C/O=C) are consistent with the partial oxidation of the surface.

In the XRD pattern (Fig. 1 j), the intense reflection at $2\theta = 44.7^\circ$, corresponds to the (110) plane of bcc metallic Fe (JCPDS No. 87-0721), and the weaker reflection at 37.7° is assignable to the Fe₃C cementite phase (JCPDS No. 76-1877). The presence of Fe₃C suggests that iron undergoes slight carburization during electrolysis, and the resulting Fe-C heterostructure is conducive to interfacial polarization. Raman spectra (Fig. 1 k) exhibit a D band at 1345 cm⁻¹ and a G band at 1580 cm⁻¹, with a corresponding I_D/I_G intensity ratio of 0.66. This value indicates that the as-prepared material features a moderate degree of graphitization. Such a graphitization level allows the material to maintain sufficient electrical conductivity for charge transport while retaining adequate structural defects to sustain polarization.

Fig. 1 l presents the current density-time curves logged during electrolysis. All the curves ascend rapidly in the first 0.5 h and then approach a steady value. The plateau current density grows with the cell voltage. A higher current density signifies faster electrochemical kinetics and thus leads to a greater Fe content and more extensive CNT growth.

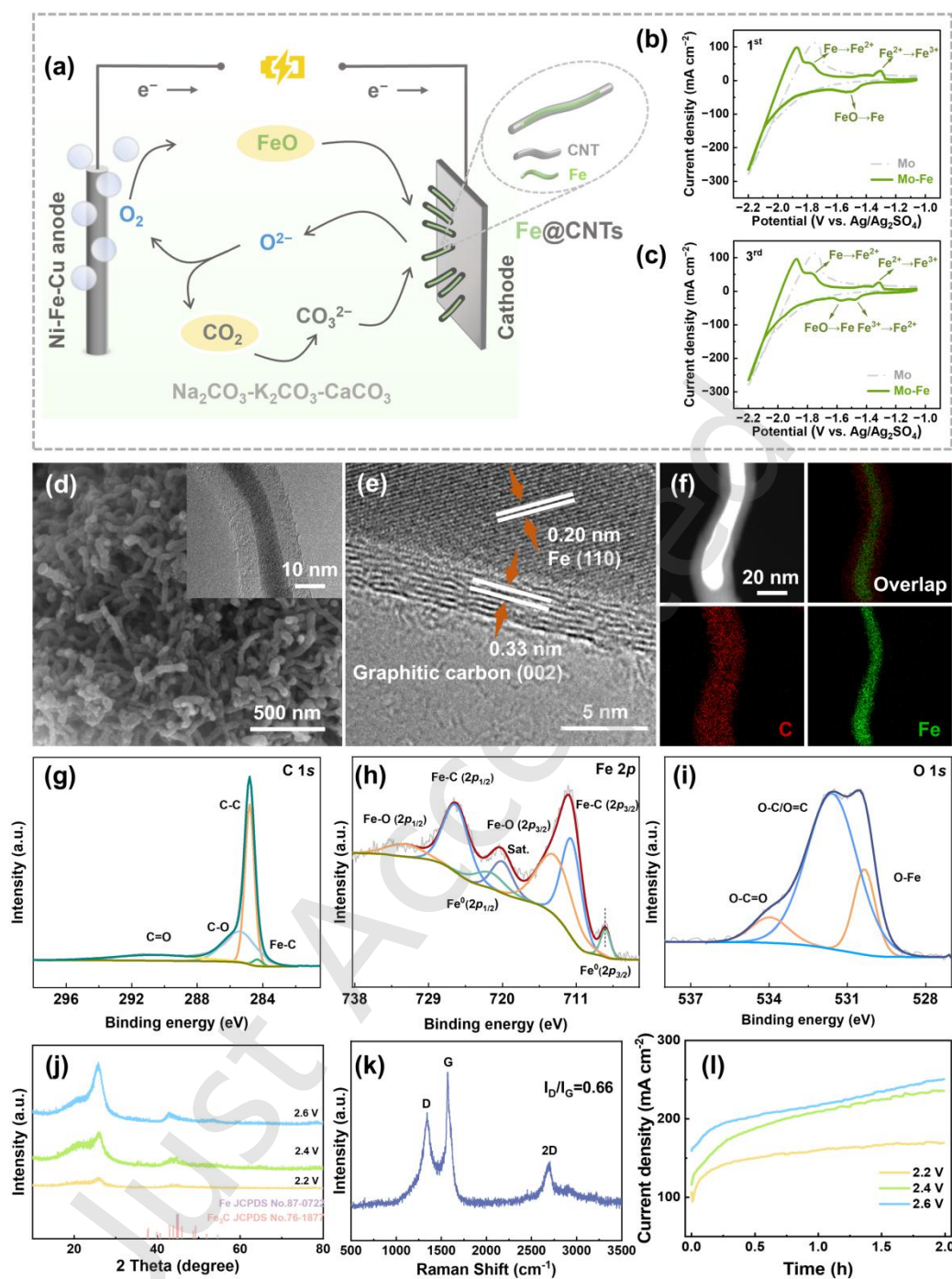


Fig. 1 Schematic and characterization of the molten salt electrochemical preparation of Fe@CNTs: (a) growth process of Fe@CNTs in the melt; (b, c) CV curves of the 1st and 3rd cycles at 50 mV/s; (d) SEM and TEM images of the Fe@CNT-2 samples; (e) HRTEM fringes of 0.33 nm graphitic (002) and 0.20 nm Fe (110); (f) EDS maps of C and Fe; (g-i) high-resolution XPS spectra of C 1s, Fe 2p, and O 1s; (j) XRD pattern with Fe and Fe₃C phases; (k) Raman spectrum with $I_D/I_G = 0.66$; (l) current density-time curves during electrolysis.

The electromagnetic parameters of samples with different concentrations were measured. Among all samples, the specimen with a filler loading of 20 wt% exhibited a broader EAB (effective absorption bandwidth, $RL < -10\text{ dB}$) over the whole frequency range compared with other concentrations. Based on the measured electromagnetic parameters, the RL values at different thicknesses were computed with Eq. 1 and 2 (Fig. 2 a-c), and the complex permittivity ($\epsilon_r = \epsilon'_r - j\epsilon''_r$) and complex permeability ($\mu_r = \mu'_r - j\mu''_r$) of the three samples over 2-18 GHz are given in Fig. 2 d-g. Fe@CNTs-1 shows an EAB of 3.7 GHz (14.3-18 GHz) at 1.4 mm, and Fe@CNTs-3 an EAB of 4.4 GHz (12.8-17.2 GHz) at 1.5 mm. The widest EAB, 5.1 GHz from 12.9 to 18 GHz, is obtained for Fe@CNTs-2 at a small thickness of 1.7 mm. Meanwhile, the RL_{min} of these samples at their corresponding thicknesses were calculated to be -14.7 dB at 16.2 GHz, -27.5 dB at 14.6 GHz, and -26.5 dB at 14.9 GHz,

respectively. Although Fe@CNTs-3 has the strongest dielectric and magnetic losses, its permittivity is excessively high, which deteriorates the impedance matching and leaves its effective bandwidth narrower than that of the better balanced Fe@CNTs-2.

With the electrolysis voltage increasing from 2.2 to 2.6 V, ϵ_r' and ϵ_r'' both rise monotonically throughout the measured band. At 10 GHz, ϵ_r' takes values of approximately 10.5, 14.2, and 18.6 for Fe@CNTs-1, Fe@CNTs-2, and Fe@CNTs-3, while the corresponding ϵ_r'' values are 3.2, 5.8, and 8.4. This trend originates from the higher Fe content and CNT yield achieved at elevated voltages, which introduce additional Fe-C interfaces (beneficial to interfacial polarization) and build a denser conductive network (raising the conductivity).

For all three samples, the real permeability μ_r' stays almost unchanged between 0.9 and 1.2, whereas the imaginary part μ_r'' shows evident resonance peaks. The largest μ_r'' values belong to Fe@CNTs-3, whose broad resonance around 8-10 GHz stems from the natural ferromagnetic resonance of the Fe nanoparticles; such an enhanced magnetic response partly counterbalances the strong dielectric loss and ameliorates the impedance matching. On the whole, Fe@CNTs-2 owns the most balanced parameter combination: its ϵ_r' and ϵ_r'' are sufficiently large to ensure strong dielectric dissipation but remain moderate enough not to impair the matching, and its μ_r' and μ_r'' furnish an appropriate magnetic loss contribution.

Fig. 2 h-i displays the dielectric loss tangent ($\tan\delta_\epsilon = \epsilon_r''/\epsilon_r'$) and the magnetic loss tangent ($\tan\delta_\mu = \mu_r''/\mu_r'$). Over the whole frequency range, $\tan\delta_\epsilon$ is much higher than $\tan\delta_\mu$ in every sample, meaning that dielectric loss accounts for the major part of the absorption. The magnetic contribution is concentrated mainly at 2-8 GHz, coinciding with the strongest magnetic resonance of the Fe nanoparticles. Broadband absorption evidently relies on the joint action of the two loss mechanisms.

In Fig. 2 j, the EAB-thickness combination of Fe@CNTs-2 is benchmarked against recently reported CNT-based absorbers and magnetic metal/CNT composites[38–49]. Positioned in the upper-right region, the present sample unites a small thickness with a broad effective band, which underlines the merit of the molten salt electrochemical strategy in fabricating high-performance absorbers.

Several key loss parameters were further examined to elucidate the absorption mechanism (Fig. 3); the relevant data were computed with Eqs. 3-7[32,33].

Derived from the relative wave impedance, the wave impedance matching coefficient M_η (Fig. 3 a) does not vary with the absorber thickness and hence reflects the intrinsic degree of matching between the material and free space. The nearer M_η approaches unity, the larger the fraction of incident waves that enters the absorber instead of undergoing reflection. Fe@CNTs-2 keeps its M_η closest to 1 across the widest frequency interval (approximately 12.9-18 GHz), which directly explains its maximum EAB. Fe@CNTs-3, on the other hand, exhibits depressed M_η values over the entire band owing to its overly high permittivity, ending up with a narrower effective bandwidth. An efficient absorber therefore calls for a high M_η and a high attenuation constant α simultaneously, because M_η governs whether the waves can enter and α governs whether they are dissipated.

The attenuation constant α (Fig. 3b) measures the capability of a material to dissipate the wave energy that has entered it. In all three samples, α rises continuously with frequency, signifying progressively stronger dissipation toward the high-frequency end. From 2 to 10 GHz, the three curves remain low and nearly overlap, with Fe@CNTs-2 slightly above the other two, in agreement with the weak absorption observed in this interval. Beyond 11 GHz, the α of Fe@CNTs-1 grows fastest and attains the maximum value, whereas Fe@CNTs-3 stays lowest; the high-frequency attenuation thus weakens with increasing electrolysis voltage. However, Fe@CNTs-1 fails to derive additional bandwidth advantage from its highest attenuation capacity, as its M_η values are markedly lower than those of Fe@CNTs-2 across the 12–18 GHz frequency band, resulting in substantial reflection of incident waves at the material surface. By contrast, Fe@CNTs-2 combines high M_η values with a moderate α value relative to the other samples. This complementary set of properties endows it with the widest EAB range among all specimens. These comparative results clearly demonstrate that broadband absorption is governed by the balance between impedance matching and attenuation capacity, rather than being determined by either factor alone.

Cole-Cole plots were employed to analyze the polarization processes within the as-prepared materials (Fig. 3 c), where each individual semicircle corresponds to one Debye relaxation process.[50–53] The curves of all samples comprise several distorted semicircles, multiple polarization relaxations take place concurrently. Compared with Fe@CNTs-1, Fe@CNTs-2 and Fe@CNTs-3 exhibit a greater number of semicircles, pointing to more diverse polarization processes that arise from interfacial polarization at the Fe-C heterojunctions together with dipole polarization of the defects distributed on the tube walls. Such synergistic polarization relaxation at engineered heterointerfaces has been identified as a general route to enhanced dielectric loss.[54–56] Consequently, the multiple Debye relaxations associated with the Fe-C heterostructure serve as an important channel for consuming electromagnetic energy.

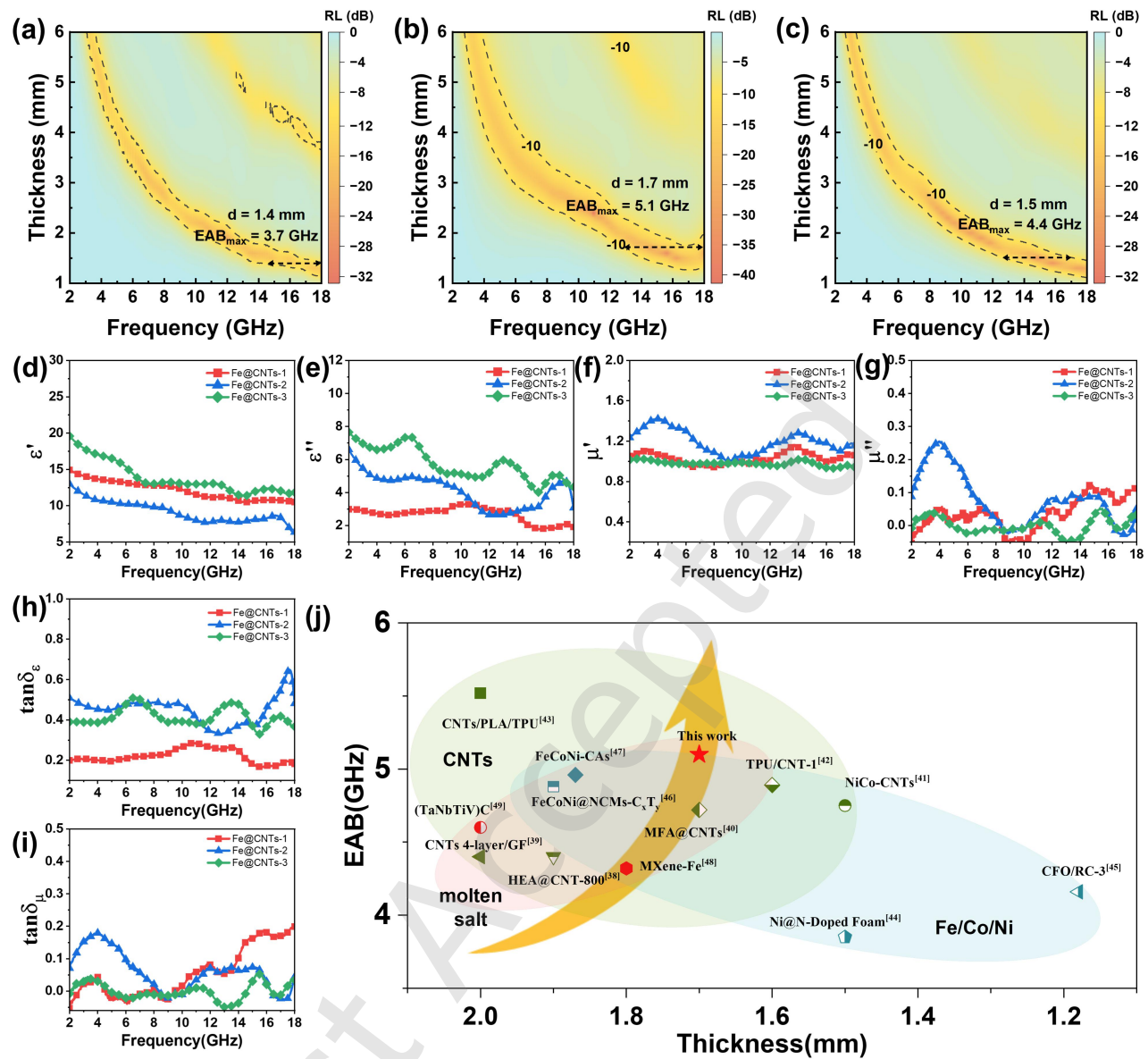


Fig. 2 Microwave absorption performance and electromagnetic parameters of the Fe@CNTs samples: (a-c) 3D RL contour maps versus frequency and thickness for Fe@CNTs-1 (a), Fe@CNTs-2 (b), and Fe@CNTs-3 (c); (d-g) complex permittivity (ϵ_r' , ϵ_r'') and complex permeability (μ_r' , μ_r'') over 2-18 GHz; (h, i) dielectric and magnetic loss tangents ($\tan\delta_\epsilon$ and $\tan\delta_\mu$); (j) EAB-thickness comparison with recently reported magnetic metal/carbon absorbers, carbon nanotube absorbers, and absorbers prepared by the molten salt route.[38-49]

The total dielectric loss (ϵ_r'') was further resolved into a conduction loss (ϵ_c'') and a polarization loss (ϵ_p'') (Eq.5-6) so as to discriminate the individual dielectric loss mechanisms (Fig. 3 d, e). The electrical conductivities of the Fe@CNTs-1, Fe@CNTs-2, and Fe@CNTs-3 samples, measured by the four-probe method, are 8.34×10^{-2} , 4.33×10^{-1} , and $4.49 \times 10^{-1} \text{ S}\cdot\text{m}^{-1}$, respectively. For the three samples, polarization loss constitutes roughly 55-65% of the total, with conduction loss supplying the remaining 35-45%; the predominance of polarization loss can be traced to the abundant Fe-C interfaces and defect sites hosted by the CNTs. The proportion of polarization loss in Fe@CNTs-2 and Fe@CNTs-3 increases within the 12-18 GHz band, which precisely coincides with the frequency range where Fe@CNTs-2 achieves its maximum EAB. This confirms that interfacial polarization plays a critical role in the superior microwave absorption performance of the as-prepared samples.

To elucidate the origin of magnetic loss, the eddy current coefficient C_0 was calculated from the experimentally measured magnetic permeability (Fig. 3 f). When eddy current loss serves as the dominant magnetic loss mechanism, the C_0 value remains constant regardless of frequency. However, the C_0 curves (Fig. 3 f) exhibit clear frequency dependence. A prominent peak located at 3-4 GHz was observed in all samples. An additional broad peak is also observed in the frequency range of 12 - 17 GHz for Fe@CNTs-1 and Fe@CNTs-2 samples. Moreover, all the C_0 values are lower than 0.015 s across the entire measured frequency band. These results demonstrate that the eddy current loss is not a constant, and magnetic loss is mainly dominated by resonance effects. This assignment is physically reasonable: these 10-30 nm Fe particles are far smaller than the skin depth

of bulk iron, and they are electrically insulated from each other by the graphitized shells. Both factors preclude the formation of eddy currents. The resonance peaks present in the μ_r'' curves (Fig. 2g) lend further support to this assignment.

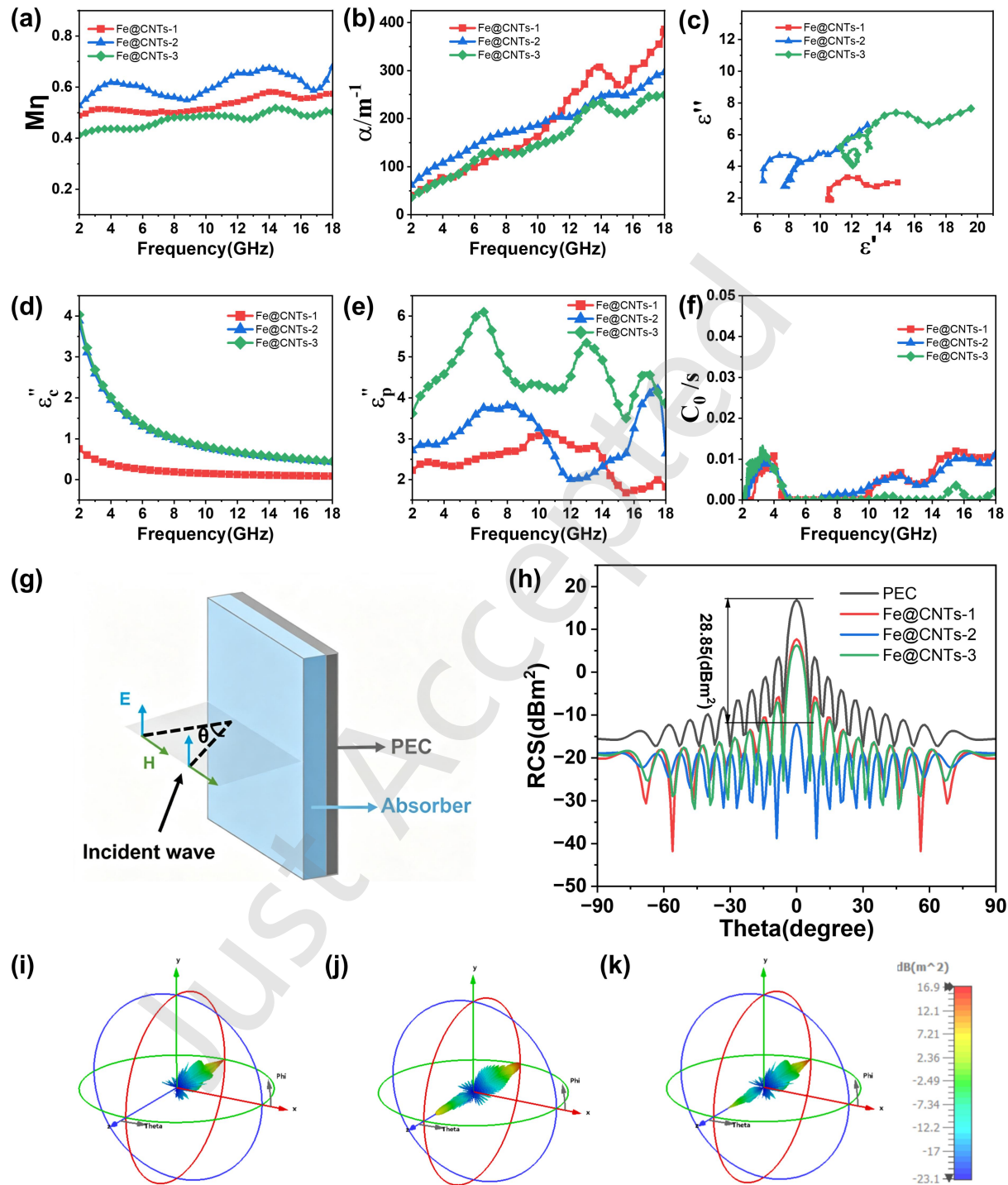


Fig. 3 Loss mechanism analysis and RCS simulation of Fe@CNTs: (a) wave impedance matching coefficient $M\eta$; (b) attenuation constant α ; (c) Cole-Cole plots with multiple polarization relaxations; (d, e) separation of the dielectric loss into conduction and polarization parts; (f) eddy current coefficient C_0 ; (g) RCS simulation geometry of the PEC-backed absorber; (h) RCS curves showing a 28.85 dB·m² reduction for Fe@CNTs-2; (i-k) 3D RCS polar distributions at 16 GHz.

RCS simulation was carried out to evaluate the radar stealth capability (Fig. 3 g, h). The RCS simulation was performed at 16 GHz under normal incidence with a linearly polarized plane wave. Relative to the bare PEC plate, the plate coated with 1.7 mm Fe@CNTs-2 shows substantially reduced RCS values across a wide angular range, and the maximum reduction reaches 28.85 dB·m₂ at normal incidence. In the three-dimensional RCS patterns at 16 GHz (Fig. 3 i-k), the scattering lobe is greatly

weakened once the absorber coating is introduced. These simulation results suggest that the Fe@CNTs absorber holds considerable promise for realistic radar cross-section reduction.

Planar absorbers are intrinsically restricted in bandwidth; to surmount this restriction, we optimized a trapezoidal pyramid metastructure composed of the Fe@CNTs-paraffin composite by CST simulation. As depicted in Fig. 4 a, the architecture places a periodic pyramid array (Component 1) above a homogeneous substrate (Component 2), with a PEC plate on the back side, and both components adopt the Fe@CNTs-2 composite. The optimized geometry comprises a bottom edge $a = 4.4$ mm, a top edge $b = 4.6$ mm (slightly exceeding the bottom edge so that the cross-section takes an inverted-trapezoid shape), a pyramid height $h_1 = 4.7$ mm, and a substrate height $h_2 = 11.7$ mm, amounting to 16.4 mm in total. The cross-section of the metastructure widens progressively from its apex to the base, giving rise to a gradient impedance transition through the material thickness. The planar Fe@CNTs-2 absorber delivers a maximum EAB of 5.1 GHz at a thickness of 1.7 mm. By contrast, the RL curves presented in Fig. 4 b demonstrate that the proposed metastructure achieves an EAB of 16.0 GHz at a total thickness of 16.4 mm. This metastructure fully covers the entire 2–18 GHz frequency band, with an absorption bandwidth more than three times that of its planar counterpart.

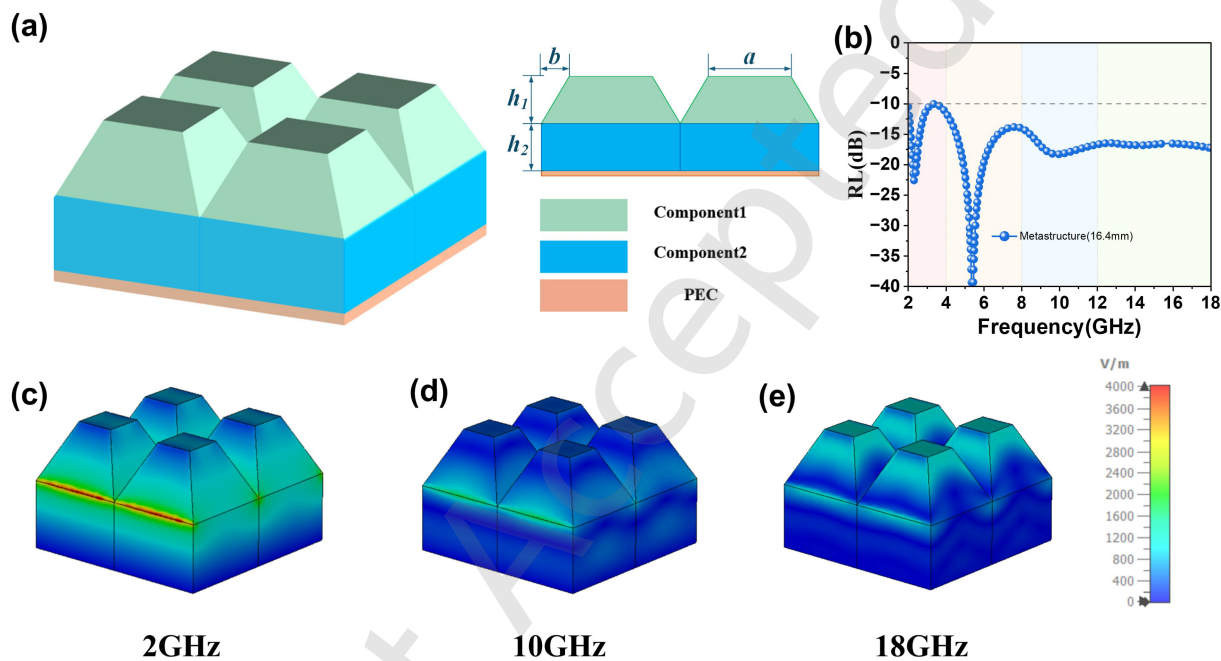


Fig. 4 Metastructure design and absorption mechanism: (a) three-dimensional schematic of the trapezoidal pyramid metastructure with geometric parameters; (b) RL comparison of the planar absorber (EAB = 5.1 GHz) and the metastructure (EAB = 16.0 GHz covering 2-18 GHz); (c-e) electric field distributions at 2 GHz, 10 GHz, and 18 GHz.

The performance of the metastructure arises from the synergy of multiple mutually reinforcing effects. The sloped pyramidal configuration facilitates a gradual impedance transition from free space to the interior of the absorber. At the top of the pyramid, the effective permittivity reaches a sufficiently low level to achieve impedance matching with free space. As the structure extends along the downward direction, the gradual increment of the cross-sectional area not only maintains satisfactory impedance matching performance, but also improves the electromagnetic attenuation capacity. Consequently, surface reflection is markedly suppressed across a broad frequency band. Furthermore, electromagnetic waves that irradiate the inclined surfaces of the structures undergo multiple reflections between adjacent pyramid units. This process extends the effective propagation distance of electromagnetic waves within the lossy medium, until the incident electromagnetic energy is completely attenuated. This multiple scattering effect is especially significant at high frequencies, at which the wavelength of electromagnetic waves is comparable to the characteristic dimension of the pyramidal units. The periodic array arrangement constructed in this design can further support the excitation of multiple resonant modes. Specifically, different regions of each trapezoidal unit resonate at distinct characteristic frequencies, and the superposition of these individual resonance responses ultimately induces a broadband absorption behavior. Meanwhile, the increase in the overall thickness of the structure compensates for the absorption performance in the 2–6 GHz frequency band, which cannot be completely covered by a single-layer pyramidal structure. Based on the above analysis, the synergistic interaction of gradient impedance matching, multiple scattering and multi-resonance superposition collectively contributes to the excellent full-band absorption performance of the

optimized metastructure. The simulated electric field distributions at representative frequencies (Fig. 4 c-e) can intuitively validate the aforementioned synergistic absorption mechanisms.

Figure 5 presents a schematic illustration of the integrated synthesis and microwave absorption process. The upper panel illustrates the electrochemical growth mechanism: Fe^{2+} ions are reduced to iron nanoparticles at the cathode, while CO_2 -derived carbon grows into carbon nanotubes with iron acting as a catalytic agent, enabling the in-situ formation of Fe-C heterointerfaces throughout the growth process. The lower panel summarizes the energy dissipation pathways in the as-prepared product. Owing to the balanced electromagnetic parameters, incident waves first penetrate the absorber readily under good impedance matching (high M_i) and are then exhausted simultaneously through conduction loss along the interconnected nanotube network, interfacial polarization at the abundant Fe-C heterointerfaces (including Fe_3C), dipole polarization of the defects and oxygen-containing groups on the tube walls, and magnetic loss, namely natural resonance and exchange resonance, of the encapsulated Fe nanoparticles. The joint operation of these mechanisms underlies the remarkable broadband absorption performance.

From an engineering standpoint, paraffin serves only as a carrier for electromagnetic parameter measurements, whereas the Fe@CNTs filler can be transferred to engineering matrices such as epoxy resin or silicone rubber for practical use. Moreover, the Fe-O signal in XPS indicates that oxidation is confined to the surface of the Fe cores; the continuous graphitic shells act as a barrier against oxygen and moisture, similar to the shell-engineering strategy used in core-shell magnetic absorbers with self-anticorrosion functionality, ensuring long-term stability in service.[57–59]

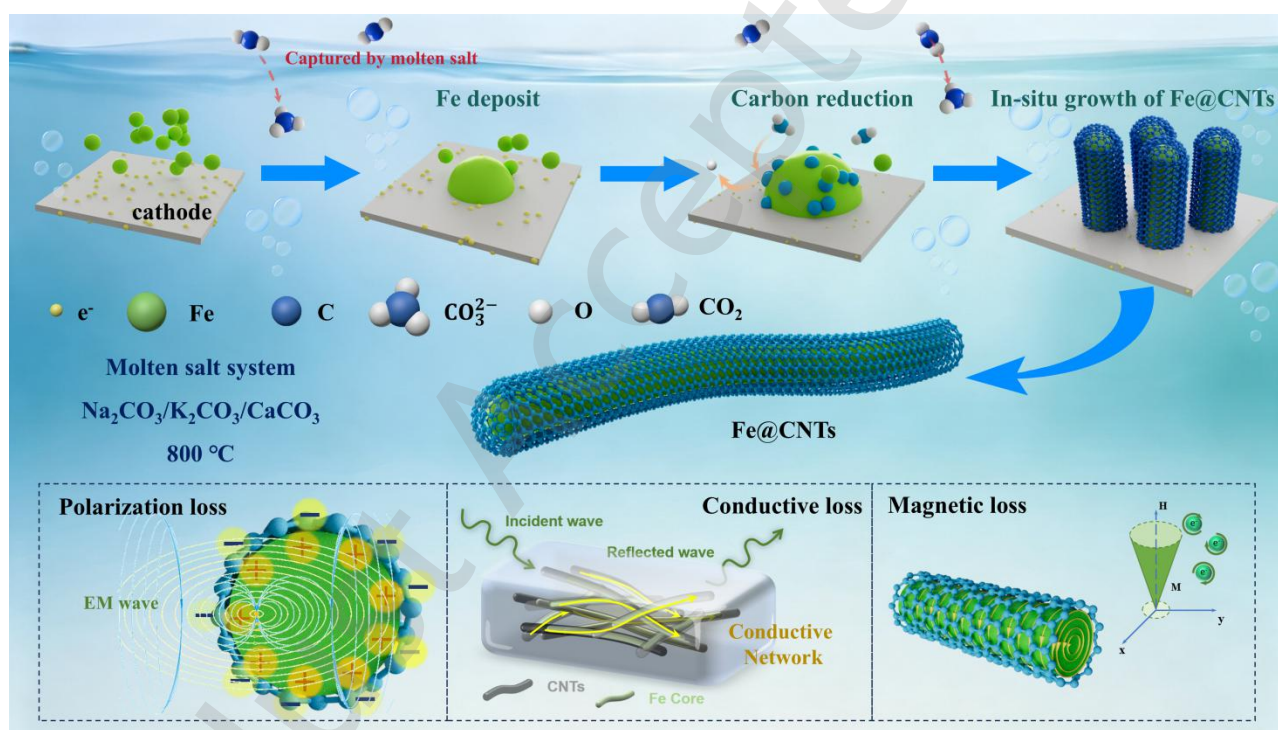


Fig. 5 Overall mechanism summary: molten salt electrochemical growth of Fe@CNTs; schematic of the electrolytic cell; cooperative absorption mechanisms including wave impedance matching, conduction loss, interfacial and dipole polarization, and magnetic loss.

4 Conclusions

In summary, Fe@CNTs composite absorbers were prepared efficiently through molten salt electrochemical reduction with CO_2 as the carbon source in a $\text{Na}_2\text{CO}_3\text{--K}_2\text{CO}_3\text{--CaCO}_3$ melt, and their bandwidth was further enlarged via metastructure design. The applied voltage exerts a strong influence on the CNT yield and the microstructure: Fe nanoparticles of 10–30 nm are uniformly encapsulated in graphitic CNTs of 20–50 nm diameter, and the abundant Fe-C heterointerfaces are corroborated by the Fe-C bonding signal in XPS. Among the three products, Fe@CNTs-2 shows the best electromagnetic balance, attaining an effective absorption bandwidth (EAB) of 5.1 GHz at a thickness of merely 1.7 mm, which compares favorably with most reported CNT-based absorbers. Mechanistic analysis reveals that this performance originates from the cooperation of conduction loss in the interconnected nanotube network, interfacial polarization at the Fe-C heterointerfaces, and magnetic loss of the iron nanoparticles. Moreover, the optimized trapezoidal pyramid metastructure ($a = 4.4$ mm, $b = 4.6$ mm, $h_1 = 4.7$ mm, $h_2 = 11.7$ mm) absorbs over the whole 2–18 GHz range at a total thickness of 16.4 mm, tripling the bandwidth of the planar absorber by virtue of gradient impedance matching, multiple scattering, and multi-resonance superposition.

The developed synthesis route is facile, scalable, and environmentally benign, and the required raw materials are inexpensive, rendering it promising for the industrial-scale production of magnetic carbon-based absorbers. Equally important, the metastructure design exemplifies a viable structural-engineering route toward ultra-broadband microwave absorption, holding potential for applications in electromagnetic stealth and interference suppression.

Data availability

All data needed to support the conclusions in the paper are presented in the manuscript and/or the Electronic Supplementary Material. Additional data related to this paper may be requested from the corresponding author upon request.

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Declaration of competing interest

All the contributing authors report no conflict of interests in this work.

Author contribution statement

Tong Wu: Conceptualization, Data curation, Methodology, Writing-original draft. **Song Bi:** Conceptualization, Formal analysis, Resources, Supervision. **Hao Li:** Investigation, Project administration, Supervision. **Hong-wei Wang:** Resources, Software, Writing-review and editing. **Zhen Wang:** Investigation, Formal analysis. **Rui Meng:** Investigation, Project administration, Resources. **Wei Xiao:** Methodology, Resources, Supervision. **Zhi-ling Hou:** Supervision, Project administration.

Informed consent

Not applicable.

Ethics statement

Not applicable.

Use of AI statement

None.

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