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Expanded graphite / Polyaniline / F & O4 Composite

Preparation and wave absorbing property

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Abstract: expanded graphite was prepared by in-situ polymerization method / Polyaniline (Eg/pani Composites , will Fe3 0 4 is loaded on Eg/pani Surface ,

to obtain a with electromagnetic absorption properties Eg/pani/fe3 0 4 Composite . by scanning the electron microscope (SEM) , X X-ray diffraction (XRD),

Fourier Transform infrared spectrometer (FTIR) and Vector Network Analyzer (VNA the appearance of composite materials , the component and absorbing properties arestudied investigate . The Wave-absorbing performance analysis results show that , When the doping concentration is 0.05 mol/l , Match thickness d = 2 mm , Minimum reflection loss for samples (rl_

in 8. $ GHz at the - Panax DB. increases with doping concentration , Minimum reflection loss peak moves to low frequency , The corresponding thickness of the is gradually thickened . Dielectric Relaxation polarization of the material , Eddy Current loss and A/4 Interference cancellation of the model Shuangfeng , makes theEg/pani/fe3 0 4 Composite in electrical The field of magnetic wave absorption has certain application prospects .

Keywords: expanded graphite ; Conductive Polymers ; Magnetic Material ; Composite ; Wave-absorbing performance
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with the wide application of electronic products , Electromagnetic Interference , electromagnetic pollution and electromagnetic leaks are getting worse , prepare for civilian and military purposes subject to concern . and a absorbing material is a functional material that is consumed by converting electromagnetic waves projected onto its surface into heat or other energy. . Composite absorbing material can effectively improve the electromagnetic environment , reduce electromagnetic leaks , is a new functional material ^,.

ferrite as a traditional nano-absorbing material , has super paramagnetic and nano effects , absorbs performance strongly , The has in the field of electromagnetic absorption Wider application [0] . But its disadvantages such as high density and easy oxidation limit its application [H] . Polyaniline (PANI) is a good stability , raw Materialcheap , synthetic Easy , conjugated conductive polymers for high temperature and antioxidant properties , The has in the field of electromagnetic absorption _ The applied price set by value [S] . The composites of both have a great concern because of their electrical losses and magnetic losses . Hou etc [6] using hard template method to prepare hollow structure F &O4/pani Minimum reflection loss for microspheres (RLm in) on GHz at -24.3 DB . Guo Yajun [7] The prepared the polyaniline / F & O4 / Carbon black composites , to C band (4~8 GHz) and Ku band (12~18 GHz) has better absorption of electromagnetic waves . and carbon black than , worm-shaped expanded graphite (EG)as a new type of carbon absorber , due

to porous network holes , to have a larger area than the table , to produce a high activity fresh surface , and an increase

in the interface layer will generate multiple layers of reflection on electromagnetic waves or Absorbs the, and to Increase the wave-absorbing ability . Zhao etc [&0] prepared eg/f&O4Nano-ring , its m in in 6. 8 GHz at -24. 8 DB . Chen , and so on [Efl] prepared EG / Pani/cofe 2 O 4 Composite , its RLm in in 13.28 GHz at -19. DB . for this , will F &O4 load on eg/panimatrix , Get loose porous structure ternary composite material , is expected to improve the electromagnetic matching effect of materials , from the To improve its wave absorbing performance .

This article uses the in-situ growth method to prepare the Eg/pani Composite , and Fe ; O 4 to Eg/pani Composite for adsorption , gets the with both electrical loss performance and magnetic loss performance. / pani/fe3O4 composite , and study different HCl Doping to EG/ pani/fe ^ Effects of absorbing properties of composite materials ,prepared by EG / pani/fe ^ Composite has a certain wave-absorbing performance , good wave performance .

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1. Experiment Section

1.1 Reagents and instruments

aniline (An), Parse Pure , Tianjin da Mao chemical reagent factory , use after decompression distillation ; ammonium persulfate (APS), Parse Pure , Tianjin City Heng Xing Chemical Reagent Manufacturing Co., Ltd. seven hydrated ferrous sulfate (FeS 0 4*7 H2 0), Parse Pure , Tianjin da Mao chemical reagent factory ; six hydrate ferric chloride(FeCl3 *6 H2 0), Parse Pure , Yantai Chemical Co., Ltd. ; Ammonia (Quality Score 25%~28%, parsing Pure , Silver Companion Chemical Reagent Co., Ltd. ; Hydrochloric acid (HCl , Quality Score 36%~38%, Parse Pure , Silver Companion chemical Reagent Limited companies ; anhydrous ethanol , Parse Pure , Tianjin Fu Yu Fine Chemical Co., Ltd. ;expanded graphite (EG), Excellent-level pure , Qingdao Tian Yuan da graphite Co., Ltd. ; two times distilled water for homemade .

S -4800 Type field emission scanning electron microscope (SEM); Japan Hitachi Corporation ;D 8/advance type X x-ray diffraction (XRD) instrument , German Brooks company ; NEXUS~670 type Fourier transform IR spectrum (FTIR) instrument , USA Hi-Power company ; PNA . 5244 A type vector Amount Network analyzer , agilent .

Eg/pani Preparation of composite materials

references [A] method to prepare the Eg/pani Composite . will 0. G [] [[]] to join ? ML The ultrasonic dispersion in distilled water is complete all , join 0.40 mol/l Hydrochloric acid and 0.50 ML aniline , Ultrasonic Scatter complete . Transfer the system to three bottles , will again contain the 1. g APS "" ML Water Solution constant pressure 0. 5 h Add to above solution , Magnetic Stirring H , centrifugal Drying , To get eg/pani composites .

Keep the amount of expanded graphite constant , The amount of other reagents is increased proportionally to (Table 1) , Repeat the above experiment as a control experiment .

eg/pani/fe_3O_4 Preparation of composite materials

references] method to prepare the Fe 3 0 4 Magnetic nanoparticles . adds a certain amount of FeS 0 4 7 H 2 0 and FeCl3 • 6H2O massage ratio for 1: 2 blending , and dissolving it to 100 mL in distilled water ultrasonic disperse evenly . Transfer the mixture to three bottles , under protection Water bath heating to ° C ° . Add ammonia reaction 2 H after , heats up to ° C And keep the warm 1 H . washed , gets after drying Fe3 0 4 Magnetic Nano granule .

will 4>ANI-3 Composite and 0. G Fe3 0 4 magnetic nanoparticles added to ML anhydrous Ethanol , Ultrasonic Dispersion completely , transfer to three-port bottle mechanical stirring 3 H , Magnetic Separation , Dry , gets the EG / PANI / Fe3 0 4 Composites .

2. Results and discussions

2.1 Sample Characterization

diagram 1 Scanning electron microscope photos for different materials . from Diagram 1 (A) to see , EG renders a loosely porous structure , with a good slice layer Structure . The that is prepared in this article EG Good expansion , has a fresh, active surface , is PANI The in-situ growth of provides the basis for . diagram 1 (B) ~ (E) is different PANI Packet-covered EG / PANI sem photos of composite materials . diagram 1 (B) The amount of aniline is 0.50 mL "" EG / PANI Composite Shape , to see , PANI in theEG Block reunion on , and accompanies PANI Fiber-like aggregates . diagram 1 (C) , when the amount of aniline is reduced to 0.25 ML , PANI The package for is larger than the figure 1 (A) is noticeably smaller , but still has PANI particle reunion in EG Surface . further reduce the amount of aniline to 0. ML, , from Diagram 1 (D) to see the clearly PANIEvenly to overwrite EG layer Structure , with better wrapping effect . also , from Diagram 1 (G) EDS The spectrogram can see , There are in composite materials N , Cl and Selement , where Cl The element originates from the HCl doped , S The element comes from an oxidizer ammonium persulfate , proved to be covered in expanded graphitesurface particles to PANI ^{M} . when the amount of aniline is lowered to the 0. ten mL when , from Diagram 1 (E) to see , PANI still enable EG End full cover , But the amount of coating is significantly higher than that of aniline 0. ML less . in the EG / PANI Composite Load Fe3 0 4 Experiment , uses aniline for 0. ML .

diagram 4 gives the EG , PANI , Fe_3O_4 , EG / PANI and EG / pani/fe $_3$ O $_4$ Composite TGA Curve . when the temperature reaches All ° C , EG and Fe_3O_4 The has a lower rate of weightlessness , is 8. 3% and 8. 5, and PANI The weight loss rate for is up to I 7%. PANI weightlessness of can be attributed to 3 phases : adsorption of water loss and reaction of aniline monomer volatilization , The volatilization of low polymers and the decomposition of polymer molecular chains . EG / PANI Composite in All C The weightlessness rate for is 28%, to calculate the in the composite PANI The mass fraction of is approximately 45.4%, is lower than the composite in PANI The theoretical content of , This is because the PANI and EG There is an interaction between , Enhanced composite material Material Thermal stability ^{022} . ternary EG / pani/fe 3 0 4 The final content of the composite is 81.0%, to calculate the in the composite Fe 3 0 4 has a mass score of + 1, slightly Lower Fe_3O_4 Add amount 50%.

2.2 Electromagnetic Wave absorption performance analysis

takes into account the HCl Doping amount to PANI Electrical Performance Impact , with 0, 0. , 0. Ten and 0. i mol/ L for HCl to PANI to mix Miscellaneous , get correspondingEG / Pani/fego # Composites , samples are recorded as S 1, S 2, S 3 and S 4. combine composites with paraffin wax by quality more than 1 : 1 Mix , and then suppress the mixed sample to the inside diameter 3. mm, , outside diameter is 7. , mm, thickness is 2. a mm coaxial ring , on 2~18 GHz The frequency range tests its absorption performance for electromagnetic waves .

diagram 5 gives the 2~18 GHz Range Samples S 1, S 2, S 3 and S 4 The electromagnetic parameters of the . from Diagram 5 (A) to see , Sample S 1 's Duplicate permittivity Real part U') The value is much larger than the other 3 Sample , from The decrements to 5. with HCl An increase in the amount of doping , ¡î value decreases , and each sample, values decrease as frequency increases ,s2, s 3 and S 4 lower rate of is smaller . same , in **Figure 5** (B) , Sample S 1 for complex permittivity imaginary part U") The value is also larger than the other 3 Sample , from the 5 Decrease to [] 6 With a larger trend , S 2, S 3 and S 4 for , The value is 9~5.46, 5.2~3 1 and 4.2~3 1, shows a trend of decreasing first and then increasing for the entire frequency range . diagram 5 (C) , - like product S1 Complex permeability of (/T) The change in is significantly different from the other 3Sample , in 2~10 and 15~18 GHz Is in a downward trend , in 10~15 GHz The scope is an increasing trend . and sample S 2, S 3 and S 4 for ¡î The value is in the 6. 5 and ten. 5GHz minimum appears near is the same as the maximum and the trend is similar . diagram 5 (D) shows the complex permeability of the sample (¡î') with frequency change relationship . Sample S 2, S 3 and S 4 a downward trend throughout the frequency range , and sample S 1 decreases before increasing , in GHz minimum value near , and 4Sample ¡î values have varying degrees of negative . a A positive part of the value is used to reflect the loss of magnetic energy . Conversely , ¡î The value is a negative representation of the magnetic energy from EG/ PANI / Fe ^ radiation out of composites . The energy emitted by the may be caused by the motion of the charge under alternating . Root According to Maxwell's equation , The carrier in an alternating magnetic field forms a vortex current under the influence of a inductive electric field. , and produce an opposite inductive power Farm , convert partial electric field to

magnetic field to radiate from inside material , cause value to drop negative [02324] .

based on the transmission line theory , reflection loss of a single layer absorbing coating at the time of vertical incidence of electromagnetic waves (RL) can be emulated with the following model

diagram 6 gives the EG / PANI / Fe 3 0 4 Reflective loss curves for different thicknesses of composites . Contrast Chart 6 to see , when the matching thickness is 1 mm ,Sample S 1 Minimum reflection loss for (RL_{min}) on , , GHz at -9. 4 DB ; Sample S 2 in D = 2 mm in 8. $ GHz Place RL_{min} reaches -37 DB , corresponds to RL <-10 DB The frequency width of the is 1. GHz (7 92~9. GHz); Sample S 3 at the matching thickness d = 4 mm on 4. A GHz Place RL_{min} reached -30. 8 DB , Bandwidth is 1. GHz (4 24~5. GHz); same ,Sample S 4 on Match thickness d = 4.5 mm on 4.24 GHz Place RL_{min} reach -40 DB, bandwidth to 0.96 GHz(3.76~7.72 GHz). contrast 4 Sample Discovery , with HC 1 An increase in the amount of doping , the minimum reflection loss peaks of the sample are gradually moving to the low-frequency . Considering the reflection loss of the sampleconsumption and matching thickness , This article with S2 as example , comparison 4 sample RL <-10 dB Bandwidth . at the matching thickness D =1. 5 mm , Sample s1,s 2, S 3and The bandwidth for the S 4 corresponds to 0, 2.24 64~ ,) GHz ,1.92 (+ 4 GHz ") and 2. (12.72~14.8GHz). to discover , by adjusting the HCl The doping amount of , to make the EG / PANI / Fe3 0 4 Composite to different frequencies The electromagnetic waves of the section are absorbed in varying degrees. .

When the electromagnetic wave hits the surface of the material , will not only encounter reflection on the surface of the absorbing material , and will also encounter an anti-on the metal substrate fire , on the 2 The same frequency of reflection waves , if 2 Wave path difference for reflection wave (4) for odd multiples of half wavelength will appear dry

When the electromagnetic wave enters the absorbing material , its wavelength A the represents the with the following expression :

with S 2 is the research object , diagram 7 the gives its minimum counter fire-loss peaks corresponding to A/4 Model . to see , when match thickness 3. 0, 3. 5,4. 0, 4. 5 and 5. 0 mm when ,

A appears under the same matching thickness 2 feature absorption peaks (divide To be 5. , 6 GHz4.72,15 GHz ;

4. , a GHz ; 3.52,11.44 GHz ; 3. ,

10.32 GHz), and with increased matching thickness , Shuangfeng The band width between decreases gradually . Description EG / PANI / Fe 3 0 4 The electromagnetic absorber at the minimum reflection loss peak of the composite material A /4 and 3 A /4 Model . The main reason for is that the absorbing material The reflection waves on the surface and the reflection waves of the matrix are eliminated . . indicates that interference is eliminated in EG / PANI / Feso ^ Composites The Electromagnetic absorption process of the plays a significant role in .

Electrical loss tangent (,/ ¡î) and magnetic loss tangent (¡î'/¡î) the reflects the electrical loss energy of the material to the electromagnetic wave Force and magnetic loss Ability [029] . from Diagram 8 to see , with EG / PANI / Fe 3 0 4 Composites HCl increases the amount of doping , The electrical of the material The loss tangent values are decreasing, and their magnetic loss tangent values are increasing . and the electrical loss tangent value (>0.) throughout the frequency The rate range is greater than the magnetic loss angle tangent (< 0), description power loss in EG /PANI/Fe 3 0 4 composites absorbing electromagnetic waves The contribution of the process is greater than the loss of the magnet . The reason for the electrical loss caused by IS : (1) EG / PANI Electronic jumps on the surface , To form a conductance [B0 '] to; (2) A conductive network may be formed inside a composite material , causing electrical loss [M] .

to study dielectric loss and magnetic loss of materials , Many scholars introduce Cole ^ Cole semicircle and Co ^^' k ')$^{-2}$. Corpse [1] theory [1] Sample S 1 as an example , by Diagram 9 (A) can be seen in the , is in the 30~45 Scope , ¡î is in the 14~23 in the range , EG / pani/fe 3 0 4 The Composite has a cole^cole semicircle . because of its , and , value decreases with frequency increase , can be inferred from the 10~18 GHz appears in the scope of cole^cole semicircle , The indicates that the material has dielectric relaxation in the High-frequency range , The is primarily made up of dipole polarization and interface poles cause ' 38 . is usually , Magnetic losses are mainly derived from domain wall

resonances , Eddy Current loss , Natural resonance and exchange resonance On , and Domain wall resonance on 2~18 GHz Band is negligible . The Magnetic loss of the sample may be caused by an exchange resonance , This is because the F & O4 The ruler of the nanoparticles in ten nm around , Small particle size , is advantageous for resonance exchange behavior [. from Diagram 9 (B) To See , for the material C 0 value is gradually with frequency Decrease , on />8 GHz Basic remain constant , This description EG/pani/ Fe^O# The absorption of electromagnetic waves by composite materials in the /<8 GHz Natural resonance in range ,/>8 GHz within range of Eddy current loss main .

3. knot theory

the uses the in-place aggregation method to prepare the Eg/pani composite , and Fe3 0 4 to eg/pani composite material adsorbed , gets the with electromagnetic performance EG/ pani/fe 3 0 4 Composite , and pass HC 1 to modify the . Research Discovery , Low Doping concentration Benefits The material's absorption of electromagnetic waves . When the doping concentration is 0. mol/l, Match thickness D = 2 mm , in 8. $ GHz at RL_{min} to reach -37 DB . increases with doping concentration , The frequency of the minimum reflection loss peaks of the sample decreases when it appears (64~4.24 GHz), match thickness gradually thicken (1~4 5 mm). by adjusting the HCl Doping concentration , to effectively adjust the frequency bands of the best absorption peaks . The results of the composite electromagnetic parameters indicate that , electrical losses contribute significantly to the electromagnetic absorption process . The material has a dielectric in the High-frequency range yu phenomenon , and 2~8 GHz The magnetic losses of materials within the range are mainly natural resonances , in 8~18 GHz Eddy Current loss in scope is the primary . above loss and A /4 Shuangfeng of interference cancellation in the model , makes the EG / pani/fe 3 0 4 Composite Pairs in 2~18 GHz Scope Partial band electromagnetic shielding within has _ Application Prospects for _ .

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