Preparation and Microwave Absorbing Properties of rGO@MoS2 Composites

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***Abstract*—We synthesized MoS2 nanoparticles by hydrothermal method and prepared the rGO@MoS2 composites by recombination with the reduction of graphene oxide (rGO). In this paper, polyvinylidene fluoroethylene is used as the matrix to regulate the dielectric constant and study the absorption properties of electromagnetic wave. The experimental results show that rGO@MoS2/PVDF with a loading of 5 wt% has the best performance of absorbing wave. Owing to high dielectric loss, extremely high surface areas and carrier mobilities with conductive network, reduced graphene oxides (rGOs) could be a candidate with respect to microwave absorption and EMI shielding. The surfaces of MoS2 spheres are wrinkled. The unique structure of rGO@MoS2 composites can transform and dissipate the energy of electromagnetic waves into heat energy.**

***Index Terms*—absorbing properties, PVDF, graphene**

I. INTRODUCTION

In recent years, many scholars have been devoted themselves to studying the transition metal sulfides MX2 (M = W, Mo, Sn, Ti, Re, Nb, Ta, Hf, Zr) [1, 2]. This kind of material is widely applied in the field of energy storage [3–6], lubrication catalysis [7], semiconductor [8-9], and high-performance protective material [10, 11]. Because of its high specific surface area and electrical performance, this kind of material has good application prospect in wave-absorbing. As a kind of typical semiconductor materials, transition metal sulfur/oxide nanomaterials varied greatly in structure and composition. This kind of materials has attracted extensive attention due to the special band structure, semiconductor or superconducting properties and good mechanical properties. Graphene, as a kind of ultra-thin two-dimensional material, has many defects and functional groups on its surface, which is considered to be an ideal material for electromagnetic wave absorbing. However, high dielectric constant of graphene can lead to impedance mismatch, thus limiting its use. Therefore, we synthesized the nanometer microsphere MoS2 using hydrothermal method, introduced it into graphene, in order to regulate dielectric constant using polyvinylidene fluoride polymer as matrix, improve the flexibility of the material, and study the electromagnetic wave absorption performance.

II. SAMPLE PREPARATION

A. Preparation of MoS2 Nanospheres

MoS2 nanospheres were synthesized by hydrothermal method. Concrete procedures can be described as follow: 0.1 g of Na2MoO4,·2H2O was dissolved in 50 mL of deionized water, with magnetic stirring for 10 min to realize full dissolving. Then, 0.2 g of L-cysteine was added, mixing for 30 min, before placing into a 100 mL reaction kettle for reaction at 200 °C for 20 h. After cooling down to room temperature, the MoS2 nanospheres can be obtained. Finally, the resulted products were repeatedly washed and precipitated several times with deionized water and ethanol, and then dried in 60 °C oven for 12 h.

B. Preparation of Composite Material rGO@MoS2

40 mg of GO and 20 mg of MoS2 nanospheres were dissolved in a flask containing 60 mL of deionized water, and then keep magnetic stirring for 1 h. After that, the flask was transferred to 90 °C oil bath. After holding for 10 min, the mixture solution was added with 32 μL of hydrazine hydrate to reduce GO. This reaction was kept for 2 h followed by ultrasonic treatment for 1 h. Finally, the sample was washed and precipitated with several times with deionized water and ethanol, and then dried in 30 °C oven for 24 h.

C. Preparation of Composite Material rGO@MoS2/PVDF

A certain amount of PVDF was dissolved in 30 mL of DMF while keeping magnetic stirring. After the PVDF was fully dissolved in DMF, a certain amount of composite rGO@MoS2 was added, followed by 2 h of ultrasonic treatment for fully dissolving. Subsequently, the mixture was transferred to a glass evaporation dish, and put into a 70 °C oven for evaporation, thus the rGO@MoS2/PVDF film was obtained.

D. Preparation of Wave-Absorbing Material rGO@MoS2/PVDF

The wave-absorbing material rGO@MoS2/PVDF was prepared by hot-pressing method. Concrete procedures are described as follow: the as-prepared rGO@MoS2/PVDF film was placed in a circular mould (outer diameter: 7.00 mm, inner diameter: 3.04 mm), heated to 220 °C, applied with pressure of 5 MPa, holding for 10 min to melt the film. After that, the melt film was cooled to room temperature and the wave-absorbing sample rGO@MoS2/PVDF was taken out,

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E. *Wave-Absorbing Performance Test*

Agilent PNA 5244 network analyzer was adopted for the test. The dielectric and magnetic permeability parameters of materials under different frequencies were measured by using air line method. The flow diagram involved in the experiment is shown in the fig. 1:

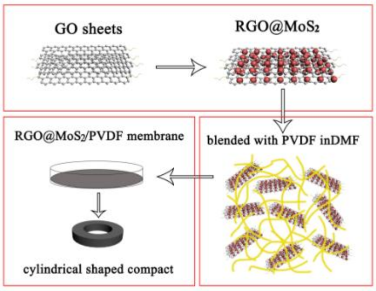

Figure 1. Schematic diagram of preparation and test flow of rGO@MoS2/PVDF.

III. RESULTS AND DISCUSSION

A. *Characterization Result*

The morphology of MoS2 is shown in fig. 2(a). MoS2 is a spherical structure with folds on the surface, with a diameter of 150 ~ 200 nm. Fig. 2 (b) shows the SEM image of rGO@MoS2. It can be seen from the figure that MoS2 nanoparticles are uniformly dispersed in graphene. Fig. 2(c) shows the cold field emission SEM image of cross section of rGO@MoS2 /PVDF film after liquid nitrogen extraction. To characterize the dispersion of rGO@MoS2 in PVDF, mapping was used to measure the distributions of Mo, S and F atoms. As shown in fig. 2(d), the uniform distribution of Mo, S and F atoms means that rGO@MoS2 is uniformly dispersed in PVDF film. The uniform distribution of these atoms facilitates the dielectric and electromagnetic wave absorption properties of the material.

B. *Wave-Absorbing Performance*

In order to study the electromagnetic wave absorption performance of the composite material, rGO@MoS2 /PVDF materials with different mass contents were prepared by hot-pressing method. Fig. 3 shows that rGO@MoS2/PVDF with mass content of 5.0 wt% has better absorbing performance than that with other mass contents. When thickness is 2 mm, the maximum reflection loss reaches −43.1 dB under the frequency of 14.48 GHz. The thickness of the sample has a great influence on the reflection loss value. Within the frequency range of 2 ~ 18 GHz, the frequency bandwidth with reflection loss greater than −10 dB is 3.6 ~ 17.8 GHz, that is, the material has absorption capacity for abroad range of electromagnetic waves.

Within the thickness range of 1–5 mm, the theoretical calculation of the RL variation of MoS2/PVDF (2.5 wt%, 5 wt%, 10 wt%, 20 wt% and 25 wt%) with frequency is shown in fig. 3(a–e). It can be clearly seen from the figure that, as the thickness of the material increases, the maximum reflection loss of the material moves towards the low-frequency direction. Moreover, most of the absolute value of reflection loss is less than 10 dB, indicating the composite material has weak absorbing capacity.

The dielectric constant is related to the material's electron polarization, ion polarization and dipole polarization. The addition of graphene actually increases the resistance type loss of the wave-absorbing material. That is, the larger the conductivity is, the larger the macroscopic current (the eddy current caused by the changes of electric field and magnetic field) caused by the carrier is, the more favorable it is for the electromagnetic energy to change into heat energy. However, the addition of too much graphene does not increase the dielectric loss, but decreases it. 25 wt% rGO@ MoS2/PVDF shows high dielectric constant and dielectric loss (but the theoretical reflection loss is very small), both of which are important electromagnetic parameters of the wave-absorbing material.

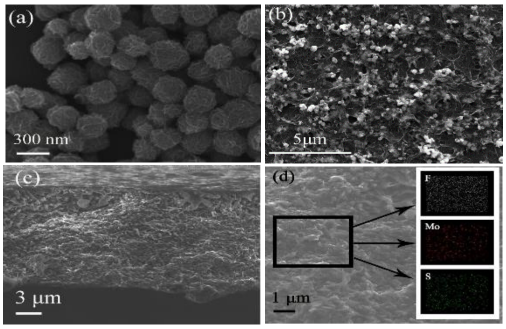

Figure 2 (**a**) SEM diagrams of MoS2 and (**b**) rGO@MoS2; (**c**) (**d**) SEM and EDS scanning elemental analysis diagrams of rGO@MoS2/PVDF films, respectively.

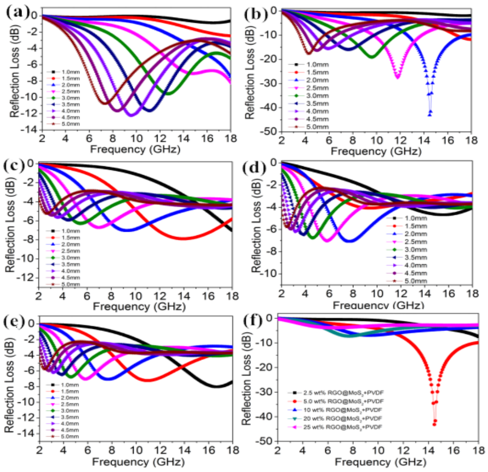

Figure 3. Reflective loss of rGO@MoS2/PVDF with different fillings at 1.0-5.0 mm thickness varies with frequency (**a**) 2.5 wt%; (**b**) 5 wt%; (**c**) 10 wt%; (**d**) 20 wt%; (**e**) 25 wt%; (**f**) reflection loss curves of different fillings at 2 mm thickness.

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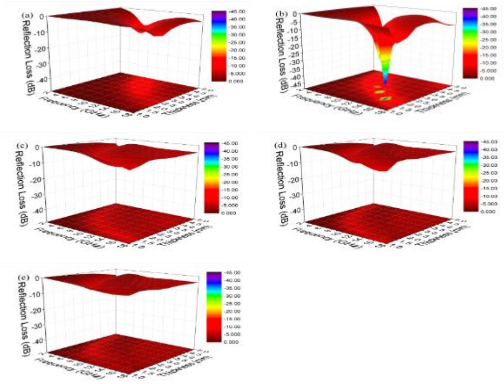

Figure 4. The filling amounts are (a) 2.5 wt% filling, (b) 5 wt% filling, (c) 10 wt% filling, (d) 20 wt% filling, (e) 25 wt% filling, and the three-dimensional absorption spectra of the reflection loss of rGO@MoS2/PVDF varying with frequency and thickness.

The three-dimensional wave-absorption diagram of reflection loss, frequency and thickness of 5 wt% rGO@ MoS2/PVDF is shown in figure 4. The wave-absorbing properties can be adjusted by adjusting the thickness of the absorber.

IV. CONCLUSION

The MoS2 nanospheres were synthesized by hydrothermal method, which was then compounded with rGO to prepare rGO@ MoS2 composite. The perfluoroethylene macromolecules were used as substrate to control the dielectric constant and improve the flexibility of the material, and the electromagnetic wave absorbing performance of the material was studied. Experimental results show that 5 wt% MoS2/PVDF has the best wave-absorbing performance. Graphene has good conductivity and high specific surface area, with a conductive network formed inside. Under the action of electromagnetic wave, polarization is generated inside the medium. MoS2 nanoparticles are nanospheres with folded structures on the surface. The graphene and MoS2 nanospheres were combined into rGO@MoS2 composite material, which is a structure that allows the energy of the electromagnetic wave to be converted into heat energy and then dissipated.

REFERENCE

[1] Z. Wang, Q. Su, G. Q. Yin, et al., “Structure and electronic properties of transition metal dichalcogenide MX2 (M = Mo, W, Nb; X = S, Se) monolayers with grain boundaries,” *Materials Chemistry and Physics*, vol. 147, pp. 1068–1073, 2014.

[2] C. Tan and H. Zhang, “Two-dimensional transition metal dichalcogenide nanosheet-based composites,” *Chemical Society Reviews*, 2015, vol. 44, pp. 2713–2731.

[3] Q. Q. Xiong and Z. G. Ji, “Controllable growth of MoS2/C flower-like microspheres with enhanced electrochemical performance for lithium ion batteries,” *Journal of Alloys and Compounds*, vol. 673, pp. 215–219, 2016.

[4] T. Stephenson, Z. Li, B. Olsen, et al., “Lithium ion battery applications of molybdenum disulfide (MoS2) nanocomposites,” *Energy & Environmental Science*, vol. 7, pp. 209–231, 2014.

[5] D. Wang, Z. Wang, C. Wang, et al., “Distorted MoS2 nanostructures: An efficient catalyst for the electrochemical hydrogen evolution reaction,” *Electrochemistry Communications*, vol. 34, pp. 219–222, 2013.

[6] D. Wang, Z. Wang, C. Wang, et al., “Distorted MoS2 nanostructures: An efficient catalyst for the electrochemical hydrogen evolution reaction,” *Electrochemistry Communications*, vol. 34, pp. 219–222, 2013.

[7] M. Chhowalla and G. A. J. Amaratunga, “Thin films of fullerene-like MoS2 nanoparticles with ultra-low friction and wear,” *Nature*, vol. 407, pp. 164–167, 2000.

[8] Y. Yuan, P. Shen, Q. Li, et al., “Excellent photocatalytic performance of few-layer MoS2/graphene hybrids,” *Journal of Alloys and Compounds*, vol. 700, pp. 12–17, 2017.

[9] D. Ovchinnikov, A. Allain, Y. Huang, et al., “Electrical Transport Properties of Single-Layer WS2,” *Acs Nano*, vol. 8, pp. 8174–8181, 2014.

[10] Y. Q. Zhu, T. Sekine, K. S. Brigatti, et al., “Shock-wave resistance of WS2 nanotubes,” *Journal of the American Chemical Society*, vol. 125, pp. 1329–1333, 2003.

[11] Y. Q. Zhu, T. Sekine, Y. H. Li, et al., “Shock-absorbing and failure mechanisms of WS2 and MoS2 nanoparticles with fullerene-like structures under shock wave pressure,” *Journal of the American Chemical Society*, vol. 127, pp. 16263–16272, 2005.

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