OPTOELECTRONICS AND ADVANCED MATERIALS – RAPID COMMUNICATIONS Vol. 9, No. 1-2, January – February 2015, p. 231 - 233

Electromagnetic and microwave absorption properties and antioxidant properties of Co-P coating on carbonyl iron

LI RONG[a,b,*], ZHOU WAN-CHENG[a], ZHOU YING-YING[a], LIU YUAN[b]
1. *State Key Laboratory of Solidification Processing, School of Materials Science and Engineering, Northwestern Polytechnical University, Xi'an 710072, China*
2. *No. 603 Faculty, Xi'an Institute of High Technology, Xi'an 710025, China*

CI@Co-P particles are prepared by electroless plating technics. The structure, morphology, and antioxidant properties analyses demonstrate that the the CI particles have been coated with intact spherical-shell Co-P coating, and the Co-P coatings, as the efficient barriers to prevent the further oxidation of the CI particles, can delay the oxidant temperature of CI about 150 °C. In terms of reflection loss (RL), the absorbing frequency band (AFB, the value of RL< -10 dB) and the matching thickness (t_m), CI particles also show effective performances after the surface modification of Co-P: minimum RL of -45.0 dB with t_m=1.6mm at 9.7 GHz, AFB of 8.2- 12.4GHz. The results indicate that Co-P coated CI particles can significantly improve the antioxidant properties of CI particles with no negative influence for RL of CI and that CI@Co-P particles could be used as an effective microwave absorption material.

(Received September 29, 2014; accepted January 21, 2015)

Keywords: Electromagnetic and microwave, Carbonyl iron, Cobalt, antioxidant

In electromagnetic and microwave (EM) absorbing research field, the materials, which possess the advantages of wide waveband strong absorption, light weight, and thin thickness, attract more attention with both commercial and defense purposes, [1-3]. Carbonyl iron (CI) particles have a wide range of applications in absorbing materials with high saturation magnetization and relative permeability at microwave frequencies [4]. However, a common drawback of the CI particles is their oxidation at high temperatures to form a non-magnetic material Fe_2O_3, which leads to the decrease in magnetic properties of CI particles, thus CI particles are generally utilized as an auxiliary absorbent in other materials or after surface treatments [5-7]. Some studies have coated Ag, Zn, Cu or Ni layers to protect iron particles against oxidation, [8-13] the results indicate that CI particles coated with Ni is an effective way to reduce oxidation. however, the courier temperature of Ni is lower than Fe. Metal Co have higer courier temperature and higher magnetic saturation, but the effects of Co on the electromagnetic and antioxidant properties of CI particles are unclear. In this letter, Co-P coated Carbonyl iron (CI@Co-P) particles are prepared by independent research and development electroless plating technics [14-15], and the morphology, composition, antioxidant and EM (electromagnetic and microwave) absorbing properties of the particles are investigated.

The preparation of Co-P electroless coating is performed in a beaker heated by thermostat water bath, chemical reagents of $NaH_2PO_2\cdot H_2O$ (reducing substance), $CoCl_2\cdot 6H_2O$ (the Cobalt ion source) and $C6H5Na_3O_7\cdot 2H_2O$ (complexing agents) as the main materials. The operating temperature is 90 °C and the deposition time is set for 60 min. The pH-value of the bath for electorless coating is adjusted to 10 with $NH_3\cdot H_2O$. Commonly, the thickness of Co-P coating can be controlled by the plating time. In this work, the thickness of the Co-P coating is below 1μm.

The raw and Co-P coated CI particles are homogeneously dispersed into paraffin wax at a mass fraction of 70% to fabricate microwave absorbing coatings. The EM parameters (complex permeability and permittivity) of the coatings are measured using a vector network analyzer (HP-8720ES). The samples for electro-magnetic measurement are of the size of 10.16×22.86×2 mm^3 and put into the brass holder (10.16×22.86 mm^2) to measure the complex permittivity and permeability, which are based on the measurements of the reflection and transmission module between 8.2 and 12.4 GHz in the fundamental wave-guide mode TE10. The structure, morphology and composition of the raw and Co-P coated CI particles are examined under a scanning electron microscope (SEM; JEOL JSM-5800 LV SKANNING) attachment with a Links Systems energy dispersive spectrometer (EDS). The antioxidant properties of the CI and CI@Co-P particles are measured by thermo-gravimetric and differential scanning calorimetry (TG-DSC), carried out on a NETZSCH Instrument STA409PC under an atmosphere of air (at a rate of 50 mL/min) with a heating rate of 20°C /min.

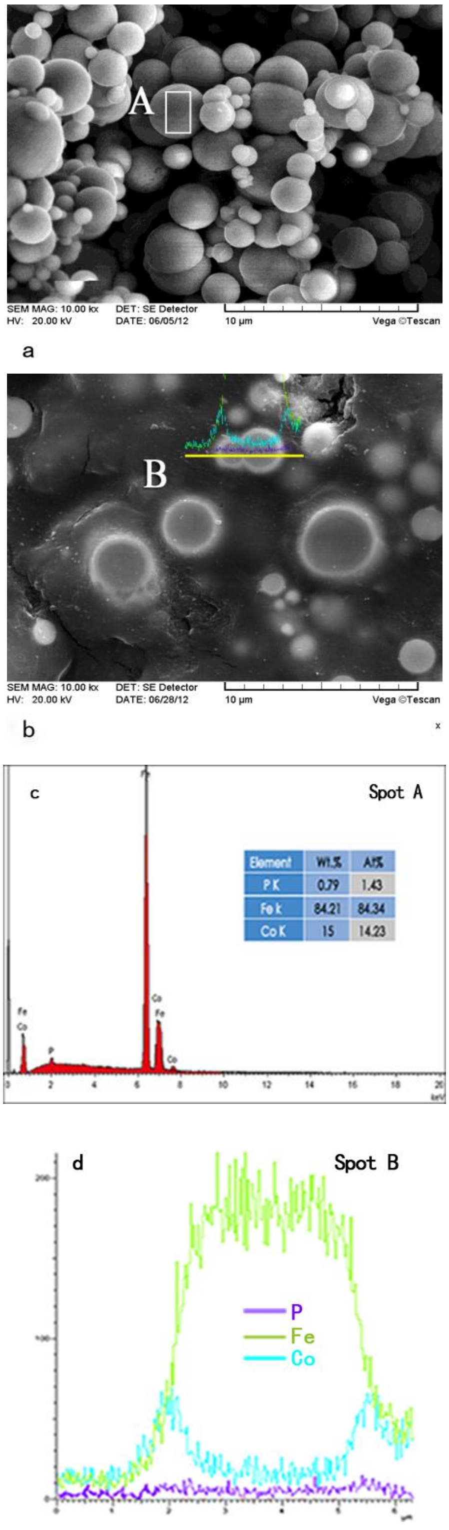

Fig. 1. The SEM images and EDS analyses of CI@Co-P particles.

Fig. 1 shows the surface and cross section SEM images of the CI@Co-P particles. After electroless Co-P plating, the CI particles are uniformly coated with Co-P coating, as shown in Fig. 1(a), and the coating is intact spherical-shell, as illustrated in Fig. 1(b), the results demonstrated that the CI@Co-P have the core-shell structure. The EDS analysis indicates the presence of Co and P on the surface of CI particles after the electroless deposition, as shown in Fig. 1(c) and (d).

Table 1. TG and DSC data for CI and CI@Co-P particles.

Sample	Te.o. /℃	Tp/℃	Tt/℃	ML/%
CI	388.62	469.08.80	530.18	29.10
CI@Co-P	533.28	623.35	674.53	34.04

Te.o = extrapolated onset; Tp = inflexion temperature of TG; Tt = ending temperature of TG; ML = mass loss in %;

Fig. 2 shows TG and DSC curves for the CI and CI@Co-P particles, the results are also summarized in Table 1. It could be seen that, after Co-P coating deposition on the surface of CI particles, the extrapolated temperature, inflexion temperature, and ending temperature of TG for CI all increase about 150 °C, whereas the mass loss just increases about 5%. The results demonstrate that the Co-P coating offers an efficient barrier to protect CI particles against oxidation due to the intactness of the coating [16].

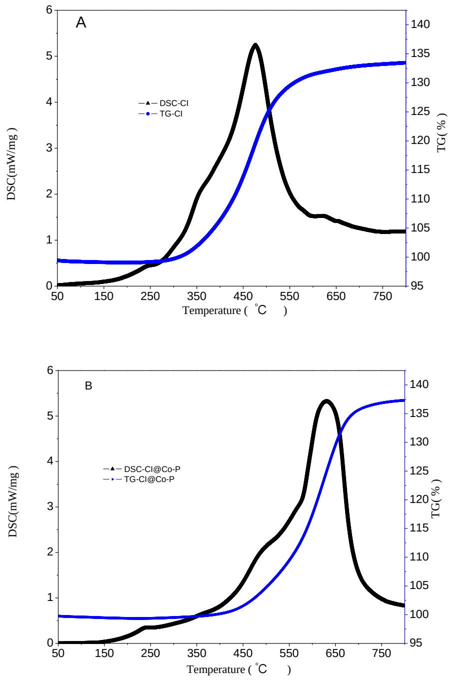

Fig. 2. TG and DSC curves for CI (A) and CI@Co-P (B) particles.

The complex permittivity and permeability of the coatings filled with the CI and CI@Co-P particles are presented in Fig. 3. From Fig. 3(a), it could be seen that the values of the real part of permittivity of the coatings filled with raw CI particles keep almost unvaried($\varepsilon' \approx 11.5$) in the frequency range 8.2–12.4GHz, the real part of permittivity increased (from $\approx$ 11.5 to $\approx$ 13.5) after Co-P coating deposition on the surface of CI particles, and the value of the imaginary part of complex permittivity (ε'') of CI particles remains unchanged before and after Co-P deposition. It can be seen clearly from Fig. 3(b) that the complex permeability of the composites between 8.2 and 12.4 GHz decrease obviously after Co-P coating deposition.

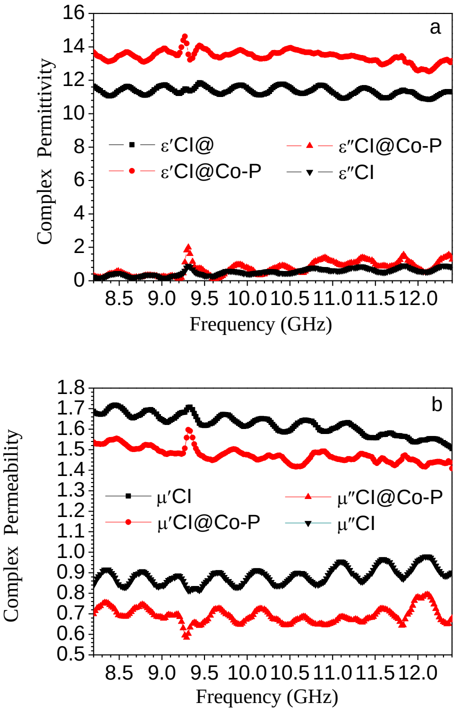

Fig. 3. Complex permittivity and permeability relative to frequency for CI, and CI@Co-P particles.

Fig. 4 shows the effect of Co-P coating on the microwave reflection loss (RL) of CI particles (the content of particles are 70wt %). It can be obviously seen that the absorbing frequency band (AFB, the value of RL$<$ -10 dB) is obtained in the whole X band (8.2-12.4GHz), for both the raw and Co-P coated CI particles filled coatings with matching thickness t_m=1.6mm, and the minimum RL increases from -35.0 dB at 9.9 GHz to -45.0 dB at 9.7 GHz after CI surface modification of Co-P. The result indicates that Co-P coating haven't weaken the effective RL of the CI particles in the X band.

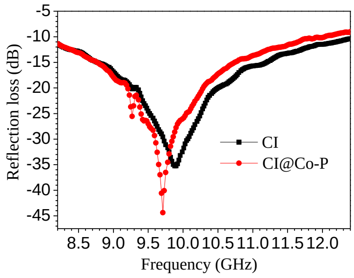

Fig. 4. Microwave reflection loss of CI, and CI@Co-P particles.

In summary, Co-P shell on CI can effectively prevent further oxidation of the CI and improve the microwave absorbing properties of CI. After surface modification of CI, the RL exceeding -10 dB is obtained in the whole X band (8.2- 12.4GHz) with minimum RL of -45.0 dB at 9.7 GHz with a matching thickness of 1.6mm. This research fabricates a potential method to obtain Co-P coated CI composites with good antioxidant and microwave absorbing properties, which may have a potential value in engineering.

References

[1] R. Sharma, R.C. Agarwala, V. Agarwala, Mater. Lett. **62**, 2233 (2008).
[2] Y. Liu, F. Luo, W. C. Zhou, D. M. Zhu, J. Alloys. Compd. **576**, 43 (2013).
[3] Z. Ma, J. B. Wang, Q. F. Liu, J. Yuan, Appl. Surf Sci. **255**, 6629 (2009).
[4] Y. Y. Zhou, W. C. Zhou, F. Luo, D. M. Zhu, Mater. Revi. **27**, 122 (2013).
[5] J. H. He, W. Wang, A. M. Wang, J. G. Guan, Acta. Metall. Sin. **25**, 201 (2012).
[6] M. Wang, Y. P. Duan, S. H. Liu, J. Magn. Mater. **321**, 3442 (2009).
[7] G. X. Tong, W. H. Wu, Q. Hu, J. Alloys. Compd. **509**, 451 (2011).
[8] X. G. Cao, H. Y. Zhang, J. Mater.Eng. **8**, 69 (2007).
[9] Q. X. Zhang, L. N. Yang, J. M. Zhang, J. G.Gua, J.Chin.Ceram.Soc. **35**, 987 (2007).
[10] M. Sarret, C. Muller, A. Amell, Surf. Coat. Technol. **201**, 389 (2006).
[11] S. Alirezaei, S. Monirvaghefi, M. Salehi, Wear. **262**, 978 (2007).
[12] Y. L Wang, W. Li, Q. X. Zhang, W. Wang, Chem. J. Chin. Univ. **10**, 31 (2010).
[13] S. Jia, F. Luo, Y. Cqing, W. C. Zhou, Physica B. **405**, 3611 (2010).
[14] R. Li, X. X. Liu, X. J. Wang, J. Solid. Rocket. Tech. **31**, 607 (2008).
[15] R. Li, X. X. Liu, X. J. Wang, J. Solid. Rocket. Tech. **34**, 754 (2011).
[16] J. Ulicny, A. Mance, Mat. Sci.Eng. A **369**, 309(2004).

---

*Corresponding author: xss760829@163.com