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# Microwave Absorbing and Magnetic Properties of the Polyaniline-$Co_{0.7}Cr_{0.1}Zn_{0.2}Fe_2O_4$ Composites

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The spinel cobalt chromium zinc ferrites ($Co_{0.7}Cr_{0.1}Zn_{0.2}Fe_2O_4$) and the polyaniline (PANI)-$Co_{0.7}Cr_{0.1}Zn_{0.2}Fe_2O_4$ composites were prepared by polyacrylamide gel and an *in situ* polymerization method, respectively. The structure of the synthesized material was characterized by X-ray diffraction (XRD) and Fourier transform infrared spectrometer (FT-IR), which shows that the spinel $Co_{0.7}Cr_{0.1}Zn_{0.2}Fe_2O_4$ ferrites and the PANI-$Co_{0.7}Cr_{0.1}Zn_{0.2}Fe_2O_4$ composites are obtained. Because a small amount $Co^{2+}$ ion of the octahedron ferrite is replaced by $Cr^{3+}$ ions, the lattice constant of the $Co_{0.8}Zn_{0.2}Fe_2O_4$ ferrites reduces from 0.8409 nm to 0.8377 nm. The magnetic properties of the two materials were investigated using vibrating sample magnetometer (VSM). The VSM results confirm that the saturation magnetization (*Ms*), remnant magnetization (*Mr*) and coercive force (*Hc*) of the PANI-$Co_{0.7}Cr_{0.1}Zn_{0.2}Fe_2O_4$ composites are 8.80 emu/g, 3.14 emu/g and 37.22 kA/m, respectively, which are smaller than those of the $Co_{0.7}Cr_{0.1}Zn_{0.2}Fe_2O_4$ ferrites. The microwave absorbing capability of the two materials were studied by wave-guide method. In the measuring frequency range of 5.0 ~ 20.0 GHz, two reflection loss maximum value of the PANI-$Co_{0.7}Cr_{0.1}Zn_{0.2}Fe_2O_4$ composites appear at 14.1 GHz and 17.9 GHz with -13.17 dB and -15.36 dB, respectively, which is obviously higher than those of the $Co_{0.7}Cr_{0.1}Zn_{0.2}Fe_2O_4$ ferrites.

*Keywords:* composites, microwave absorbing properties, magnetic properties.

## 1. INTRODUCTION

With the electronic devices and electromagnetic signals widely used, the electromagnetic radiation and electromagnetic interference have led to a growing impact on people's daily life. Thus, the microwave absorption material which have properties such as: light weight, strong absorbing properties and absorbing bandwidth have attracted a great deal of attention [1, 2]. The spinel ferrites ($MFe_2O_4$, M=Fe, Co Ni, Zn, etc.) are especially important microwave absorbing materials because of its good chemical stability and the strong magnetic loss at high frequencies. Sun et al [3] prepared MnZn ferrites by solid-phase, and then investigated the effects of different temperature on magnetic loss ($P_L$) of ferrites. However, the ferrites have some disadvantages, such as its small dielectric loss and large areal density. As a conductive polymer, PANI has some characteristics such as light weight, corrosion resistance and good environmental stability, but PANI is dielectric material, and its magnetic loss is close to zero, so the pure PANI is difficult to use as a single microwave absorbing material [4 – 7].

The conductive polymer-ferrite composites have attracted more and more attention due to its synergetic and complementary behavior between ferrite and polymer [8 – 10]. Xiao et al [8] have discussed the development of polypyrrol doped with magnetic crystals compound over the past 20 years, explored the synthetic methods, physical properties and application prospects of the electromagnetic polypyrrole composites, and discovered that the electromagnetic properties and microwave absorbing properties are closely related to the amount of the crystals with magnetic properties. Gairola et al [9] synthesized the PANI-$Mn_{0.2}Ni_{0.4}Zn_{0.4}Fe_2O_4$ composites using the mechanical blending method, and studied the effect of the thickness of the composite material on the electromagnetic shielding performance. Yang et al [10] prepared the PANI-$BaFe_{12}O_{19}$ composite by an in situ polymerization, and found that there were two reflection loss bands at 7.8 GHz and 24.2 GHz, which was -12.5 dB and -11.5 dB respectively.

In this paper, PANI-$Co_{0.7}Cr_{0.1}Zn_{0.2}Fe_2O_4$ composites were synthesized by an in situ polymerization. The dielectric, magnetic and microwave absorbing properties of the polyaniline-$Co_{0.7}Cr_{0.1}Zn_{0.2}Fe_2O_4$ were investigated, which has not been reported before.

## 2. EXPERIMENTAL

### 2.1. Sample Preparation

The $Co_{0.7}Cr_{0.1}Zn_{0.2}Fe_2O_4$ ferrites were prepared by the polymer gel method [11]. The PANI-$Co_{0.7}Cr_{0.1}Zn_{0.2}Fe_2O_4$ composites were prepared by an in situ polymerization. In a typical procedure, 1.86 g aniline monomer, 3.44 g dodecylbenzene sulfonic acid (DBSA), 0.186 g $Co_{0.7}Cr_{0.1}Zn_{0.2}Fe_2O_4$ ferrites and 100 ml distilled water were added into a three-necked flask equipped with a mechanical stirrer and ultrasonic dispersed for 1h. After that a large amount of ice was used to let the temperature of the reaction atmosphere at about 0 °C. 4.56 g ammonium persulfate (APS) was dissolved in 100 ml distilled water, and then added dropwise to the reaction system in 30 min. Stirred for 8 h to ensure that the polymerization was complete. All the reaction was carried

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out at about 0 °C. Finally, the mixture was precipitated in an excess volume acetone for 24 h. After that the precipitate was filtered and washed with acetone and distilled water, until the washing solution became clear. Then the filter cake was dried under vacuum at 60 °C for 3 h, and the dark green powder obtained was the PANI-$Co_{0.7}Cr_{0.1}Zn_{0.2}Fe_2O_4$ composite.

## 2.2. Sample characterization

Phase formation of the synthesized product was identified by X-ray diffraction (XRD, with $CuK_{\alpha}$ radiation, $\lambda = 0.154178$ nm, using $\theta$-$2\theta$ step scan mode with a scanning rate 0.02°). Fourier transform infrared (FT-IR, AVATAR-360) spectrometer was used for monitoring the infrared absorption changes of the product. The magnetic properties of the samples were measured by vibrating sample magnetometer (VSM-220) at the maximum applied 20KOe. The vector network analyzer (E5071C) was used to test the permittivity (ε' and ε'') and reflection loss of the samples.

## 3. RESULTS AND DISCUSSION

Fig. 1 shows the X-ray diffraction patterns of PANI-$Co_{0.7}Cr_{0.1}Zn_{0.2}Fe_2O_4$ (a), $Co_{0.7}Cr_{0.1}Zn_{0.2}Fe_2O_4$ (b) and $Co_{0.8}Zn_{0.2}Fe_2O_4$ (c). The peaks appeared at around $2\theta$ = 18.18°, 30.08°, 35.40°, 43.04°, 53.40°, 56.90°and 62.42° are well corresponded to the crystal planes of spinel ferrite (111), (220), (311), (400), (422), (511), (440), respectively, which match well those of JCPDS card 22-1086 for $Co_{0.8}Zn_{0.2}Fe_2O_4$ according to Fig. 1 c. Compared with Fig. 1 c, the location of diffraction peaks of Fig. 1 b slightly move to the right. The location of crystal plane (311) of $Co_{0.8}Zn_{0.2}Fe_2O_4$ and $Co_{0.7}Cr_{0.1}Zn_{0.2}Fe_2O_4$ is 35.40° and 35.54°, respectively. The lattice parameter has been calculated using the classical formula [12]:

$$\alpha = \frac{\lambda(h^2 + k^2 + l^2)^{1/2}}{2\sin\theta}, \tag{1}$$

where $a$ is the lattice parameter; $\lambda$ (0.154178nm) is the wavelength of CuKα; h, k, l are the Miller indices. The value of lattice parameter of $Co_{0.8}Zn_{0.2}Fe_2O_4$ and $Co_{0.7}Cr_{0.1}Zn_{0.2}Fe_2O_4$ is 0.8409 nm and 0.8377 nm, respectively. The radius of $Cr^{3+}$ and $Co^{2+}$ ions is 0.0615 nm and 0.0745 nm, respectively, a small amount $Co^{2+}$ ions of the octahedron ferrites are replaced by $Cr^{3+}$ ions, which results in the reduce of lattice constant of the $Co_{0.7}Cu_{0.1}Zn_{0.2}Fe_2O_4$ ferrites. Apart from this, there is a small diffraction peak appearing around $2\theta = 33.30°$ in Fig. 1 b, which is the diffraction peaks of $Cr_2O_3$. The authors not only see the $Co_{0.7}Cr_{0.1}Zn_{0.2}Fe_2O_4$, but also see that there are two broad diffraction peaks at about $2\theta$ = 19.8° and 25.6°, which are the characteristic peaks of PANI in Fig. 1 a. Because the chain of PANI contains a large number of rigid benzene ring, the PANI has not only the properties of periodic parallel and perpendicular but a certain degree of crystallinity. So the PANI has two relatively X-ray broad diffraction patterns [13, 14]. All these reveal the formation of the PANI-$Co_{0.7}Cr_{0.1}Zn_{0.2}Fe_2O_4$ ferrite composite. Fig. 2 a and b show the FT-IR spectra of the PANI-$Co_{0.7}Cr_{0.1}Zn_{0.2}Fe_2O_4$ composites and $Co_{0.7}Cr_{0.1}Zn_{0.2}Fe_2O_4$ ferrites, respectively.

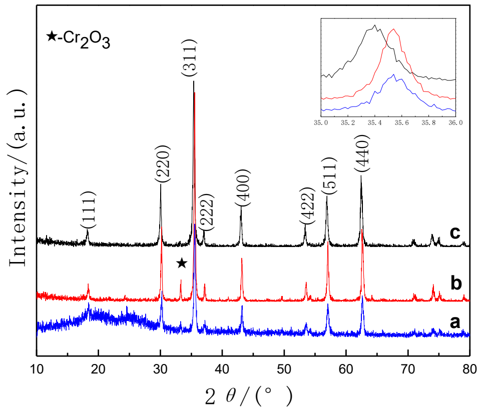


**Fig. 1.** X-ray diffraction patterns of the samples: a – PANI-$Co_{0.7}Cr_{0.1}Zn_{0.2}Fe_2O_4$, b – $Co_{0.7}Cr_{0.1}Zn_{0.2}Fe_2O_4$; c – $Co_{0.8}Zn_{0.2}Fe_2O_4$

In the Fig. 2 b, the trough at the 572cm$^{-1}$ is attributed to the stretching vibration of the ferrite metal-oxygen ion (M-O), and belongs to the vibration absorption peak of $V_1$, which is the characteristic of ferrites in tetrahedral sites [15]. In Fig. 2 a, the characteristic troughs of PANI locate at the 1566 cm$^{-1}$, 1491 cm$^{-1}$, 1387 cm$^{-1}$, 1344 cm$^{-1}$, 1237 cm$^{-1}$, and 1124 cm$^{-1}$, respectively. The troughs at 1566 cm$^{-1}$ and 1491cm$^{-1}$ are attributed to stretching vibration of C = C of the benzenoid rings and quionoid, the trough at 1344 cm$^{-1}$ and 1387 cm$^{-1}$ are attributed to stretching vibration of N-H, the trough at 1237 cm$^{-1}$ is assigned to stretching vibration of C-N group on the macromolecular chains of PANI, and the peak at 1116 cm$^{-1}$ is the vibration of N=Q=N(Q) represents a quinone ring) [16, 17]. At the same time the major troughs of ferrites also appear in Fig. 2 a, but the intensity of the peaks has been weakened, which indicates that the PANI-$Co_{0.7}Cr_{0.1}Zn_{0.2}Fe_2O_4$ has been obtained.

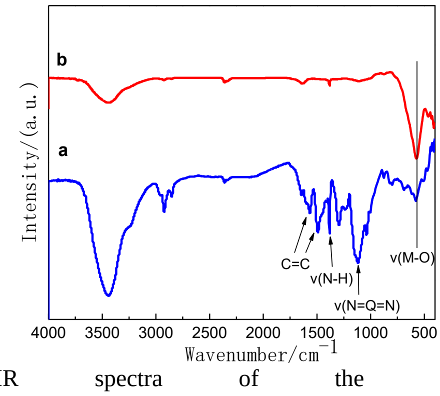


**Fig. 2.** FT-IR spectra of the samples: a – PANI-$Co_{0.7}Cr_{0.1}Zn_{0.2}Fe_2O_4$; b – $Co_{0.7}Cr_{0.1}Zn_{0.2}Fe_2O_4$

Fig. 3 a and b show the hysteresis loops for the PANI-$Co_{0.7}Cr_{0.1}Zn_{0.2}Fe_2O_4$ composites and $Co_{0.7}Cr_{0.1}Zn_{0.2}Fe_2O_4$ ferrites, respectively. The magnetic parameters of the saturation magnetization (*Ms*), residual magnetization (*Mr*) and coercivity (*Hc*) of the two materials determined by the hysteresis loops are given in Table 1. From Table. 1, the values of *Ms* and *Mr* of PANI-$Co_{0.7}Cr_{0.1}Zn_{0.2}Fe_2O_4$ composites were 8.80 emu/g and 3.14 emu/g, respectively, which are much smaller than those of the $Co_{0.7}Cr_{0.1}Zn_{0.2}Fe_2O_4$ ferrites. At the same time, the value of *Hc* of PANI-$Co_{0.7}Cr_{0.1}Zn_{0.2}Fe_2O_4$ composites is 37.22 KA/m, which is similar to that of

$Co_{0.7}Cr_{0.1}Zn_{0.2}Fe_2O_4$ ferrites. The $Co_{0.7}Cr_{0.1}Zn_{0.2}Fe_2O_4$ ferrite is magnetic material, and the PANI is dielectric material, so the magnetic properties of the PANI-$Co_{0.7}Cr_{0.1}Zn_{0.2}Fe_2O_4$ composites mainly depend on the ferrites, so the magnetic parameters of the PANI-$Co_{0.7}Cr_{0.1}Zn_{0.2}Fe_2O_4$ composites are less than that of the $Co_{0.7}Cr_{0.1}Zn_{0.2}Fe_2O_4$ ferrites.

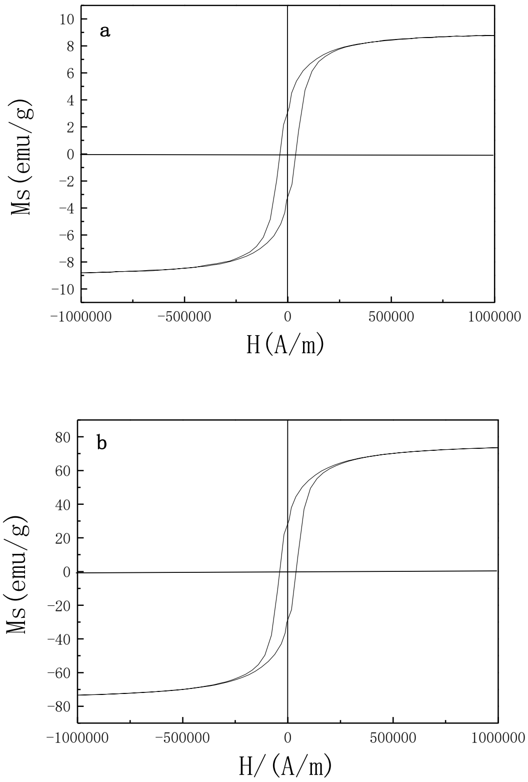


**Fig. 3.** The Hysteresis loops of the samples: a – PANI-$Co_{0.7}Cr_{0.1}Zn_{0.2}Fe_2O_4$; b – $Co_{0.7}Cr_{0.1}Zn_{0.2}Fe_2O_4$

**Table. 1.** The magnetic parameters of the PANI-$Co_{0.7}Cr_{0.1}Zn_{0.2}Fe_2O_4$ and $Co_{0.7}Cr_{0.1}Zn_{0.2}Fe_2O_4$

| Magnetic parameters | PANI-$Co_{0.7}Cr_{0.1}Zn_{0.2}Fe_2O_4$ | $Co_{0.7}Cr_{0.1}Zn_{0.2}Fe_2O_4$ |
|---|---|---|
| *Ms*/(emu/g) | 8.80 | 73.84 |
| *Mr*/(emu/g) | 3.14 | 28.43 |
| *Hc*/(kA/m) | 37.22 | 38.91 |

Fig. 4 a and b show the dielectric loss ($\tan\varepsilon=\varepsilon''/\varepsilon'$) for the PANI-$Co_{0.7}Cr_{0.1}Zn_{0.2}Fe_2O_4$ composites and $Co_{0.7}Cr_{0.1}Zn_{0.2}Fe_2O_4$ ferrites in the frequency of 5 ~ 20GHZ, respectively. And they show $\tan\varepsilon$ for the PANI-$Co_{0.7}Cr_{0.1}Zn_{0.2}Fe_2O_4$ composites ranging from 0.67 ~ 0.80, that for the $Co_{0.7}Cr_{0.1}Zn_{0.2}Fe_2O_4$ ranging from 0.10 ~ 0.36. The value of $\tan\varepsilon$ for the PANI-$Co_{0.7}Cr_{0.1}Zn_{0.2}Fe_2O_4$ composites is greater than that of the $Co_{0.7}Cr_{0.1}Zn_{0.2}Fe_2O_4$ ferrites. The value of dielectric loss for materials mainly depends on the polarization of electric dipole [18]. According to the theory of complex permittivity, because of the influence of the electric field, when the metal surface is irradiated by electromagnetic wave, there are two forms of current will be produced in the material, which is the conduction current and displacement current. The interaction between the surface of ferrite and PANI chain [19] results in a conduction current and a displacement current, and causes the dielectric relaxation effect and space charge polarization effect, which leads to the dielectric loss of PANI-$Co_{0.7}Cr_{0.1}Zn_{0.2}Fe_2O_4$ greater than that of the single ferrite.

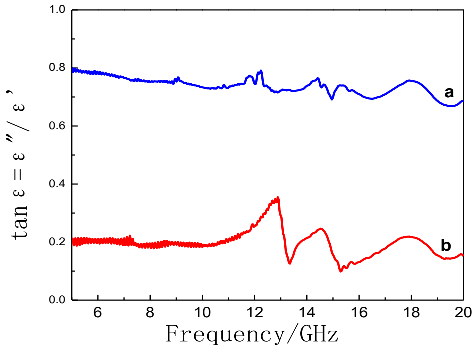


**Fig. 4.** The dielectric loss curves of the samples: a – PANI-$Co_{0.7}Cr_{0.1}Zn_{0.2}Fe_2O_4$; b – $Co_{0.7}Cr_{0.1}Zn_{0.2}Fe_2O_4$

Fig. 5 show the reflection loss curves for the PANI-$Co_{0.7}Cr_{0.1}Zn_{0.2}Fe_2O_4$ composites and $Co_{0.7}Cr_{0.1}Zn_{0.2}Fe_2O_4$ ferrites, respectively. In the frequency range of 5 ~ 20GHz, there are two great reflection losses appearing at 14.1 GHz and 17.9 GHz for the PANI-$Co_{0.7}Cr_{0.1}Zn_{0.2}Fe_2O_4$ composite, and the value is -13.17 dB and -15.36 dB, respectively. And the value of reflection loss for $Co_{0.7}Cr_{0.1}Zn_{0.2}Fe_2O_4$ is -8.2 dB and -8.1 dB. The reflection loss of the PANI-$Co_{0.7}Cr_{0.1}Zn_{0.2}Fe_2O_4$ composites is obviously higher than that of the $Co_{0.7}Cr_{0.1}Zn_{0.2}Fe_2O_4$ ferrites. Therefore, the composite material shows good microwave absorbing properties. The magnetic loss of ferrite mainly depends on the influence on the complex permeability by domain wall resonance, and the spin resonance plays a major role. The microwave absorption properties of PANI can be explained by the strong polarization and interfacial relaxation effects which were caused by the localized bound charges. The microwave absorbing property of the PANI-$Co_{0.7}Cr_{0.1}Zn_{0.2}Fe_2O_4$ nanocomposites is a combination of the dielectric loss originated from the dipolar polarization and relaxation effects of PANI, the spin-rotation resonance of $Co_{0.7}Cr_{0.1}Zn_{0.2}Fe_2O_4$, the interfacial polarization and relaxation effects between PANI and ferrites [20], which leads to the increase of the reflection loss.

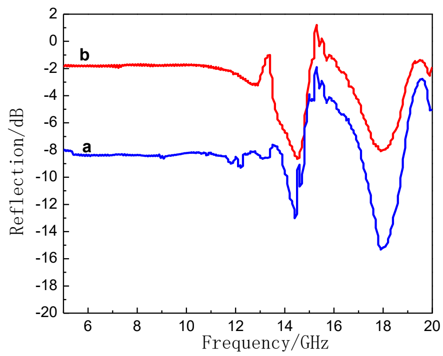


**Fig. 5.** The reflection loss curves of the samples: a – PANI-$Co_{0.7}Cr_{0.1}Zn_{0.2}Fe_2O_4$; b – $Co_{0.7}Cr_{0.1}Zn_{0.2}Fe_2O_4$

## 4. CONCLUSIONS

The values of Ms, Mr and Hc for PANI-$Co_{0.7}Cr_{0.1}Zn_{0.2}Fe_2O_4$ composites are 8.80 emu/g, 3.14 emu/g and 37.22 kA/m, which are less than that of the $Co_{0.7}Cr_{0.1}Zn_{0.2}Fe_2O_4$ ferrite.

In the measuring frequency range of 5.0 ~ 20.0 GHz, two reflection loss maximum value of PANI-$Co_{0.7}Cr_{0.1}Zn_{0.2}Fe_2O_4$ composites appear at 14.1GHz and 17.9 GHz with -13.17 dB and -15.36 dB, respectively, which is obviously higher than those of the $Co_{0.7}Cr_{0.1}Zn_{0.2}Fe_2O_4$ ferrites.

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