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Review

# Electromagnetic and microwave-absorbing properties of Co-based amorphous wire and $Ce_2Fe_{17}N_{3-\delta}$ composite





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#### ABSTRACT

A radar microwave absorbing material was designed by integrating Co-based amorphous microwires and Ce<sub>2</sub>Fe<sub>17</sub>N<sub>3-δ</sub> composites. The complex permittivity and permeability of the Co-based amorphous microwires gridding was measured by Vector Network Analyzer (VNA), which display magnetic resonance and dielectric resonance near 10 GHz. The microwave absorption behavior of the Co-based amorphous microwires gridding tested by VNA shows that absorbing bandwidth below –10 dB was broadened to 3.6 GHz in X-band as integrated with Ce<sub>2</sub>Fe<sub>17</sub>N<sub>3-δ</sub> composites. The mechanism of efficient microwave absorption in X-band were also discussed, which incorporated the microwave absorption characters of electromagnetic resonance and  $\lambda/4$  wavelength matching absorption.

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#### 1. Introduction

Contraposing the advances in radar-related research and electromagnetic radiation pollution induced by microwave devices and apparatus, microwave absorbing materials (MAMs) have a wide range of applications in military radar stealth, prevention of electromagnetic radiation and electromagnetic compatibility (EMC) [1–4]. Consequently, the study of high-performance MAMs with thin, light, and broad absorbing bandwidths (BWs) attracts a lot of attentions in gigahertz (GHz) frequency ranges [5,6]. Among those

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MAMs, the microwave absorptive character of amorphous ferromagnetic microwires is of considerable interest owing to their magnetic anisotropy, a variety of magnetoelectric resonance effects and excellent high-frequency soft magnetic properties [7–10]. For example, P. Marín et al. [11] prepared the absorbent sheet made up of a dielectric material containing amorphous magnetic Fe<sub>89</sub>Si<sub>3</sub>BC<sub>3</sub>Mn<sub>4</sub> microwires, the bandwidth with reflection loss (RL) below -10 dB was about 3 GHz, and the absorption mechanism was related not only with ferromagnetic resonance but also with electric resonance.

In addition, orthogonal structure based on ferromagnetic microwires was theoretically predicted that can obtain metamaterial characteristics [12]. Hence, it would be much more useful to deploy magnetic microwires to construct such microwire composites with orthogonal arrangement [13]. The absorbers with orthogonal periodic geometric features in a layer absorb energy through resonating property determined by the shape and size of the pattern as well as the distance between the unit cell of the pattern, the feature of which is called frequency selective surface (FSS) [14]. Moreover, the amorphous magnetic microwires FSSs patterned on a thin film were employed to broaden bandwidth, and mainly focused on design and evaluation of shape and size of the periodic pattern [15].

In order to enhance microwave absorption, the multi-layered radar absorbing structures (MRAs) with a dielectric material onto the FSS have been studied. Lee et al. [16] prepared the radar absorber by integrating inductive frequency-selective carbon fiber fabric composite and multiwalled carbon nanotube-loaded glass fabrics, and the composite showed two separated absorption peaks with RL less than -10 dB covered whole X and Ku bands with thickness of 3.63 mm.

In this work, a matching pattern absorber Ce<sub>2</sub>Fe<sub>17</sub>N<sub>3- $\delta$ </sub> powders/ Silicone composites placed onto amorphous magnetic microwires of orthogonal gridding structure was proposed, and the structure exhibits multiple absorption peaks in the microwave regime. The bandwidth of the composite with RL < -10 dB was 3.6 GHz in Xband, which indicates that this structure would enable a promising candidate for electromagnetic microwave absorber.

#### 2. Experimental

The Co<sub>63</sub>Fe<sub>4</sub>B<sub>22.4</sub>Si<sub>5.6</sub>Nb<sub>5</sub> amorphous microwires is prepared by melt extraction method as described in Ref. [17]. The high purity cerium and iron react by induced-melting in an argon atmosphere following by nitridation to produce Ce<sub>2</sub>Fe<sub>17</sub>N<sub>3- $\delta$ </sub> powders. Then Ce<sub>2</sub>Fe<sub>17</sub>N<sub>3- $\delta$ </sub>/silicone composite with volume fraction of 28% was prepared to form thin film.

The Co-based amorphous microwire of orthogonal structure was prepared, and fixed on polyethylene terephthalate (PET) film



Fig. 1. Schematic cross-section of each layer illustration of the MRAs.

with glue, as schematically shown as layer (2) and (3) of Fig. 1. And its' thickness is 0.24 mm. The RASs in this study consist of  $Ce_2Fe_{17}N_{3-\delta}$ /silicone composite onto the front and the back of Cobased microwires gridding of orthorhombic structure stuck on a layer of polyethylene terephthalate (PET) film, as shown in Fig. 1. Finally, the back material denoted as perfectly electrical conductor which is normally made of highly conductive metallic layers, prohibiting microwave transmission to penetrate through the RASs.

Static magnetic property of Co-based amorphous microwire was measured by Vibrating Sample Magnetometer (VSM, 7304, USA) under an applied magnetic field of 20 kOe. And morphology of Cobased amorphous microwire was observed by scanning electron microscopy imaging (SEM, Hitachi S-4800). Microwave behavior of the microwire composites was tested by waveguide method. The test set is mainly composed of a Vector Network Analyzer (VNA, N5225 A, Agilent Technologies), a standard WR90 waveguide (HD-100WAL75, Xian Hengda Microwave Tech. Co., China). The waveguide has an inner cross section of 22.86  $\times$  10.16 mm, which fully covered by the sample to prevent leakage of EM waves. For a typical measurement, the response of the waveguide is first calibrated in the range of 8–12 GHz by a thick aluminum block. Then, the sample is mounted on the waveguide port, with the  $Ce_2Fe_{17}N_{3-\delta}$  composite side facing to the waveguide. The measured S11 is transferred to a computer connected to the VNA. The complex permeability and complex permittivity was extracted via the obtained S-parameters by an implanted computer program: Reflection/Transmission Epsilon Fast Model [18].

#### 3. Results and discussion

Fig. 2 shows magnetization curves of Co-based amorphous microwire at room-temperature. The magnetic hysteresis loops of microwires exhibit good soft-magnetic properties, the saturated magnetization  $B_s$  is 0.54 T. The inset figure in Fig. 2 present cross section image of the microwire with diameter about 60  $\mu$ m.

The complex permittivity ( $\varepsilon = \varepsilon' - i\varepsilon''$ ) and complex permeability ( $\mu = \mu' - i\mu''$ ) of the Co-based amorphous microwires gridding of different wire-space were measured by vector network analyzer in the 8~12 GHz frequency ranges. Three layers of amorphous mesh composites with the same specification were superimposed with thickness of 0.72 mm and used for EM performance test, as waveguide test has the requirement of thickness [19]. As shown in Fig. 3,



Fig. 2. Magnetic hysteresis loops of the  $Co_{63}Fe_4B_{22,4}Si_{5,6}Nb_5$  microwires, Inset shows the SEM image of microwire.



Fig. 3. The frequency dependence of complex permittivity and complex permeability of the Co-based amorphous microwires gridding composite with different microwires spacing, d = 2.0, 2.4, 2.8, 3.2 and 4.0 mm, respectively.

the frequency dependence of the complex permittivity and complex permeability both displays obvious resonance. The permeability curve of amorphous microwires gridding of different microwire space was almost accordant. So, wire-space has little impact on magnetic performance of the composite. The real part of permeability  $\mu'$  maintains constant about 2 with increasing frequency and then appears a peak at about 10 GHz. The shape anisotropy characteristic and fibers alignments induced high permeability [5]. The imaginary part of permeability  $\mu''$  exists a peak at 10 GHz, which implies that the magnetic resonance occurred and is responsible for high frequency EM wave absorption [20]. As can be observed, the permittivity curve has same trend with the different microwire spacing. With the space decreasing from 4.0 mm to 2.4 mm, the dielectric resonance peak get stronger and moves to high frequency. Denser amorphous microwires result in larger internal boundary layer capacitance and conductive network, and cause high complex permittivity [21]. Take microwires gridding of space d = 0.24 for discussion, the real part of permittivity  $\varepsilon'$  increase from 24 to 42 and decreases to 14 in the range of 9.5-10.0 GHz, then goes up again. The imaginary part of permittivity  $\varepsilon''$  exhibits one clear dielectric resonance near 9.8 GHz. The resonance mechanism has been reported to originate in the interfacial polarization due to accumulation of charges near the interfaces between the insulating and conducting phases. This interfacial polarization is referred to Maxwell-Wagner-Sillars (MWS) polarization [22]. According to the experimental results, the Co-based amorphous microwire gridding present strong electromagnetic resonance in X-band and tunable permittivity properties.

Then, microwave absorbing performance of specimens was tested with the space of wire-wire (d) of 2.0 mm, 2.4 mm, 2.8 mm, 3.2 mm, 4.0 mm respectively. Fig. 4 shows a reflection loss of -6 dB occurs at about 10.5 GHz, when d = 2.4 mm. Meanwhile, dielectric resonance and magnetic resonance both occur near 10 GHz, which enhance the microwave absorbing performance. With the decrease of d, reflection of Co-based microwires gridding plays a dominant role, which induce low RL. On the contrary, while d is increases to more than 2.4 mm, microwave absorbing performance of Co-based microwires turns to weaker due to the decrease of the long-range dipolar interactions between the microwires [23]. Although the



**Fig. 4.** RL curves for Co-based amorphous microwire gridding composites with different microwires spacing, d = 2.0, 2.4, 2.8, 3.2 and 4.0 mm, respectively.

dynamic interactions are not strictly limited by the spacing, the interaction peaks weaken when the wire spacing is increased due to the influence of the lack of strong electromagnetic excitation between the microwires and the electromagnetic performance of wires [24]. Particularly, in the X-band frequency range, a wire spacing of 2.4 mm led to a higher absorption. Thus, it should be noted that the electromagnetic (EM) properties of such microwire composites are dependent not only on the intrinsic properties of microwires, such as the ferromagnetic resonance, but also on mesostructure such as wire-spacing.

However, the Co-based amorphous microwire gridding provide a sharp but narrow absorption peak. In order to enhance absorption in X-band, the Ce<sub>2</sub>Fe<sub>17</sub>N<sub>3- $\delta$ </sub> composite was proposed in this experience. As we know, high absorption can be achieved by minimized reflectance and transmittance of the structure. So the incident layer material impedance must well matched to free space impedance for getting low reflectance. Fig. 5 shows the RL curves for Ce<sub>2</sub>Fe<sub>17</sub>N<sub>3- $\delta$ </sub> composite of different thickness. Ce<sub>2</sub>Fe<sub>17</sub>N<sub>3- $\delta$ </sub> composite has the



Fig. 5. RL curves for  $Ce_2Fe_{17}N_{3-\delta}$  composites with various thickness.

best microwave absorption bandwidth below -10 dB reaches 3 GHz in X-band with thickness of 1.44 mm. Its' microwave absorption mechanism can be explained by impedance matching condition and interface-reflection model [25]. So, the introduction of Ce<sub>2</sub>Fe<sub>17</sub>N<sub>3- $\delta$ </sub> composite makes the structure has a good match for air.

The Ce<sub>2</sub>Fe<sub>17</sub>N<sub>3- $\delta$ </sub> composite with volume fraction of 28 vol% and thickness of 1.36 mm was used here as a matching layer. The complex permittivity and complex permeability of Ce<sub>2</sub>Fe<sub>17</sub>N<sub>3- $\delta$ </sub> composite almost maintain constant as presented in Fig. 6(a). Contrasted with the flat EM curve obtained from Ce<sub>2</sub>Fe<sub>17</sub>N<sub>3- $\delta$ </sub> composite, the imaginary part of permeability  $\mu''$  and permittivity  $\epsilon''$  of MRAs remarkably increase (Fig. 6(b)). And the real part of permeability  $\mu'$  increase at 8–10.8 GHz, and pulled down arousing from frequency selective character of wires. The real part of permittivity  $\epsilon'$  also increases and appears a peak at 9.2 GHz. Therefore, it can be concluded that the orthogonal Co-based microwire composite enhances the complex permittivity and permeability of the Ce<sub>2</sub>Fe<sub>17</sub>N<sub>3- $\delta$ </sub> composite.

In order to enhance microwave absorption,  $Ce_2Fe_{17}N_{3-\delta}$  composite and Co-based microwires gridding composites was integrated as MRAs. Fig. 7(a) shows the RL curves for MRAs at different thickness of  $Ce_2Fe_{17}N_{3-\delta}$  composites. Among them, MRAs has wider absorption cover 3.6 GHz and stronger absorption of -20.9 dB at thickness of 1.36 mm. As reported in recent year, there are a lot



Fig. 7. RL curves for MRAs (a) with various matching thickness; (b) Frequency dependence of matching thickness  $t_m$  simulated by using  $\lambda/4$  interface-reflection model.



Fig. 6. Frequency dependencies of complex permittivity and complex permeability of Ce<sub>2</sub>Fe<sub>17</sub>N<sub>3-δ</sub> composition (a) and MRAs(b).

works about absorber structures. Somak Bhattacharyya et al. prepared a triple band polarization-independent metamaterial absorber [26] and a dual layer polarization-insensitive metamaterial absorber [27] exhibit mult-band absorption, with peak absorptivities over 90%, but their thickness is not ideal as a metamaterial. Mayank Agarwal et al. [28] proposed a closed-ring resonator-based metamaterial absorber with thickness of 1.6 mm. and the absorber shows above -10 dB RL from 8.50 to 11.13 GHz. So, the structure in this experience has better absorbing performance. There are two obvious absorption peaks in RL curve, which assumed that the Ce<sub>2</sub>Fe<sub>17</sub>N<sub>3-ô</sub> composite and Co-based microwires gridding composites both devote to microwave absorption. With the thickness of  $Ce_2Fe_{17}N_{3-\delta}$  composite increasing, the absorption peaks move to the lower frequency. As a dielectric material onto the Co-based microwire gridding is attached to result in resonance peak shift to a lower frequency, which is called the dielectric loading effect [29]. Hence, it was observed in Fig. 7(b) that very thin dielectric layer red shifted the resonating frequency of the first peak to the neighborhood of  $f_{res}/\sqrt{\varepsilon_r}$ , where  $\varepsilon_r$  is a dielectric constant [30]. Consequently, the first peak arouses from Co-based amorphous microwires, as well as shall be the responsibility of electromagnetic resonance.

And as shown in Fig. 7(b), the second RL peak appears at a certain frequency for a given absorber thickness. The matching thickness  $t_m$  simulated by using  $\lambda/4$  interface-reflection model matches perfectly with the matching pattern absorber thickness, which has been studied by Li et al. [31]. According to interface reflection model, phase matching phenomenon will occur when the thickness of the microwave absorbing materials  $t_m$  and the frequency of incident EM f<sub>m</sub> satisfy equation as follows [32],

$$t_m = \frac{n}{4} \frac{c}{f_m \sqrt{|\mu_r \varepsilon_r|}}, \quad n = 1, 3, 5 \tag{1}$$

where  $t_m$  and  $f_m$  are matching thickness and matching frequency, respectively. Plots of second peak related to matching thickness corresponds to have a good match with calculated value  $t_m$ , which indicates that the second peak is caused by  $\lambda/4$  wavelength matching absorption. And the deviation aroused from the optical path difference of EM wave, makes the decrease desire of matching pattern absorber thickness.

Among the three curves, while  $t_m = 1.36$  mm, the MRAs is suitable for X-band microwave absorption, with absorb bandwidth of 3.6 GHz, and the lowest RL of -20.86 dB. In light of the above, Cobased microwire gridding patterned on a thin film have been employed to broaden bandwidth, and synergistic with matching pattern absorber, which induced a wide microwave absorb in X-band.

#### 4. Conclusions

The synergistic effect between  $Ce_2Fe_{17}N_{3-\delta}$ /silicone composite and Co-based microwires result in the enhancement of electromagnetic character and microwave absorption. According to the observed experimental results, Co-based microwires gridding shows excellent electromagnetic character, and  $Ce_2Fe_{17}N_{3-\delta}$ /silicone composite exists good microwave absorption. The reflection loss of a microwave absorber consisted of  $Ce_2Fe_{17}N_{3-\delta}$ /silicone and Co-based microwires was wider and shows the absorb characters of the two materials. As a result, this structure supposed an idea for materials design in EM wave absorption.

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