Electromagnetic Wave Absorption Properties of Cobalt-Containing Polymer-Derived SiCN Ceramics

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Abstract. PDCs-SiCN(Co) were composed by polymer-derived technique from polysilazane and Co nano-powder. XRD, SEM and hysteresis loops patterns show that there are magnetic Co nanoparticles and newly formed magnetic Co₃C particles in the compound, which makes the composite possess the required impedance matching due to the combination of dielectric and magnetic properties, thus having certain microwave absorption properties. Electromagnetic wave absorbing performance of the PDCs-SiCN (Co) ceramics was discussed when the frequency range is 2-18GHz. PDCs-SiCN (Co) ceramics mixed with 5wt% of Co nanoparticles provides best electromagnetic wave absorption performance compared with other Co contained samples when the thickness is 2mm. The minimum R_L is -8.3 dB at 16.7 GHz, this means that 85% of the incident electromagnetic waves enter the material at this frequency and are further depleted. The minimum reflection loss (R_L) of all samples was decreased and its corresponding frequency was also changed with the increase of sample thickness. Bandwidth of microwave absorptivity exceeding 88% (RL< -9 dB) of sample which doped with 5wt% cobalt reaches 0.35 GHz when the thickness is 7mm. The results indicated that the prepared materials have certain microwave absorption properties and have potential application prospects in the field of microwave absorbing materials.

Introduction

Nowadays, with the burgeoned of wireless electronic device, electromagnetic wave pollution and information safety problems are exploding, for instance the harm to human health and military security, microwave absorbing materials were able to take in incident electromagnetic wave power, then converted it to heat power or other types of power, so, it is pressing necessary to fabricate microwave absorbing materials that possessed excellent absorbing ability, light weight and wide impactful frequency bandwidth[1-7].

In recent years, PDCs-SiCN ceramics comprising of Si-C-N amorphous matrix and free-carbon nano-domains have gained extensive attention due to the admirable functional performances, such as light weight, excellent thermal stability and electrical property, thus, polymer-derived SiCN ceramics (PDCs-SiCN ceramics) can be used as dielectric loss material in microwave absorbing filed [8-12].
However, its conductive and permittivity are too high to satisfy the requirement of impedance match [12,13]. Accordingly, adding magnetic loss particles into PDCs-SiCN ceramics should be a feasible method to solve it, which can tune the EM parameters and improve microwave absorption property [14, 15]. Cobalt as ferromagnetic materials, due to its large snoek limit and high permeability, probably a highly valuable candidate for oncoming generation of microwave absorption materials [16-21]. But, independent magnetic loss materials reveal poor microwave absorption property [22]. Moreover, the density of cobalt metal is so high that it cannot meet the requirement of lightweight material under the condition of single use, which greatly limits its value. Consequently, some studies have been combined Co with dielectric loss material to obtain excellent microwave absorption properties [23, 24]. Zhang et al. [23] studies the properties of Co/C nanofiber materials and found the high product of permittivity and permeability and their appropriate matching contributed to improve microwave absorption. Xing et al. [24] studied the microwave absorption of Hexagonal Co/C composites and demonstrated that Hexagonal Co/C composites have gigantic potential in absorption materials.

In this article, Cobalt-containing SiCN ceramics (PDCs-SiCN (Co) ceramics) were fabricated by a polymer-derived technique. The phase structure, magnetic properties of samples prepared with various Co mixing amount were discussed, the electromagnetic parameters and reflection loss of each sample were discussed among frequency range of 2-18GHz, the effect of thickness on the microwave absorption properties of samples prepared at measured frequencies was also discussed.

Experimental

PDCs-SiCN (Co) Ceramics discussed in this article have been successfully constructed by the polymer-derived method. The polysilazane (PSZ, Shanghai Haiyi Technology Trade Co., Ltd, China) was cross-linked 2h in the nitrogen atmosphere of 600°C, and the buff crosslinking polymer was obtained. The cobalt nano-powders (Co nano-powders, 50nm, Shanghai Macklin Biochemical Co., Ltd, China) were mixed into prepared French grey powders in a certain proportion (5 wt%, 10 wt%, 20 wt%, 30 wt%, 40 wt%) and adhesives (polysilazane here) were added to obtain the mixture. Grinding the resulting mixture. The uniform grinding mixture was pressed into a disc-shaped green compact under the condition of holding pressure for 2 minutes at 16 MPa. Then the Green body pyrolysis 2h in a nitrogen atmosphere of 1100°C to form ceramics.

The crystal structure of obtained sample was tested by X-ray diffraction (XRD, D/MAX-Ultima IV, Rigaku, Japan). The microstructure and chemical composition were investigated by scanning electron microscope (SEM, EVO18, Zeiss, German). The magnetic property was studied with a vibration sample magnetometer (VSM, JDAW-2000D, Yingpu, China). The complex permittivity and permeability were derived from the vector network analyzer (5244 A, Agilent-N, USA) in the frequency range of 2-18 GHz. In order to prepare samples used for electromagnetic parameters survey, a sample containing 71.4% of the obtained PDCs-SiCN (Co) ceramics was pressed into a coaxial ring with an external diameter of 7.0 mm, an internal diameter of 3.0 mm and a thickness of about 2.0 mm in which paraffin wax was play the part of adhesive.

Results and discussion

Phase compositions

The phase composition of the samples prepared with different amount of cobalt added and pure PDCs-SiCN were confirmed by XRD patterns, the peaks of fcc Co (JCPDS 15-0806) in Figure 1 at around 51.52° exhibited by PDCs-SiCN(Co) ceramics (10wt%, 20wt%, 30wt%, 40wt%) could be corresponding with (200) crystal planes. Diffraction peaks of magnetic compound [25] CoC (JCPDS 26-0450) appeared in all samples with cobalt addition, and the strength of the diffraction peaks enhanced with the augmented of cobalt content. The results prove that Co and C form a new compound in the PDCs-SiCN(Co) ceramics systems. When the content of Co was over 10%, a large number of peaks of Si3N4 (JCPDS 73-1210) were appeared in the materials. Si3N4 is an insulating phase, which has neither conductivity nor magnetism. Its appearance will reduce the dielectric properties and permeability of
materials, next, the absorbing properties of the material were further reduced. This is why the reflection loss of samples with cobalt content exceeded 10 wt% in the thickness range of 2-7mm is greater than -8 dB in subsequent analysis. At $\theta = 26.43^\circ$, the diffraction peak of carbon (JCPDS 08-0415) is occurred, and its intensity is enhanced with the increase in cobalt content, this suggests that not all C is fused in the Co$_3$C compound and it also proves an increase in the crystallization of carbon.

![Figure 1. XRD patterns of PDCs-SiCN (Co) ceramics with different amount of cobalt added.](image)

![Figure 2. Raman spectra of PDCs-SiCN (Co) ceramics with different amount of cobalt added.](image)

The existence state of carbon atoms also has a great influence on the electromagnetic parameters, which further affect the absorption properties [26, 27]. In order to further characterize the existence state of the carbon phase, the chemical component of PDCs-SiCN(Co) ceramics is further demonstrated by Raman. **Figure 2** shows the Raman spectra of samples prepared. The lattice defects and structural disorder of graphite are characterized by D-band which appeared at near 1350cm$^{-1}$, the
stretching vibration of C atom SP² hybridization in graphite lattice plane is characterized by G-band which appeared at near 1580cm⁻¹. This phenomenon demonstrating the existence of free carbon in PDCs-SiCN(Co) ceramics. The ratio of intensities of D-band to G-band (I_D/I_G) could be used to prove the degree of crystal structure disorder of carbon in PDCs-SiCN(Co) ceramics. The smaller the value of I_D/I_G, the greater the disorder degree of free carbon, the higher the crystallization degree of free carbon. As can be seen form Table 1, The position of D peak fluctuates between 1336 cm⁻¹ and 1363 cm⁻¹ and G peak fluctuates between 1564 cm⁻¹ and 1616 cm⁻¹, the figure of I_D/I_G has overall downward trend with the increasing of Co content that demonstrated the carbon disorder degree was increased. The reason may be that the addition of magnetic metal Co promotes the crystallization degree of carbon[28].

Table 1. Raman spectra parameters of PDCs-SiCN (Co) ceramics with different amount of cobalt added.

| Sample | I_D/I_G | ω_D(cm⁻¹) | FWHH_D(cm⁻¹) | ω_G(cm⁻¹) | FWHH_G(cm⁻¹) |
|--------|---------|------------|---------------|------------|---------------|
| SiCN   | 0.931   | 1401.774   | 353.447       | 1615.184   | 851.808       |
| 5wt%   | 1.842   | 1354.525   | 191.250       | 1564.046   | 128.074       |
| 10wt%  | 1.694   | 1355.526   | 209.430       | 1565.029   | 132.688       |
| 20wt%  | 1.294   | 1336.736   | 142.047       | 1565.145   | 109.891       |
| 30wt%  | 0.853   | 1362.669   | 177.033       | 1583.998   | 120.424       |
| 40wt%  | 1.025   | 1352.293   | 138.564       | 1568.161   | 105.622       |

Figure 3. SEM image (a), EDS spectra (b) and mapping(c) of PDC-SiCN(Co) ceramics with 10wt% of cobalt added
Microstructures
The morphology dimension and main elements of PDCs-SiCN (Co) ceramics with 10wt% content were investigated by The SEM and EDS. Combining with Figure 3(a), We can realize that the whole material consists of two parts: the matrix and the small and bright particles distributed on it. The matrix is gray, dense and amorphous. Uniting the EDS images Figure 3(b), it is known that the small particles distributed on the matrix are Co nanoparticles. It can be observed from the mapping Figure 3(c) that Co elements are uniformly distributed in the material. Since Polysilazane do not contain oxygen (The molecular structure of Polysilazane is shown in Figure 4), it can be seen from EDS images that the elements of the matrix are Si, C and N, the three elements were evenly distributed in the matrix. Oxygen may be introduced during crosslinking or pyrolysis.

![Figure 4. Molecular structure of polysilazane.](image)

Magnetic properties
The test results of magnetic properties of samples prepared with different cobalt content were shown in Figure 5. By comparison, the following conclusions can be drawn: (1) Each sample obtained shows a complete hysteresis loop, indicating that each sample has typical ferromagnetic characteristics at room temperature. (2) With the increase of Co content, the coercivity hardly changed but the saturation magnetization increases gradually, which indicates that good ferromagnetic properties of materials at room temperature can be attributed to the addition of Co nanoparticles. (3) Each sample exhibits relatively small coercivity, saturation magnetization and residual magnetization, indicating that each sample is a soft magnetic material. Soft magnetic materials are easy to get magnetism in the
magnetic field, and when the magnetic field withdraws, the magnetism is also easy to disappear, in this process, soft magnetic material itself receives thermal energy and emits thermal energy[29]. These characteristics makes samples prepared have the ability to convert electromagnetic energy into heat energy and then to dissipate. Thus, contributing further to electromagnetic wave attenuation of materials.

Figure 6. The real (a) and imaginary (b) parts of the complex permittivity of PDC-SiCN(Co) ceramics with Different amount of cobalt added.

Electromagnetic parameters

Relatively complex permittivity \( (\varepsilon' = \varepsilon' - j\varepsilon'') \) and relatively complex permeability \( (\mu' = \mu' - j\mu'') \) are meaningful data to characterize the properties of microwave absorbing materials. Real part of the relative complex permittivity \( (\varepsilon') \) and relatively complex permeability \( (\mu') \) characterizes the storage capacity of materials for electric and magnetic energy. Imaginary part of the relative complex permittivity \( (\varepsilon'') \) and relatively complex permeability \( (\mu'') \) characterizes material's loss ability to electric and magnetic energy. \( \varepsilon' \) and \( \varepsilon'' \) of samples prepared with various Co content and pure SiCN ceramics were measured at 2-18 GHz, as showed in Figure 6(a) and Figure 6(b). Figure 6(a), in the range of 2-8 GHz, there was no obvious fluctuation of \( \varepsilon' \) values of all samples. \( \varepsilon' \) values fluctuate in the range of 3 to 15 at 8-18GHz and all samples doped with cobalt near 17GHz have a resonance peak. The \( \varepsilon' \) of PDC-SiCN (Co) ceramic (5wt%) is smaller than other Co contained samples at 2-18 GHz and it is worth noting that a smaller \( \varepsilon' \) is beneficial[30]. while \( \varepsilon'' \) values fluctuate in the range of -6 to 4 at 8-18GHz, the resonance peaks appeared in all cobalt contained sample in the range of 7-12GHz and 16-18 GHz, each resonance peaks in the \( \varepsilon'' \) curve indicates a relaxation loss process. Both \( \varepsilon' \) and \( \varepsilon'' \) change with the difference of cobalt content., indicating that the dielectric performance can be improved by controlling Co content. For further comprehends the dielectric loss mechanism of materials, the \( \varepsilon' - \varepsilon'' \) image of samples with cobalt content of 10wt% was drawn. According to Debye's dielectric relaxation theory, links of \( \varepsilon' \) and \( \varepsilon'' \) can explain by the formula (1)[31].

\[
(\varepsilon' - \varepsilon_\infty)^2 + (\varepsilon'')^2 = (\varepsilon_\infty - \varepsilon_\infty)^2
\]

Where \( \varepsilon_\infty \) and \( \varepsilon_0 \) are the relative dielectric permeability and static permittivity, respectively. According to this formula, the \( \varepsilon' - \varepsilon'' \) image should show a Cole-Cole semicircle. From Figure 7 (a), we can see two distinct semicircles, which represent two forms of dielectric relaxation processes in composites [32],it also fits perfectly with the two resonance peaks on the \( \varepsilon'' \) image. Relaxation processes of composites may arise from the interfacial polarization between magnetic cobalt metal particles, magnetic Co3C particles and SiCN ceramics. In addition, defects in materials can produce polarization relaxation, thus contributing to microwave attenuation [32].
Figure 7. Cole-Cole semicircle curve and frequency dependence of $\mu''(\mu')^{-2}f^{-1}$ for PDC-SiCN(Co) ceramics with 10 wt% cobalt added.

Figure 8. The real (a) and imaginary (b) parts of the complex permeability of PDC-SiCN(Co) ceramics with different amounts of cobalt added.

Figure 8(a) and Figure 8(b) show the $\mu'$ and $\mu''$ of the complex permeability of samples prepared with various Co content and pure ceramics, respectively. We can see that the variation range of $\mu'$ value of all samples in 2-18 GHz is very small and there is almost no change in the range of 2-8 GHz, but there is a large fluctuation (0.7-1.3) at 16-18 GHz. The $\mu'$ value of samples with 30 wt% cobalt content in the range of 12-16 GHz is larger than that of the other five samples, and the maximum value is 1.3, which indicates that the samples have a strong magnetic energy storage capacity. $\mu''$ value of all materials prepared fluctuates between 0.1-0.5 within 2-18 GHz. Similar to $\mu'$, the fluctuation range of $\mu''$ was very small (< 0.1) in the range of 2-8 GHz. But when the frequency exceeds 16 GHz, the $\mu'$ of all samples fluctuate greatly. When content of Co is 30 wt%, the maximum value of $\mu''$ reaches 0.5, which shows excellent magnetic loss ability. Magnetic loss originates mainly from magnetic hysteresis, the eddy current loss, domain-wall resonance, natural resonance and exchange resonance. Magnetic hysteresis could be disregarded owing to the weak applied field [33]. Domain-wall resonance happens in a small frequency range (1-100 MHz) generally [31], so hysteresis loss and domain-wall resonance could be ignored when the frequency is between 2 - 18 GHz. The existence of eddy current loss can be expressed by value $\mu''(\mu')^{-2}f^{-1}$ [31,34], if eddy current loss exists, this value varies with frequency as a constant whose value is equal to $2\pi\sigma d^2\mu_0$ (Here $d$ is the thickness of the material, $\sigma$ is the electrical conductivity of the material) and is independent of the frequency.
conductivity, $\mu_0$ is the vacuum permeability). In order to figure out the magnetic loss mechanism of the prepared samples, we made the $\mu''(\mu')^{-2f^{-1}} - f$ curve. We can see clearly from Figure 8(b) that this value fluctuates with frequency and peaks appear at 16-18 GHz. This shows that magnetic loss of samples mainly comes from natural resonance loss.

Dielectric loss tangent ($\tan\delta_{\varepsilon}$) and the magnetic loss tangent ($\tan\delta_{\mu}$) are powerful data to characterize electromagnetic energy loss of electromagnetic wave absorbing materials. Using the $\varepsilon'$ and $\mu'$, $\varepsilon''$ and $\mu''$, According to the following formula

$$\tan\delta_{\varepsilon} = \frac{\varepsilon''}{\varepsilon'}$$

$$\tan\delta_{\mu} = \frac{\mu''}{\mu'}$$

$\tan\delta_{\varepsilon}$ and $\tan\delta_{\mu}$ of each sample could be calculated [35]. From Figure 9(a), It can be seen that the variation trend of $\tan\delta_{\varepsilon}$ is the same as that of $\varepsilon''$, all samples with cobalt contained exhibit extremely small fluctuations in the range of 2-8 GHz and show resonance peaks at 8-12 GHz and 16-18 GHz, respectively. For the sample with 30 wt% cobalt content, When the frequency is at 16-18 GHz, the $\tan\delta_{\varepsilon}$ values fluctuate widely (-0.4 ~ 0), and there are resonance peaks. When the sample with cobalt content is 40 wt%, the maximum value of $\tan\delta_{\varepsilon}$ can reach 0.35, exhibited certain dielectric loss properties. The relationship between $\tan\delta_{\varepsilon}$ and frequency in all samples are shown in Figure 9(b).

Similar to $\tan\delta_{\varepsilon}$, $\tan\delta_{\mu}$ and $\mu''$ have the same trend of change. $\tan\delta_{\mu}$ has a small fluctuation range (-0.1 ~ 0.1) in 2 ~ 14 GHz but a large fluctuation range (-0.1 ~ 0.45) in 16 ~ 18 GHz. The $\tan\delta_{\mu}$ values of samples with cobalt content of 30 wt% and 40 wt% reached 0.45 and 0.15 at around 17 GHz, respectively. Proving excellent magnetic loss ability of the prepared material. By comparing $\tan\delta_{\varepsilon}$ and $\tan\delta_{\mu}$ of all samples, We can see that the $\tan\delta_{\mu}$ of samples with different Co content is close to $\tan\delta_{\varepsilon}$ in most frequency ranges. Which indicates that the electromagnetic wave loss of samples prepared in this paper can give the credit to the interaction of electric and magnetic energy losses.

![Figure 9](image-url)

Figure 9. The dielectric (a) and magnetic loss tangents (b) of PDC-SiCN(Co) ceramics with Different amount of cobalt add.

**Microwave absorption properties**

Reflection loss ($R_L$) could be used to characterize microwave absorption performance of absorbers and could be calculated by using $\varepsilon_r$ and $\mu_r$ and transmission line theory [36].
The reflection loss ($R_L$) is given by:

$$R_L (dB) = 20 \log \left( \frac{Z_{in} - 1}{Z_{in} + 1} \right)$$

(4)

$$Z_{in} = \frac{\mu_r \text{tanh} \left( \int \frac{2\pi}{c} f d\varepsilon_r \mu_r \right)}{\varepsilon_r}$$

(5)

Here, $\mu_r$ is relative complex permeability, $\varepsilon_r$ is relative complex permittivity, $f$ is the microwave frequency, $c$ is light velocity, $d$ is thickness of the sample.

**Figure 10** indicates the relationship between $R_L$ and frequency of PDCs-SiCN (Co) ceramics with different Co content in 2-18GHz. From the graph, we can realize that $R_L$ changed significantly with the change of cobalt content. It is shown that the absorption property of the material can be controlled by controlling the cobalt content. PDCs-SiCN (Co) Ceramics mixed with 5wt% of Co nanoparticles provides best electromagnetic wave absorption performance compared with other Co contained samples. The optimal $R_L$ is -8.3 dB at around 17 GHz with a thickness of 2mm. Bandwidth with microwave absorptivity greater than 84% (RL<-8dB) is 0.1GHz. It is demonstrated that the material has certain microwave absorption performance. Combined with the analysis results of electromagnetic parameters, we can see that this performance is the result of the interaction of dielectric loss and magnetic loss.

Using transmission line theory, **Figure 11** reveals the Reflection Loss ($R_L$) of different thickness PDCs-SiCN (Co) ceramics. With the increase of sample thickness, it shows us that the minimum reflection loss ($R_L$) of all samples was decreased and its corresponding frequency was also changed. For example, for a sample with 5 wt% cobalt, when its thickness is 2 mm, its minimum reflection loss is -8.3 dB at 16.7 GHz, when its thickness is 3 mm, its minimum reflection loss is -9.1 dB at 17 GHz. When the thickness is 7 mm, the sample has the best absorbing property, and the minimum reflection loss can reach -9.4 dB at 13.8GHz, Bandwidth of microwave absorptivity exceeding 88% ($R_L$< -9 dB) reaches 0.35 GHz, showing it certain microwave absorption performance. In addition to the sample with 5 wt% cobalt content, the samples with cobalt content of 10 wt% are the same, When the thickness is 2 mm, the minimum RL is – 7.9 dB at 16.7 GHz. When the thickness is 5 mm, the minimum $R_L$ is -8.7 dB at 17 GHz and bandwidth of microwave absorptivity exceeding 84% ($R_L$< -8 dB) reaches 5.5 GHz. It has a minimum $R_L$ value of -8.8 dB at 17 GHz and bandwidth of microwave absorptivity exceeding 84% ($R_L$< -8 dB) reaches 5.8 GHz when the thickness is 7mm, which also...
shows relatively good absorption performance. However, the minimum reflective loss of other samples (cobalt content 20 wt%, 30 wt% and 40 wt%) is higher than -8 dB, which shows poor microwave absorption performance. Presumably because they contain a large amount of Si$_3$N$_4$ which is neither conductive nor magnetic, which reduces the conductivity and permeability of materials and further lead to the impedance matching of materials cannot meet the required requirements. It can be seen that the $R_L$ values of materials are closely related to their thickness. This means that the excellent absorbing properties of materials can be achieved by controlling thickness.

Figure 11. The reflection loss ($R_L$) of PDCs-SiCN (Co) ceramics with different amount of cobalt added at different thicknesses
Conclusions
PDCs-SiCN (Co) Ceramics have been successfully constructed by the polymer-derived method. SEM picture proved that the whole material consists of two parts: SiCN ceramics matrix and the small and bright Co particles distributed on it. Raman image and Gauss fitting data showed that the prepared samples contain free carbon, and the degree of crystallization of free carbon enhanced with the increased of cobalt content. XRD, SEM and hysteresis loops patterns showed together that there are magnetic Co nanoparticles and newly formed magnetic Co$_3$C particles in the compound, which makes the composite possess the required impedance matching due to the combination of dielectric and magnetic properties, thus having certain microwave absorption properties. Electromagnetic wave absorbing performance of the PDCs-SiCN (Co) Ceramics was discussed when the frequency range is 2 ~ 18 GHz. PDCs-SiCN (Co) Ceramics mixed with 5wt% of Co nanoparticles provides best electromagnetic wave Absorption performance compared with other Co contained samples when the thickness is 2mm. The optimal R$_L$ is -8.3 dB at around 17 GHz, this means that 85% of the incident electromagnetic waves enter the material at this frequency and are further depleted. The minimum reflection loss (R$_r$) of all samples was decreased and its corresponding frequency was also changed with the increase of sample thickness. Bandwidth of microwave absorptivity exceeding 88% (R$_L$ < -9 dB) of sample which doped with5wt% cobalt reaches 0.35 GHz when the thickness is 7mm. This shows that the material has certain microwave absorption performance and can be used as a candidate for microwave absorbing materials.

Acknowledgment
This research was financially supported by the National Science Foundation of China (No.51572154).

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