Mechanical and microwave absorption properties of Ti-filled SiC_f/SiC composites via precursor infiltration and pyrolysis

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Abstract

SiC fiber reinforced SiC ceramic matrix composite is a kind of promising high-temperature structure absorbing integrated material because of its good temperature resistance and high-temperature stability. However, the mechanical properties of the composites are poor and it is difficult to adjust the electromagnetic parameters. In this study, Ti powders were used as active fillers to improve the mechanical and microwave absorption properties of SiC_f/SiC composites fabricated by polymer infiltration and pyrolysis (PIP) process. The compositions of SiC matrix derived from polycarbosilane (PCS) precursor modified by various content Ti powders were investigated by XRD and Raman spectroscopy. With the incorporation of Ti powders, free carbon decomposed from PCS was consumed due to the formation of TiC phase. The effect of Ti fillers on the mechanical and dielectric properties of SiC/SiC composites was also investigated. The flexural strength of SiC_f/SiC composite was gradually improved with increasing Ti content owing to the improved density and TiC particle strengthening. Besides, consumption of free carbon in SiC matrix may impair the conductive network of free carbon, leading to decreased conductivity and the imaginary part of complex permittivity of the composite. Therefore, an optimum microwave absorption performance of SiC_f/SiC composite with an RL_m value of - 37 dB at 10.51 GHz and an effective absorption bandwidth of 3.28 GHz was obtained at the thickness of 2.5 mm and the Ti content of 5 wt%. The Ti-filled SiC_f/SiC composite with superior microwave absorption property is a promising candidate in the high-temperature absorbing field.

1 Introduction

In recent years, with the rapidly development of information technology and high-frequency electronic technology, electromagnetic radiation pollution has done harmful to people's health. To solve radiation pollution problems, electromagnetic wave absorbing materials have attracted extensive attention [1-5]. Particularly, lots of interests have been focused on studying the high-temperature microwave absorbing materials due to applications in extremely harsh environments [6, 7]. Compared with carbon and

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ferromagnetic absorbing materials, SiC_{f}/SiC composites have been recognized as promising structural and electromagnetic absorbing materials due to their superior properties such as high strength, oxidation and corrosion resistance [8–10].

For fiber reinforced ceramic matrix composites, in order to ensure the integrity and reinforcing effect of fibers, the preparation method of ordinary composites cannot be used. Nowadays, SiC_f/SiC composites are commonly fabricated by precursor infiltration and pyrolysis (PIP) method and chemical vapor infiltration (CVI) method. Many researchers have shown that the dielectric constant of the composites prepared by CVI method was very large and difficult to adjust, and, so, CVI method is not suitable to obtain SiC_f/ SiC composites with excellent microwave absorbing properties [11]. PIP is another mainstream method for fabricating SiC_f/SiC composites because of its low preparation temperature, convenient preparation process, low consumption and easy control of matrix composition. As well known, polycarbosilane (PCS) is used as the precursor of SiC matrix for PIP process and the components of SiC matrix derived from PCS are amorphous SiC and free carbon [12]. The



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SiC_f/SiC composites fabricated by PIP method show a poor microwave absorbing property owing to the high conductivity caused by free carbon generated during the pyrolysis of PCS, leading to impedance mismatch and a strong reflection of microwave on the composite surface [13]. Besides, it is considered that free carbon can form a conductive network and the destruction of network leads to the decrease of conductivity for composites [14].

Generally speaking, heat treatment at 600 °C is the most convenient and simple process for carbon removal. The disadvantage of this way to remove carbon is obvious. It will reduce the composite density, provide oxygen channel during service at high-temperature and hence the fiber degradation. As a result, the mechanical properties of SiC_f/ SiC composites decrease and the load-bearing capacity of composites fails. Another way to reduce the free carbon in the matrix is thermal cross-linking of precursors. Mu et al. [15] prepared SiC_f/SiC-Al₂O₃ composites modified by thermal cross-linking procedure and the result indicated that composites possessed an excellent microwave absorption property owing to the consumption of carbon and SiC by the incorporated oxygen. However, the thermal cross-linking procedure can damage the fibers to a certain extent and lead to a decline of the mechanical properties of composites. The attention should be paid to the preparation of protective coatings in the research of thermal cross-linking carbon removal process to weaken the influence of thermal cross-linking on fibers and enhance the mechanical properties of composites.

Introducing active fillers into precursors is another way to remove carbon. Active fillers, such as Ti, Cr, Mo, Al, CrSi₂, etc., can react with free carbon during the pyrolysis process of precursors and hence adjust the dielectric property of matrix [16–21]. For active fillers Cr and CrSi₂, the reaction temperature with free carbon is above 1200 °C while the reaction temperature of Mo and free carbon needs to be as high as 1500 °C. Therefore, considering that SiCf/ SiC composite is prepared by PIP method in this work, the selected active fillers should be able to consume free carbon at low temperature (~1000 °C). Liu et al. [22] prepared nickel-containing polymer-derived SiCN ceramics (PDC-SiCN) by polymer-derived method and it was shown that SiCN with magnetic property can be synthesized by mixing nickel nanopowders with liquid-phase polysilazane. It can be concluded that the dielectric and magnetic properties of PDC-SiCN ceramics can be tuned by varying the amount of nickel adding. In our previous work [23], we have prepared aluminum-containing SiC_f/SiC composites by mixing aluminum powders with PCS successfully. Results showed that free carbon generated during the pyrolysis process of PCS can be removed, that is accompanied with an improvement in mechanical and microwave absorption performance of SiC_f/SiC composites. It should not be neglected that the reaction product of free carbon and aluminum is aluminum carbide, which reacts rapidly with water in hot and humid environment, thus limiting the application of composite materials in hot and humid environment. Titanium powders have been investigated as active fillers during the pyrolysis process of polymers in the last 20 years [24, 25]. But no study has been focused on the effect of titanium on the dielectric property of polymer-derived ceramics. Besides, the reactions among active fillers and pyrolysis products of precursors will lead to a certain volume expansion, which alleviates the volume shrinkage during pyrolysis and helps to improve the mechanical properties of materials. Therefore, active filler-controlled pyrolysis (AFCP) is an efficient carbon removal method without degradation to the mechanical properties of SiC_f/SiC composites.

In the present work, we used PCS as precursor, SiC fibers as reinforcement and Ti powders as active fillers, which can optimize the dielectric properties of PDC-SiC matrix, to fabricate SiC_{f}/SiC composites with various Ti content by PIP method. The composition evolution of SiC matrix at different Ti contents during pyrolysis process was characterized. Besides, the density, porosity, flexural strength and cross-section morphology of composites were investigated to analyze the effect of Ti content on the mechanical property. Moreover, the dielectric property and calculated reflection loss values were investigated to evaluate the microwave absorption properties of composites with various content Ti fillers.

2 Experimental procedure

2.1 Materials

The volume fraction of 2D SLF SiC fabrics, which were used as reinforcement of composites, is 40% and the polycarbosilane (PCS) power, supplied by Sailifei Ceramics Fiber Co. Ltd., Su Zhou, China is taken as the precursor of SiC matrix. General properties of the SLF SiC fibers and PCS can be found elsewhere [26]. The active Ti fillers were provided by Shanghai Xiangtian Nanomaterial Co., Ltd, Shanghai, China. As shown in Fig. 1, the average particle size of Ti powders is about 1 μ m, as tested by scanning electron microscopy (SEM; Tescan Vega3 SBH, Brno, Czech).

2.2 Fabrication and characterization of SiCf/SiC composites with various content Ti powder

In present study, we used precursor infiltration and pyrolysis (PIP) method to fabricate $SiC_{f'}SiC$ composites with Ti filler. The detail scheme preparation process of $SiC_{f'}SiC$ composites with Ti fillers is shown in Fig. 2 and specific process parameters can be found in our previous work [23]. The first step is the decarbonization of SiC fibers to remove the



Fig. 1 The morphology of purchased Ti powder

sizing agent. Second, the SiC fibers were infiltrated in PCS/ xylene solution and pyrolyzed after drying and hot mold pressing. Subsequently, the composite preforms were infiltrated in different mass ratio PCS/Al/xylene slurries listed in Table 1 and pyrolyzed in vacuum sintering at 1000 °C for 2 h to adjust the dielectric properties of SiC matrix. As the final stage, the composites were densified through further infiltration and pyrolysis using PCS/xylene solution as precursor until the mass gain of composites is no more than 1%. For convenience, the SiC_f/SiC composites were designated as S0, S2, S5, S10 respectively, according to the content of Ti filler in second PIP process.

X-ray diffraction (X'Pert PRO MPD, PANalytical. Almelo, The Netherlands), using Cu K α (λ = 1.54 Å)

Table 1 Composition of mixed precursor solution

Number	Xylene/g	PCS/g	Ti/g	Ti ratio
1	50	50	0	0
2	50	50	1.02	2%
3	50	50	2.63	5%
4	50	50	5.56	10%

radiation, was used to characterize the phase compositions of pyrolyzed products for PCS with various Ti content and the graphitization degree of carbon was tested by Raman spectroscopy (LabRAM HR800). The fracture surface morphologies of SiC_f/SiC composites with various content Ti fillers were examined by SEM. The open porosity and density of composites were measured by Archimedes method and the flexural strength was measured by a threepoint bending test with a specimen size of $40^l \text{ mm} \times 4^w$ $mm \times 3^t$ mm according to the general guidelines of ASTM standard C 1341. The complex permittivity was measured by the waveguide method using a vector network analyzer (E8362B) in the frequency range of 8.2-12.4 GHz (X band) and the DC electrical conductivity was obtained by a resistance tester. The detail measurement was narrated in our previous work [23].



Fig. 2 The preparation rote of 2D SiCf/SiC composites with Ti filler



Fig.3 XRD patterns of PCS with various Ti content pyrolyzed at 1000 $^\circ C$ for 2 h

3 Results and discussion

3.1 The influence of titanium powder content on the characteristics of PCS/Ti derived SiC matrix

The XRD patterns of polycarbosilane (PCS) with different Ti contents pyrolyzed at 1000 °C for 2 h are shown in Fig. 3. It is apparent that only a broad peak can be detected for SiC matrix derived from pure PCS, indicating that the pyrolyzed product is mainly amorphous SiC. When Ti powders are doped in the precursor, crystallized TiC and β -SiC can be clearly characterized. As the Ti content increased to 10 wt%, new phases Ti₅Si₃ and Ti₃SiC₂ were detected due to excess Ti powder.

Accordingly, Ti powder as active fillers can react with gaseous carbonaceous species (e.g., CH_4) released from the decomposing PCS at ~400 °C [19, 24, 27]. As the pyrolysis temperature increases to 800 °C, Ti powder will react with the free carbon generated from PCS to form titanium carbide. In addition, the free Si, which will be generated during the pyrolysis when active Ti fillers were introduced in the silicon-based polymers [24], has not been detected due to its low content. The excess Ti powder may react with amorphous silicon carbide to form Ti₅Si₃ and react with free Si and TiC to form Ti₃SiC₂ according to the XRD results. The possible reactions of PCS with active filler Ti based on the XRD patterns can be express as follows:

$$\mathrm{Ti} + \mathrm{CH}_4 \to \mathrm{Ti}\mathrm{C} + 2\mathrm{H}_2 \tag{1}$$

$$Ti + C \rightarrow TiC$$
 (2)

$$8\text{Ti} + 3\text{SiC} \rightarrow 3\text{TiC} + \text{Ti}_5\text{Si}_3$$
 (3)

$$2\text{TiC} + \text{Ti} + \text{Si} \rightarrow \text{Ti}_3\text{SiC}_2 \tag{4}$$

Thus, it can be concluded that the conductive network formed by free carbon in the matrix can be destroyed by a small amount of Ti powder. Besides, the Ti fillers addition in present work is aimed to decrease the amount of free carbon in the pyrolyzed products and obtain the PDC-SiC matrix with a lower conductivity. The conductivities of Ti₅Si₃ (~1.5 × 10⁶ S/m) and Ti₃SiC₂ (~4.5 × 10⁶ S/m) are much higher than that of SiC and excessive Ti filler introduced will certainly generate amount of Ti₅Si₃ and Ti₃SiC₂. Therefore, the content of Ti fillers should be controlled no more than 10 wt%.

Raman spectroscopy is an important tool for characterizing the state of free carbon and the recorded Raman spectrum are shown in Fig. 4. Two obvious peaks at around 1350 and 1600 cm⁻¹, namely, D and G peaks of free carbon, represent the degree of amorphous and graphited states. It is obvious that the intensity of D and G peaks decreased sharply with the Ti filler incorporation, indicating that a large amount of free carbon was consumed by adding Ti filler in the precursor.

From the XRD and Raman results, the Ti filler introduction can result in a destruction of conductive network formed by free carbon and reach the purpose of the initial experimental design to adjust the dielectric property of SiC matrix. The determination of optimal Ti filler content needs for further experimental exploration about mechanical and dielectric properties.



Fig. 4 Raman spectrum of pure PCS and PCS with 5 wt% Ti powder pyrolyzed at 1000 $^\circ\!C$ for 2 h

Table 2 Properties of fabricated $\mathrm{SiC}_{\mathrm{f}}/\mathrm{SiC}$ composites with various Ti content

Sample	Porosity (%)	Flexural strength (MPa)	Displacement (mm)	Electrical conductivity (S/m)
S0	16.2	157±9	0.14 ± 0.03	4.9
S2	12.7	285 ± 11	0.27 ± 0.02	3.4
S5	10.6	316±8	0.32 ± 0.03	2.3
S10	10.2	330 ± 10	0.35 ± 0.04	7.2



Fig. 5 Stress-displacement curves of SiCf/SiC composites with different Ti content

3.2 The mechanical properties of SiC_f/SiC composites with various Ti content

The properties of SiC_f/SiC composites with different Ti contents are listed in Table 2. It can be observed that the open porosity decreases and the density slightly increases with the Ti content increase that is accompanied with a gradually enhancement of flexural strength of SiC_f/SiC composites. The relationship between flexural strength and displacement of SiC_f/SiC composites with different Ti content, tested by three-point bending method, is shown in Fig. 5. It is apparent that the fracture mode of SiC_f/SiC composites changes from brittle fracture mode to pseudoplastic fracture mode with the incorporation of Ti filler. When the applied force reaches the maximum value, the curvilinear trend drops off gradually for composite S2, S5 and S10, indicating that the introduction of Ti filler is conducive to crack deflection. The cross-section morphologies of SiC_f/SiC composites with different Ti contents are shown in Fig. 6. It is obvious that macropores in the intro-bundle and the pore size decrease with the Ti content increase, resulting in more compact composites. As shown in Fig. 6c, d, when the Ti filler content increased to 5%,

there were almost no macro-holes in the matrix, only some small micro-holes existed between the monofilaments, which proved that the Ti filler introduction was beneficial to the densification of SiC_f/SiC composites. Thus, the improved flexural strength is possible due to the reaction between Ti filler and gaseous hydrocarbons to compensate for partial volume shrinkage and the strong combination of titanium reaction products with SiC matrix [19, 24].

3.3 The dielectric properties of SiC_f/SiC composites with various Ti content

In Fig. 7, the complex permittivity and tangent loss of SiCf/ SiC composites with different Ti contents are presented. It is observed that the ε' values almost show a rising trend, as the Ti content increases from 0 to 10 wt%. While the ε'' values decrease firstly and increase dramatically, as the Ti content increases to 10 wt%. Besides, both ε' and ε'' show a declining trend, as the frequency increases in the whole X band, indicating the frequency response effect of the complex permittivity for the SiC_f/SiC composites. The tangent loss, as seen in Fig. 7c, follows the same trend with ε'' . The average value of tangent loss for the SiC_f/SiC composites without filler is about 0.9. After introducing 2 wt% Ti, the average value of tan δ decreases sharply to 0.55 and the ε'' decreases from 7.8 to 4.9. When the Ti content is 5 wt%, ε'' drops to 3.8 resulting in a valley value of tan δ . As the Ti content increases to 10 wt%, the average value of tan δ reaches 0.9 and ε'' increases to 10.2.

The trend of ε' values can be explained by the Lichtencker logarithmic equation [28]:

$$\operatorname{Ln} \varepsilon' = \sum_{i} V_{i} \times \operatorname{Ln} \varepsilon'_{i} \tag{5}$$

where V_i is the volume fraction of each component of the composites, ε'_i is the ε' vaule of each component of the composites. The components of SiC_f/SiC composites with Ti filler are the SLF fibers, PDC-SiC matrix (amorphous SiC and free carbon), reaction products of Ti and PCS (e.g., TiC, Ti_5Si_3 , Ti_3SiC_2) and the pores. In the present work, the SiC fiber volume fraction for composites is 40 vol%. Thus, the change of ε' is mainly caused by PDC-SiC matrix, reaction products of Ti and PCS and the pores. As the pores decrease slightly with the Ti content increase and all the reaction products of Ti have a higher ε' than SiC matrix, the ε' of SiC_f/SiC composites with Ti fillers increases with the increasing Ti content. Besides, plenty of interfaces are formed among the mixed matrix due to the incorporation of Ti. Free electrons will be gathered in the interfaces to form space-charge layer when an external electric field is applied with and it will help to enhance the ε' value [1–3, 14, 22].



Fig. 6 Cross-section morphologies of SiCf/SiC composites with various Ti content a without filler; b 2 wt%; c 5 wt%; d 10 wt%

The ε'' represents the material ability to dissipate electromagnetic wave energy, which can be calculated by the following equation according to Debye theory [29]:

$$\varepsilon'' = \frac{\sigma}{\omega\varepsilon_0} = \frac{\sigma}{2\pi\varepsilon_0 f} \tag{6}$$

where σ is the electrical conductivity, ε_0 is the dielectric constant in vacuum and f is the frequency of electromagnetic wave. It can be asserted that ε'' is positively correlated with σ . The electric conductivity shows a downward trend firstly and then rises rapidly with the Ti filler content increasing, as displayed in Table 2, giving rise to the variation of ε'' values.

When the Ti content is less than 5 wt%, the reaction products of Ti is mainly composed of TiC according to the XRD results, indicating the main reaction of Ti is (1) and (2). The consumption of free carbon in the second PIP process will destroy the network of carbon in the matrix, which decreases the conductivity of composites. Besides, it is unavoidable that TiC will be formed when free carbon is consumed and the formation of high conductivity TiC did not lead to a sharp increase in conductivity of composites, which can be explained by the percolation theory [30-32]. The DC conductivity of composites can be also described as the following equation:

$$\sigma_{\rm dc} = \sigma_p \left(V - V_c \right)^{\lambda} \tag{7}$$

where σ_p and V is the conductivity and volume fraction of filler in the composites, respectively, V_C is the critical volume fraction or percolation threshold and x is the exponent related to the dimension of composites. With the content of Ti fillers increasing, the grain size of TiC increases gradually as it can be concluded from XRD patterns. When the Ti filler content is not above than 5 wt%, both the grain size and content of TiC particles are small, resulting in a long distance between TiC particles and making it difficult to form an effective conductivity network. However, Ti₅Si₃ and Ti₃SiC₂ begin to appear and TiC content increases, as the Ti content increases to 10 wt%. These conductivity particles get closer between each other, leading to a significant improvement of conductivity as well as the imaginary part of complex permittivity.



Fig. 7 Complex permittivity and tangent loss of SiC_f/SiC composites with different Ti content at the frequency of 8.2–12.4 GHz

3.4 The microwave absorption properties of SiC_f/SiC composites with various Ti content

The reflection loss (RL) is used to characterize the microwave absorption properties of materials, which can be calculated by the complex permittivity and permeability according to the following equations:

$$RL(dB) = 20 \log \left| \frac{Z_{in} - Z_0}{Z_{in} + Z_0} \right|$$
(8)

$$Z_0 = \sqrt{\frac{\mu_0}{\varepsilon_0}} \tag{9}$$

$$Z_{in} = Z_0 \sqrt{\frac{\mu_r}{\epsilon_r}} \tanh\left[j\left(\frac{2\pi ft}{c}\right)\sqrt{\mu_r \epsilon_r}\right]$$
(10)

where Z_{in} and Z_0 represent the input impedance of the materials and free space, respectively, μ_r , ε_r and t are the measured relative permeability, permittivity and thickness of materials, respectively, f is microwave frequency and c is the velocity of light in the vacuum. For nonmagnetic SiC_f/SiC composites, RL value is mainly relative to complex permittivity ($\varepsilon = \varepsilon' - j\varepsilon''$) and material thickness t. The calculated RL values of SiC_f/SiC composites with various Ti content at 2.5 mm thickness are exhibited in Fig. 8a. It can be concluded that RL value increases firstly and then decreases with the increasing Ti content. The SiC_f/SiC composites with 5 wt% Ti filler shows the optimal microwave absorption property whose RL_m reaches to - 37 dB at 10.51 GHz and bandwidth below - 10 dB is



Fig.8 a Calculated frequency-dependence reflection loss (RL) values of SiC_f/SiC composites with various Ti content at a thickness of 2.5 mm; b The RL values of SiC_f/SiC composites with 5 wt% Ti fillers at various thicknesses

3.28 GHz. The RL values of SiC_f/SiC composites with 5 wt% Ti filler at various thicknesses are shown in Fig. 8b. It is observed that absorption bandwidth can be tailored by changing the thickness and the peak of RL values shifted to a lower frequency with the increasing thickness of composites, which can be explained by quarter-wavelength cancellation model [33]. Thus, considering the peak value of RL and bandwidth below – 10 dB, the optimum thickness of SiC_f/SiC composites with 5 wt% Ti fillers is 2.5 mm.

As well known, an excellent absorption material should not only possess high absorbing efficiency, but also possess good impedance matching [34]. The attenuation coefficient, which is used to evaluate the attenuation ability of microwave absorption materials, can be calculated as follows [35]:

$$\alpha = \frac{\sqrt{2\pi f}}{c} \sqrt{\mu'' \varepsilon'' - \mu' \varepsilon' \sqrt{\left(\mu'^2 + \mu''^2\right) \left(\varepsilon'^2 + \varepsilon''^2\right)}} \quad (11)$$

The attenuation coefficient of SiC_f/SiC composites with various Ti content is shown in Fig. 9a. The attenuation coefficient shows the same trend with imaginary part of complex permittivity, as well as conductivity, indicating that composites with higher conductivity have better attenuation ability of EM wave. However, excessive conductivity is not conducive to impedance matching and will causes serious reflection of incident EM wave at the interface between air and material.



Fig. 9 a Attenuation constants b input impedances of SiC_f/SiC composites with various Ti contents

The relation of input impedance and frequency for all samples with a thickness of 2.5 mm is presented in Fig. 9b. According to Eq. (10), the absolute value of RL increases when the input impedance Z_{in} is closer to free space impedance Z_0 of 377 Ω . The input impedance increased firstly and decreased sharply when the Ti content increases to 10 wt%, which is contrary to the trend of attenuation coefficient. In other word, although the introduction of excessive titanium powder will improve the attenuation ability of composite, it will reduce its impedance matching ability. According to the RL value results, the superior microwave absorption property for SiC_f/SiC composites with 5 wt% Ti filler is manly ascribed to the good impedance matching because sample S5 possesses the lowest attenuation coefficient.

4 Conclusions

In this work, considering that the tangent loss of SiC_f/SiC composites is 0.9, adding Ti powders as active filler is an efficient way to decrease the free carbon generated from the pyrolyzed PCS and hence decrease the tangent loss of the SiC matrix. By adjusting the content of Ti powders, the SiC_f/ SiC composite with 5 wt% Ti filler possesses an optimum microwave absorption performance with an RL_m value of - 37 dB at 10.51 GHz and an effective absorption bandwidth of 3.28 GHz at the thickness of 2.5 mm. It is mainly attributed to the good impedance matching by consuming free carbon and destroying its conductive network. Besides, the flexural strength increases from 157 to 330 MPa with the increasing Ti content, indicating the SiC_f/SiC composites with Ti fillers can be used as a structural material. Further study should be focused on the high-temperature absorbing property of SiC_f/SiC composites with Ti fillers.

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