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Electromagnetic wave absorption and mechanical properties of SiC nanowire/low-melting-point glass composites sintered at 580°C in air

Ranran Shi[1)], *Wei Lin*[2)], *Zheng Liu*[3)], *Junna Xu*[4)], *Jianlei Kuang*[1),✉], *Wenxiu Liu*[1)], *Qi Wang*[1),✉], *and Wenbin Cao*[1),✉]

1) Department of Inorganic Nonmetallic Materials, School of Materials Science and Engineering, University of Science and Technology Beijing, Beijing 100083, China
2) National Demonstration Center for Materials Education, Institute for Advanced Materials and Technology, University of Science and Technology Beijing, Beijing 100083, China
3) Beijing Institute of Aeronautical Materials, Aero Engine Corporation of China, Beijing 100095, China
4) School of Materials and Environmental Engineering, Hunan University of Humanities, Science and Technology, Loudi 417000, China
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Abstract: SiC nanowires are excellent high-temperature electromagnetic wave (EMW) absorbing materials. However, their polymer matrix composites are difficult to work at temperatures above 300°C, while their ceramic matrix composites must be prepared above 1000°C in an inert atmosphere. Thus, for addressing the abovementioned problems, SiC/low-melting-point glass composites were well designed and prepared at 580°C in an air atmosphere. Based on the X-ray diffraction results, SiC nanowires were not oxidized during air atmosphere sintering because of the low sintering temperature. Additionally, SiC nanowires were uniformly distributed in the glass matrix material. The composites exhibited good mechanical and EMW absorption properties. As the filling ratio of SiC nanowires increased from 5wt% to 20wt%, the Vickers hardness and flexural strength of the composite reached HV 564 and 213 MPa, which were improved by 27.7% and 72.8%, respectively, compared with the low-melting-point glass. Meanwhile, the dielectric loss and EMW absorption ability of SiC nanowires at 8.2–12.4 GHz were also gradually improved. The dielectric loss ability of low-melting-point glass was close to 0. However, when the filling ratio of SiC nanowires was 20wt%, the composite showed a minimum reflection loss (RL) of −20.2 dB and an effective absorption (RL ≤ −10 dB) bandwidth of 2.3 GHz at an absorber layer thickness of 2.3 mm. The synergistic effect of polarization loss and conductivity loss in SiC nanowires was responsible for this improvement.

Keywords: SiC nanowires; glass composite; flexural strength; dielectric properties; microwave absorption

1. Introduction

With the comprehensive application of wireless communication technology, the harm of electromagnetic waves (EMW) has also received considerable attention. Correspondingly, high-performance EMW-absorbing materials have been extensively studied in recent years [1–2]. Among several EMW-absorbing materials, SiC materials exhibit great application potential because of their moderate electric resistance, high-temperature resistance, and chemical inertness [3]. Moreover, one-dimensional (1D) SiC can build a three-dimensional conductivity loss network while enhancing multiple reflection and scattering effects, thereby achieving excellent EMW absorption performance [4–9]. However, SiC/polymer matrix composites are difficult to work above 300°C, which limits the application of SiC-based EMW-absorbing materials. Therefore, a series of SiC/ceramic matrix EMW-absorbing composites was prepared on the basis of different forms of 1D SiC, such as fiber, whisker, and nanowire [10–21]. However, such ceramic matrix composites are usually prepared by high-temperature sintering at 1000–1600°C, and inert gas is used as a protective atmosphere to avoid SiC oxidation. Huge energy consumption and complex processes and equipment are necessary. Thus, SiC/inorganic matrix composites with lower sintering temperatures and moderate high-temperature resistance must be developed.

Compared with ceramic matrix composites, glass matrix composites are easier to sinter at low temperatures. Li *et al.* [22] prepared a dense SiC–Al_2O_3–glass composite coating at 900°C using a potassium silicate matrix material. Zhang *et al.* [23] fabricated an Al_2O_3/SiC nanowires/glass composite at 900°C by using the Bi–B–Si–Zn–Al glass. Doo *et al.* [24] prepared a SiC whisker-reinforced ceramic tape at 850°C by using calcium aluminoborosilicate glass as the matrix material. In addition, the sintering temperature of the re-

✉ Corresponding authors: Jianlei Kuang E-mail: jlkuang@ustb.edu.cn; Qi Wang E-mail: wangqi15@ustb.edu.cn; Wenbin Cao E-mail: wbcao@ustb.edu.cn

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duced graphene oxide/glass EMW absorption composite was reduced to 700°C in an argon atmosphere [25–26]. However, in our previous study, the initial oxidation temperature of SiC nanowires (SiCnw) was approximately 650°C [27]. Therefore, a low-melting-point glass with a sintering temperature lower than 600°C was selected as an inorganic matrix material in this work. Such a low sintering temperature can be used to prepare SiCnw/glass composites in air without a complex inert atmosphere or vacuum furnace. Meanwhile, it can also effectively reduce the oxidation of SiC to avoid the decline in the EMW absorption performance. In general, the mechanical properties of glass matrix materials are weaker than those of ceramic matrix materials. However, as a typical 1D material, SiC nanowires can also effectively enhance the mechanical properties of glass matrix composites [23–24,28–29]. Therefore, SiC nanowires/low-melting-point glass composites were sintered at 580°C in an air atmosphere in this work, which is significantly lower than the reported sintering temperature of SiC nanowires/inorganic matrix composites. Moreover, the effects of the filling ratio of SiC nanowires on the dielectric parameters, EMW absorption performance, and mechanical properties of the composites were also investigated.

2. Experimental

2.1. Materials

Low-melting-point glass powders (YFX-1273) with a softening temperature of 580°C were purchased from Fuzhou Invention Photoelectrical Technology Co., Ltd. Their chemical composition is shown in Table 1. SiC nanowires (SiC content ≥ 99wt%) were prepared by microwave heating and concentrated by using a gravity method [30]. The glass powders and SiC nanowires were ball milled in a polyethylene milling jar with agate balls for 30 min, and ethanol was used as the liquid medium. The weight content of SiC nanowires in the raw materials was set at 0, 5%, 10%, or 20%, which were labeled as GS0, GS5, GS10, and GS20, respectively. The mixed slurry was dried at 110°C in a vacuum drying oven. The powder mixture was pressed into a rectangular green body under 50 MPa and then sintered at 580°C for 30 min in an air atmosphere. Afterward, the sintered samples were ground to 22.86 mm × 10.16 mm × 2 mm (length × width × height) for dielectric parameter measurement by using the waveguide method.

Table 1. Chemical composition of glass powders wt%

ZnO	B_2O_3	SiO_2	K_2O	ZrO_2	Fe_2O_3	Al_2O_3	CuO	Y_2O_3	NiO
41.00	33.20	10.96	13.26	0.23	0.05	1.24	0.03	0.02	0.01

2.2. Characterization

The chemical composition was determined by Inductively Coupled-Plasma Optical Emission Spectrometry (ICP-OES, VARIAN 715-ES, USA). The composites were analyzed by using an X-ray diffractometer with Cu K_α radiation (XRD, Bruker D8 Advance, Germany). Field-emission scanning electron microscopy (FESEM, ZEISS Ultra 55, Oberkochen, Germany) equipped with an energy dispersive spectroscopy (Oxford Instruments X-Max, Oxford, UK) was used to characterize the micro-morphology of composites. The hardness of the composites was determined by using a micro-indenter equipped with a diamond Vickers indenter. Flexural strength measurements were performed on bar specimens (3 mm × 4 mm × 36 mm) using a three-point bend fixture with a span of 30 mm. Dielectric permittivity at 8.2–12.4 GHz was measured by using a Keysight PNA-L N5232A vector network analyzer (Palo Alto, Canada) with the waveguide method.

3. Results and discussion

Fig. 1 shows the phase composition of low-melting-point glass and SiCnw/glass composites with different SiC nanowire filling ratios. The low-melting-point glass is primarily composed of an amorphous phase, with a small amount of ZnO and SiO_2 crystal phases. Based on the XRD patterns of the composites, sharp diffraction peaks correspond to the (111), (200), (220), (311), and (222) crystal planes and stacking faults (SF) of 3C-SiC (cubic crystalline). With the increase in the SiC nanowire filling ratio, the diffraction peak intensity of SiC is significantly enhanced relative to the amorphous diffraction peak. Therefore, SiC nanowires are not oxidized when sintered at 580°C.

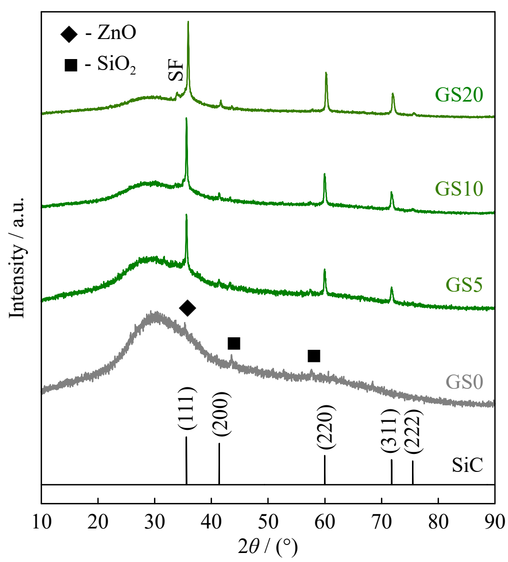

Fig. 1. XRD patterns of the SiCnw/glass composites sintered at 580°C.

Fig. 2(a) shows the micro-morphology of the powder mixture of low-melting-point glass powders and SiC nanowires. The particle size of low-melting-point glass powders is 1–5 μm, and the diameter and length of SiC nanowires are 50–200 nm and tens of microns, respectively. Fig. 2(b)–(d)

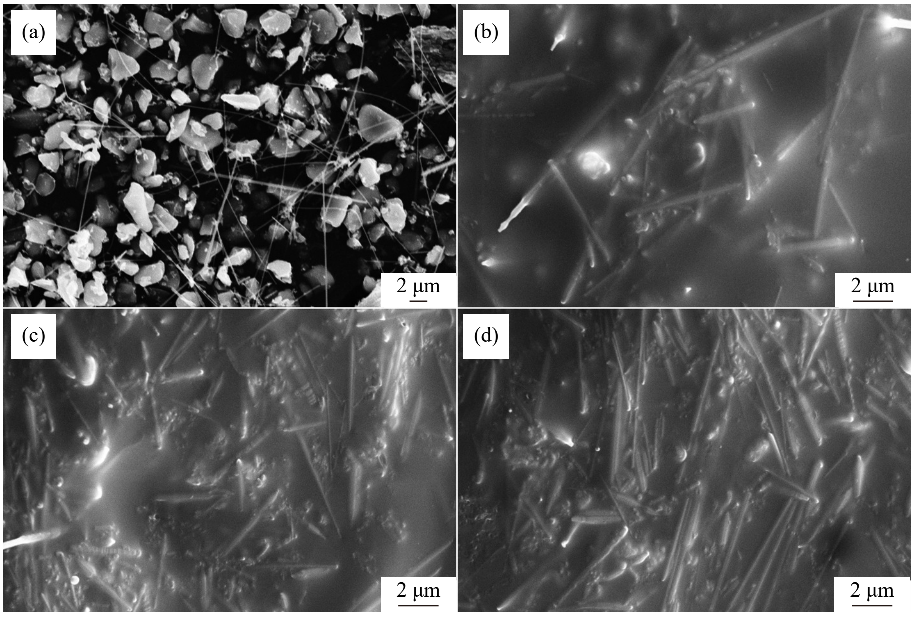

Fig. 2. SEM images of (a) the powder mixture of raw materials, (b) GS5, (c) GS10, and (d) GS20.

shows the typical surface morphology of the SiCnw/glass composite with different SiC filling ratios. A large number of SiC nanowires are randomly and uniformly embedded in the glass matrix. After sintering in air, the morphology of the SiC nanowires did not significantly change. In addition, the content of SiC nanowires in the composite gradually increases with the increase of the filling ratio.

Fig. 3 shows the mechanical properties of the SiCnw/glass composite. After the introduction of SiC nanowires, the Vickers hardness and flexural strength of the low-melting-point glass were significantly enhanced. The hardness value increased from HV 442 of the GS0 sample to HV 564 of the GS20 sample, with an increase of 27.7%. Meanwhile, samples GS5 (179 MPa), GS10 (207 MPa), and GS20 (213 MPa) showed 45.7%, 68.6%, and 72.8% improvements, respectively, in the flexural strength compared with sample GS0 (123 MPa), which can be attributed to the enhancement mechanism of SiC nanowires, including bridging, pullout, and crack deflection. The abovementioned results indicate that the SiCnw/glass composites have good mechanical properties.

Fig. 4(a)–(c) shows the dielectric properties of SiCnw/glass composites in the frequency range of 8.2–12.4 GHz. In the whole measurement frequency range, the relative dielectric constant (ε'), dielectric loss (ε''), and loss tangent ($\tan\delta = \varepsilon''/\varepsilon'$) of the low-melting-point glass (GS0 sample) are ~5.418, ~0.015, and ~0.003, respectively, which indicates a dielectric loss ability of nearly zero. By contrast, SiCnw/glass composites exhibit higher dielectric properties. In addition, as the filling ratio of SiC nanowires increased from 5wt% to 20wt%, ε', ε'', and $\tan\delta$ of composites increased from 5.83–6.08, 0.74–0.86, and 0.12–0.14 to 9.73–10.17, 2.65–3.21, and 0.27–0.32, respectively. The dielectric loss tangent ($\tan\delta$) is a key index of EMW-absorbing materials, which is used to indicate the ability of materials to dissipate EMW. Moreover, a higher $\tan\delta$ value represents a stronger dissipative attenuation ability of an EMW. The $\tan\delta$ value of the GS20 sample has increased by approximately 100 times compared with low-melting-point glass. Therefore, the introduction of SiC nanowires has dramatically enhanced the dielectric loss ability of the glass. This remarkable improvement in dielectric properties can be attributed to the polarization loss and conductance loss induced by SiC nanowires [31–33]. On the contrary, the real (μ') and imaginary (μ'') rel-

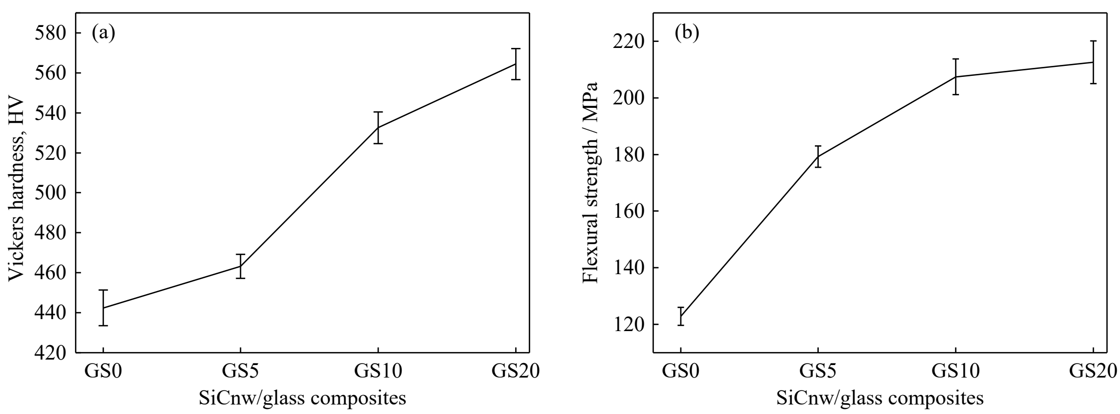

Fig. 3. (a) Vickers hardness and (b) flexural strength of SiCnw/glass composites.

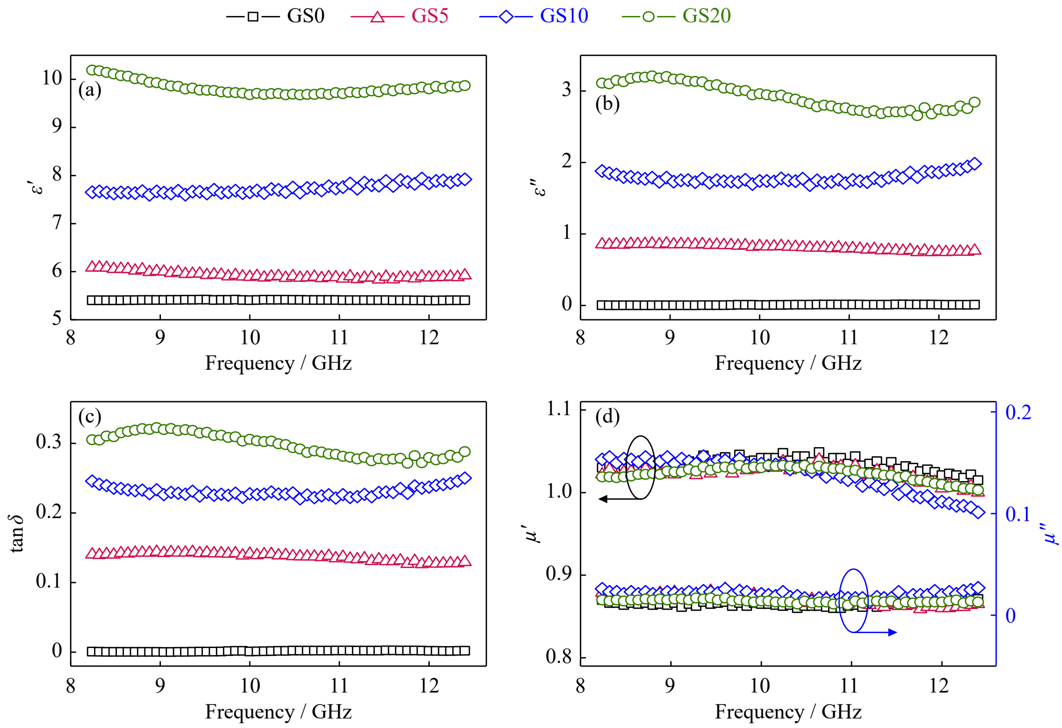

Fig. 4. Permittivity and permeability properties of SiCnw/glass composites as a function of frequency: (a) ε'; (b) ε''; (c) tan δ; (d) μ' and μ''.

ative permeability of all samples are lower than 1.049 and 0.027, respectively (Fig. 4(d)). This result indicates that the magnetic loss ability of the composite material is weak. Fig. 5 shows the Cole–Cole curves of the composites, which can illustrate the loss mechanism of the EMW. In this curve, a semicircle represents a kind of polarization relaxation loss. The Cole–Cole curve of the low-melting-point glass is irregular, which corresponds to its poor dielectric loss ability. After the introduction of SiC nanowires, a clear semicircle appears in the GS5 sample, which indicates that the polariza-

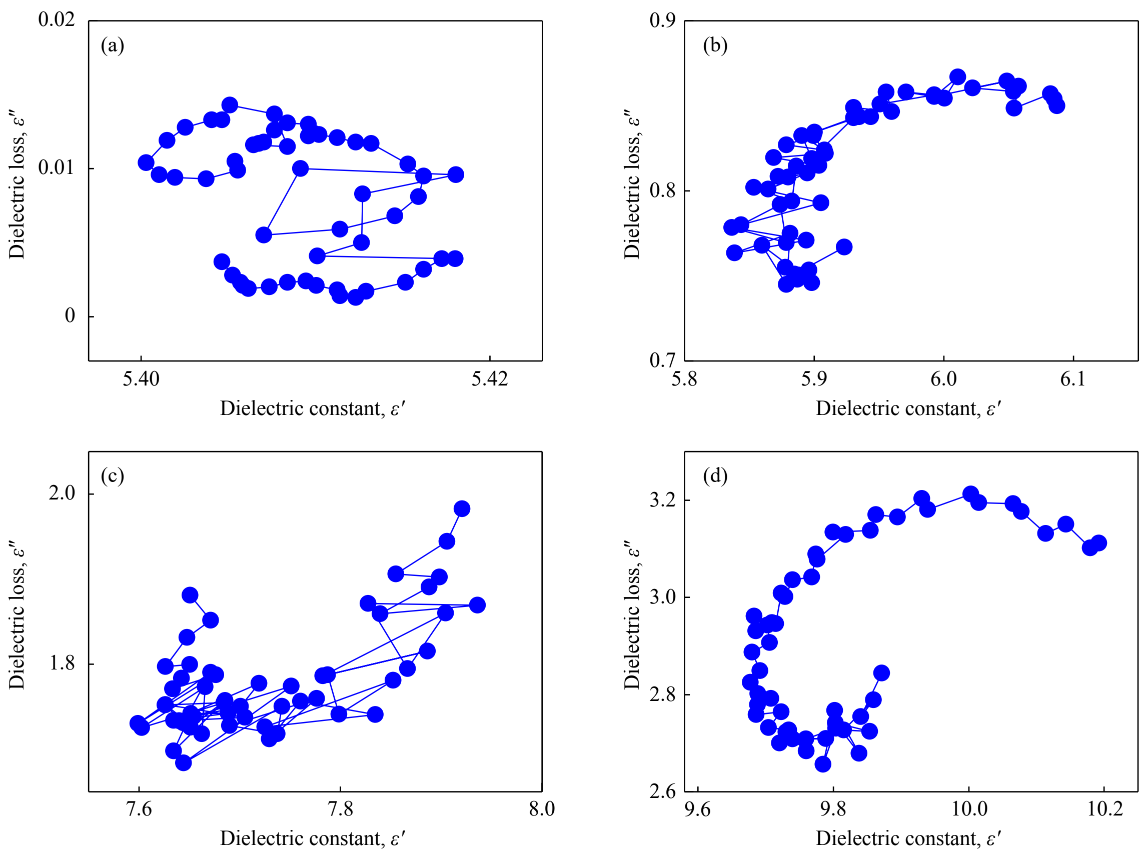

Fig. 5. Cole–Cole curves of samples (a) GS0, (b) GS5, (c) GS10, and (d) GS20.

tion loss contributes to its dielectric loss ability. This phenomenon is primarily due to the lattice defects of SiC nanowires [32]. With the further increase of the filling ratio of SiC nanowires, a tail appears in the curve, and its length gradually increases. Therefore, considerable conductance loss occurs in the composite [34]. As mentioned previously, at a higher filling ratio of SiC nanowires, an electrical conduction network can be easily formed, thereby enhancing the conductance loss ability.

The reflection loss (RL) was calculated in accordance with the transmission line theory [35], which is used to evaluate the EMW absorption performance of materials.

$$Z_{\rm in} = \sqrt{\frac{\mu_{\rm r}}{\varepsilon_{\rm r}}}\tanh\left(j\frac{2\pi f d}{c}\sqrt{\mu_{\rm r}\varepsilon_{\rm r}}\right) \tag{1}$$

$$\mathrm{RL} = 20\lg\left|\frac{Z_{\rm in}-1}{Z_{\rm in}+1}\right| \tag{2}$$

where $Z_{\rm in}$ is the input impedance of the absorber, $\varepsilon_{\rm r}$ and $\mu_{\rm r}$ are the complex permittivity and permeability of the absorber, respectively, f is the frequency of the incident microwave, d is the absorber thickness, and c is the velocity of light. At the same thickness of absorbing material, lower RL represents a stronger EMW absorption ability. Fig. 6 shows three-dimensional plots of the RL of SiCnw/glass composites versus the absorber thickness (1–5 mm) and frequency (8.2–12.4 GHz). The RL calculation results are completely consistent with the dielectric measurement results. The low-melting-point glass has almost no EMW-absorbing performance in the whole measured frequency and thickness range, in which its minimum RL was larger than −1 dB. With the formation of the SiCnw/glass composites, the RL value of the composites decreases significantly, which indicates that their EMW absorption performance has been effectively enhanced. However, when the filling ratios of SiC nanowires in the composites are 5wt% and 10wt%, their minimum RL values are only −5.3 and −11.0 dB, respectively. As the filling ratio further increases to 20wt%, the composite shows a minimum RL of −20.2 dB for thickness of 2.8 mm (−20.2 dB@2.8 mm) and an effective absorption (RL ≤ −10 dB) bandwidth of 2.3 GHz (9.8–12.1 GHz) at an absorber layer thickness of 2.3 mm (2.3 GHz@2.3 mm). This improved EMW absorption ability is primarily attributed to the synergistic effect of polarization loss and conductivity loss of SiC nanowires. In addition, compared with the representative 1D SiC inorganic composite (Table 2), the SiCnw/glass composite exhibits remarkable EMW absorption properties. Notably, its preparation conditions are simple. Thus, the abovementioned results indicate that the SiCnw/glass composite has good mechanical and EMW absorption properties, and it can be prepared at a low temperature in air atmosphere, making it a potential high-performance EMW absorption material. Moreover, this well-designed SiC/glass composite provides a novel insight into EMW-absorbing materials other than polymer and ceramic matrix composites. By reducing the sintering temperature of the glass matrix material, this EMW-absorbing material can be easily transformed into a coating and pre-

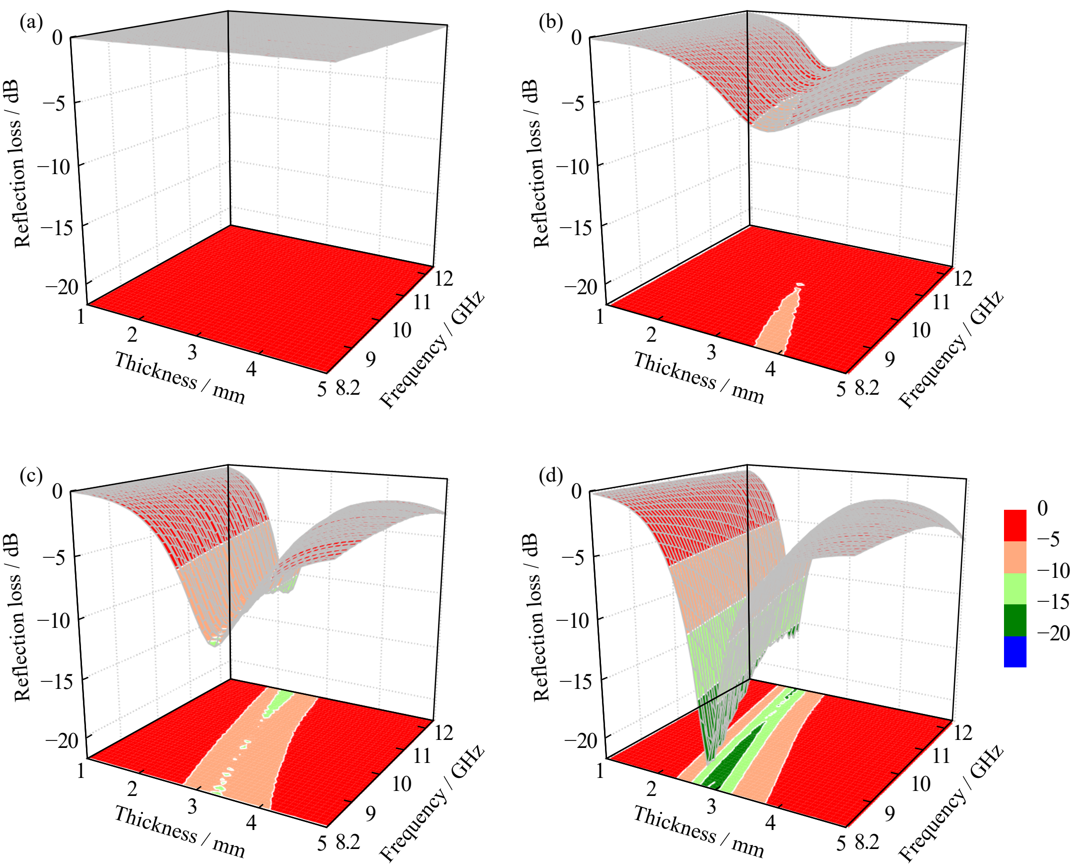

Fig. 6. Three-dimensional patterns of the reflection loss of SiCnw/glass composites versus the absorber thickness and frequency: (a) GS0, (b) GS5, (c) GS10, and (d) GS20.

Table 2. EMW absorption properties of 1D SiC inorganic composites

Materials	Preparation condition	SiC ratio	Minimum RL@thickness	Effective absorption bandwidth@thickness
SiCf/mullite composite [10]	1000°C in vacuum	—	−38 dB@2.9 mm	4.2 GHz@3.4 mm
SiCf/Si_3N_4 composite [11]	800°C, CVI process	43vol%	−13.3 dB@4.2 mm	—
SiCf/SiC composites [12]	1000°C in vacuum	—	−25 dB@2.3 mm	3.72 GHz@2.7 mm
SiCnw/SiCf/SiC composites [15]	900°C in N_2	—	−16.5 dB@4.5 mm	2.9 GHz@3.5 mm
SiCnw/SiOC ceramic [16]	1400°C in Ar	—	−10 dB@3.8 mm	—
SiCnw/SiO_2/$3Al_2O_3\cdot 2SiO_2$ porous ceramic [17]	1400°C in Ar	—	−35 dB@5.5 mm	4.2 GHz@5 mm
SiCw/porous SiC skeleton [18]	1500°C in Ar	—	−29 dB@2.2 mm	3.2 GHz@2.2 mm
SiCnw/$Ba_{0.75}Sr_{0.25}Al_2Si_2O_8$ ceramic [20]	1450°C, hot pressing	20vol%	−24.7 dB@1.85 mm	2.18 GHz@2.4 mm
This work	580°C in air	20wt%	−20.2 dB@2.8 mm	2.3 GHz@2.3 mm

Note: SiCf, SiCw, and CVI represent SiC fibers, SiC whiskers, and chemical vapor infiltration, respectively.

pared on a variety of substrate surfaces, which may have better corrosion resistance and mechanical properties than the polymer matrix EMW-absorbing coating [22].

4. Conclusion

SiC nanowires/low-melting-point glass composites were sintered at 580°C in an air atmosphere. Based on the XRD results, SiC nanowires were not oxidized during low-temperature sintering. Therefore, these composites exhibit good mechanical and EMW absorption properties. With the increase of the filling ratio of SiC nanowires from 5wt% to 20wt%, the Vickers hardness and flexure strength of the composite increased by 27.7% and 72.8% compared with the glass, reaching HV 564 and 213 MPa, respectively. In addition, the dielectric permittivity and EMW absorption properties of the composites in the frequency range of 8.2–12.4 GHz are gradually enhanced. When the filling ratio of SiC nanowires reaches a maximum of 20wt% in this experiment, the composite material also achieves a minimum RL of −20.2 dB. Meanwhile, it shows an effective absorption (RL ≤ −10 dB) bandwidth of 2.3 GHz at the absorber layer thickness of 2.3 mm. This improvement can be attributed to the synergistic effect of polarization loss and conductivity loss of SiC nanowires. Moreover, this well-designed SiC/glass composite provides a novel insight into EMW-absorbing materials other than polymer and ceramic matrix composites.

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Conflict of Interest

Wenbin Cao and Qi Wang are the editorial board member and the youth editorial board member for this journal, respectively, and were not involved in the editorial review or the decision to publish this article. All authors have no financial/commercial conflicts of interest.

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