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High-efficient production of SiC/SiO$_2$ core-shell nanowires for effective microwave absorption

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ABSTRACT

In the current report, we have demonstrated that the high-efficient production of SiC/SiO$_2$ core-shell nanowires can be achieved through the introduction of trace of water vapor during the chemical vapor deposition process. The yield of the SiC/SiO$_2$ core-shell nanowires is dramatically improved due to the introduction of water vapor. The SiC/SiO$_2$ core-shell nanowires exhibit an excellent microwave absorption property in the frequency range of 2.0–18.0 GHz with a very low weight percentage of 0.50 wt.% in the absorbers. A minimum reflection loss value of -32.72 dB (> 99.99% attenuation) at 13.84 GHz has been observed with the absorber thickness of 3.0 mm. Moreover, the SiC/SiO$_2$ core-shell nanowires based absorber can reach an effective absorption bandwidth (< -10 dB) of 5.32 GHz with the absorber thickness of 3.5 mm. Furthermore, a possible absorption mechanism is also proposed in detail for such

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effective attenuation of microwave which can be attributed to the dielectric loss and magnetic loss of SiC/SiO₂ core-shell nanowires.

Keywords: SiC/SiO₂ core-shell nanowires, High efficient production, Chemical vapor deposition, Microwave absorption, Low weight percentage, Polarization

1. Introduction

Recently, electromagnetic pollution, which can perturb biological immunity systems and induce miscellaneous diseases, has become an anxious social issue due to the rapid development of information technology, such as wireless and satellite communications, and extensive applications of electronic devices in our daily life [1-3]. Therefore, high-performance microwave absorption (MA) materials with a good absorption capacity that can effectively reduce adverse microwaves are highly desired [4-14]. Additionally, the application of high-performance MA materials in national defense security, such as in stealth technology, synthetic aperture radar and satellite, has further highlighted the requirement for the high-performance MA materials [15-17]. Generally, the microwave absorption property of MA materials is related closely to their dielectric properties (complex permittivity; \( \varepsilon_r = \varepsilon' - j\varepsilon'' \)), magnetic properties (complex permeability; \( \mu_r = \mu' - j\mu'' \)), and the electromagnetic microwave (EM) impedance match [18]. An effective approach to satisfy the above requirements lies in designing and fabricating hybrid dielectric and magnetic materials. However, there are still some drawbacks which restrict its practical applications. For example, most of the magnetic absorption materials are metals or metal oxides which possess high density that cannot be used in large quantity as filler of absorbers [19-26]. For practical civil and military applications, an ideal MA material should have not only low density, low filler loading ratio, good corrosion resistance, and high thermal stability, but also broad MA bandwidth and tunable absorption frequency range [27, 28].
Silicon carbide (SiC) nanomaterials, as one of the most important semiconductors, have been considered as promising candidates for remarkable MA materials due to their unique properties, such as low density, high thermal stability, excellent chemical resistivity, and good mechanical strength [29]. These unique properties are highly beneficial in the field of spacecrafts and aircrafts [30]. However, the poor dielectric properties caused by single polarization and low conductivity limit their applications as ideal MA materials [30, 31]. Great efforts have been made to improve the MA performance of SiC materials in the past decades, and diverse SiC based materials have been fabricated. These materials include SiC/Co hybrid nanowires [24], SiC nanowires [29, 32], polypyrrole@SiC nanocomposites [30], SiC/C composite ceramics [33], SiC microtubes [34], stacking fault SiC nanowires [35], SiC/Co@SiO\(_2\) nanowires [36], and doped SiC nanomaterials [37-39]. During the preparation process of SiC nanomaterials, some undesired SiO\(_2\) is usually formed as a by-product on the surfaces of SiC nanomaterials due to the existence of trace of oxygen. However, SiO\(_2\) is regarded as an electromagnetic transparent material in electromagnetic research field, and can significantly improve the MA performance for the nanoscale MA absorbers [40-45]. C.Y. Liang demonstrates that the introduction of SiO\(_2\) as a shell layer of SiC/Co nanowires can effectively improve the MA performance of SiC/Co nanowires [36]. After coating a transparent SiO\(_2\) shell, electrons can transfer between SiO\(_2\) shell and Co layer. This leads to the formation of gradient \(\varepsilon\) field within SiO\(_2\) and more incident microwave enters into the absorbers, thus enhancing the MA property of SiC/Co nanowires [36].

Herein, in the present research, SiC/SiO\(_2\) core-shell nanowires were fabricated via a one-step method without using any catalysts. The SiC/SiO\(_2\) core-shell nanowires exhibited a significantly improved EM absorption ability in the frequency range of 2–18 GHz. The growth process of SiC/SiO\(_2\) core-shell nanowires involved a catalyst-free vapor–solid (VS) growth process. Based
on the growth mechanism, water vapor was introduced during the preparation process to improve the growth rate of SiC/SiO$_2$ core-shell nanowires. The composition and structure of these as-synthesized SiC/SiO$_2$ core-shell nanowires were confirmed by X-ray diffraction (XRD), scanning electron microscope (SEM), transmission electron microscope (TEM), Raman spectrum, and energy dispersive X-ray spectroscopy (EDS). The MA properties of these as-synthesized SiC/SiO$_2$ core-shell nanowire based absorbers with different thicknesses were systemically investigated in the frequency range of 2-18 GHz. Moreover, a mechanism for microwave transparent SiO$_2$ shell enhancing MA performance of SiC/SiO$_2$ core-shell nanowires was also proposed.

2. Experimental

2.1. Materials

The silicon powders ($\geq$ 99%, analytical) were obtained from Liaoning Nitride Compound Ltd. China. Sodium hydroxide (NaOH, $\geq$ 98%, analytical) and ethanol were supplied by Tianjin Shuangchuan Chemical Reagent Company, China.

2.2. Synthesis of SiC/SiO$_2$ core-shell nanowires

The SiC/SiO$_2$ core-shell nanowires were synthesized via a facile chemical vapor deposition process. The process is based on the reaction between silicon and active carbon in the absence of catalysts. Typically, 6 g silicon powders were put into an alumina crucible ($7 \times 3 \times 2$ cm) which was lined with graphite paper. The crucible was covered by a graphite plate and placed into a tube furnace for further heat treatment process. Before the chemical vapor deposition process, N$_2$ gas (99.99%) was introduced into the furnace at a flow rate of 100 ml/min for 20 minutes to purge the tube under atmospheric pressure. The N$_2$ gas was kept during all the following experimental process with the flow rate of 100 ml/min. Afterwards, the furnace was first heated
to 800 °C with a heating rate of 10 °C/min and then to the target temperature of 1200 °C with a heating rate of 3 °C/min. The temperature was maintained for 2 h at 1200 °C. It is worth to note that trace of water vapor was introduced into the tube furnace (by changing the Line I to Line II as show in Fig. 1 a) during the temperature maintaining process. When the heat treatment process was terminated, the furnace was cooled down to 800 °C with a cooling rate of 4 °C/min, followed by naturally cooling down to room temperature. The samples were also synthesized without the introduction of water to compare with the modified ones. Finally, white wool like product was found in the inner walls of graphite paper and cover.

2.3. Structural characterization

XRD spectra of the as-synthesized products were obtained on a Rigaku X-ray diffractometer with Cu Kα radiation, and the diffraction points were recorded in the range of 2θ from 10° to 80°. Raman spectra were recorded from 600 to 1100 cm⁻¹ on a microscopic confocal Raman spectrometer (Renishaw) using a 532 nm laser (intensity 10%). SEM images of the as-synthesized products were performed on TESCAN VEGA II electron microscopy equipped with an energy dispersive X-ray spectroscopy (EDS). TEM and high-resolution TEM (HRTEM) images of the as-synthesized products were collected on a JEM-2100 electron microscopy.

2.4. Electromagnetic parameter measurements

The microwave absorption performance of the as-synthesized products was evaluated by a vector network analyzer (VNA; Agilent N5245A) in transmission-reflection mode in the frequency range of 2-18 GHz. The electromagnetic parameters of the as-synthesized products were calculated via HP85071 software. The SiC/SiO₂ core-shell nanowires/paraffin absorbers were prepared by uniformly mixing of SiC/SiO₂ core-shell nanowires and paraffin with a weight
ratio of 1:200 (0.50 wt.%). The mixtures were then pressed into toroidal shaped samples with an inner diameter of 3.0 mm, outer diameter of 7.0 mm, and a thickness of 3 mm, respectively.

3. Results and discussion

3.1. Growth mechanism of SiC/SiO\textsubscript{2} core-shell nanowires

Based on the characterization results and consideration of possible reactions among the intermediate gas phases, the reaction steps, and the surface energy minimization, R.B. Wu proposed a VS growth mechanism for preparation of SiC/SiO\textsubscript{2} core-shell nanowires in the absence of catalysts [46]. The growth process involves following reactions. First, SiO vapor will generate through the reaction of small amount of oxygen with evaporated silicon at high temperature:

\[
2\text{Si}(s) + \text{O}_2(g) \rightarrow 2\text{SiO}(g) \quad (1)
\]

where (s) and (g) stand for solid and gaseous states, respectively.

The generated SiO has unsaturated chemical bonds, and could be easily absorbed on the inner surface of graphite crucible, which also generated many chemically active sites at high temperature [46, 47]. As a consequence, the following reactions take place to form SiC nuclei so as to reduce the system energy [46]:

\[
\text{SiO}(g) + 2\text{C}(s) \rightarrow \text{SiC}(s) + \text{CO}(g) \quad (2)
\]

\[
\text{SiO}(g) + 3\text{CO}(g) \rightarrow \text{SiC}(s) + 2\text{CO}_2(g) \quad (3)
\]

\[
3\text{SiO}(g) + \text{CO}(g) \rightarrow \text{SiC}(s) + 2\text{SiO}_2(s) \quad (4)
\]

\[
2\text{SiO}(g) + \text{O}_2(g) \rightarrow 2\text{SiO}_2(s) \quad (5)
\]

Reaction (2) is mainly responsible for the SiC nuclei formation as well as providing the reactant gas CO for the subsequent reactions (3) and (4). These reactions are more possibly responsible for the growth of SiC nanowires [47]. SiC nanowires can grow steadily as long as the
adequate supplement of SiO and CO gases are there. Meanwhile, SiO$_2$ is also formed by the reactions (4) and (5) and can easily deposit and condense into small clusters on the surfaces of SiC nanowires due to the good fluidity of SiO$_2$ [47]. This will lead to the final formation of SiC/SiO$_2$ core-shell nanowires [47, 48].

Based on the above description, the generated CO which participates in the reactions plays a very important role in the formation of SiC/SiO$_2$ core-shell nanowires. Herein, we hypothesize that the moderate introduction of CO may improve the growth rates of SiC and SiO$_2$, and this will result an increase in the yield of SiC/SiO$_2$ core-shell nanowires. In the current research, consideration of the potential risk of leakage and toxicity of CO, water vapor is selected as a precursor to react with C at high temperature to produce CO, and the reaction is as follows:

$$\text{H}_2\text{O}(g) + C(s) \rightarrow \text{CO}(g) + \text{H}_2(g) \quad \text{……………… (6)}$$

The modified setup for the synthesis of SiC/SiO$_2$ core-shell nanowires is shown in Fig. 1a. It is noteworthy that the temperature of water was kept at 25 °C during the synthesis process to ensure the amount of water vapor introduced into the reactions per unit time is constant. The growth of the SiC/SiO$_2$ core-shell nanowires will follow the steps proposed by R.B. Wu after generation of enough CO (Fig. 1b to d). Fig. 2 shows the photographs of the SiC/SiO$_2$ core-shell nanowires synthesized with and without water vapor introduction. Obviously, much more white wool like SiC/SiO$_2$ core-shell nanowires (Fig. 2a and b) are obtained after introducing water vapor into the reactions, indicating an increase in the yield of the SiC/SiO$_2$ core-shell nanowires.

3.2. Composition and microstructure of the as-synthesized SiC/SiO$_2$ core-shell nanowires

Fig. 3a and b respectively display the XRD pattern and Raman spectrum of the as-synthesized products synthesized with water vapor introduction. The diffraction peaks observed at 36°, 60° and 72° in Fig. 3a agree well with the known values for 3C-SiC, and can be indexed to be the
planes of (111), (220), and (311) reflections of cubic 3C-SiC (JCPDS Card No.29-1129) [47]. Additionally, the broad XRD peak at low diffraction angle (10 - 30°) can be assigned to the amorphous phase of SiO$_2$ [49]. Raman spectrum (Fig. 3b) shows three peaks located approximately at 795, 913 and 971 cm$^{-1}$. The two peaks at 795 and 971 cm$^{-1}$ can be assigned to the transverse optical (TO) phonon mode and longitudinal optical (LO) phonon mode of 3C-SiC [50], respectively. The weak peak centered at 913 cm$^{-1}$ corresponds to the peak of amorphous SiO$_2$, which is similar to the value reported by Z.J. Li [50]. The results of XRD pattern and Raman spectrum clearly indicate the existence of SiC and SiO$_2$ phases.

Fig. 4 shows the representative SEM images and corresponding EDS spectra of the as-synthesized products with and without water vapor introduction. Fig. 4a and d, show the overall appearance of the as-synthesized products at a low magnification. It can be seen from these figures that the as-synthesized products display either a curved or straight one-dimensional like structure, up to several hundreds of micrometers long. This indicates the large-scale growth of nanowires with a uniform size distribution for both samples. However, the diameter of the samples synthesized with and without water vapor introduction are obviously different which can be clearly seen from the higher magnification SEM images as illustrated in Fig. 4b and e. The diameter of the as-synthesized products with water vapor introduction is in the range of 300-500 nm (Fig. 4b) while the diameter of the as-synthesized products without water vapor introduction is in the range of 10-50 nm (Fig. 4e). Clearly, the introduction of water vapor has effectively increased the growth rate of the nanowires. The elemental composition of both as-synthesized products were confirmed by using EDS analysis attached to the SEM as illustrated in Fig. 4c and f, revealing that both as-synthesized products are composed of C, O and Si elements. Moreover, the atomic ratios of C:O:Si calculated from EDS analysis shown in Fig. 4c and f are 1:5.3:3.3
and 1:5.0:3.3, respectively. The EDS results suggest that both as-synthesized products might comprise of SiC/SiO$_2$ core-shell heterostructures, which can be further confirmed by TEM analysis.

Fig. 5 presents the typical TEM images and corresponding selected area electron diffraction (SAED) patterns of the as-synthesized nanowires synthesized with and without water vapor introduction. Fig. 5a and d respectively depict the low magnification TEM images of both as-synthesized nanowires, representing that both nanowires possess a smooth surface with uniform diameter along the axial direction. However, the diameters of the nanowires are obviously different synthesized with and without water vapor introduction. This is well matched with the findings observed by SEM images as discussed in earlier section. A diameter of about 400 nm (Fig. 5b) for the nanowire synthesized with water vapor introduction is observed while the diameter of the nanowire synthesized without water vapor introduction is observed only 50 nm (Fig. 5e). Moreover, the phase contrast is deep in the core region for both nanowires, indicating the existence of inner cores (dark) and light outer shells. Inset in Fig. 5e is the representative HRTEM image of the nanowire synthesized without water vapor introduction, implying an imperfect structure with few defects. The interplanar spacing of two neighboring lattice fringes is calculated to be 0.25 nm, and is matching well with the d-spacing of (111) plane of 3C-SiC [51]. Fig. 5c and f present the corresponding SAED patterns recorded from the central parts of the nanowires in Fig. 5b and e, respectively, further confirming that the cores of the resultant products are 3C-SiC [51]. In addition, both the SAED pattern and the HRTEM image suggest that the nanowires synthesized with and without water vapor introduction grow along the [111] direction. Here, the HRTEM image of the nanowire synthesized with water vapor introduction is not discussed because the shell of the nanowire is too thick as shown in Fig. 5b and cannot be
clearly identified by HRTEM. In order to get more information about the cores of the nanowires synthesized with water vapor introduction, the shells of the nanowires are removed by 2 mol/L NaOH solution at 70 °C (Supporting information). Fig. 6 exhibits the corresponding SEM, TEM, and HRTEM images as well as the SAED pattern of the nanowires after complete removal of the SiO$_2$ shells. The EDS and Raman spectra confirm that the SiO$_2$ shells have been completely removed after NaOH solution treatment (Fig. S1 and S2). Fig. 6a and b present the SEM and TEM images of the nanowires after complete removal of the SiO$_2$ shells. The diameter of the nanowires after removal of SiO$_2$ shells obviously decreases (100 nm) as compared with the ones without removal of the SiO$_2$ shells. Fig. 6c displays the corresponding SAED patterns recorded from the central part of the nanowire (Fig. 6b). The diffraction spots become much clear in Fig. 6c, which confirms that the cores of nanowires synthesized with water vapor introduction are also 3C-SiC. This result can be further demonstrated by the HRTEM image as shown in Fig. 6d. The interplanar spacing of two neighboring lattice fringes is 0.25 nm in accordance to the d-spacing of (111) plane of 3C-SiC [51]. Additionally, the crystallinity of the SiC cores synthesized with water vapor introduction is also imperfect, and few defects can be found in the HRTEM images as marked with white lines in Fig. 6d.

3.3. Microwave absorption properties of the as-synthesized SiC/SiO$_2$ core-shell nanowires

Fig. 7 illustrates the MA properties of the as-synthesized SiC/SiO$_2$ core-shell nanowires synthesized with water vapor introduction using a weight percentage of only 0.50% in the absorbers in the frequency range of 2-18 GHz. The MA properties of the sample are calculated according to the following equations [25]:

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

where the normalized input impedance ($Z_{in}$) is given by the following equation:
\[ Z_{in} = Z_0 \sqrt{\frac{\mu_r}{\varepsilon_r}} \tanh \left[ j \left( \frac{2\pi f d}{c} \right) \sqrt{\frac{\mu_r \varepsilon_r}{c}} \right] \]

where \( d \) is the thickness of the absorber, \( f \) is the frequency of the electromagnetic microwaves, \( c \) is the velocity of light, \( Z_0 \) is the impedance of free space, and \( Z_{in} \) is the input impedance of the absorber, respectively [52]. As shown in Fig. 7 and tabulated in Table 1, the minimum reflection loss (RL) values of the as-synthesized SiC/SiO\(_2\) core-shell nanowires are changed with the increase of the absorber thickness, suggesting that the range of absorption frequency can be tuned by adjusting the thickness of the absorbers. When the absorber thickness is less than 2.0 mm, the RL is always more than -10 dB (90% microwave absorption) in the frequency range of 2 to 18 GHz. However, when the absorber thickness is in the range of 2.5 to 5.5 mm, the RL below -10 dB (90% microwave absorption) can be obtained in the frequency range of 5.96-14.80 GHz. Especially, a minimum RL value of -32.72 dB is achieved at 13.84 GHz when the absorber thickness is 3.0 mm.

<table>
<thead>
<tr>
<th>Thickness (mm)</th>
<th>Absorption peak (GHz)</th>
<th>RL (dB)</th>
<th>Absorption bandwidth (GHz, &lt; -10 dB)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.0</td>
<td>14.08</td>
<td>-2.54</td>
<td>-</td>
</tr>
<tr>
<td>1.5</td>
<td>14.32</td>
<td>-3.95</td>
<td>-</td>
</tr>
<tr>
<td>2.0</td>
<td>14.08</td>
<td>-8.28</td>
<td>-</td>
</tr>
<tr>
<td>2.5</td>
<td>14.00</td>
<td>-15.68</td>
<td>1.22 (12.58-14.80)</td>
</tr>
<tr>
<td>3.0</td>
<td>13.84</td>
<td>-32.72</td>
<td>4.32 (10.98-15.30)</td>
</tr>
<tr>
<td>3.5</td>
<td>13.76</td>
<td>-16.43</td>
<td>5.32 (9.53-14.85)</td>
</tr>
<tr>
<td>4.0</td>
<td>9.20</td>
<td>-11.95</td>
<td>1.98 (8.34-10.32)</td>
</tr>
<tr>
<td>4.5</td>
<td>8.00</td>
<td>-11.49</td>
<td>1.31 (7.36-8.97)</td>
</tr>
<tr>
<td>5.0</td>
<td>7.20</td>
<td>-11.19</td>
<td>1.24 (6.60-7.84)</td>
</tr>
<tr>
<td>5.5</td>
<td>6.48</td>
<td>-10.96</td>
<td>1.08 (5.96-7.04)</td>
</tr>
</tbody>
</table>

Generally, a MA material with RL value less than -30 dB is regarded as an excellent absorber, because this value corresponds to 99.999% RL or absorption of microwaves [53]. Additionally,
the absorption bandwidth with the RL less than -10 dB is 5.32 GHz (between 9.53 and 14.85 GHz) when the absorber thickness increases to 3.5 mm. This result indicated that the as-synthesized SiC/SiO\textsubscript{2} core-shell nanowires exhibit not only high RL values but also wide absorption bandwidth. Compared to the representative SiC nanomaterial based composites recently reported, the as-synthesized core-shell SiC/SiO\textsubscript{2} nanowires exhibit an excellent ability to absorb electromagnetic microwave. Table 2 outlines that SiC nanowires can be used as lightweight microwave absorbers with a low weight percentage as compared with other nanostructured composites.

<table>
<thead>
<tr>
<th>Materials</th>
<th>RL (dB)</th>
<th>Thickness (mm)</th>
<th>Bandwidth (GHz)</th>
<th>Weight percentage (%)</th>
<th>References</th>
</tr>
</thead>
<tbody>
<tr>
<td>SiC/Co nanowires</td>
<td>-25.0</td>
<td>2.5</td>
<td>6.6</td>
<td>25.1</td>
<td>[24]</td>
</tr>
<tr>
<td>SiC nanowires</td>
<td>-31.7</td>
<td>2.0</td>
<td>2.5</td>
<td>35</td>
<td>[29]</td>
</tr>
<tr>
<td>Polypyrrole /SiC nanowires</td>
<td>-20.0</td>
<td>2.5</td>
<td>6.52</td>
<td>10</td>
<td>[30]</td>
</tr>
<tr>
<td>SiC nanowires</td>
<td>-17.4</td>
<td>2.0</td>
<td>2.5</td>
<td>30</td>
<td>[32]</td>
</tr>
<tr>
<td>SiC microtubes</td>
<td>-23.9</td>
<td>1.0</td>
<td>-2.0</td>
<td>20</td>
<td>[34]</td>
</tr>
<tr>
<td>Stacking fault SiC nanowires</td>
<td>-30.0</td>
<td>4.6</td>
<td>3.7</td>
<td>50</td>
<td>[35]</td>
</tr>
<tr>
<td>SiC/Co@SiO\textsubscript{2} nanowires</td>
<td>-25.0</td>
<td>2.1-2.5</td>
<td>6.6</td>
<td>50</td>
<td>[36]</td>
</tr>
<tr>
<td>Ni doped SiC powders</td>
<td>-22.57</td>
<td>2.0</td>
<td>3.0</td>
<td>33</td>
<td>[54]</td>
</tr>
<tr>
<td>Si\textsubscript{3}N\textsubscript{4}-SiC composite ceramic</td>
<td>-27.1</td>
<td>2.5</td>
<td>2.8</td>
<td>3</td>
<td>[55]</td>
</tr>
<tr>
<td>Yttria-stabilized zirconia/SiC</td>
<td>-26.6</td>
<td>5.0</td>
<td>5.0</td>
<td>100</td>
<td>[56]</td>
</tr>
<tr>
<td>SiC/SiO\textsubscript{2} core-shell nanowires</td>
<td>-32.74</td>
<td>3.0</td>
<td>4.32</td>
<td>0.5</td>
<td>This work</td>
</tr>
<tr>
<td>SiC/SiO\textsubscript{2} core-shell nanowires</td>
<td>-16.43</td>
<td>3.5</td>
<td>5.32</td>
<td>0.5</td>
<td>This work</td>
</tr>
</tbody>
</table>

To investigate the MA mechanism of the as-synthesized SiC/SiO\textsubscript{2} core-shell nanowires, the relative complex permittivity ($\varepsilon_r = \varepsilon' - j\varepsilon''$) and permeability ($\mu_r = \mu' - j\mu''$) of the products are considered. Generally, the microwave absorption properties of MA materials are closely associated with their relative complex permittivity and complex permeability. Real permittivity ($\varepsilon'$) and real permeability ($\mu'$) represent the storage ability related to electric and magnetic energy,
while the imaginary permittivity ($\varepsilon''$) and imaginary permeability ($\mu''$) are in regards to the dissipation of electric and magnetic energy [17, 25, 57, 58], respectively. The complex permittivity and permeability of the as-synthesized SiC/SiO$_2$ core-shell nanowires have been evaluated on the basis of the transmission line theory [59]. Fig. 8 shows the frequency dependences of the real part ($\varepsilon'$) and imaginary part ($\varepsilon''$) of the complex permittivity (Fig. 8a), the real part ($\mu'$) and imaginary part ($\mu''$) of the complex permeability (Fig. 8b), the dielectric loss tangent ($\tan\delta\varepsilon$) (Fig. 8c) and the magnetic loss tangent ($\tan\delta\mu$) (Fig. 8d). As shown in Fig. 8a, the initial value for $\varepsilon'$ is 7.05 at 2 GHz, and slightly decreases to 4.92 from 2 to 12 GHz. In the range of 12 to 18 GHz, the $\varepsilon'$ exhibits broad multiresonance peaks. It is well known that the $\varepsilon'$ is an expression of the polarizability of a material, which consists of electric polarization and dipolar polarization at microwave frequencies [60]. The interface is formed between SiO$_2$ and SiC phases in the as-synthesized SiC/SiO$_2$ core-shell nanowires, and the crystal planes along the interface are normally different. This difference will cause disorder on the crystalline interfaces [61, 62]. As a result, defects and dangling bonds are commonly formed along the interface for charge accumulation [63], thus enhancing the polarization along the interfaces. In addition, the defects are also found in the interior of the SiC cores as shown in the Fig. 6d. The different electronic structures of different phases (defects and perfect SiC phase), such as different conduction and valence band positions, can cause a mismatch between the electronic structures of the different phases along the interfaces. This will result in charge imbalance and redistribution along the interface to introduce a polarization effect [64]. The values of $\varepsilon''$ increased slightly with little variation in the range of 2-12 GHz (Fig. 8a), and the $\varepsilon''$ also exhibits broad multiresonance peaks in the range of 12-18 GHz as $\varepsilon'$. According to the free electron theory, $\varepsilon'' = \frac{1}{2\pi\varepsilon_0\rho}$, where $\varepsilon_0$ is dielectric constant and $\rho$ is the resistivity of the MA absorption
materials [65]. The existence of the defects formed inside the SiC cores can effectively increase the resistivity of SiC nanowires, thus increasing the value of $\varepsilon''$. Fig. 8b shows the frequency dependence of the real and imaginary parts of the complex permeability of the as-synthesized SiC/SiO$_2$ core-shell nanowires. It is observed that the $\mu'$ (1.07) and $\mu''$ (0) values almost have no change in the frequency range of 2-12 GHz. In the frequency range of 12-18 GHz, the values of $\mu'$ decrease sharply from 1.3 to 0.65 and then slight increase to about 1.0. For $\mu''$, in the frequency range of 12-18 GHz, the values increase sharply from 0 to 0.6 and then decrease sharply from 0.6 to -0.2 followed by smoothly increase to about 0. Some values of $\mu''$ are negative in the frequency range of 12-18 GHz, which is meaningless from the physics point of view, and these negative values of $\mu''$ might be due to noise [29]. Fig. 8c and 8d exhibit the dielectric tangent loss and magnetic tangent loss of the as-synthesized SiC/SiO$_2$ core-shell nanowires, respectively. Generally, the dielectric tangent loss ($\tan\delta_\varepsilon = \varepsilon''/\varepsilon'$) is commonly used to describe microwave absorption [66]. It is observed that the values of $\tan\delta_\varepsilon$ exhibit three variation tendencies in the frequency range of 2-18 GHz: (I) increase from 0.27 to 0.92 in the frequency range of 2-13.36 GHz, (II) sharp decrease from 0.92 to 0.14 in the frequency range of 13.36-15.92 GHz, and (III) smooth increase from 0.14 to 0.41 in the frequency range of 15.92-18 GHz. Fig. 8d shows the magnetic loss of the SiC/SiO$_2$ core-shell nanowires. As shown in Fig. 8d, the values of $\tan\delta_\mu$ exhibit four variation tendencies in the frequency range of 2-18 GHz: (I) almost close to 0 in the frequency range of 2-12 GHz, (II) sharp increase from 0.021 to 0.70 in the frequency range of 12-13.76 GHz, (III) sharp decrease from 0.70 to -0.27 in the frequency range of 13.76-15.76 GHz, and (IV) smooth increase from -0.27 to -0.018 in the frequency range of 15.76-18 GHz. Compared to the values of dielectric loss, the values of magnetic loss are relatively low in the frequency range of 2-12 GHz. This indicates that the main contributor to RL
is dielectric loss in the frequency range of 2-12 GHz. From 12 to 18 GHz, the good MA properties can be attributed to the coupling effects of dielectric loss and magnetic loss.

4. Conclusions

The yield of the SiC/SiO₂ core-shell nanowires can be dramatically improved by the introduction of trace water vapor during the synthesis process which involves a VS growth mechanism. The characterization results of XRD, Raman, SEM, TEM, HRTEM and SAED demonstrate that SiC/SiO₂ core-shell nanowires with core (SiC) diameter of about 100 nm and shell (SiO₂) thickness of about 150 nm are obtained. The diameter of the SiC/SiO₂ core-shell nanowires synthesized with water vapor introduction obviously increases in comparison to the SiC/SiO₂ core-shell nanowires which are synthesized without water vapor introduction. This indicates the improvement of growth rate of SiC/SiO₂ core-shell nanowires after water vapor introduction. The microwave absorption measurements show that the SiC/SiO₂ core-shell nanowires exhibit an excellent microwave absorption property in the range of 2.0–18.0 GHz with a very low weight percentage of 0.50 wt.%. A minimum RL value of -32.72 dB (> 99.99% attenuation) is obtained at 13.84 GHz with the absorber thickness of 3.0 mm. More important, the absorption bandwidth (< -10 dB) can go up to 5.32 GHz when the absorber thickness increases to of 3.5 mm. This result is superior to the other SiC based microwave absorption materials reported recently. The excellent MA properties could be attributed to the dielectric loss and magnetic loss of the SiC/SiO₂ core-shell nanowires. The advantages of the low density, high thermal stability, excellent chemical resistivity, and outstanding mechanical strength of SiC, the SiC/SiO₂ core-shell nanowires are promising candidates as MA materials in practical applications, especially in the field of specific spacecrafts and aircrafts.

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Figure captions

**Fig. 1.** (a) The schematic of the setup used for the heat treatment process, (b-d) the growth process of the SiC/SiO$_2$ core-shell nanowires.

**Fig. 2.** The photograph of the products synthesized with and without water vapor introduction: (a) top view and (b) side-view of the sample synthesized with water vapor introduction, (c) top view of the sample synthesized without water vapor introduction.

**Fig. 3.** The XRD pattern (a) and Raman spectrum (b) of the SiC/SiO$_2$ core-shell nanowires synthesized with water vapor introduction.

**Fig. 4.** The SEM images and EDS spectra of the SiC/SiO$_2$ core-shell nanowires synthesized with (a, b, and c) and without (d, e and f) water vapor introduction.

**Fig. 5.** The TEM images and corresponding SAED patterns of the SiC/SiO$_2$ core-shell nanowires synthesized with (a, b, and c) and without (d, e and f) water vapor introduction. Inset in e is the corresponding HRTEM image.

**Fig. 6.** (a) SEM image, (b) TEM image, (c) SAED pattern and (d) HRTEM image of the SiC/SiO$_2$ core-shell nanowires synthesized with water vapor introduction after removal of SiO$_2$ shell by 2 mol/L NaOH solution.

**Fig. 7.** Frequency dependence of RL for SiC/SiO$_2$ core-shell nanowires synthesized with water vapor introduction with different thickness in the frequency range of 2–18 GHz.

**Fig. 8.** Frequency dependence of the complex relative permittivity (a), permeability (b), dielectric tangent loss (c) and magnetic tangent loss (d) of SiC/SiO$_2$ core-shell nanowires synthesized with water vapor introduction in the frequency range of 2–18 GHz.
Fig. 1
Fig. 2
Fig. 3
Fig. 4
Fig. 5
Fig. 6
Fig. 7
Fig. 8
Graphical abstract
HIGHLIGHTS

- SiC/SiO₂ core-shell nanowires can be prepared via a facile catalyst-free chemical vapor deposition process.

- The yield of the nanowires is effectively improved by introducing trace of water vapor during the preparation process.

- The SiC/SiO₂ core-shell nanowires show excellent microwave absorption properties.