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Incremental Preparation and Absorbing Properties of SiC Nanowires

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Abstract

With the rapid development of science and technology, the requirements for absorbing materials in various fields are more and more high. SiC material, as a kind of absorbing materials, has a variety of excellent characteristics, and it is one of the key research absorbing materials. This experiment chose activated carbon powder and silicon powder to grow the SiC nanowires on the graphite paper. In the process, activated carbon atoms on the surface of the graphite paper and coarse porous structure are more likely to contribute to formation of SiC crystal nucleus. In addition, increasing the water vapor in the experiment can improve the output of SiC nanowires, which is a highlight of preparing and improving the yield of SiC nanowires in this experiment. The microstructure and morphology of the SiC nanowires were characterized by XRD and SEM, and the microwave absorbing properties of the SiC nanowires was tested. When the molar C/S ratio is 2: 1 and the reaction sintering temperature is 1300℃, the SiC nanowires have smooth and uniform surface and high aspect ratio. At the same time, by studying the wave absorption properties, it is found that the SiC nanowires prepared have good dielectric properties and high reflection loss rate.

Keywords

SiC; Nanowires; Core-shell Structure; Wave Absorption Properties.

1. Introduction

Modern science and technology are developing with each passing day, bringing convenience to all aspects of people's production and life. Among them, the development of electromagnetic wave is widely used in military defense, communication, navigation, medical treatment, energy and other aspects, and plays a very important role in human society [1-4]. However, there are two sides of a coin. Electromagnetic wave brings benefits and harms to people at the same time[8].

As a kind of ceramic absorbing material, silicon carbide has these great features: high-temperature stability, oxidation resistance, high frequency dielectric resonance characteristics, and can polarize electron dipoles. All these features determine that silicon carbide can absorb electromagnetic waves. [9-10]. At present, the preparation methods of silicon carbide nanowires mainly include template method, carbothermal reduction method, chemical vapor deposition method, etc.[11]. However, these methods have more or less problems, such as high cost, low purity, low yield and complex process.

In this experiment, silicon powder and activated carbon were used as the main raw materials, and graphite paper was used as the growth platform to prepare SiC nanowires with less structural defects and large aspect ratio. In order to increase the yield of SiC nanowires, the tube furnace was modified to increase the content of gaseous water in the furnace and make it participate in the solid phase reaction. It provides an important theoretical basis for the preparation of SiC nanowires with low cost and high yield to further promotes the application of SiC nanowires in the field of absorbing materials.

2. Experimental Process

The main raw materials of this experiment include silicon powder, activated carbon, graphite paper, alcohol lamp. The specific experimental process: silicon powder and activated carbon powder were mixed and ground in different proportions. The graphite paper was attached to the inner wall of the graphite crucible. The mixed powder of silicon powder and activated carbon was tiled inside the graphite crucible, and a layer of graphite paper was covered on the upper layer of the mixed powder as a growth platform for silicon carbide nanowires. The sample was placed in a tube furnace, and high-purity nitrogen gas was introduced as a protective gas. At the same time, in order to study the effect of water vapor on the yield of silicon carbide, nitrogen gas was passed through a gas cylinder with distilled water, allowing nitrogen gas to carry water vapor into the tube furnace. Set a certain sintering temperature, heating and cooling procedures, then scraped gray SiC nanowires which are on the graphite paper. The microstructure and morphology of SiC nanowires were characterized by XRD and SEM, and the microwave absorbing properties of the samples were tested.

3. Results and Discussion

Fig. 1 shows the XRD patterns and SEM images of the prepared samples. The four peaks of XRD correspond to the (111), (200), (220) and (311) diffraction peaks of β-SiC, respectively. When the reaction sintering temperature is 1300°C and the ratio of carbon to silicon is 2:1, the product can be clearly seen to contain elongated continuous SiC nanowires, showing a nest-like structure.

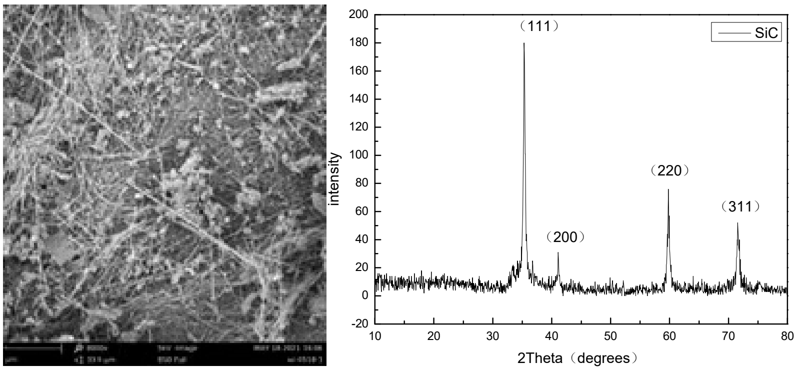

Fig 1. XRD and SEM patterns of SiC nanowires with core-shell structure

3.1. Analysis of Microwave Absorbing Properties of Sic Nanowires Prepared at Different Sintering Temperatures

SiC is a kind of material that does not have the ability of magnetic loss. It mainly relies on dielectric loss to complete the loss of electromagnetic waves. Fig. 2 is the real part parameter diagram of the dielectric constant of SiC nanowires prepared at different sintering temperatures. The real part value of dielectric constant sintered at 1200°C is relatively high, all above 10. Reaction sintering at 1300°C can be clearly observed that there is a certain frequency so that the real part of the dielectric constant is less than 10. From the change of the imaginary part of the dielectric constant in Fig. 3, it can be seen that the imaginary part of the dielectric constant of the sample is not much different when the sintering temperature is 1200°C or 1400°C. At 1300°C, the imaginary part of the dielectric constant is higher and more stable than that at 1200°C and 1400°C. According to the formula, the complex dielectric constant can be written as ε=ε+iσ/ω, ε represents the real part of the dielectric constant. It can be concluded that the imaginary part is related to the conductivity. The larger the imaginary part, the greater the conductivity, the greater the dielectric loss, and the stronger the ability to absorb electromagnetic waves[10]. Therefore, according to the imaginary part of the dielectric constant, the product prepared at the sintering temperature of 1300°C has the strongest ability to attenuate electromagnetic waves.

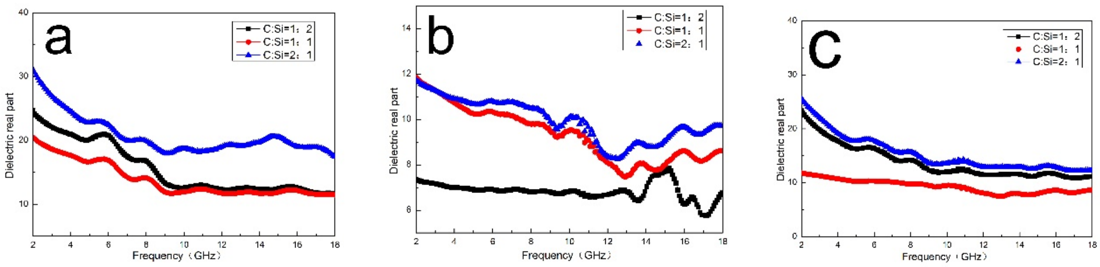

Fig 2. Real part of dielectric constant at different sintering temperatures(a:1200℃; b:1300℃; c:1400℃)

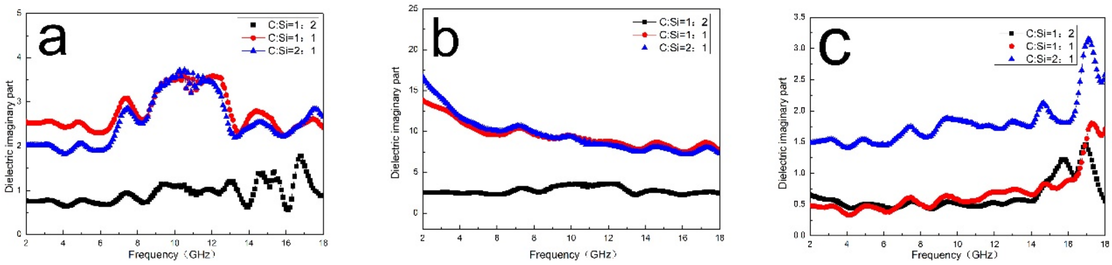

Fig 3. Imaginary part of dielectric constant at different sintering temperatures(a:1200℃; b:1300℃; c:1400℃)

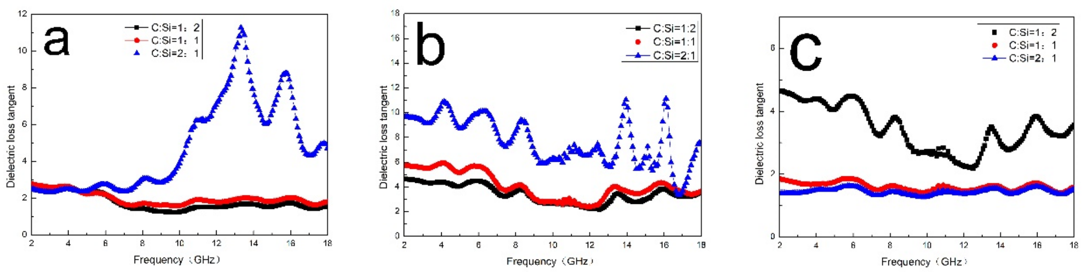

Fig 4. Dielectric loss tangent at different sintering temperatures(a:1200℃; b:1300℃; c:1400℃)

Fig. 4 shows the dielectric loss tangent of SiC nanowires prepared at different sintering temperatures. The tangent value of dielectric loss angle is obviously higher at 1300°C.This is because the structure of SiC material is diversified by sintering at 1300°C.SiC whiskers with high aspect ratio, core-shell structure attached to the surface and porous structure of carbon fiber can increase energy dissipation. The SiC whiskers generated by itself have high aspect ratio and uniform thickness, and are easy to lap each other to form conductive network structure, shortens the electron moving path. Thus, these SiC whiskers improve the conductance loss and further increasing the electromagnetic wave loss, to enhance the absorbing performance of the material.

Fig. 5 shows the Cole-Cole semicircle curves of SiC nanowires with core-shell structure prepared at different temperatures and the same molar ratio of carbon to silicon. The figure shows that the graph is composed of multiple rings, and each Cole-Cole semicircle corresponds to a dipole relaxation process. When the sintering temperature is 1400°C, the semicircles are relatively few and the structure is simple. When the sintering temperature is 1300 °C, the semicircles increase and the structure becomes more complicated (Fig.5(b)), which indicates that the SiC nanowires have multiple polarization relaxation modes when the temperature is 1300°C. Increase the consumption capacity of electromagnetic waves, thereby enhancing the absorption of electromagnetic waves.

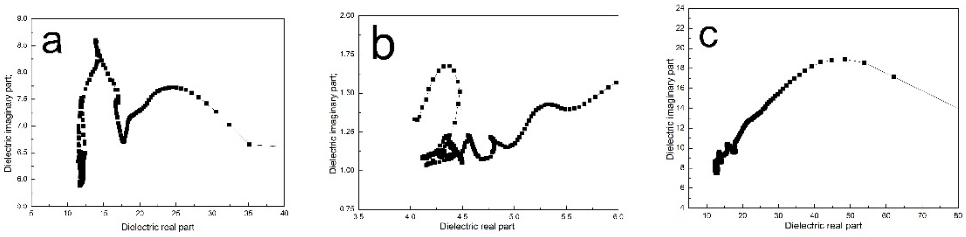

Fig 5. Cole-cole semicircles sintered at different temperatures(a: 1200℃; b:1300℃; c:1400℃)

There are two main indexes to evaluate the excellent absorbing properties of absorbing materials : reflection loss strength (RL) and absorption bandwidth[10]. In general, the higher the reflection loss, the wider the absorption band, the better the absorbing performance. When the reflection loss rate RL<-10dB, the absorption of electromagnetic waves can reach more than 90%, showing excellent impedance matching ability[15]. Fig.6 is the reflection loss rate of the core-shell structure SiC nanowires absorbing materials sintered at different temperatures.

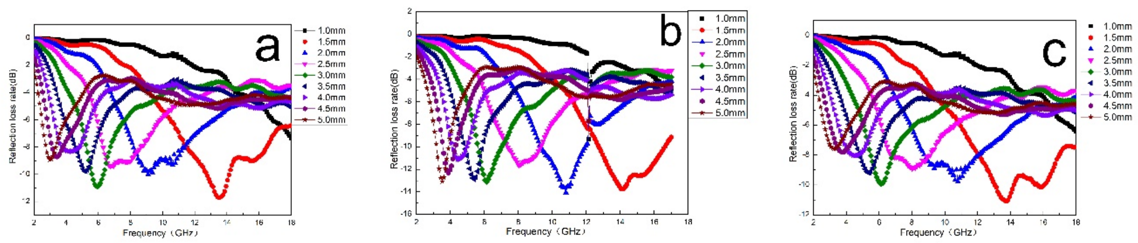

Fig 6. Reflection loss rate of SiC nanowires sintered at different temperatures (a:1200℃; b:1300℃; c:1400℃)

When the sintering temperature is 1300°C, the reflection loss of SiC nanowires is less than -10dB except for 1.0mm, and the minimum reflection loss is -14dB when the coating thickness is 2.0mm. Compared with 1200°C and 1400°C, when the sintering temperature is 1300°C, the absorption capacity of electromagnetic wave is stronger and the absorption bandwidth is wider. With the increase of thickness, the effective absorption bandwidth covers 3-18GHz.

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3.2. Analysis of Microwave Absorbing Properties of Sic Nanowires Prepared by Different Ratios at 1300°C

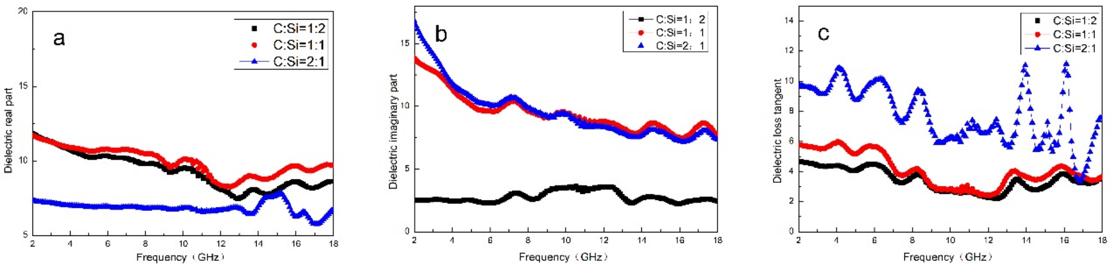

Fig 7. Dielectric constant of different molar carbon silicon ratio

Fig. 7 shows the permittivity of SiC nanowires with core-shell structure prepared by different molar ratios of carbon to silicon.When the molar ratio of carbon to silicon is 2:1, the value is the lowest, the imaginary part is the largest, the dielectric loss tangent is relatively large, and the dielectric loss capacity is the strongest. At this time, the generated SiC nanowires have a high aspect ratio and are easy to lap into a three-dimensional conductive network structure, which shortens the electron movement path and increases the loss of electromagnetic waves, thereby enhancing the absorbing properties of the material

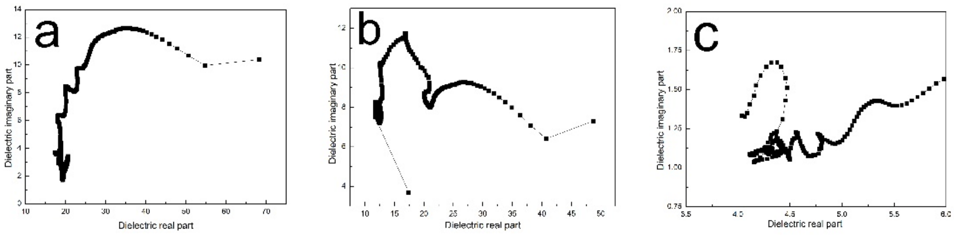

Fig 8. Cole-cole semicircles with different molar carbon-silicon ratios(a: C: Si=1:2; b:C: Si=1:1; c:C: Si=2:1)

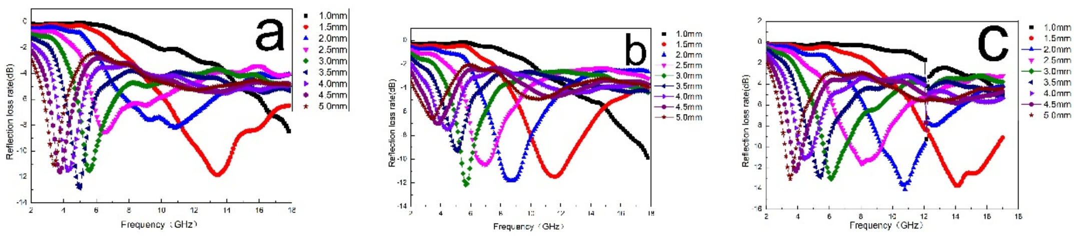

Fig 9. Reflection loss rate of SiC nanowires sintered at different molar ratios of carbon to silicon(a:C: Si=1:2; b:C: Si=1:1; c:C: Si=2:1)

From the Cole-Cole curve in Fig. 8,with the increase of carbon-silicon ratio, the semicircle curve is relatively complex and the arcs increase. Because each Cole-Cole semicircle corresponds to a dipole relaxation process, this shows that when the molar ratio of carbon to silicon is 2:1 during the preparation process, SiC nanowires have multiple polarization relaxation modes, which increases the consumption capacity of electromagnetic waves, thereby enhancing the absorption of electromagnetic waves.

Fig. 9 shows the reflection loss rate of SiC nanowires sintered at 1300°C with different molar ratios of carbon to silicon.The absorbing properties of SiC nanowires are relatively better than others when the molar ratio of carbon to silicon is 2:1. At the moment, SiC nanowires have the highest effective bandwidth. Except for 1.0mm, all other coating thickness is less than-10dB, and has a wider effective bandwidth, loss intensity is stronger.

3.3. Absorbing Properties of Sic Nanowires (1300°C,C:Si=2:1) Prepared by Introducing Water Vapor

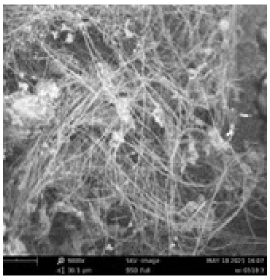

Fig 10. SEM image of SiC nanowires formed after vapor injection

In fig. 10, the sintering temperature is 1300°C. In the tube furnace reaction sintering, the inert protective nitrogen gas is first passed through the cylinder containing distilled water, so that the gas is entrained with water vapor into the tube furnace to promote the formation of nanowires. The morphology and structure of SiC have changed after water vapor is introduced. Compared with Fig.1, the nanowires begin to change from an irregular shape such as size and thickness to a uniform and smooth surface with uniform thickness, which is connected to each other to present a bird 's nest structure. The yield of SiC nanowires has also increased. From 0.08g before water vapor to 0.12g (increased by 50%), the expected goal of changing the morphology and yield of SiC nanowires by water vapor was achieved. On this basis, we discuss the change of its absorbing properties.

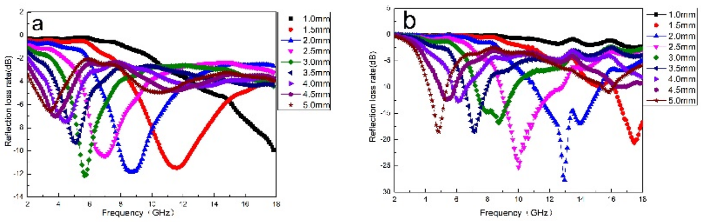

Fig 11. Curves of reflection loss rate of SiC nanowires versus frequency before and after passing water vapor (a : before water vapor ; b : After water vapor)

As shown in Fig.11, after the reactants are treated with water vapor during the sintering process, the electromagnetic wave absorption capacity is significantly improved. When the thickness is 2.0mm, the minimum reflection loss rate increases from the lowest -12dB to the lowest-27.5dB (the absorbing performance is increased by 1.3times). When the thickness is greater than 1mm, the absorption of electromagnetic waves can reach more than 90 %, and the effective frequency bandwidth increases.

4. Conclusion

In this paper, SiC nanowires were prepared by activated carbon, silicon powder and nitrogen gas protection. The phase and morphology of SiC nanowires were characterized by XRD and SEM. The effects of different molar ratios of silicon to carbon, different reaction sintering

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temperatures, and whether water vapor was introduced during the reaction sintering process on the growth of SiC nanowires were studied. The effects of water vapor and other factors on the growth of SiC nanowires were discussed, as well as their absorbing properties were discussed. The SiC nanowires prepared by sintering at 1300°C and the ratio of carbon to silicon was 2:1 had Smooth surface and uniform thickness, and the reflection loss rate was the lowest. The yield of water vapor reaction increased by 50%, and the absorbing performance increased by 1.3times.

Acknowledgments

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