Experimental study on dielectric properties of SiC material and temperature distribution of rubber materials containing silicon carbide coated metal via microwave heating

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Abstract. Microwave heating has not been widely used in the heating process of the rubber products containing metal skeleton, because the metal reflects microwaves that leads to a large amount of heat in the metal-rubber contact surface due to the skin effect and the eddy current effect in the microwave field. This experiment investigated the effects of microwave frequency and temperature on the dielectric parameters of silicon carbide coating materials. The microwave heating process of rubber composites containing metal skeleton coated with SiC mixture was also investigated experimentally. The results are as follows: As the microwave frequency increases, the dielectric parameters of the SiC coating material increase slowly first, when the frequency reaches a certain point, the dielectric parameters increase sharply to a maximum after that fall rapidly; Based on microwave frequency of 2450MHz, the dielectric constants of three different compositions of SiC coating materials decrease with increasing temperature; The temperature of the rubber composite containing the steel wire coated with the SiC is lower than the rubber composite containing the steel wire naked. The larger the content of SiC, the more uniform the temperature distribution, and the more effective the weakening of the sparks phenomenon caused by the skin effect.

Key words: rubber composites; microwave heating; metal skeleton; dielectric parameters; silicon carbide.

1. Introduction

Rubber is widely used due to its beneficial proprieties like strength, long lasting, water resistance and heat resistance [1]. In conventional vulcanization process, rubber is heated by hot air or water. Because of the lower conductivity of rubber material, inner temperature of rubber products is frequently less than surface temperature, which leads to the nonuniformity in temperature distribution of rubber material [2]. Finding more efficient ways to heat rubber material is essential in research and particular industry.

Microwave heating is one of new advancing techniques for heating rubber material. Its major advantages are rapidly emerging, precise control, fast start-up, and so-called volumetric heating, because the microwave energy that penetrates into the material will be absorbed and converted into internal energy in the material [3]. In recent years, microwave technology has been used increasing rapidly in
rubber industry, including drying, vulcanization and preheating, but these rubber products can only be without metal skeleton [4].

The metals can reflect microwave radiation, where delocalized electrons are free to move in relatively broad regions. The kinetic energy of some electrons may increase enabling them to jump out of the material, resulting in the ionization of the surrounding atmosphere. At a macroscopic level, this phenomenon is perceived as sparks or electric arcs formation. The problem of metal sparks must be dealt with when microwave technology is used in heating rubber products containing metal skeleton materials. One way to solve this problem is to coat the metal surface with absorbing materials.

The materials which interact with microwaves to generate heat are called microwave absorbers. The ability to heat material in the presence of a microwave field is defined by its dielectric loss tangent: $\tan \delta = \varepsilon''/\varepsilon'$. The dielectric loss tangent is composed of two parameters, the dielectric constant (or real permittivity), $\varepsilon'$, and the dielectric loss factor (or imaginary permittivity), $\varepsilon''$; i.e., $\varepsilon = \varepsilon' - i \varepsilon''$, where $\varepsilon$ is the complex permittivity. The dielectric constant ($\varepsilon'$) determines the amount of reflection and absorption of incident energy, while the dielectric loss factor ($\varepsilon''$) measures the electric energy dissipation in form of heat within the material [5].

Silicon carbide (SiC) is a typical semiconductor material. Due to its low density, good oxidation resistance and chemical stability, excellent mechanical strength and adjustable electrical conductivity, it is an ideal choice for lightweight, high temperature resistant microwave-absorber. Hongyu Wang[13] et al. compared the microstructure, mechanical properties, composite dielectric constant and microwave absorption properties of two different types of silicon carbide and epoxy composites of KD-ISiCf and SLF-SiCf. It was found that the absorption rates of the two composites were not high.

Yongshan Wei [14] et al. proposed a new method to improve the microwave absorption properties of SiC fibers. The properties of FeCo/SiCf composites in low temperature and high vacuum environments were investigated by RF magnetron sputtering. Compared with SiC fiber, FeCo/SiCf composites have good microwave absorption properties in the microwave frequency range.

Jae-Hun Choi [15] et al. mainly studied the mechanical properties and electromagnetic properties of SiC/epoxy composites, and measured the electromagnetic properties of epoxy-based composites under different pressures. The microwave absorption properties of single-layer and multi-layer composites were analyzed and found to have excellent microwave absorption properties.

Wei Zhou [16] et al. used nickel nanoparticles as catalysts to carry out catalytic chemical vapor deposition (CCVD) on the surface of carbon fibers to form silicon carbide nanofibers (SiCNFs). And the test found that the microwave absorption characteristics were effectively improved compared with pure carbon fibers.

The aim of the study is to investigate dielectric properties of SiC mixture and it’s effects on the temperature distribution of rubber composites containing steel wire during microwave heating.

2. Experimental

2.1. Materials and apparatus

Abrasion resistant compound; Micron-sized silicon carbide (Average particle size 10 $\mu$ m); Round iron sheet (d=25mm, $\delta=1$mm); Phenolic resin powder; N, N-Dimethylformamide.

The microwave heater(Mls1200, operating frequency is 2450MHz); Fiber optic temperature sensor(Temperature range -50 ℃~200 ℃). Compared with traditional temperature sensor, optical fiber temperature sensor has the advantages of high sensitivity, small volume, light weight, easy bending, no electromagnetic interference and good corrosion resistance; Impedance Analyzer (Keysight E4991B Impedance Analyzer, Frequency range 1MHz~3GHz); Scanning electron microscope (JSM-7001F); The SZQ type tetrahedral preparator(Tianjin Kexin Testing Machine Factory); Electric oven(KWS0709J-02H(XP)).
2.2. Preparation
Silicon carbide coating solution: The phenolic resin and the solvent N,N-dimethylformamide (DMF) were mixed uniformly, and micron-sized silicon carbide powder was added to make silicon carbide solutions with 200g/L, 400g/L and 600g/L respectively.

Silicon carbide coating sample: A suitable amount of silicon carbide solution was dropped from a dropper onto a Teflon plate to form a circle with a diameter greater than 12 mm.

Iron sheet coated with silicon carbide: First, stir the pre-configured silicon carbide coating solution evenly with a stirrer, then, the 200g/L, 400g/L, and 600g/L silicon carbide coating solutions are respectively applied to a plurality of iron sheets by the SZQ type applicator. Then heat them in an electric oven. Set 5 groups for each composite due to different coating thickness. The coating solution of 200g/l is set as group A; the coating solution of 400g/l is set as group B; and the coating solution of 600g/l is set as group C. Grouped as shown in the following table:

| Number | A1    | A2    | A3    | A4    | A5 | B1   | B2   | B3   |
|--------|-------|-------|-------|-------|----|------|------|------|
| Thickness (mm) | 0.1032 | 0.1553 | 0.2022 | 0.2543 | 0.3019 | 0.1041 | 0.1562 | 0.2013 |

2.3. Testing of dielectric performance parameters.
The test process is carried out in two cases, at a normal temperature of 19 ℃ and temperature rise of 30 ℃~120 ℃.

Before the normal temperature test, the RF coaxial extension cable needs to be removed from the heating furnace; The measurement frequency range from 1MHz to 3GHz, the interval is 5MHz, and the data is tested and recorded.

In the heating experiment, the furnace is heated to a specified temperature for a period of time. When the temperature sensor measures the temperature of the sample to reach the test temperature, the scanning measurement starts at the set frequency; after the measurement is completed, continue to heat up, maintain temperature, scan and measure until the last temperature point. After the measurement is completed, save the measurement data. The temperature rise is measured at a temperature interval of 5 ℃, and the measurement frequency is 2450 MHz. Test and record the data.

2.4. Study on Temperature Field of Microwave Heating Rubber Composite
The rubber sheets are made into sheets of 70 mm in length, 70 mm in width and 6 mm in thickness. Take two pieces of rubber sheet for each test, the iron piece is placed in the center of the two rubber pieces, and two pieces of rubber piece are wrapped around the piece of iron. The temperature measurement point is located at the outer edge of the iron piece in the two rubber sheets. The rubber sheets are placed in the microwave generator, and turn on the fiber temperature online monitoring device, then start measuring and recording the temperature data. The heating power is 100W, and the data is recorded every second. The heating is stopped when the temperature sensor detects that the temperature reaches 100 ℃. Observing whether there is sparking during the experiment, and study the temperature rise law of the iron carbide rubber composite containing different compositions of silicon carbide.
3. Results and discussion

3.1. Effect of Microwave Frequency on Dielectric Properties of Silicon Carbide Materials

![Figure 1](image1.png)

**Fig. 1** The $\varepsilon'$ of different content silicon carbide coating material

As can be seen from Figure 1, at low frequency of the microwave, the $\varepsilon'$ is relatively stable; when the frequency increases to a certain value, the $\varepsilon'$ increases sharply and a peak appears. And the higher the silicon carbide content, the earlier the peak appears. As the frequency continues to increase, the value drops sharply and then gradually stabilizes.

![Figure 2](image2.png)

**Fig. 2** The $\varepsilon''$ of different content silicon carbide coating materials

As can be seen from Figure 2, as the microwave frequency increases, three curves are roughly the same. At the low frequency of the microwave, the $\varepsilon''$ rises slowly, and the value of the 600g/L silicon carbide coating material is slightly larger than the other two. When the frequency increases to a specific value, since the polarization gradually cannot keep up with the electric field change, the loss gradually becomes larger, and thus the value increases sharply, and a peak appears. And 600g/L silicon carbide coating material peaks earlier than the other two. As the frequency continues to increase, the value drops abruptly and then slowly returns to a plateau value.
Fig. 3 The tanδ of different content silicon carbide coating material

As can be seen from Figure 3, the trend of the three contents of the silicon carbide coating material is consistent. First, it is relatively stable. As the frequency continues to increase, the value of tanδ increases abruptly and a peak appears, and the higher the silicon carbide content, the earlier the peak appears. This indicates that as the content of silicon carbide in the coating material increases, the tanδ of the silicon carbide coating material becomes larger.

3.2. Effect of Temperature on Dielectric Properties of Silicon Carbide Materials

Fig. 4 The effect of temperature on ε of three different contents of silicon carbide coating at 2450MHz

As shown in Figure 4, at the microwave frequency of 2450 MHz, the ε of three different contents of SiC coating materials are decreasing from 30 °C to 120 °C.

The ε of 200g/l SIC coating material is higher than the other two.
Fig. 5 The effect of temperature on $\varepsilon^*$ about three different components of silicon carbide coating at 2450MHz

As can be seen from Figure 5, at the microwave frequency of 2450MHz, the $\varepsilon^*$ of three different contents of SiC coating materials decrease with increasing temperature. Among them, 200g/L coating material curve has the largest slope, indicating that the $\varepsilon^*$ of the coating material of this composite is sensitive to temperature change at this frequency. The $\varepsilon^*$ of the coating material of 200 g/L is greater than the other two, it is indicated that it has a good microwave dissipation capability.

Fig. 6 The effect of temperature on $\tan\delta$ about three different components of silicon carbide coating at 2450MHz

It can be seen from Fig. 6 that when the microwave frequency is 2450MHz, the $\tan\delta$ of three different compositions of silicon carbide coating materials increase with increase of temperature. The smaller the content of silicon carbide coating, the larger the value. That is, at the microwave frequency of 2450MHz, 200 g/L silicon carbide coating material absorbs microwaves more easily, and 600 g/L silicon carbide coating material is more difficult to absorb microwaves.

3.3. Temperature field study of rubber composite
Microwave heating experiment was carried out on the rubber composites containing iron sheets. During the first few seconds of the experiment, there was an arc sound in the cavity of the microwave generator. At the same time, the ventilator of the microwave generator emits a pungent smell of scorching. The
fiber optic thermometer detected a sharp up and down fluctuation in temperature. At this time, stop heating and took out the rubber sheet, and it was found that the rubber was obviously scorched. In combination with the arc sound heard and the smell of the burnt rubber smelled before, it can be judged that the rubber composite has a "discharge and spark" phenomenon. The charred rubber in microwave field can be seen in figure 7.

Fig. 7 The charred rubber in microwave field

The temperature rise law of the rubber composites containing silicon carbide coated iron sheet measured by the experiment was obtained. The results are as follows:

Fig. 8 Temperature rise curve of rubber composites with different thickness silicon carbide coating  
(The content of silicon carbide is 200g/L)

Fig. 9 Temperature rise curve of rubber composites with different thickness silicon carbide coating  
(The content of silicon carbide is 400g/L)
Fig. 10 Temperature rise curve of rubber composites with different thickness silicon carbide coating (The content of silicon carbide is 600g/L)

It can be seen that there is no “ignition” phenomenon in the rubber composites coated with the silicon carbide coating during microwave heating process. And the thickness of the silicon carbide coating has a significant effect on the temperature of the rubber composites: The thicker the silicon carbide coating, the higher the temperature of the composites. This is because the silicon carbide coating can absorb microwave energy and convert it into heat energy. The thicker the coating, the higher the silicon carbide content, the more the microwave absorption, so the larger the microwave energy to be converted, the higher the temperature of the composites.

4. Conclusion
The purpose of this study is to investigate the effect of silicon carbide coating materials on the temperature distribution of rubber composites. The conclusions obtained are as follows.

(1) At room temperature, in the microwave frequency range of 1MHz to 3GHz, the dielectric properties of the silicon carbide coating material do not change much at low frequencies. When the frequency increases to a certain value, the dielectric parameter peaks and then fall sharply, and finally stabilize. And the higher the silicon carbide content, the larger the parameter.

(2) At the microwave frequency of 2450MHz, in the temperature range of 30 °C to 120 °C, the $\varepsilon'$ and the $\varepsilon''$ of three different compositions of silicon carbide coating materials are continuously decreasing. And to a certain extent, the larger the silicon carbide content, the smaller the value. And the tan$\delta$ increases with increasing temperature, indicating that the ability of the material to absorb microwaves increases with increasing temperature, and to a certain extent, the lower the content of silicon carbide coating, the stronger the ability of the coating material to absorb microwaves.

(3) The rubber composites coated with silicon carbide coating material heating by microwave is studied and the conclusions are as follows:
There is no “ignition” phenomenon in the rubber composite coated with the silicon carbide coating during microwave heating. It is indicated that the absorbing material coated with silicon carbide can effectively prevent the phenomenon of "ignition". And the thicker the silicon carbide coating, the higher the temperature of the composite.

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