Research on the Influence of Powder and Its Surface Modification to the Flame Retardant, Electric Performance and Mechanical Properties of Silicone Potting Material

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Abstract. A silicone resin as base material, composite powder as filler, and the electronic potting silicone material was prepared using a silane coupling agent γ-3(trimethylsilyl) propyl methacrylate (KH-570) to do surface treatment. The influence of surface modification and its amount on the flame retardant, electric performance and mechanical properties of silicone potting material was studied in this paper. The developed organic silicon electronic potting materials with good flame retardant, electric performance, and mechanical performance, could be used in the high voltage grade of products, and meet the electrical performance requirements of an arrester.

Introduction

With the rapid development of microelectronics technology, electronic components have been developed towards miniaturization, integration, ultra thin and high performance. In order to ensure the normal work of the electronic components from the interference of outside dust, moisture, shock, vibration, lightning and chemical substances, electronic components need package or insulation sealing protection[1-3]. Therefore, a lot of researches have been carried on flame retardant, electric performance and mechanical performance of electronic potting material [4-6], and the requirement of comprehensive performance is still increasing.

A silicone resin as base material, composite powder as filler, and the electronic potting silicone material was prepared using a silane coupling agent γ-3(trimethylsilyl) propyl methacrylate (KH-570) to do surface treatment. The influence of surface modification and its amount on the flame retardant, electric performance and mechanical properties of silicone potting material was studied in this paper. The developed organic silicon electronic potting materials with good flame retardant, electric performance, and mechanical performance, could be used in the high voltage grade of products, and meet the electrical performance requirements of an arrester.

Experiment

Materials and Equipment

501 silicone rubber and M, N components are from Zhonghao Chenguang Research Institute; alumina powder, aluminum nitride powder are from Foshan Victor chemical materials Co. Ltd.; high-speed mixing dispersion machine(ESJ-300) is from Shanghai Yi Le Mechanical and Electrical Equipment Co., Ltd.; vacuum drying box(DZ-BC) is from Tianjin Yin Sai Technology Development Co. Ltd.; thermal conductivity meter (HC-110) is from EKO Company; rotary viscometer(NDJ-4) is from Shanghai Ping Xuan Scientific Instrument Co. Ltd.; shaw A hardness tester is from Shanghai Luchuan measuring tool Co., Ltd.; universal testing machine INSTRON 1185, Instron corporation;

impedance analyzer(E4991A) is from USA Agilent Technology Co. Ltd..

Performance Test

Viscosity was tested by rotary viscometer according to GB/T 2794-1995; thermal conductivity was measured according to GB/T 11205-2009; the tensile strength and elongation at break were measured according to GB/T 528-2009; the relative dielectric constant was measured according to GB/T 1409-2006; volume resistivity was measured according to GB/T 1410-2006.

Surface Treatment of Powder

After two hours' preliminary drying at 110 °C, a certain amount of alumina, aluminum hydroxide powder, quartz powder and silica powder were respectively added into high-speed mixing dispersion machine. Under high speed stirring, 0.26% ethanol solution of KH - 570 (the mass ratio of KH-570 to ethanol was 1:1) was added in the form of spray to do surface treatment, the powder is then placed in a vacuum drying oven at 100 °C for 40min to obtain modified powder.

Different mass ratio of alumina powder, aluminum hydroxide powder, quartz powder and silica powder were mixed to obtain a composite powder, and composite proportion of modified powder was shown in Table 1.

Table 1 Composite proportion

	Quartz powder	Aluminum hydroxide powder	Alumina powder	Silicon powder
Composite powder1	1	1	1	1
Composite powder2	1	2	1	1
Composite powder3	2	2	1	1

After surface modification, the modified composite powders 1,2,3 were denoted by C_i and the unmodified composite powders 1,2,3 were denoted as W_i.

Preparation of Two-component Mixture

A certain amount of composite powders C_i and W_i was added to 100 parts of M, N components in silicone rubber, even high-speed dispersion, then made of a matrix material are referred to as base material C_i' and W_i'.

Sample Preparation

The base material C_i' and W_i' were blended and mixed uniformly according to the quality ratio of the 1:1 and the bubble was taken off. Curing conditions was a temperature of 80°C±1, a relative humidity of 50%±5%, whereby the potting material A_i and B_i were obtained. Potting compound A_i was prepared by the composite powders after surface modification, and potting compound B_i was prepared by the unmodified composite powders.

Result and Conclusion

Effect of Powder Content on the Mechanical Properties of Silicone Electronic Potting Material

As shown in Fig.1, with the increase of the dosage of the composite powder, the tensile strength of the potting material was first increased and then decreased, and a maximum value occured when the composite powder was 190 parts. This was due to the interaction between the powder and silicone rubber increased with the increase of the amount of powder, so the tensile strength of the potting material improved. But when the powder amount was more than 190 copies, due to the increased viscosity of potting material and encapsulant, resulting in local agglomeration of powder body, leading to a decrease in the tensile strength of the silicone potting material. In the same amount of powder, the tensile strength added composite powders after the surface modification was higher than that added unmodified composite powders. This was due to the powder after surface treatment, the compatibility of the powder and silicone rubber was improved obviously, the dispersion of the powder was improved, and the interaction force of the interface was enhanced.

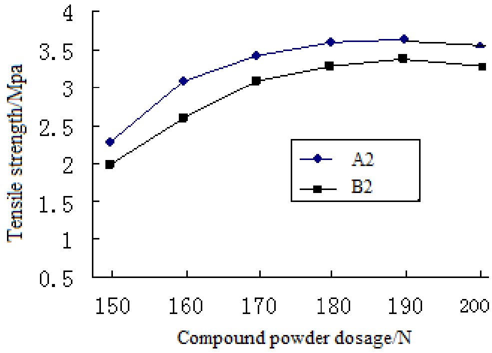

Fig.1. The relationship between the composite powder content and tensile strength of silicone potting material

Effect of Powder on the Electrical Properties and Flame Retardant of Electronic Potting Material

In 100 parts of M, N silicone components, composite powders were added to the test study, and Table 2 was the test results of the cured product, which was added 190 copies modified composite powders C_2.

Table 2 The measurement results of electrical properties and flame retardant of potting material

Performance	Measurement results	Measurement basis
Appearance	White/gray elastomer	Visualization
Dielectric loss tanW	$(1.21\sim3.0)\times10^{-3}$	GB1410-89
Flame retardant grade (Vertical method)	FV-0	GB/ T13448-89
Volume resistivity (Ψ· cm)	$(1.7\sim2.4)\times10^{14}$	GB/T1692-92
Relative permittivity	2. 1~2. 5	GB1409-88
Breakdown strength (kV /mm)	18~21	GB1408-89

Experimental studies have shown that, since H_2O molecules was produced by $Al(OH)_3$ decomposited at high temperature, which could absorb the heat generated in combustion, $Al(OH)_3$ were helpful to improve flame retardant effect of the potting material. The study also showed that $Al(OH)_3$ had less effect on the dielectric loss of silicone potting material, but rubber thickening effect of $Al(OH)_3$ was obvious, while the thickening effect of quartz powder filler was lesser. Rubber vacuum exhaust bubble was easy by adding some quartz powder filler, at the same time, the flame retardant performance was better, and the influence on basic material of dielectric loss was less. In conclusion, the study demonstrated potting material prepared by powder composite C_2 could better satisfy the product for filling material electrical properties and process performance requirements.

Insulation is an important performance of silicone potting material, which is usually characterized by a volume resistivity. The higher volume resistivity of potting materials, the better the insulation. Effect of powder amount on volume resistivity of the potting material as shown in Fig. 2.

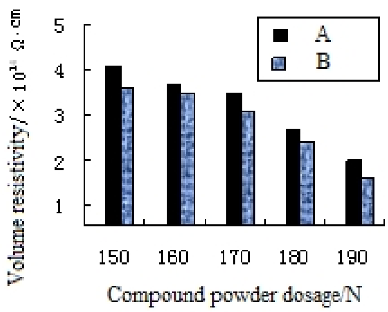

Fig.2. Effect of powder content on volume resistivity of potting material

As shown in Fig.2, along with the increase of the powder content, volume resistivity of potting material was gradually reduced. The reason was that the volume resistivity of the silicon powder was lower than silicon rubber. In the same amount of powder, the volume resistivity of the potting material prepared by powder after surface modification was higher than that of the potting material prepared by unmodified powder. The reason was that the powder after coupling agent surface treatment could increase interface bonding of powder and the base material, so that the silicone resin inter-molecular force was enhanced. The activity of segment was reduced, while the conductivity decreases, and the volume resistivity increases.

Influence of Surface Modification of Powder on Viscosity and Stability of Potting Material
The influence of modified and unmodified powder and their contents on the viscosity of potting materials as shown in Fig.3.

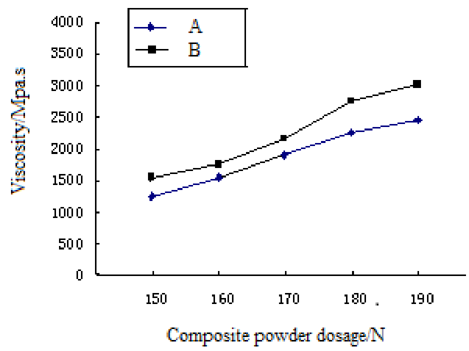

Fig.3. The relations between the content of addition of modified and unmodified powder and viscosity of potting material

Fig.3 shows that with the increase of the content of the powder, the viscosity of the potting material increases, the viscosity of the potting material prepared by the powders after surface treatment was significantly lower than that prepared by the unmodified powder under the same power content. The reason was that there was hydroxyl on the surface of the powder, and chemical bonding and physical adsorption occurred between hydroxyl and silicone of potting compound. Therefore, the viscosity of potting compound was increased.With the increase of the content of the powder, the interaction between powder and silicon rubber also increased, resulting in rising viscosity of potting material. After power modified by silane coupling agent KH-570, its surface active hydroxyl decreased significantly, the powder surface energy and surface polarity was reduced, and the interaction between the powder and silicon rubber force was reduced. As a result, the viscosity of the potting compound decreased. At the same time, the storage stability of the potting material increased.

Effect of Composite Powder Content on the Thermal Conductivity of Electronic Potting Material
Effect of composite powder content on thermal conductivity of silicone potting material as shown in Fig.4. With the increase of the volume fraction of the composite powder, the thermal conductivity of potting material increased significantly. Due to the thermal conductivity of matrix was far less than that of filler, filling volume was low, the increasement of thermal conductivity rate of the potting material was not significant. Because of the low quantity of fillings, most powder particles failed to contact directly, the high thermal conductivity of the filler powder could not fully played. With a further increase in the content of the composite powder, the distance reduced between the particle, the heat transfer resistance reduced and thermal conductivity rate of potting material was growing faster. Because high filling amount of the powder, effective thermal conductive network which made by powder particles formed in the resin, and heat could be passed along the network. At this time the high thermal conductivity of composite powder filler full played, further increasing the amount of composite powders, the thermal conductivity of the potting material growed slowly. From Fig.4 also showed that, in the same amount of powder, the thermal conductivity of the potting compound A_i was higer than that of the potting compound B_i, this is because the powder surface coupling modification,

which improved the interfacial compatibility of the silicone rubber and potting material, and reduced the interface defects, the possibility of the gap, and the thermal resistance of the system.

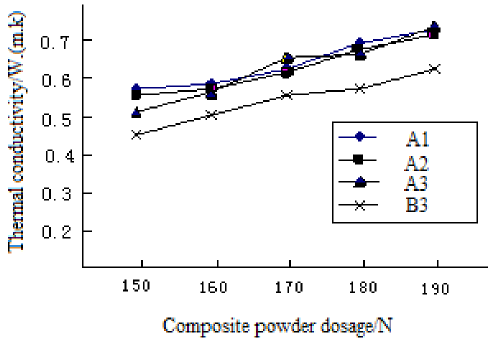

Fig.4. Effect of composite powder content on thermal conductivity of silicone potting material

Conclusions

Powder after surface modification can improve insulating, tensile strength, thermal conductivity rate, and storage stability of silicone potting material. With the increasing amount of composite powder, viscosity and thermal conductivity of silicone potting material increases, volume resistivity decreases. With the increasing amount of composite powders, tensile strength increases at first and then decreases. The silicon electronic potting materials after adding 190 copies compound powder modified by KH-570 have good flame retardant property, electric performance and mechanical performance, which can be used in the high voltage grade of products, and meet the electrical performance requirements of the arrester.

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