

Study on Fire Resistance Performance of Fireproof Glass for Flame Retardant

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Abstract. For mica, low-melting glass powder and flame retardant added to silicone rubber fireproof glass, the fire resistance of fireproof glass after adding different materials was studied. It was found that the cross linking effect was the best when the amount of DCP added with vulcanizing agent was 1wt%. After adding mica, the tensile strength of glass burning decreased, and the elongation at break was the maximum at 30wt%, reaching 97.9%. At the same time, after the burning of the ceramic material, the strength is more rigid. After the combustion of the silicone rubber system, it gradually changes from the collision state to the contracted state, and the critical point of the morphological change is 50 wt%, mica. At the same time, the layered eutectic structure was observed by scanning electron microscopy. The mica content was higher and the eutectic state was denser. For this reason, the mica content was 40 wt%. After the addition of the low-melting glass flit, the mechanical properties are worse, and the volume of the ceramics shrinks faster, but the strength is increased and the heat resistance is better. The test data found that after the addition of the flame retardant, the content of MH decreased after the addition of the flame retardant synergism, although the mechanical properties decreased slightly, but the flame retardant properties remained unchanged. In addition, the TGA test and solvent resistance test data show that the thermal stability and solvent resistance of the synergistic flame retardant system with APP addition is better than other synergistic systems.

1. Introduction

Silicone rubber burns slowly, there are no droplets, and no toxic gases are released [1]. Because of its good combustion characteristics, silicone rubber has great advantages in fire protection and flame retardant applications. As a new type of fireproof material, dramatized silicone rubber material has the same properties as ordinary polymer materials at room temperature, and can form a dense and hard ceramic layer at high temperatures, which can hinder the further spread of flame [2]. In recent years, the research of dramatized silicone rubber materials has attracted the interest of many experts and scholars [3]. Wang Zhe et al. [4] selected mica and glass powder as fillers and found that glass powder greatly improved the ceramic properties of silicone rubber. To this end, this paper by adding inorganic mineral fillers, flame retardants and additives to silicone rubber. Optimize the processing conditions and the best ratio of various added fillers to obtain fire-retardant silicone rubber cable materials. At the same time, the UV-ray technology was used to crosslink the silicone rubber/polyolefin composite cable material.



By optimizing the optimal conditions of UV cross linking, the structure and properties of the silicone rubber before and after cross-linking were studied. A good flame retardant applied to building fire glass materials.

2. Experimental materials

2.1. Experimental raw materials

Mixed silicone rubber (MVQ, grade XT141); dicumyl peroxide (DCP); magnesium hydroxide (MH); mica, grade GA-5; low melting glass powder (softening point 350°C, melting point 500°C, mesh Number 3000, grade DZ855A); melamine cyanurate (MCA); melamine polyphosphate (MPP); polyphosphoric acid according to C APP; expanded graphite (EG).

2.2. Sample production

A certain amount of silicone rubber is placed on a SK-110 two-roll refining (glue) machine for mixing, and an inorganic filler and a vulcanizing agent dicumyl peroxide (CDCP) are added thereto, and the mixture is uniformly kneaded at room temperature, and then placed. On the flat vulcanizer, at 180°C, vulcanization for 10 min, the samples of 1 mm, 2mm and 3 mm required for the experiment were prepared and tested for relevant properties.

3. Experimental methods

Oxygen Index (LOI): Performed on an HC-2 type oxygen indexer according to ASTM D2863-77, sample size 100×6.5×3mm³.

Vertical Combustion (UL-94): One of the most widely used test methods for internationally recognized flame retardant materials. The test was carried out on a CZF-1 vertical burner according to the ASTM D625-77 standard, and the sample size was 127×12.7×3 mm³.

Scanning Electron Microscopy (SEM): The SEM image was plated with a gold film before observation on a JEOL JSM-6700E scanning electron microscope [5].

Gravimeter Analysis (TGA): The TGA curve was obtained on a TGA-SOH thermometer from room temperature to 900°C at a temperature ramp rate of 10°C / min with an air flow rate of 6×10⁻⁵ m³ /min.

Solvent resistance test: About 1 g of the silicone rubber material sample was placed in a 50 ml toluene solution, and placed at a temperature of 25°C for 120 hours, and the mass change before and after the placement was compared.

4. Analysis of experimental results

4.1. Influence of the content of modicum peroxide (DCP) of flame retardant on the mechanical properties of glass silica gel

Different amounts of DCP were added to the glass silica gel, and a 1 mm sample was vulcanized on a flat plate vulcanization to test the mechanical properties [6]. The results are shown in Figure 1 below.

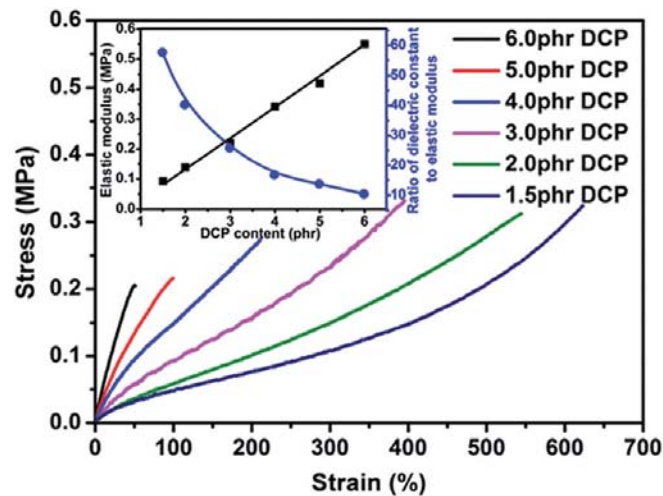


Figure 1. Effect of DCP content of vulcanizing agent on mechanical properties of silicone rubber

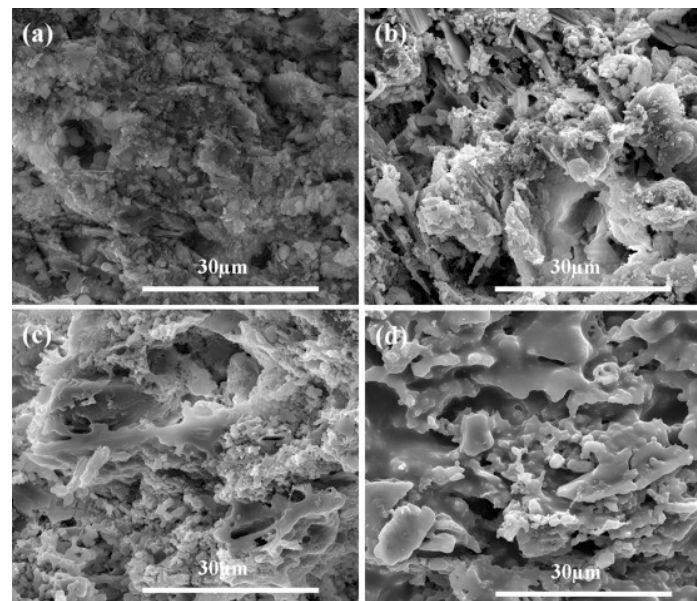
It can be seen from Fig. 1 that the tensile strength of the glass silica gel first increases with the increase of the DCP content of the vulcanizing agent. When the DCP is 1.5 wt%, the tensile strength of the glass silica gel reaches a maximum of 0.2 M Pa. Because under the action of DCP, the system forms a three-dimensional network structure, which makes it difficult to cause relative slip between macro molecular chains, resulting in an increase in the interaction force between the polymer material chains, and an increase in tensile strength, DCP [7]. After the content exceeds 3 wt%, the tensile strength decreases significantly. This is because the glass silica gel material cross links to form more cross-linking points, which greatly hinders the movement of the molecular chain. Therefore, the tensile strength first rises and then falls. It can be seen from Fig. 1 that as the content of DCP increases, the elongation at break of the glass silica gel material tends to increase slowly. This is because as the amount of DCP of the vulcanizing agent increases, the degree of cross linking of the material is increased, the density of the cross-linking point of the material is increased, and the chain is shortened, which restricts the movement of the chain. Therefore, the elongation at break of the material tends to decrease. It can be seen that the cross-linking effect is best when DCP is 3 Wt%.

4.2. Effect of mica content on the properties of glass silica gel

It can be seen from Table 1 that the strength of the residue after bantering of the glass silica gel system to which mica is added is increased, and the hardness of the residue after bantering of the glass silica gel system after the mica content exceeds 30% by weight is hard [8]. This is because at low levels of mica, the bantering effect is not obvious, and the continuity of the residue is not high; when the mica content is high, the bantering effect is remarkable, the continuity of the residue after bantering is improved, and the hardness of the residue is improved. The change in the size of the residue, as the mica content increased, the size expansion of the residue decreased from 4.9 to 0.2, and the residue changed from expansion to contraction at a mica content of 50% by weight. This is because at a high temperature (e.g., 1000 °C, the silica produced by the combustion of the glass silica gel or the added inorganic filler forms a eutectic with the melting of the mica edge portion.

Table 1. Effect of different content of mica on the strength and size change of silicone rubber after firing

project name	Mica content (Wt %)					
	0	10	20	30	40	50
Before burning (mm)	21.4	21.5	22.0	21.5	21.0	21.5
After burning (mm)	26.3	24.5	23.0	22.2	21.2	20.5
Secondary contraction	+4.9	+3.0	+1.0	+0.7	+0.2	-1.0
Residual strength	Poor strength	hard	hard	hard	hard	hard

**Figure 2.** Scanning electron demographic of the residue after bantering of different content of mica

As can be seen from Fig. 2, in the absence of the addition of the mica inorganic filler, no peeling layered eutectic structure was observed in (a). When the mica content is 1% by weight, the layered eutectic structure with peeling can be clearly seen in (b), and the density of the stripped eutectic structure increases as the mica content increases, (d) the graph shows that the eutectic structure has the highest density when the mica content is 40% by weight. This also explains that as the content of mica increases, the size expansion of the residue decreases [9].

4.3. Effect of low melting point glass powder on the related properties of glass silica composites

Different amounts of low-melting glass flit were added to the silicone rubber material containing 30wt% of mica, wherein the vulcanizing agent DCP content was 1 wt%. Vulcanization is used to prepare the samples required for the experiment. The porcelain performance test includes three aspects: porcelain temperature measurement, porcelain thermal shock resistance and compactness test. (1) Porcelain temperature: The prepared sheet was cut into a sample of about 2 cm×2.5 cm, and the temperature was maintained for 10 min in a muffle furnace, and then taken out, and judged by thermal shock results and strength. Whether it has been porcelain [10]. (2) Thermal shock resistance: The same size method is used for the porcelain treatment. The sample is taken out from the muffle furnace and placed directly in the tap water to observe whether the sample is broken or not. Determine the thermal shock resistance of the material. (3) Declassification: The water droplets were dropped on the treated sample, and the penetration speed of the water was observed to judge the compactness of the sample.

Table 2. Effect of the amount of low-melting glass powder on the thermal shock resistance of porcelain

Temperature / °C	Low melting glass powder content (wt %)					
	0	10	20	30	40	50
600	A	A	A	A	A	A
650	A	A	A	A	A	A
700	A	A	A	B	B	B
750	A	A	A	B	B	B
800	A	A	A	B	B	B
850	A	A	B	B	B	B
900	A	A	B	C	C	C
950	A	B	C	C	C	C
1000	B	C	C	C	C	C

Table 2 shows the effect of the amount of low-melting glass flit on the thermal shock resistance of the porcelain. When the heat treatment temperature is low, the samples of each system are thermally shocked and easily pulverized. The glass powder system without adding a low melting point appeared to be water-free at 1000°C. This is the mica added in the system that begins to melt and hinder at 1000°C. The 10wt% low-melting glass powder system was added to the water at a temperature of 950°C. This is because the low-melting glass flit is low in content, the bantering effect is not significant, and the interbreed porcelain is discontinuous. When the amount of the glass flit at 700°C was 30 wt%, 40 wt% and 50 wt%, the sample was not pulverized, and the silicone rubber system containing 20 wt% of the low-melting glass flit was still pulverized [11]. After that, at 800°C, the samples of each system were not powdered. However, after the temperature exceeds 900°C, the sample will be brittle when the water strength is reduced; at 950°C, the sample will start to burst; at 1000°C, the sample will burst more. This is because under a relatively small amount of low-melting glass flit, the liquid phase formed under low temperature conditions has less bantering effect, so that the bonding effect on SiO₂ is weak, and the residue after bantering is easy to be pulverized and strength. Smaller. When the bantering temperature is increased, the formed liquid phase is more interbreed, and the strength of the interbreed ceramics is increased and no longer pulverized. Under the condition of very high temperature, the thermal shock damages the interbreed porcelain, and the interbreed porcelain is easily broken.

4.4. Effect of magnesium hydroxide content on mechanical properties and flame retardation of silicone rubber

Different amounts of magnesium hydroxide were added to the silicone rubber system, and 1 wt% of the vulcanizing agent DCP was added to prepare samples of 1 mm and 3 mm for performance test.

Table 3. Effect of MH content on vertical combustion of silicone rubber

project name	MH content (Wt %)					
	0	40	45	50	55	60
UL-94	fail	V-1	V-0	V-0	V-0	V-0

It can be seen from Table 3 that the silicone rubber system without MH cannot pass the UL-94 test, and the silicone rubber system with 40 wt% MH can pass the V1 rating in the UL-94 test. The silicone rubber system after the MH content exceeds 45 wt% can pass the V-0 rating of the UL-94 test.

4.5. Effect of different allergists on mechanical properties and flame retardation of silicone rubber composites

Table 4. Formulation of different flame retardant allergists

sample name	Different flame retardant content (Wt %)				
	MH	APP	MCA	MPP	EG
SMG-0	0	0	0	0	0
SMG-1	50	0	0	0	0
SMG-2	44	6	0	0	0
SMG-3	44	0	6	0	0
SMG-4	44	0	0	6	0
SMG-5	44	0	0	0	6

Table 5. Flame retardant properties of silicone rubber composites

sample name	Flame retardant performance	
	LOI (%)	UL-94
SMG-0	34	Fail
SMG-1	53	V-0
SMG-2	46	V-0
SMG-3	43	V-0
SMG-4	44	V-0

It can also be seen from Table 2.7 that the SMG-0 sample without flame retardant has an LOI value of 34%, cannot pass the UL-94 test, and can pass the UL-94 test after adding the flame retardant. The LOI value increased from 34% to 53% after MH was added. The same amount of synergism APP was added. The LOI values of the samples after MCA and MPP were 46%, 43% and 44%, and the LOI values did not change much. However, after adding EG, the sample has a LOI value of more than 60 and a good flame retardant effect.

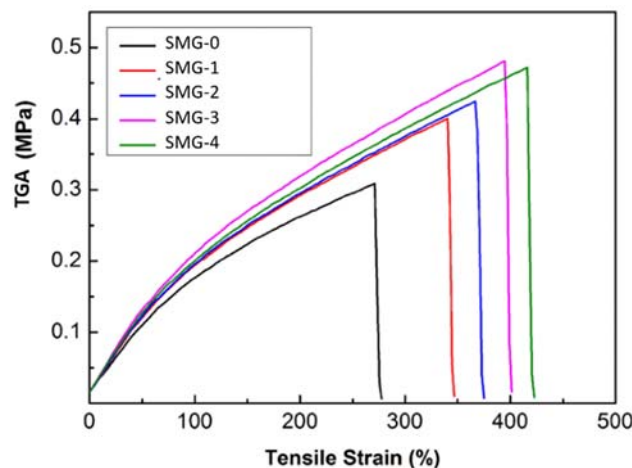


Figure 3. TGA curve of the sample

When the mixture is heated, the decomposition of APP and MCA occurs first, and the generated hydrolysis product reacts with MH, which hinders the hydrolysis process of APP and MCA, thus affecting the hydrolysis of MH itself. It is currently accepted that the reaction product of MH and APP, MCA, or both, is magnesium phosphate. It is the further thermal decomposition of magnesium

phosphate that produces the second-order weight loss on the TGA curve of the sample, which is also the reason for the high efficiency flame retardant effect. The thermal degradation peak temperature of APP is (366°C) 11°C higher than the thermal degradation temperature (355°C) of MCA, indicating that they can improve the thermal stability of silicone rubber composites, but the thermal stability of APP in the synergistic system. The best.

5. Conclusion

The paper found through experiments that the content of low-melting glass powder is 30wt%. In terms of low-temperature flame retardation, the addition of MH of the flame retardant deteriorates the mechanical properties, but the flame retardant properties are improved. It is most suitable at a 50 wt% a content. In addition, the effects of different allergists on the flame retardation and mechanical properties of the system were analyzed. It was found that the flame retardant performance was improved and the mechanical properties were lower than those without any flame retardant, but compared with the addition of flame retardant MH system. The mechanical properties did not change much, and the flame retardant performance changed little. Therefore, the addition of the synergism decreased the MH content. Although the mechanical properties decreased slightly, the flame retardant properties remained unchanged. Through TGA test, it was found that the thermal stability of APP added in the synergistic system was the best. The solvent resistance test found that the solvent resistance of the system with different flame retardants was reduced, and the system with APP added had the smallest reduction.

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