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Significant improvement of urethane-containing silane on the tracking and erosion resistance of silicone rubber/silica nanocomposite by enhancing the interfacial effect



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ABSTRACT

Rapid crosslinking of silicone chains on the nanosilica surface during arc discharge is proposed as an efficient strategy to improve the tracking and erosion resistance of silicone rubber. Urethane-containing silane (URS) was synthesized by using (3-isocyanatopropyl) triethoxysilane, isopropyl alcohol and allyl alcohol via nucleophilic addition and trans-etherification. The suppression effect and mechanism of URS on the tracking and erosion of addition-cure liquid silicone rubber/silica (ALSR/SiO₂) nanocomposite were investigated. It was found that URS could significantly enhance the tracking and erosion resistance of ALSR/SiO₂. When only 2.5 phr URS was added, all the ALSR/SiO₂/URS specimens passed the inclined plane test at 4.5 kV and the erosion rate decreased from 3.44% to 0.25%. This might be because URS tightly constrained silicone chains on the SiO₂ surface by strong hydrogen-bonding interactions. During arc discharge, URS at the interphase enhanced the catalytic activity of platinum compound to promote the radical crosslinking of silicone chains absorbed on the SiO₂ surface. Hence, a compact and intact ceramic layer was quickly formed on the ALSR surface and protected the underlying materials from further arc ablation. Our finding provides a new method and theoretical basis for the design, preparation and application of the novel high-performance polymer insulating material.

1. Introduction

Silicone rubber (SR) is increasingly used in the high-voltage insulation due to its light weight, high vandal resistance, excellent insulating property, superior hydrophobicity and self-recovery of hydrophobicity [1–3]. However, when subjected to dry band arcing, SR inevitably degrades and even forms a carbonaceous track or erosion on the surface [4]. Once tracking occurs, the electrical insulation of SR is damaged, which seriously threatens the security of the power system [5,6]. Incorporating micron-sized aluminum hydroxide (ATH) is the common method to improve the tracking and erosion resistance of SR [7–10]. However, to achieve satisfactory tracking and erosion resistance for outdoor insulation, 40–60 wt% ATH is necessary, which severely deteriorates the hydrophobicity, mechanical properties and processability of SR [11,12].

How to effectively enhance the tracking and erosion resistance of SR is an enormous challenge in the high voltage outdoors insulation field [13]. The development of nanotechnology provides a new way to solve this issue. Among different nano fillers, nano silica (SiO₂) has attracted

the most concern due to its superior physical reinforcement effect for SR. Liu found that incorporating 1 wt% nano SiO₂ could improve the tracking resistance of SR [14]. During arc discharge, the SR/SiO₂ nanocomposite could form a ceramic-like layer on the SR surface, which inhibited the further degradation of silicone chains to some extent. However, with increasing the SiO₂ content, the tracking and erosion resistance of the SR/SiO₂ nanocomposite had a limited improvement [15]. This was mainly attributed to the two following aspects. On the one hand, when suffering from dry band arcing, some local temperature on the SR surface exceeds 400 °C. Accordingly, most of the interfacial interactions between SiO₂ and SR were destroyed [16]. On the other hand, with the filler addition increasing, SiO₂ agglomerates more and more seriously due to the strong interaction of the nanofillers [17]. Therefore, when being struck by high-voltage arc, the ceramic layer formed on the SR surface was too weak to protect underlying material.

During arc discharge, thermal crosslinking of silicone chains also plays a vital role to improve the tracking and erosion resistance of SR [18–20]. It is an efficient strategy to improve the tracking and erosion resistance of SR by catalyzing the thermal crosslinking of silicone

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chains at the interphase between SR and nano silica. Our previous work has found that platinum (Pt) and nitrogen-containing silane had good synergism to promote the thermal crosslinking and suppress the degradation of silicone chains [21]. In addition, many researches have reported that nitrogen-containing compounds, especially urea- and urethane-containing compounds, could enhance the interfacial interactions between SiO₂ and heterochain polymers, such as epoxy resin and polypropylene glycol, by strong hydrogen-bonding interactions [22,23].

In this work, urethane-containing silane (URS) was synthesized to enhance the interfacial interactions between addition-cure liquid silicone rubber (ALSR) and SiO₂ by strong hydrogen-bonding interactions. The synergism of URS and platinum compound could also promote the thermal crosslinking of silicone chains. The effects of URS on the dispersion, the interfacial interactions of SiO₂, the thermal stability and the tracking resistance of ALSR/SiO₂ composite were intensively studied. A suppression mechanism of URS on the tracking and erosion of ALSR is also proposed. This work provides a new strategy to effectively improve the tracking and erosion resistance of SR for high-voltage insulation.

2. Experimental

2.1. Materials

(γ -Isocyanatopropyl) triethoxysilane (ICPES) was offered by Jiangsu Huasheng Fine Chemical Co., Ltd., China. Isopropyl alcohol (IPA) and allyl alcohol (AA) were obtained from Xiya Chemical Reagent Co., Ltd., China. Tetrabutyl titanate (TBT) was purchased from Tianjin Fuchen Chemical Reagent Factory, China. Vinyl-terminated poly(dimethylsiloxanes) (VPDMS, viscosity was 20,000 mPa·s and vinyl content was 0.23 wt%), poly(hydromethylsiloxane) (PHMS, viscosity was 160 mPa·s and hydride content was 0.75 wt%) and platinum (0)-1,3-divinyl-1,1,3,3-tetramethyldisiloxane complex (Karstedt's catalyst, 2000 ppm) were obtained from Guangzhou Xiyou Technology Co., Ltd., China. Fumed silica with the average diameter of 12 nm was supplied by Tokuyama Co., Ltd., Japan. 1-Ethynylcyclohexanol as inhibitor was offered by Shenzhen Xinzeye Technology Co., Ltd., China.

2.2. Synthesis of urethane-containing silane

24.7 g ICPES (0.1 mol) and 6.6 g IPA (0.11 mol) were mixed in a 150 mL four-neck flask with stirring, and the reaction was maintained at 60 °C for 3 h under nitrogen atmosphere. After the reaction was finished, the unreacted IPA was removed by rotary evaporation at 50 °C to obtain (γ -isopropylurethanepropyl) triethoxysilane (IPUPTES), and the yield of IPUPTES was 97.3%.

17.4 g AA (0.3 mol) and 0.2 g TBT were mixed in a 150 mL four-neck flask with stirring for 15 min, and the mixture was heated to 70 °C. Subsequently, IPUPTES was added into the mixture and the reaction was kept at 70 °C for 5 h. Then, the reaction was maintained for another 1 h under reduced pressure. The unreacted AA and the byproduct ethanol were removed to obtain the urethane-containing silane (URS). The yield of URS was 89.6%. The vinyl content in URS was 17.3 wt%, which was determined by iodometric titration [24]. The synthetic scheme of URS is shown in Fig. 1 and the chemical structure of URS was determined by FTIR and ¹H-NMR, as shown in Fig. S1 and Fig. S2.

2.3. Preparation of ALSR/SiO₂/URS composites

V-PDMS, SiO₂ and URS were intensively mixed in a kneading machine at 30 °C for 6 h to obtain V-PDMS/SiO₂/URS mixture. Subsequently, PHMS, 1-ethynylcyclohexanol and Karstedt's catalyst (Pt) were added to the mixture and stirred vigorously for 20 min by an electric mixer. Finally, the mixture was vulcanized at 120 °C for 10 min under 8 MPa by a press vulcanizer to obtain the ALSR/SiO₂/URS

samples. The formula of the ALSR/SiO₂/URS samples is listed in Table 1. In consideration of the practical application in the insulation field [25], the SiO₂ content in ALSR was set to 40 phr. For comparison, ALSR and ALSR/URS samples without SiO₂ were also prepared.

2.4. Characterization

2.4.1. Inclined plane test

The tracking and erosion performance was carried out by an inclined plane tracking and erosion resistance test apparatus (DX8427, Dongguan Daxian Instruments Co., Ltd., China) according to IEC 60587–2003. A schematic diagram of inclined plane (IP) test setup is shown in Fig. 2. Five specimens were tested for each formulation. Each test specimen with the dimension of 120 × 50 × 6 mm³ was mounted at an inclination of 45°. The two electrodes were fixed on the surface of each test specimen at a distance of 50 mm. During the test, a constant AC voltage of 4.5 kV was applied to each specimen, along with a flow rate of 0.6 mL·min⁻¹ of standardized conductive solution (0.10 wt% NH₄Cl and 0.02 wt% isooctylphenoxypolyethoxyethanol). When the leakage current exceeded 60 mA for 2 s, the test apparatus would recognize this moment as the time to failure. After 6 h of IP test, a specimen without excess current would be regarded as passing the test. After the IP test, the eroded portion of the test specimen was cleared away, and the decreased mass of the specimen was recorded as the eroded mass. The erosion rate of the specimen was the ratio of eroded mass to the mass of the specimen.

2.4.2. Rheological measurements

Rheological measurements were carried out on a stress controlled rheometer (HR-2, TA Instruments, US) with a plate geometry (diameter 20 mm) at 25 °C. For frequency sweeps, temperature and strain were 30 °C and 1%, respectively. For temperature sweeps, frequency and strain were 10 rad/s and 1%, respectively.

2.4.3. Transmission electron microscopy (TEM)

The dispersion of SiO₂ in the nanocomposite was investigated using transmission electron microscopy (JEM-2100 F, Japan). The ultra-thin sections of the samples were prepared by ultramicrotomy (Leica Ultracut UCT) at -140 °C.

2.4.4. Swelling experiments

Swelling experiments were performed according to ASTM D 6814–02. The volume fraction of the rubber in the swollen gel (V_r) was determined on the basis of a simple additive rule of volumes according equation (1):

$$V_r = \frac{(D - fm_0) / \rho_r}{(D - fm_0) / \rho_r + A_0 / \rho_s} \quad (1)$$

where m_0 was the initial weight of the samples before swelling, D was the weight of the de-swollen test specimen, f was the weight fraction of the insoluble components, A_0 was the weight of the absorbed solvent, ρ_r was the density of the samples, and ρ_s was the density of the solvent.

2.4.5. Fourier transform infrared spectrometry (FTIR)

The liquid samples were painted on the surface of KBr pellets and spectra were recorded with a FTIR spectrometer (Tensor 27, Bruker Optics, Germany). The measurements were carried out in the region of 4000–400 cm⁻¹ and scanned 16 times for each sample. The resolution was 4 cm⁻¹.

2.4.6. Thermogravimetry (TG)

The thermogravimetry was carried out by using a TG analyzer (TG 209, Netzsch Instruments, Germany) from 30 to 900 °C at a linear heating rate of 20 °C min⁻¹ and an air flow rate of 30 mL·min⁻¹. The samples with a weight of 5–10 mg were measured in alumina crucibles.

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