

Enhancing UV Resistance in RTV: Preparation and Performance Evaluation of Nano ZnO-RTV Composites



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Abstract

In order to improve the UV resistance of room-temperature vulcanized silicone rubber (RTV), this study introduces a novel approach involving the modification of nano zinc oxide (ZnO) with a titanate coupling agent (PN). The modified nano ZnO was then incorporated into RTV to prepare nano ZnO-RTV composites at varying mass fractions. UV accelerated aging experiments were conducted on both pure RTV and nano ZnO-RTV composites to evaluate the impact of nano ZnO on RTV's UV resistance. The findings demonstrate a significant improvement in the UV shielding performance of RTV with the addition of nano ZnO; specifically, nano ZnO-RTV composites with a 1.0% mass fraction exhibited an increase in UV shielding efficiency of 72.63%. Additionally, the electrical strength of RTV was observed to slightly increase upon the addition of nano ZnO. After 2500 hours of UV aging, the reduction in static contact angle and the degree of aging reflected by the surface microstructure, were substantially less in nano ZnO-RTV composites compared to pure RTV. These observations align with the alterations in RTV molecular chains, including Si- $(CH_3)_2$, Si- CH_3 , and C=O, before and after aging. Furthermore, the study revealed that the thermal stability of the nano ZnO-RTV composite material post 2500 hours of UV aging markedly surpasses that of pure RTV.

Keywords

Nano ZnO, Room temperature vulcanized silicone rubber, Nano composite materials, UV resistance

Introduction

Room temperature vulcanized silicone rubber, known for its excellent hydrophobicity and hydrophobicity recovery migration characteristics, is widely used as an anti-fouling flashover coating in power systems [1]. However, during actual service, RTV materials coated on porcelain insulators, glass insulators, and transmission and transformation equipment suffer from degradation due to heat, electricity, acid rain, and ultraviolet radiation, consequently reducing their operational lifespan. In the southern region of China, high humidity, factors like high humidity, heat, and strong ultraviolet radiation accelerate the aging of RTV materials, leading to detachment and pollution flashover accidents. Therefore, enhancing the UV resistance of RTV materials is crucial for prolonging their UV aging life.

Research indicates that nano zinc oxide (ZnO), a broadband gap semiconductor material with a bandgap value of approximately 3.37 eV, exhibits an outstanding UV shielding effect [2-5]. Unlike

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other UV light shielding agents, nano ZnO not only protects against medium to long wavelength ultraviolet radiation, but also demonstrates robust thermal stability. Consequently, nano ZnO is frequently employed as a UV shielding agent in polymer materials. In recent years, extensive research has been conducted in the realm of nano ZnO/organic polymer composites to investigate nano ZnO's UV shielding capabilities. XU B, et al. [6] performed UV aging experiments on nano ZnO-PP composites and found that nano ZnO has significant anti UV aging and nucleation effects on polypropylene materials; ZHANGCH, et al. [7] studied the effect of nano ZnO-EP composite coatings on the UV aging resistance of PBO fibers, revealing that the tensile strength of PBO composite fibers under the same UV aging conditions was double that of pure PBO fibers; CUICW, et al. [8] developed a nano ZnO-PMMA composite material with high UV light shielding properties, finding that after 200 hours of UV aging, the fracture elongation of pure PMMA decreased by up to 52%, whereas the nano ZnO-PMMA composite material only showed a 3% decrease, maintaining excellent mechanical properties. However, reports on using nano ZnO to enhance the UV resistance of RTV are scarce.

This study utilized nano ZnO as an inorganic filler, incorporating it into the RTV matrix after surface chemical modification with a titanate coupling agent, to create nano ZnO-RTV composites with mass fractions of 0, 0.5%, 1.0%, and 2.0%. We conducted UV accelerated aging experiments on each sample. The study compares the changes in surface static contact angle, infrared characteristic absorption peak, surface microstructure, and thermal decomposition characteristics of nano ZnO-RTV composites with varying contents over time, aiming to elucidate the mechanism by which nano ZnO influences the UV resistance of RTV.

Experiment

Primary raw materials

107 glue (SR, 107[#]B2000), Shenzhen Hongyejie Technology Co., Ltd; Dibutyl February tin silicate (industrial grade), Jinan Mingyu Chemical Co., Ltd; Ethyl orthosilicate (analytical pure), nano ZnO (particle size 30 ± 10 nm), Aladdin Reagent Co., Ltd; Titanate coupling agent (PN-105, industrial grade), Changzhou Jiulong Additives Co., Ltd; Acetone (analytical pure), Guangzhou Tongyuan Chemical Technology Co., Ltd; Anhydrous ethanol (analytical pure), deionized water, M cLean Reagent Co., Ltd.

Preparation of nano ZnO-RTV composite materials

Weigh 10g of nano ZnO and dry it thoroughly. Then disperse it ultrasonically in 200 mL of acetone. Prepare a mixture of acetone and deionized water in a 3:1 volume ratio. Weigh 1g of titanate coupling agent and add it to the mixture. After mixing evenly, incorporate this into the nano ZnO ultrasonic dispersion and sonicate for 20 minutes. Subsequently, stir and reflux the above mixed solution at 70 °C for 8 hours, then let it stand at room temperature for 24 hours. Follow this by washing repeatedly with anhydrous ethanol, acetone, and deionized water. Finally, dry it under vacuum at 100 °C for 12 hours. The modified nano ZnO obtained is referred to as PN ZnO.

For the uniform dispersion of nanoparticles in the RTV matrix, initially dilute the RTV in a suitable volume of acetone. Then, add PN-ZnO to this mixture and stir mechanically at 40 °C for 6 hours. Vacuum heat the mixture to evaporate the acetone. Slowly add 4.0% ethyl orthosilicate and 0.5% dibutyltin dilaurate drop wise in a nitrogen atmosphere Vacuum stir this mixture, then quickly pour it into a mold. Allow it to rest for 8 hours to form the nano ZnO-RTV composite materials. Prior to performance testing, clean the samples with anhydrous ethanol and dry them in a 25 °C dryer for 24 hours.

UV accelerated aging experiment

In order to simulate the outdoor exposure process of RTV as much as possible, this study selected a QUV accelerated aging testing machine, Q-Lab Company in the United States, and used UVA-340 lamp tubes (irradiation wavelength 290-400 nm) according to the GB/T14522-2008 test method for testing. The specific experimental conditions are shown in Table 1.

Performance testing

The chemical structure of nano ZnO, both pre- and post-modification, was analyzed using a VERSEX70 fourier transform infrared spectroscopy (FTIR) device, manufactured by Bruker Company, Germany. This device operates within a wave number range of 400-4000 cm⁻¹. For observing the dispersion of nano ZnO in the RTV matrix, Merlin-type Field Emission Scanning Electron

Exposure cycle	UV lamp model	Irradiance/(W/m ² × nm)	Wavelength/nm	Blackboard temperature/°C
8 h drying	UVA-340	0.76 ± 0.020	340	60 ± 3
4 h condensation				50 ± 3

Table 1: Experimental conditions for fluorescent UV lamp exposure.

Microscopy (SEM) by Carl Zeiss AG, Germany, was employed. The absorption characteristic spectrum of the samples in the UV-visible region was detected using the Lambda 950 Ultraviolet-Visible Spectrophotometer (UV-Vis) from PerkinElmer, USA, which has a scanning wavelength range of 200-400 nm. A custom-built AC electrical strength testing platform was utilized for sample testing. The samples were immersed in transformer oil and subjected to 7 breakdown tests, with a voltage increase rate of 0.5 kV/s for each type of sample.

Upon completion of the UV accelerated aging experiments, FTIR tests are conducted on the surfaces of each sample, both pre- and post-aging, to characterize the changes in their characteristic functional groups. These tests are performed with a wave number range of 500 to 3750 cm⁻¹. Additionally, the surface microstructure of each sample is examined before and after aging using SEM. To assess the static contact angle of each sample's surface, both before and after aging, we utilize the OCA 40 Micro Surface Tension Surface Contact Angle Tester from Data Physics, Germany. Furthermore, the thermal stability and decomposition characteristics of the samples are evaluated using a TG 209F1 Libra Thermo gravimetric Analyzer from NETZSCH, Germany. This analysis is carried out in a nitrogen atmosphere with a heating rate of 10 °C/min, across a temperature range from room temperature to 800 °C. The sample mass was 10 mg.

Performance Analysis of Composite Materials

Chemical structure of nano ZnO

Fourier transform infrared spectroscopy (FTIR) is commonly used for characterization and analysis of chemical functional groups. In this paper, FTIR detection is used to study the surface grafting effect of nano ZnO. Figure 1 shows the infrared spectra of nano ZnO, nano ZnO-PN, and titanate coupling agent (PN).

From Figure 1, it can be seen that nano ZnO

exhibits characteristic absorption peaks of Zn-O and -OH at 480 cm⁻¹ and 3435 cm⁻¹, respectively. After PN modification, the Zn-O absorption peak intensity of nano ZnO-PN remained unchanged, but the -OH absorption peak was significantly weakened. At the same time, the characteristic absorption peaks of PN, Ti-O-P (1028 cm⁻¹), -CH₃ (2963 cm⁻¹, 2870 cm⁻¹ ¹), and -CH₂- stretching vibration peaks (2926 cm⁻ ¹), also appeared, indicating that the nano ZnO surface was successfully grafted onto PN. This is because the alkyl hydroxyl groups generated by PN hydrolysis react with the hydroxyl groups on the surface of nano ZnO, ultimately achieving the chemical grafting of nano ZnO onto PN. Considering that during the preparation process, the modified nanoparticles were washed multiple times with anhydrous ethanol and vacuum dried at 100 °C for 24 hours, the coupling agent physically adsorbed on the surface of the modified nanoparticles can be ignored.

Microscopic morphology analysis of composite materials

SEM was used to observe the microstructure of the liquid nitrogen brittle fracture surface of nano ZnO-RTV composite materials to determine whether nano ZnO is uniformly dispersed in the RTV matrix. The test results are shown in Figure 2. From Figure 2, it can be seen that the modified nano ZnO is uniformly dispersed in the RTV matrix, with a particle size of 20-40 nm. In Figure 2c, the majority of ZnO particles with a mass fraction of 1.0% have particle sizes within the nanoscale range and do not exhibit agglomeration. The particle morphology is irregular spherical. After the mass fraction of nano ZnO increases to 2.0% (Figure 2d), some particles will undergo obvious agglomeration, and the particle size of the agglomerated particles has reached the micrometer level. Overall, at low filling amounts, the modified nano ZnO can achieve uniform dispersion in the RTV matrix, but as the filling amount continues to increase, it becomes increasingly challenging to prevent the nanoparticles from agglomerating.









UV shielding performance of composite materials

To characterize the UV shielding performance of nano ZnO-RTV composite materials, UV Vis spectrophotometer was used to detect each sample, and the UV transmission spectrum obtained is shown in Figure 3. In addition, to further quantify the UV shielding efficiency of the sample, equations (1) to (2) were used to calculate the arithmetic mean of the transmittance of the sample in the irradiation band (290-400 nm) of the UV accelerated aging experiment.

$$T(\mathrm{UV})_{i} = \frac{1}{m} \sum_{\lambda=290}^{400} T_{i}(\lambda)$$
(1)

$$A(UV) = 1 - T(UV)$$
⁽²⁾

In equations (1) to (2), *m* represents the number of measurements between the 290-400 nm wavelength band; $T_i(\lambda)$ is the spectral transmittance of sample *i* at wavelength λ ; T (UV) is the transmittance of ultraviolet light; A (UV) is the UV light shielding rate.

According to equations (1) to (2), the variation pattern of UV shielding efficiency of nano ZnO-RTV composite materials with different filling amounts can be obtained, as shown in Figure 4.

From Figure 4, it can be seen that the UV shielding rate of pure RTV is only 25.22%. With the increase of nano ZnO filling amount, the UV shielding rate of the sample rapidly increases. At a mass fraction of 1.0%, the shielding rate of the sample reaches its maximum value, which is 97.85%, and then shows a downward trend. It can be seen that the addition of nano ZnO can significantly improve the shielding efficiency of RTV against ultraviolet light, with an increase of up to 72.63%. Analysis suggests that the significant UV shielding performance of nano ZnO-RTV is mainly due to its strong absorption, easy refraction, and scattering effects on UV light. On the one hand, as a wide bandgap semiconductor material, nano ZnO has a bandgap width of about 3.37 eV at room temperature and an exciton binding energy of up to 60meV. The corresponding free exciton response absorption band starts at 387 nm, which is beneficial for absorbing ultraviolet light [9]. On the other hand, nano ZnO particles have a very small particle size and excellent interface reflection performance. When incident light irradiates onto nano ZnO, there will be continuous diffuse reflection at the interface of the nano ZnO-RTV matrix. In addition, the size of nano ZnO is smaller than the wavelength of ultraviolet light, so ultraviolet light scatters around the particles in various directions, further weakening







the intensity of incident ultraviolet light. However, when the mass fraction of nano ZnO reaches 2.0%, the UV light shielding efficiency of nano ZnO-RTV does not increase but decreases. This may be due to the aggregation of nano ZnO into micrometer particles, which cannot effectively absorb or scatter UV light. This is consistent with the SEM test results mentioned above.

Electrical strength of composite materials

Using nano modification technology to fill RTV with nano ZnO to enhance the UV shielding performance of RTV, it is also necessary to explore the impact of nano ZnO on the electrical strength of RTV to ensure that nano ZnO-RTV composite materials are suitable for power systems. Therefore, the electrical strength test was conducted on the sample, and the specific data is shown in Table 2.

From Table 2, it can be seen that the electrical strength of pure RTV is 17.06 kV/mm. The electrical strength of nano ZnO-RTV gradually increases with the increase of nano ZnO filling amount, and reaches the maximum value of 19.52 kV/ mm when the mass fraction is 2.0%. Analysis suggests that it may be due to the use of nano ZnO as a promoter in the curing process of RTV prepolymers [10-13], which leads to the formation of a denser cross-linking system in the nano ZnO-RTV composite material. Moreover, nano ZnO is more evenly dispersed in RTV. Therefore, electrons in the composite material need to overcome higher potential barriers to undergo transitions, and their carrier directed migration activity is constrained, resulting in greater electrical strength. It can be seen that an appropriate amount of nano ZnO filling can increase the electrical strength of RTV.

Aging Experiment Results and Analysis

SEM analysis of aged samples

Using SEM to detect the surface microstructure of nano ZnO-RTV composite materials with different mass fractions, in order to explore the effect of UV

Table 2: The electric strength of nano ZNO-RTVcomposite.

Mass fraction/%	Electrical strength/(kV/mm)
0	17.06
0.5	18.96
1.0	19.33
2.0	19.52

aging on the surface microstructure of each group of samples, and to verify the weakening effect of nano ZnO on the UV aging effect of RTV surface.

The microstructure of the surface layer of pure RTV under UV aging conditions of 0-2500 hours is shown in Figure 5. From Figure 5, it can be seen that the surface of the unaged RTV is relatively flat and smooth, but there are a few micro scale clusters, which may be dust adhering to the surface of the sample during the RTV preparation process. After 1000 h of UV aging, the surface of the pure RTV sample began to show a slight protrusion of sheet-like silicone rubber, but this phenomenon was not particularly obvious. When the UV aging time reaches 2000 hours, not only block shaped silicone rubber peels off but also curled strip shaped silicone rubber appears on the surface of the sample, indicating that the RTV UV aging effect is more obvious. Until the aging time reaches 2500 h, more fine and fragmented blocks are exposed on the surface of the sample. Analysis suggests that prolonged ultraviolet radiation is the fundamental cause of the surface detachment and aging phenomenon of RTV samples.

After aging for 2500 hours, the surface microstructure of nano ZnO-RTV composites with different mass fractions is shown in Figure 6. From Figure 6, it can be seen that the surface morphology of the pure RTV sample is different. The surface of the 0.5% mass fraction nano ZnO-RTV sample shows a decrease in blocky shedding and a small number of exposed nanoparticles. When the mass fraction of nano ZnO increases to 1.0% and 2.0%, the blocky and spherical nanoparticles on the micro surface of the sample gradually decrease after an aging period of 2500 hours, it is observed that the impact of aging on the surface microstructure of RTV diminishes progressively with the increase of nano ZnO filling amount.

Surface static contact angle analysis of aged samples

To investigate the effect of UV aging on the surface hydrophobicity of nano ZnO-RTV composite materials with different mass fractions, surface static contact angle tests were conducted on each group of samples, and the results are shown in Figure 7.

From Figure 7, it can be seen that the surface hydrophobicity of the unaged nano ZnO-RTV



Figure 6: SEM images of nano ZnO-RTV composite with different mass fraction after aging for 2500 h.



composite material is excellent, and its static contact angle is around 107°; After UV aging, the static contact angle on the surface of each group of samples decreased to varying degrees. Among them, the decline trend of pure RTV is the most obvious. After 2500 hours of UV aging, its static contact angle decreases to 74°, which is 30% smaller than the initial static contact angle; After 2500 hours of UV aging, the static contact angles of each group of nano ZnO-RTV composite material samples were 93°, 96°, and 100°, respectively, and they still maintained good hydrophobicity. Overall, after UV aging, the loss of hydrophobicity of pure RTV is much greater than that of nano ZnO-RTV composites.

Analysis suggests that the side methyl Si-(CH₃)₂ on the RTV molecular chain acts as a strong polar group, which has a shielding effect on the main chain and endows RTV with excellent hydrophobicity. However, the C-H bond energy in Si-(CH_3), is relatively small. Long term ultraviolet radiation tends to weaken the C-H bonds, leading to their facile breakage and the generation of free radicals. These free radicals further react with oxygen, forming hydrophilic groups such as C=O. This reaction contributes to a progressive reduction in the shielding effect provided to the polymer's main chain, altering its properties. Macroscopically, the static contact angle of RTV is significantly reduced, and the hydrophobicity is significantly weakened. In addition, after 2500 hours of UV aging, the surface static contact angle of each group of nano ZnO-RTV composite material was much higher than that of pure RTV, which is consistent with the SEM test results in section 3.1. This is because nano ZnO, as a UV light shielding agent, can absorb or scatter some UV light, which not only weakens the direct UV radiation damage to the RTV side methyl group, but also suppresses the generation of hydrophilic group C=O.

Discussion on the UV Aging Mechanism of Nano ZnO-RTV Composite Materials

The main component of RTV is polydimethylsiloxane (PDMS), whose molecular structure is shown in Figure 8, and the corresponding chemical bond energy is shown in Table 3. After the UV accelerated aging experiment, RTV underwent varying degrees of photo-oxidative aging. The underlying reason is that the UV energy is greater than many chemical bond energies in PDMF,



Table 3: The chemical bond energy ofpolydimethylsiloxane.

Chemical bond	Bond energy/(kJ/mol)
Si-O	447
О-Н	463
C-H	413
Si-C	318

leading the chemical bonds to break, generating different types of free radicals. Under the action of oxygen, further free radical reactions occur to generate new chemical substances.

The photon energy of 320 ~ 400 nm ultraviolet radiation is 380 ~ 299 kJ/mol (the shorter the wavelength, the greater the photon energy), and the photon energy of the main wave peak of the irradiated light at 340 nm is 352 kJ/mol. According to Table 3, the radiation band energy of the ultraviolet aging experiment is similar to the bond energy of Si-C, C-H, and O-H. Therefore, long-term ultraviolet irradiation can cut off Si-C and C-H on the PDMS molecular chain.

Ultraviolet light can trigger free radical reactions in the main chain of RTV. As the free radical reaction is triggered, the above chemical bonds gradually weaken or even break, and the PDMS molecular chain undergoes partial cleavage, generating active free groups~Si-CH₂·,~Si·, H· and CH₃·. In addition, when CH₃· seizes the hydrogen atom of the PD-MS side methyl group to generate~Si-CH₂·, small molecule segments of siloxane and CH₄ gas will also be generated, resulting in changes in the microstructure of RTV, such as the appearance of many blocky shedding substances on the surface of the sample. Furthermore, under UV irradiation conditions, \sim Si-CH₂· continued to undergo photooxidation reaction with oxygen, generating C=O hydrophilic groups, which exhibited a significant decrease in the surface contact angle of RTV at the macroscopic level.

Due to the ability of nano ZnO to eliminate the photo reactive radicals generated by RTV main chain free radical reactions, further photoreaction of the main chain is inhibited. Therefore, the filling of nano ZnO can significantly enhance the UV resistance of RTV.

Conclusion

In addressing the challenge of poor UV resistance of RTV coatings in power equipment, this study utilizes titanate coupling agents to modify the surface of nano ZnO This approach has successfully led to the preparation of nano ZnO-RTV composite materials. We conducted UV accelerated aging experiments to assess the impact of nano ZnO on various performance aspects of RTV, including surface microstructure, static contact angle, and electrical properties. The key conclusions are as follows:

- 1) Nano ZnO can markedly improve the UV shielding performance of RTV. Specifically, nano ZnO-RTV composites with a mass fraction of 1.0% demonstrate an increase in UV shielding efficiency of up to 72.63%.
- 2) The UV shielding efficiency of the composite materials initially increases and then decreases as the nano ZnO filling amount rises. Furthermore, incorporating an optimal amount of nano ZnO can also improve the electrical strength of RTVs.
- 3) Extended UV aging leads to the fracturing and subsequent oxidation of hydrophobic groups Si- $(CH_3)_2$ and Si- CH_3 in RTV into the hydrophilic group C=O. This process results in the detachment of blocky substances from the RTV surface, significantly diminishing its hydrophobicity at a macro level.
- 4) The addition of nano ZnO can significantly inhibit the aging and detachment of the RTV surface, maintaining the static contact angle of the nano ZnO-RTV composite material's surface above 90°.

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