

Review

Research Progress on Aging Mechanisms and Performance Improvements of Silicone Rubber Insulating Materials

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Abstract: Although traditional outdoor insulation materials (such as ceramic and glass) have the advantages of stable chemical properties and excellent insulation performance, they are difficult to deal with the occurrence of pollution flashover in the wet and dirty environment of outdoor operation. The application of organic outdoor insulation materials (such as silicone rubber) has become a solution to this problem. Their good hydrophobicity enable them to maintain a long time of normal operation in dirty areas. However, due to long-term complex outdoor working conditions, silicone rubber will also be aging under the impacts of a variety of factors, resulting in poor performance or even unable to use. Up to now, the research of improving and strengthening the performance of organic outdoor insulation materials is still a research hotspot. This paper reviews the researches on the aging mechanisms of silicone rubber insulation materials in recent years and the achievements of performance improvement work, and summarizes the effects of various improvement methods on its performance.

Keywords: silicone rubber; aging mechanism; fillers filled silicone rubber; surface fluorination

1. Introduction

As a basic energy source, electric energy is economical, clean and easy to use. It plays an irreplaceable role in various industries and people's life. Currently, electric energy accounts for 27% of end-energy consumption in China, which is higher than the world average. The value is expected to exceed 30% by 2025 according to the China Electricity Council. With the increase of power demand and the expansion of the scale of the power grid, people's requirements on the reliability of the power supply are also increasing, and there are higher requirements on the insulation performance of the transmission equipment and lines. Outdoor insulation is a field prone to failure in the power system. The normal operation of insulators ensures the safety of the power grid. Improving the performance of outdoor insulating materials has always been an important research direction in the field of high voltage and insulation.

The traditional outdoor insulating materials are mainly inorganic materials, such as ceramic and glass. Although they have the advantages of stable chemical properties and good insulation performance, it is difficult to deal with the occurrence of pollution flashover in the humid and dirty environment of outdoor work, which will seriously affect the safety of the power grid. The invention of organic insulating materials and composite insulators has provided a solution to this problem. The insulator made of organic insulating materials, such as glass fiber resin, epoxy resin, silicone rubber (SR), has excellent flashover resistance, erosion resistance and aging resistance, as well as superior mechanical properties and light weight [1-3]. Compared with traditional insulators, composite insulators have greater application value.

Under the impacts of various external factors, such as intense electric field, high temperature, ultraviolet radiation and so on, SR insulating materials will still experience varying degrees of aging, resulting in poor performance [3-5]. Therefore, the improvement of SR insulating materials has been one of the research emphases in the field of electrical engineering and power industry. In this paper, the aging mechanisms of outdoor insulation silicone rubber and the research progress of silicone rubber performance improvements are summarized and discussed.

2. Aging of Silicone Rubber Insulating Materials

Even if the silicone rubber material has excellent insulation performance, good thermal stability and hydrophobicity and many other advantages, but in the long-term complex outdoor working environment, under the impacts of various external factors, there will be complex aging phenomena, resulting in the decline of properties of silicone rubber.

2.1. Aging Caused by High Temperature

High temperature is a major factor leading to the aging of silicone rubber. Under normal operating conditions, the maximum allowable temperature of the cable is 90 °C. Generally speaking, the operating temperature of DC crosslinked polyethylene cable is 70 °C. However, when the cable is under overload or short circuit, the maximum allowable temperature of the cable in the short term is about 250 °C [2,3]. The power system often has a large load capacity, so the insulating materials work at a high temperature for a long time. In this case, the low molecular substances in SR will accelerate the migration to the surface, and cause chemical reactions, such as cross-linking and oxidation of molecules inside the material. After the molecular side functional groups of SR are damaged by the reaction, the hydrophobicity of the material is reduced, and the destruction of the whole internal structure also makes the SR material to hardening and embrittlement.

Chen et al. [2] carried out an artificial accelerated thermal aging experiment on liquid silicone rubber (LSR) samples. They conducted experiments on the LSR samples at 120 °C and recorded the data every 10 d. The experimental results showed that the mechanical properties of LSR decreased significantly after aging for about 20 d. The tensile strength of LSR was down by 44.6% and elongation at break was down by 66.9% after 60 d aging. The decreasing range increased with the aging time. The conductivity of the LSR sample increased after thermal aging as the system became dense and the ionic mobility decreased. It also made the breakdown field strength increase to a certain extent, but the figure decreased slightly after 40 d of aging time. The reason for this situation is that in the early stage of thermal aging, the cross-linking reaction was the main reaction. The cross-linking density in the system increased and the free travel of electrons was shortened. While in the late stage of thermal aging, the degradation reaction was the main reaction. The system structure was damaged and the breakdown field strength was reduced.

2.2. Aging Caused by Ultraviolet Light

Another environmental factor that is hard to avoid when working outdoors is UV exposure. Ultraviolet light has short wavelength and high photon energy, which is easy to cut the chemical bonds inside the material, causing the molecular chains of SR polymer to break and further chemical reactions, such as oxidation, crosslinking and degradation, which will affect the mechanical and electrical performances of the material. After ultraviolet irradiation, some bonds, such as C-H, Si-C and Si-O-Si in SR, are broken to form free radicals, and there will be hydrophilic -COOH and -CH₂OH [4]. In the aerobic environment, Si crosslinks with O to form SiO₃ and SiO₄ [4].

Wang et al. [5] studied the impacts of ultraviolet exposure on surface appearances, mechanical

properties and hydrophobicity of high temperature vulcanized silicone rubber (HTV SR). After the sample was irradiated with UV-A (wavelength 315-400 nm) for 1000 h, it was found that the interaction between the fillers and the SR matrix became loose, the degree of crosslinking of the matrix increased, and the hydrophobicity decreased with the aging time. The fracture of Si-C in SR material led to the destruction of macromolecular network structure and, to some extent, material degradation, which caused the increase of surface defects resulting in material deterioration. Ahmadi-Joneidi et al. [6] studied the effects of ultraviolet on the electrical performances of SR materials. With the extension of UV exposure time, the hydrophobicity and flashover voltage of the materials decreased.

2.3. Aging Caused by Electricity

Electrical aging is an unavoidable aging factor of insulating materials. In this process, the insulation capacity and electrical strength of SR insulating materials will be affected. Under the action of long-term high electric field intensity, the insulator will have corona discharge, arc discharge and other surface discharge, resulting in dielectric deterioration damage, tracking and erosion, causing the permanent reduction of insulation performances.

Wang et al. [7] used the pine-plate electrode to simulate the non-uniform electric field environment, and took the HTV SR sheets as the test samples to explore the effects of corona on the aging properties of the materials. The results showed that corona caused the decrease and temporary loss of hydrophobicity of the materials. The higher the temperature, the faster the hydrophobicity recovery time, and the greater the hydrophobic angle after recovery. At the same time, the tensile strength and hardness of the material decreased, the conductivity decreased, the dielectric loss angle increased, the dielectric constant decreased, it can be known that corona discharge greatly affected the electrical performances of SR materials. In another study [8], needle-plate electrode and SR materials were used to conduct experiments. It was found that AC corona caused the hydrophobicity loss and recovery of clean test pieces to be rapid, while the loss of hydrophobicity of contaminated test pieces was relatively slow and the recovery was faster. Wang et al. [9] carried out an AC corona test on high temperature vulcanized SR material. According to the results, black powdery corona rings were generated on the surface of the material after corona discharge, which gradually changed from surface damage to depth. Both the surface layer and inner layer of the material suffered different degrees of erosion, and the molecular structure of SR was destroyed, resulting in cracking and oxidation. Hydrophobicity, mechanical and electrical properties all decreased to varying degrees. Ma et al. [10] compared the effects of AC corona and DC corona on SR, and found that AC corona produced higher dose of ozone, made the surface oxidation of SR more serious, and had a greater impact on its electrical properties than DC corona.

Corona discharge is an important factor leading to erosion and aging of SR on insulators. In order

to reduce the effects of SR aging caused by this reason, many scholars have carried out researches. Venkatesulu and Thomas [11] carried out corona tests on the unfilled SR samples and different weight percentages (1%, 2%, 3%) of nano silica filled samples. The results showed that compared with the unfilled samples, when the weight was 3%, the surface roughness was reduced, the crack width was reduced by 7 times, and the hydrophobicity loss was also reduced. With the addition of fillers, the degree of erosion of materials was reduced. With the increase of the amount of aluminum hydroxide in SR, the electric marking resistance and vertical combustion grade of SR were improved [12].

2.4. Aging Caused by Other Factors

The working environment of outdoor insulators is complex and the aging of SR insulating materials in practical application is a long process, so its aging is often affected by a variety of environmental conditions, such as salt spray, moisture, and pollution. Insulation equipment in coastal areas has long been working in salt spray and damp environment. Under such oceanic atmospheric conditions, salt spray particles will settle on the surface of SR materials, rapidly absorb moisture and form salt solution, which penetrates into the silicone rubber materials to aging SR in electrical and chemical forms. Peng et al. [13] conducted a study on the effects of salt spray and relative ambient humidity on the electrical properties of HTV SR, and the experimental result showed that the combination of humidity and salt deposition on the material surface aggravated corona aging of SR. Under the condition of high humidity, the material's hydrophobic property gradually decreased with aging, and under the condition of low humidity, posthydrophobic recovery after 4 d played a dominant role, making it restore its partial hydrophobicity. Meanwhile, the corona power generation process caused degradation and damage to the surface of SR material, aggravated the penetration of salt solution, and decreased the surface resistivity of the material.

Zhao et al. [14] conducted corona tests under different pressure conditions in a pressure-relief vessel in order to simulate the aging phenomenon in high altitude and low pressure areas. According to the test results, the damage degree of corona discharge on the surface hydrophobicity of SR materials accelerated with the decrease of pressure, and the hydrophobicity recovery rate of synthetic insulators in high altitude area was much less than the loss rate after corona aging. Wang et al. [15] pointed out that even though SR inhibited pollution flashover in a short time due to its hydrophobicity, flashover would still occur under some conditions (such as long-term humid environment) if the surface of the SR aggravated the accumulation of pollutants and the pollution hydrophobicity fluctuated greatly.

Tatsuya Sakoda et al. [16] studied the flashover voltage and discharge behavior of SR when it was polluted by fog, red clay or carbon particles. For SR samples polluted with carbon, partial discharges occurred between water droplets and form interspersed dry bands. For SR samples polluted with red clay,

the leakage current induced uniform dry bands and dry-band arc discharges. Because flashover occurred through the dry bands, the noticeable difference in flashover voltage between the polluting materials was not seen. The pollution deposit changed the surface state and caused partial discharges which led large leakage current, but the partial discharges did not significantly lower insulation performance.

3. Performance Improvements of Silicone Rubber Insulating Materials

In order to improve the performance of SR, expand the application range of organic composite insulators and extend their service life, researchers and technicians of various countries have never stopped the research in this field. The main objectives of these studies include further improving the hydrophobicity of SR insulating materials and their long-term stability under electrical, mechanical, thermal and various environmental factors. At present, most of researches focus on improving the performances of SR by adding functional fillers and chemical modification.

3.1. Adding Functional Fillers to Improve the Performances of Silicone Rubber

SiO₂ and Al(OH)₃ (ATH) are two common fillers of SR for insulation. SiO₂ can improve the mechanical strength, wear resistance and elongation of rubber. ATH is a kind of flame retardant with a wide range of applications. The mechanism is that ATH is decomposed by heat into water and alumina with certain thermal conductivity. This process is a strong endothermic reaction, which cools the polymer and plays a flame retardant effect.

It is found that SiO₂ and ATH also affect the hydrophobic and electrical performances of SR. Many experiments and applications have proved that appropriate filling of SiO₂ and ATH fillers is conducive to improving the tracking and erosion resistance of SR materials [17-19]. Xue et al. [20] studied the effects of ATH, irregular shape of SiO₂ and spherical SiO₂ on the mechanical, electrical and thermal performances of SR. The experimental results showed that the spherical silica filled silicone rubber had better electrical and mechanical performances, and its tensile strength was nearly twice that of ATH filled silicone rubber. Meanwhile, its dielectric constant and dielectric loss were the lowest among the three materials. As SiO₂ fillers filled SR has good thermal stability and thermal conductivity at high temperatures, it also has better resistance to arc aging. Fan et al. [21] pointed out that the introduction of ATH fillers resulted in the increase of -OH and the decrease of Si(CH₃)₂, which limited the crosslinking of SR. Tuğrul Akkoyun et al. [22] studied the effects of ATH filling amount on the hydrophobicity of SR after corona aging, and found that SR filled with 50% wt ATH had the best effects under several different filling amounts.

In addition to the content of the fillers, the size of the particles also affects the performances of SR.

Alhaytham et al. [23] studied the effects of fumed silica and ground silica fillers on the DC erosion of SR. In the experiment, fumed silica particles size of 7 nm and ground silica 10.5 μm were used as fillers. The 5% wt fumed silica filled SR sample and 30% wt ground silica filled SR sample were prepared respectively. The experimental results showed that even though the silica filling content of the former was lower, its erosion resistance was higher.

Apart from the above fillers, common SR insulating material fillers also include various metal oxides and nitrides. Nitride fillers have the advantages of high thermal conductivity, high temperature resistance and good electrical insulation performance, and are widely used in polymer insulating materials. In recent years, the research on boron nitride (BN) as SR filler for insulation is not few. Mrudul et al. [24] prepared 1%, 3%, 5% wt nano-scale BN fillers filled SR and conducted performance tests. The results showed that the corona inception voltage of SR material was improved under the influence of the fillers. By comparing the three different filling content samples, it could be found that the erosion and liquid permeation damage of 3% wt BN fillers filled SR were the least. The 3% wt could be used as the optimal filling weight percentage for outdoor high voltage environments to minimize degradation due to leakage current, heat and liquid diffusion. Zhou et al. [25] modified the BN nanosheet fillers with polydopamine to improve the dispersion of the fillers in the SR matrix and make them evenly filled in the matrix. The short-term performance test showed that the DC breakdown field strength and tensile strength of SR had the greatest improvement when the filling amount was 15% wt, which were 23.2% and 38.3% higher than that of pure SR. The dispersion of the fillers in the matrix was also significantly improved by the action of polydopamine.

3.2. Surface Fluorination to Improve the Performance of Silicone Rubber

Using chemical ways to introduce new atoms in SR and change its chemical structure and properties, can also play a role in enhancing the performances of SR. As is known to us, in addition to having similar properties to SR, fluoro silicone rubber also has better erosion resistance and solvent resistance. However, due to its high cost, its actual production and market share are relatively low. The advantages of SR in the field of outdoor insulating materials are mainly related to its surface electrical properties, so in the field of electrical insulation, many studies have been carried out on the introduction of fluorine on the surface of SR to improve its insulation properties.

Surface fluorination modification methods include direct fluorination, plasma fluorination, fluorination reagent surface modification and so on. In electrical engineering research field, epoxy resin, SR and other insulating materials are usually treated by direct fluorination. Direct fluorination is an efficient and economical method for preparing fluorinated polymers invented in the last century. The surface of the material is fluorinated with F_2 gas. Due to its high electronegativity and reinforcing

chemical reactivity, F_2 can also generate a large number of fluorine free radicals at room temperature, so it is easy for F_2 to engage in substitution or addition reactions with organic compounds. F atoms enter the polymer surface in the form of C-F and cause cross-linking reactions, which improve the surface density of the polymer and strengthen the surface performances. A certain proportion of F_2/N_2 mixture is usually used in industry for direct fluorination [26]. First, the material to be fluorinated is placed in the container, and the container is heated. After reaching the required temperature, high purity nitrogen is introduced several times to replace the gas in the container, and then the reaction gas is slowly introduced for fluorination, and a certain pressure of gas is maintained until the end of the reaction.

Due to the excellent properties of fluorinated polymers, many scholars have studied the fluorinated modification of materials in recent years. Kim Seokjin et al. [27] proposed that direct surface fluorination of melamine sponge could significantly enhance the oil-water separation performance of the fluorinated material, which could be used as separation and purification adsorbent in industry. Jiang et al. [28] fluorinated a series of carbon molecular sieves, and the resulting samples had better electrochemical performance and lower cost than the fluorinated graphite, which was believed to have great potential applicability in the field of lithium primary batteries. Xie et al. [29] used F_2/N_2 mixed gas to directly fluorinate the surface of polydimethylsiloxane (PDMS), which greatly changed the physical and chemical characteristics of the surface of the experimental material. After fluorination and subsequent annealing treatment, a highly hydrophobic and partially conductive fluorine-containing surface was obtained.

Hydrophobicity is an index reflecting the water penetration resistance of materials. The surface tension of materials with strong hydrophobicity is small, and water exists in the form of spherical droplets on the surface. When the volume of droplets increases to a certain extent, they will slide off the surface of the material. The leakage current of outdoor insulators is related to surface contaminants and environmental humidity [30], so the improvement of hydrophobic performance of insulating materials can also reduce the influence of environmental humidity to a certain extent. There are studies which have demonstrated that fluoridation can improve the hydrophobicity of organic outdoor insulating materials. Du et al. [31] conducted direct fluorination treatment on room temperature vulcanized silicone rubber (RTV SR) samples, and found that after 10 min of fluoridation, the contact angle of the samples without corona aging increased from 107.9° to 116° , and decreased after 10 min, which was mainly due to the reduction of material surface roughness under the influence of fluorination. The contact angle of the samples that lost hydrophobicity due to corona aging increased from 42.4° to 68° . When the contact angle was greater than 90° , the material was regarded as hydrophobic. The experimental results showed that the hydrophobicity of RTV SR would be improved by proper fluorination.

In addition, many studies have been carried out to investigate the effects of surface fluorination on

the electrical performances of SR. Du et al. [32] studied the accumulation and attenuation of surface charge of RTV SR caused by direct fluorination, and found that the initial surface potential of the fluorinated sample was lower than that of the unfluorinated sample, which inhibited the accumulation of surface charge. The reason was that the high electronegativity of fluorine made C-F bond highly polarized, and the fluorinated layer captured charge to form a shielding layer, preventing further injection of charge. Direct fluorination also significantly accelerates the attenuation of surface charges. At the same time, some researchers have pointed out that there are abundant deep traps caused by fluorination for a long time, resulting in a large amount of space charge accumulation in SR, which reduces the effects of inhibiting charge accumulation [33,34].

In recent years, Zhenlian An and his research team have made many achievements in the fluorination modification of SR insulating materials. The team studied the mechanism of direct fluorination of SR, analyzed the chemical components of fluorinated liquid silicone rubber (LSR) by attenuated total reflection infrared spectroscopy (ATR-IR) and X-ray photoelectron spectroscopy (XPS). The experimental results showed that there were bonds Si-F and C-F in the fluorinated layer of SR and that the fluorination of SR did not simply replace parts of hydrogen atoms on methyl atoms or the whole methyl atoms with fluorine atoms, and other reactive substances (such as methyl derivatives and their fluoride) would also be attached to Si atoms or methyl through substitution reactions [35]. The team tested the DC flashover voltage of SR samples before and after fluorination and found that the surface conduction of SR samples increased due to fluorination and the DC flashover voltage increased by 64% [36].

In order to know the influence of temperature on the fluorination effect of SR, An et al. investigated the influence of fluorination at different temperatures on the moisture barrier properties of SR and found that the fluorinated samples at 30 °C had higher moisture absorption rate, fluorine content and compactness than those at 60 °C although the thickness was small [37]. The team also studied the influence of fluorination on the corona discharge resistance of SR at different temperatures [38]. It was found through experiments that compared with fluorination at 25 °C and 55 °C, fluorination at 85 °C for 30 min could obtain a thick enough fluorination layer, which significantly improved the corona resistance performance. After corona test, the chemical properties, surface morphology and thickness of the surface layer did not change significantly, but the fluorinated samples at 55 °C showed worse corona resistance than those at 25 °C. The unsatisfactory fluorination effect at 55 °C was caused by two competing effects, the thermal activation of the substitution reaction and fluorine diffusion through the surface layer and the steric hindrance by the fluorinated methyl groups to the fluorination and fluorine diffusion [39].

An et al. also investigated the effect of direct fluorination on the tracking and erosion resistance of SR [40]. Inclined plane tracking and erosion tests were carried out on samples before and after

fluorination. Among the 15 unfluorinated samples, 7 failed to pass the test due to combustion, while the fluorinated samples all passed the test. Meanwhile, compared with the unfluorinated samples, the fluorinated ones had a larger average erosion depth but a smaller average erosion length or area. Through observation and analysis, it is concluded that the improvement of the resistance by fluorination is due to the changes of the composition, structure and interface of the SR matrix and fillers, and also affected by the change of surface appearances.

4. Conclusions

This paper summarizes the causes of aging of silicone rubber outdoor insulating materials and related research results in recent years. At the same time, it introduces several hot improvement directions of silicone rubber for insulation, and summarizes the research results. The aging of outdoor insulating SR is a long and complex process under the comprehensive actions of many factors. At present, the researches on its aging mechanism are still in progress. Among them, surface discharge has the most serious effect on SR, and some other factors (such as contamination, salt deposition) have obvious influence only when discharging. It is necessary to explore the aging mechanism of SR to improve the performance of composite insulator. In this review, adding functional fillers and surface fluorination to improve the performances of SR are introduced. Although there have been many remarkable results, it is still difficult to find an optimal scheme for the type, size and content of fillers. At the same time, fluorination modification of organic insulating materials (silicone rubber, graphene, epoxy resin, etc.) has research value and potential economic benefits in the field of electrical insulation. Composite insulators made of silicone rubber have been developed for decades and will continue to play a huge role in the field of outdoor insulation in the future. In addition, the research of its performance improvements will still be a vital focus in the electrical industry.

Potential Conflicts of Interest

The author declares no conflict of interest.

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Int. J. Nano & Matl. Sci. **2023**,10(1): 1-14

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