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RTV Silicone Rubber Degradation Induced by Temperature Cycling

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Abstract: Room temperature vulcanized (RTV) silicone rubber is extensively used in power system due to its hydrophobicity and hydrophobicity transfer ability. Temperature has been proven to markedly affect the performance of silicone rubbers. This research investigated the degradation of RTV silicone rubber under temperature cycling treatment. Hydrophobicity and its transfer ability, hardness, functional groups, microscopic appearance, and thermal stability were analyzed using the static contact angle method, a Shore A durometer, Fourier transform infrared spectroscopy (FTIR), scanning electron microscopy (SEM), and thermogravimetry (TG), respectively. Some significant conclusions were drawn. After the temperature was cycled between $-25\text{ }^{\circ}\text{C}$ and $70\text{ }^{\circ}\text{C}$, the hydrophobicity changed modestly, but its transfer ability changed remarkably, which may result from the competition between the formation of more channels for the transfer of low molecular weight (LMW) silicone fluid and the reduction of LMW silicone fluid in the bulk. A hardness analysis and FTIR analysis demonstrated that further cross-linking reactions occurred during the treatment. SEM images showed the changes in roughness of the RTV silicone rubber surfaces. TG analysis also demonstrated the degradation of RTV silicone rubber by presenting evidence that the content of organic materials decreased during the temperature cycling treatment.

Keywords: room-temperature vulcanized silicone rubber; temperature cycling; hydrophobicity; transfer of hydrophobicity; hardness; functional group; microscopic appearance; thermal stability

1. Introduction

Pollution flashover accidents threaten the reliability and the safe operation of power systems, and they have been intensively studied in recent years [1–3]. Due to the promising properties, such as hydrophobicity and transfer of hydrophobicity, room temperature vulcanized (RTV) silicone rubber coatings are widely used to prevent pollution flashover, which has been proven to be effective in the presence of pollution and humidity [4–6]. However, numerous aging problems exist in the long-term use of RTV silicone rubbers, which can cause changes in their physical and chemical properties [7,8]. Since testing methods for evaluating the long-term reliability have not been completely standardized, it becomes vitally necessary to assess the effect of environmental factors and operating conditions on the properties of RTV silicone rubbers.

The degradation of silicone rubbers has been extensively studied and some valuable conclusions have been drawn. There were several factors responsible for the degradation, including corona, ultraviolet, liquid absorption, etc. [9–12], in all of which temperature was considered to be one of the

most crucial factors. Kumagai et al. [7] investigated the chemical changes of RTV silicone rubbers aged under thermal stress (250 °C to 300 °C), and the results showed that silicone oligomers of MD6M, MD7M and D8 disappeared and flammable byproducts were formed. Patel et al. [13] studied the extractable matter of RTV silicone rubbers heated at 190 °C and suggested that the thermal aging caused a significant increase in octamethylcyclotetrasiloxane (D4), which indicated the depolymerisation processes within the polymer network. Wu et al. [14] reported that the side chain reaction and the crosslinking reaction are the main reactions during the thermal oxidation aging of silicone rubbers in the temperature range from 120 °C to 250 °C. All of these studies concentrated on the degradation of RTV silicone rubbers at constant temperatures. However, so far few studies have explored the degradation of RTV silicone rubbers under temperature cycling, which has proven to be a major cause of the aging and failure of materials [15–17].

In the present study, temperature cycling between -25 °C and 70 °C was selected to investigate the degradation of RTV silicone rubbers. Experiments were carried out for 20, 40, and 60 days. Hydrophobicity, transfer of hydrophobicity, and hardness were analyzed, and Fourier transform infrared spectroscopy (FTIR), scanning electron microscopy (SEM), and thermogravimetry (TG) were employed to investigate the chemical and morphological changes. This research is intended for contributing to the better understanding of the aging of RTV silicone rubber materials, especially for those operating in areas with large difference in temperature between day and night, such as the Qinghai area in China [18].

2. Experimental Setup

Figure 1 shows the flowchart of the experiment. Pretreatment, temperature cycling treatment, and performance analysis were performed in sequence.

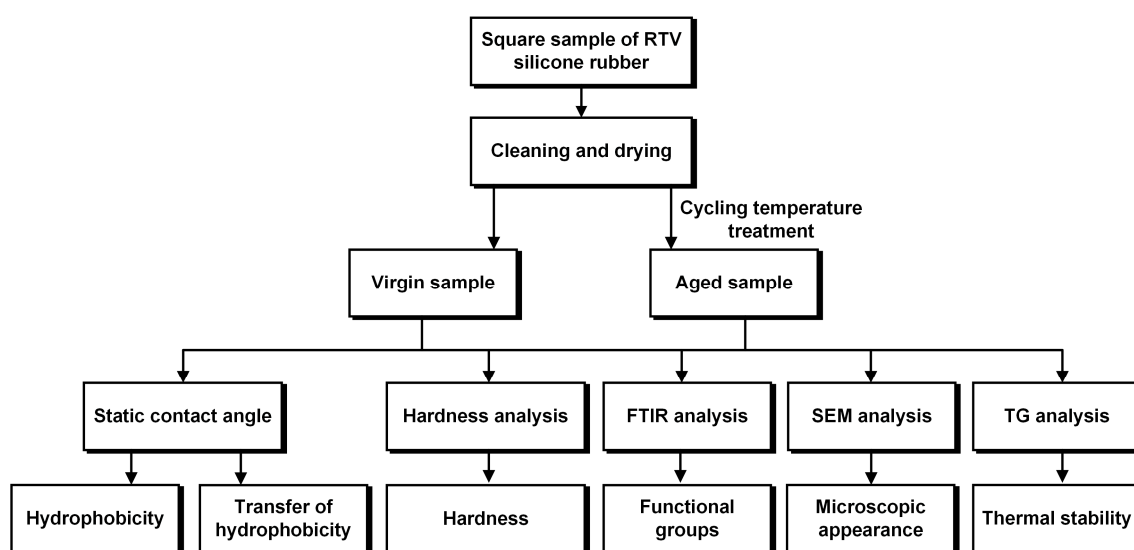


Figure 1. Flowchart of the experiment.

2.1. Materials

To reach some general conclusions, two types of commercial RTV silicone rubber coatings were used, denoted as Sample 1 and Sample 2. RTV silicone rubber plates were prepared by vulcanizing the coatings in a mold. To ensure the uniformity of sample preparation, samples were prepared simultaneously and the air temperature was at approximately 27 °C. Then they were initially cut into 60 mm × 60 mm × 2 mm square samples, cleaned with ethyl alcohol and deionized water in sequence, and then placed in a dust-proof container for 24 h for natural drying. For each type of RTV silicone rubber, four groups of samples were prepared, in which one group had no further treatment, namely

virgin samples, and the other three groups of samples were placed in a chamber for temperature cycling tests.

2.2. Temperature Cycling Test

To reflect the true state under temperature cycling and avoid unilateral destruction caused by high or low temperature, the crystallization temperature of RTV silicone rubber materials and the boiling point of silicone oligomers were not covered in the temperature range [7,19]. Thus, a temperature range from $-25\text{ }^{\circ}\text{C}$ to $70\text{ }^{\circ}\text{C}$ was selected, and the tests were performed in an XR-HWHS-225 constant temperature humidity chamber (Shenzhen Teelong Intelligent Equipment Co., Ltd., Shenzhen, China). The humidity was regulated automatically by a built-in combined system with a piece of gauze and a sink. As shown in Figure 2, the time for each temperature cycle was set to 24 h, in which cooling for 2 h, remaining at $-25\text{ }^{\circ}\text{C}$ for 10 h, heating for 2 h, and remaining at $70\text{ }^{\circ}\text{C}$ for 10 h were included. Aged samples after 20, 40, and 60 cycles were analyzed. It is noted that all of the aged samples were removed from the chamber at $27\text{ }^{\circ}\text{C}$ during the cooling process.

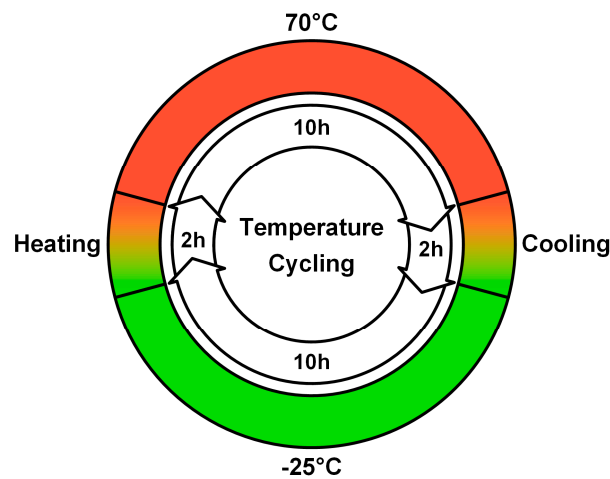


Figure 2. Diagram of a temperature cycle.

2.3. Performance Analysis Methods

For silicone rubber aging experiments, the hydrophobicity of samples is often characterized by the static contact angle using the $\theta/2$ method, as illustrated in Figure 3 [20], which assumed that the water drop profile is a circle.

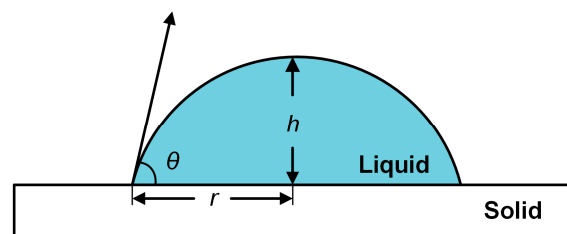


Figure 3. Static contact angle measurement using the $\theta/2$ method.

The value of the contact angle can be calculated using the following equation:

$$\theta = 2 \tan^{-1} (h/r), \quad (1)$$

the accuracy of such a method tends to be greatly influenced by the volume of the water drop and the hydrophobicity, because their increase would make the influence of gravity become obvious [21]. Given this, distilled water with a volume of 4 μL was used [22]. The measurement was carried out with an SL 200B contact angle measuring device (Shanghai Solon Information Technology Co., Ltd., Shanghai, China) at a temperature of 23 ± 2 $^{\circ}\text{C}$, and the contact angle of five points was obtained. After the minimum and maximum were removed, the average was plotted.

When hydrophilic contaminants are deposited on the surface of RTV silicone rubber materials, the surface of the pollution layer can gradually become hydrophobic. Several explanations can be advanced regarding the mechanism of the hydrophobicity transfer ability of silicone rubbers. The contribution of low molecular weight (LMW) silicone fluid and silicone rubber matrix was widely debated, and that of LMW silicone fluid was widely recognized [23]. As shown in Figure 4, small amounts of LMW silicone fluid transfer from the bulk to the surface of the pollution layer, resulting in the increase of hydrophobicity of the surface. As with the measurement of hydrophobicity, the transfer of hydrophobicity can be depicted by the static contact angle. A mixture of kieselguhr and NaCl was used to simulate the hydrophilic contaminant [24], and the weight of kieselguhr and NaCl was determined by the non-soluble deposit density of $1 \text{ mg}/\text{cm}^2$ and salt deposit density of $0.1 \text{ mg}/\text{cm}^2$ [25], respectively. Then, a small amount of distilled water was added to the mixture to prepare the contamination suspensions. With a soft bristle brush, the suspensions were deposited on the surface of samples, and the samples were placed in a dust-proof container for hydrophobicity transfer. The static contact angle of the contamination was measured at different transfer times.

The hardness of the silicone rubber samples was obtained using a LX-A Shore A durometer (Shanghai Liuling Instrument Factory, Shanghai, China). For each measurement, the hardness of five points was acquired and their average was plotted.

FTIR was utilized to analyze the change of the molecular structure and functional groups in the surface of samples [26], and specific molecules and groups located in the surface layer, typically 1 μm to 10 μm , could be identified by the attenuated total reflection technique. The FTIR measurements were performed with a Nicolet 5700 FTIR spectrometer (Thermo Fisher Scientific, Waltham, MA, USA). Samples were scanned from 675 cm^{-1} to 4000 cm^{-1} at a resolution of 0.5 cm^{-1} .

A Quanta 200 SEM (FEI Company, Eindhoven, The Netherlands) was used to capture SEM photographs at $1000\times$ magnification for the analysis of the surface condition of the samples.

To check the thermal stability of the samples, TG analysis was carried out with a Hitachi 7300 series TG-DSC analyzer (Hitachi, Ltd., Tokyo, Japan) at a heating rate of $20 \text{ }^{\circ}\text{C}/\text{min}$ under a N_2 atmosphere, and the TG curves for the samples in the temperature range of $100 \text{ }^{\circ}\text{C}$ to $700 \text{ }^{\circ}\text{C}$ in N_2 were plotted.

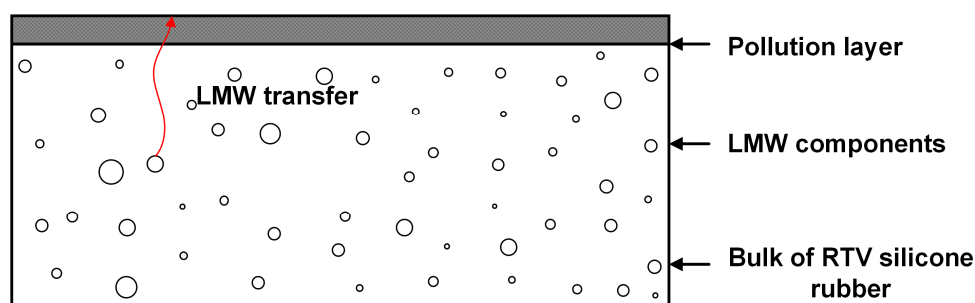


Figure 4. Transfer of LMW components from the bulk to the surface of the pollution layer [27].

3. Results and Discussion

3.1. Hydrophobicity

As shown in Figure 5, after the aging time of 20 days, 40 days, and 60 days, the static contact angles of both Sample 1 and Sample 2 change modestly in comparison with the virgin ones. This implies that the surface of the samples always remains hydrophobic, and temperature cycling has a slight effect on the hydrophobicity of the RTV silicone rubber surface.

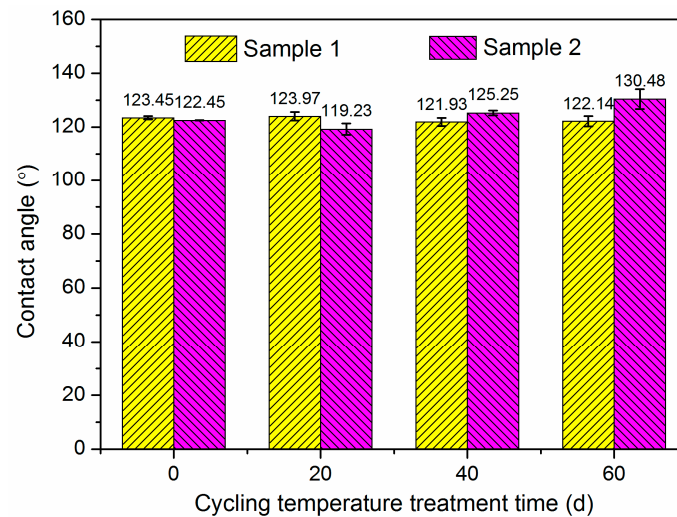


Figure 5. Contact angle of virgin samples and samples at different aging times.

3.2. Transfer of Hydrophobicity

Through measurement of the static contact angle of the pollution layers, which were formed by the drying of the suspensions, the hydrophobicity transfer ability of the samples was depicted. As shown in Figure 6, the temperature cycling can evidently influence the transfer of hydrophobicity of the samples. During the transfer time, the contact angle of the surface of the pollution layers increases for all samples. However, in comparison with the contact angle of the virgin samples, the contact angle for samples aged for 20 days is larger, that aged for 40 days is approximately equal, and that aged for 60 days is smaller.

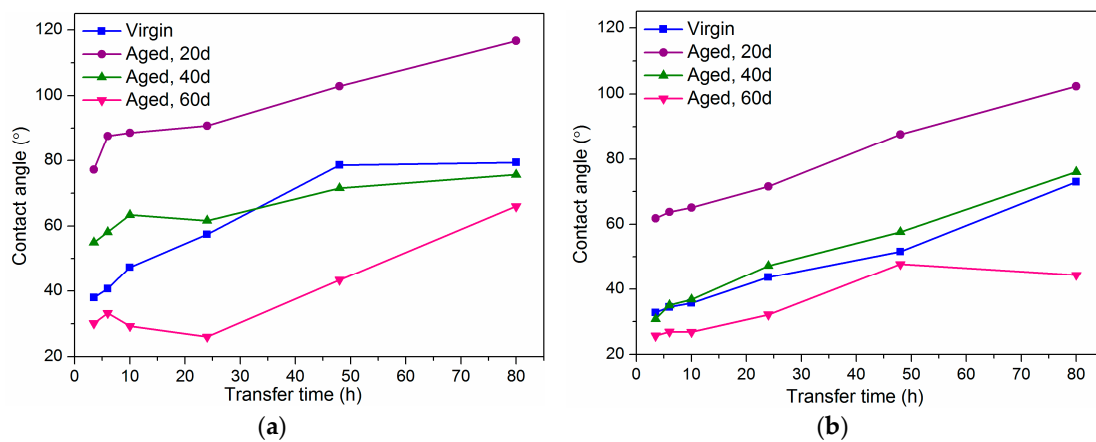


Figure 6. Contact angle of pollution layer covered on samples at different transfer times. (a) Sample 1; and (b) Sample 2.

As stated previously, the effect of temperature cycling treatment on the hydrophobicity of RTV silicone rubbers is slight. However, their transfer ability is strongly influenced. By considering the theory that the hydrophobicity transfer ability is contributed by LMW silicone fluid [23], the analysis results for hydrophobicity and its transfer ability can be explained as follows: During the temperature cycling treatment, more channels for transfer of LMW silicone fluid form and facilitate the evaporation and diffusion of LMW silicone fluid, leading to a competitive mechanism of the easier transfer for LMW silicone fluid and the reduction of LMW silicone fluid in the bulk. When the treatment time is short, the easier transfer for LMW silicone fluid dominates the transfer process because there are enough available LMW components. With the increase of the treatment time, the reduction of LMW silicone fluid is dominant.

3.3. Hardness Analysis

Figure 7 presents the hardness of virgin and aged samples. After temperature cycling, the hardness of the samples considerably increase. Notably, the hardness increases sharply in the first 20-day period, but slowly in the second 20-day period and the last 20-day period. Due to the difference in formula, the hardness of Sample 2 changes greater than that of Sample 1.

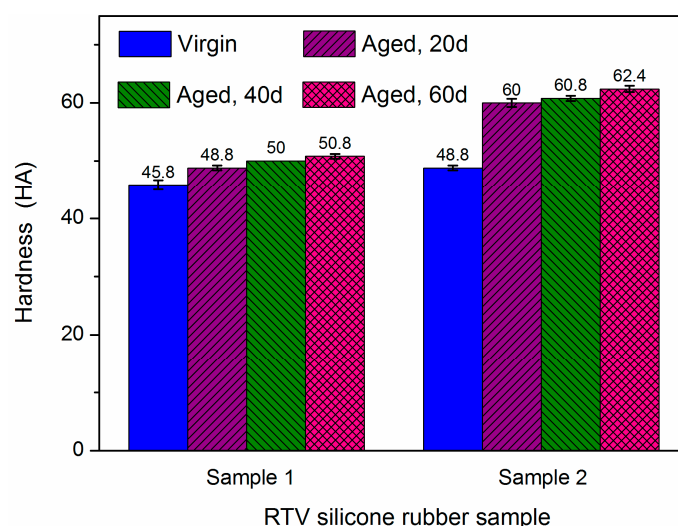


Figure 7. Hardness of virgin samples and aged samples at different aging times.

3.4. FTIR Analysis

The FTIR spectra of the virgin and aged samples are shown in Figure 8. By comparing the typical characteristics of the IR absorption for polydimethyl siloxanes [28,29], changes of groups in samples can be analyzed. The difference in the peak intensities can signify the difference before and after temperature cycling treatment for both types of samples. As shown in Table 1, the region from 3700 cm^{-1} to 3200 cm^{-1} indicates the formation of the O–H bond, which is attributed to the presence of alumina trihydrate (ATH), usually added as filler in silicone rubber materials. It can be observed that there is no ATH in Sample 2, but peaks with a low height appear. Similarly, for Sample 1, a slight change due to O–H is seen. This is a clear sign of mild hydroxylation of the RTV silicone rubber surface. The bond of CH_3 in Si-CH_3 also shows a slight increase. Most significantly of all, peaks due to Si–O–Si increase. Combining the results of hardness analysis, it can be explained that temperature cycling treatment may result in further cross-linking reaction. Studies performed by Schneider et al. [16] also showed similar results. In their experiments, two commercially-available silicones, namely RTV 615 and Sylgard 184, were exposed to temperature cycles ranging from $-40\text{ }^\circ\text{C}$ to $150\text{ }^\circ\text{C}$. Higher elastic moduli of aged samples compared with unaged samples was observed, and the crosslinking aging mechanisms leading to tighter polymer networks was emphasized [17]. These results presented in

this study may be attributed to the reaction of RTV silicone rubber with moisture in the air, which is controlled by the temperature.

Table 1. Characteristic FTIR absorption bands for PDMS [29].

Wavenumber (cm^{-1})	Bond
3700–3200	O–H
2960	C–H in CH_3
1280–1240	Si– CH_3
1100–1000	Si–O–Si
840–790	$\text{Si}(\text{CH}_3)_2$
700	$\text{Si}(\text{CH}_3)_3$

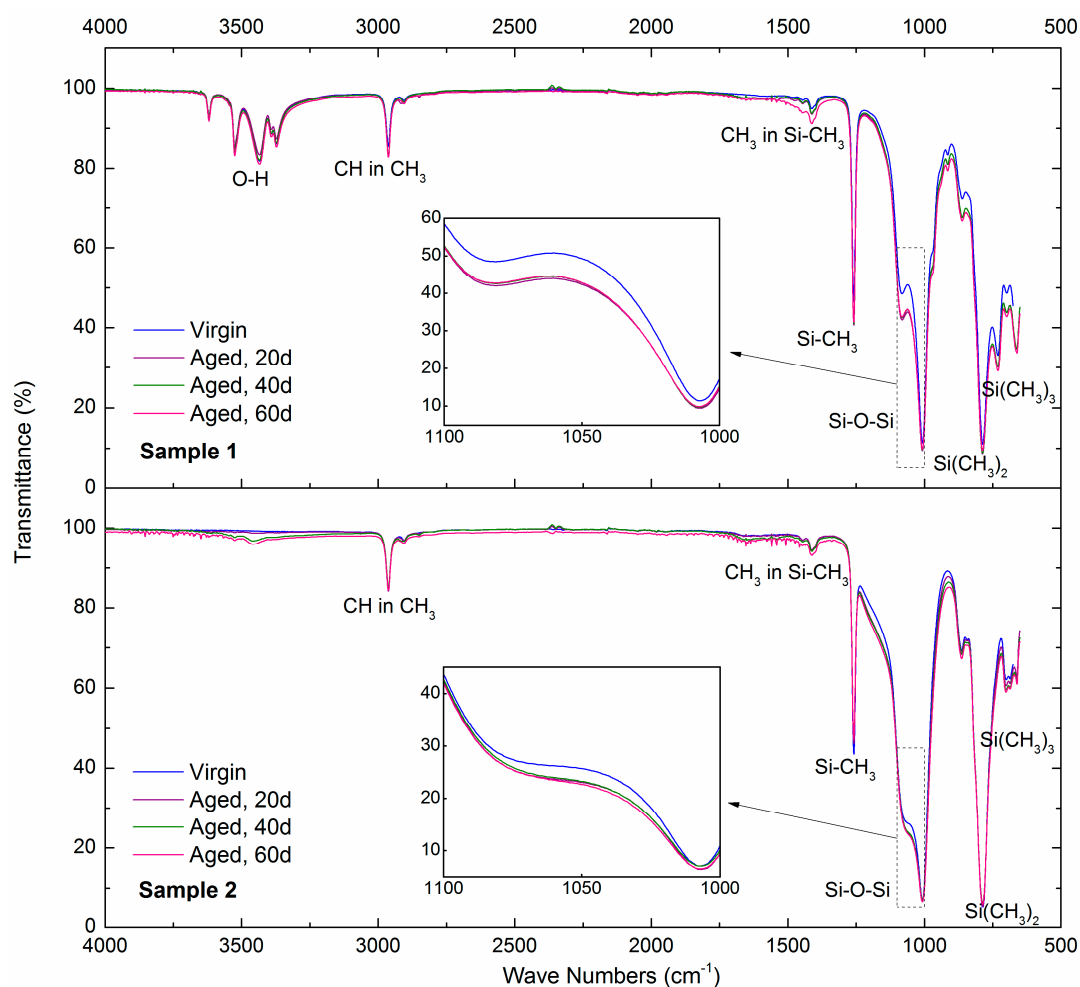


Figure 8. FTIR spectra of samples.

3.5. SEM Analysis

To study the microscopic appearance of virgin and aged samples, SEM analysis was conducted. The representative SEM images of virgin samples and samples aged for 20 days, 40 days and 60 days are presented in Figure 9. Figure 9a–d, represent the SEM images of virgin and aged samples for Sample 1, and Figure 9e–h represent those for Sample 2.

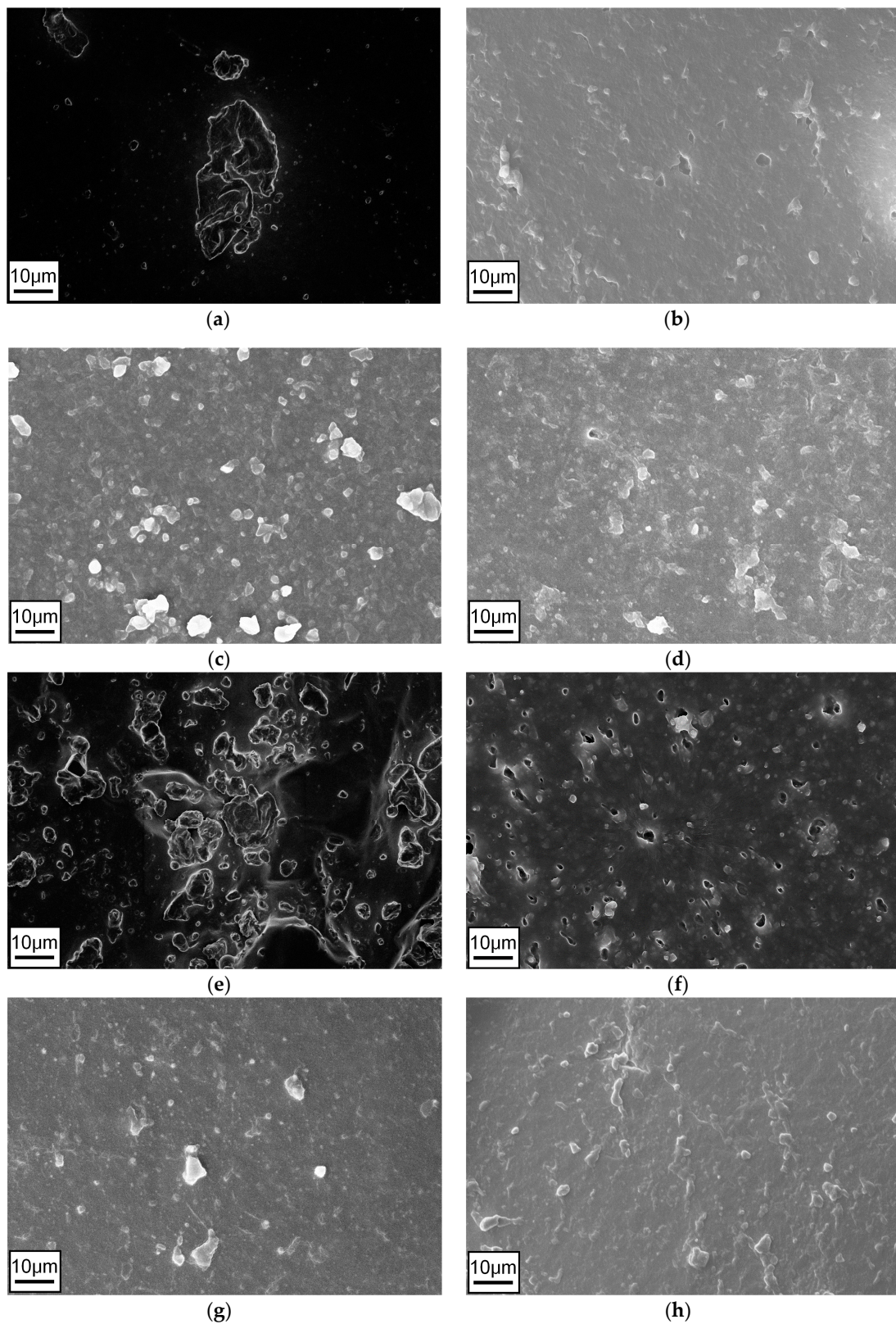


Figure 9. SEM images of RTV silicone rubber—1000× magnification. (a) Sample 1, virgin; (b) Sample 1, aged for 20 days; (c) Sample 1, aged for 40 days; (d) Sample 1, aged for 60 days; (e) Sample 2, virgin; (f) Sample 2, aged for 20 days; (g) Sample 2, aged for 40 days; and (h) Sample 2, aged for 60 days.

It can be observed from the representative images that the clustered fillers are dispersed in the RTV silicone rubber matrix for the virgin samples [30]. After the temperature cycling treatment, the surface microstructure of the samples is altered, and the roughness of their surfaces changes.

3.6. TG Analysis

For analysis of the thermal stability of the samples, TG curves were plotted in Figure 10. The TG curves differ dramatically between the two types of samples. With the increase of temperature, the weight declines rapidly at two portions of the curves for Sample 1, and the corresponding temperatures are approximately 340 °C and 484 °C. However, the weight declines rapidly only at approximately 545 °C for Sample 2. The occurrence of the weight loss stage at approximately 340 °C implies the dehydration of ATH for Sample 1 [31]. The residual weight of the aged samples is higher than that of the virgin samples. For instance, it can be seen from Figure 11 that residue of Sample 1 at 700 °C increases from 32.5% to 35.4%, 36.1%, and 36.9% for temperature cycling treatment time of 20 days, 40 days, and 60 days, respectively. Similarly, for Sample 2, it increases from 33.5% to 39.0%, 40.4%, and 42.7%, respectively. It can be concluded that a certain amount of the organic materials had already been lost in the air during the temperature cycling treatment time, leaving a large amount of inorganic filler materials in RTV silicone rubber samples. The boiling point of silicone oligomer for each size is tabulated in Table 2, in which M means $(\text{CH}_3)_3\text{SiO}_{1/2}-$ and D means $-(\text{CH}_3)_2\text{SiO}-$. The different boiling temperatures for the linear and cyclic silicone oligomers signify that a heat treatment at adequate temperature can remove the desired groups of silicone oligomers [8]. Since the cycling temperature range is considerably lower than the boiling points of linear and cyclic silicone oligomers, the loss of the organic materials may be the result of temperature cycling.

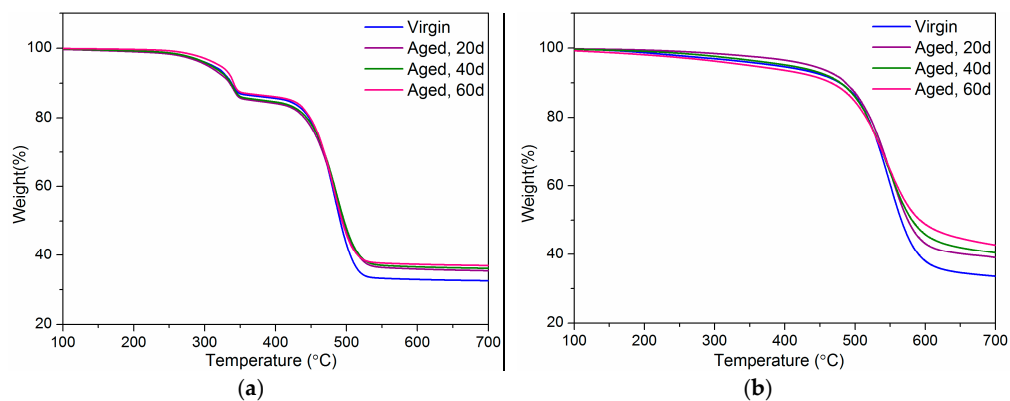


Figure 10. Thermogram of the decomposition of RTV silicone rubber samples before and after temperature cycling treatment. (a) Sample 1; and (b) Sample 2.

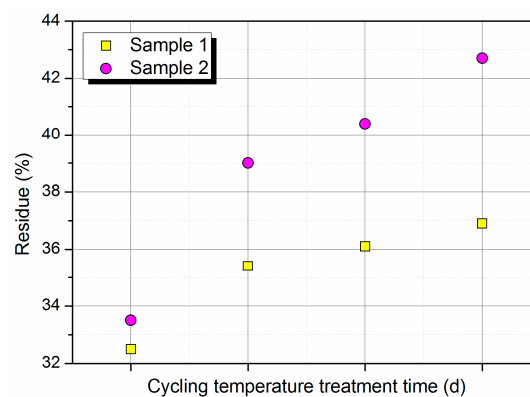


Figure 11. Residue data of samples from TG curves at 700 °C.

Table 2. Boiling points of linear and cyclic silicone oligomers. M: $(\text{CH}_3)_3\text{SiO}_{1/2}$ -, and D: $-(\text{CH}_3)_2\text{SiO}-$ [8].

Linear Structure	Boiling Point (°C)	Cyclic Structure	Boiling Point (°C)
MM	100	-	-
MDM	153	D ₃	135
MD ₂ M	194	D ₄	173
MD ₃ M	229	D ₅	207
MD ₄ M	242	D ₆	245
MD ₅ M	267	D ₇	254–265
MD ₆ M	287	D ₈	275–290
MD ₇ M	305	-	-

4. Conclusions

The present work investigated the degradation of RTV silicone rubbers under temperature cycling between -25 °C and 70 °C. Hydrophobicity and its transfer ability, hardness, functional groups, microscopic appearance, and thermal stability were analyzed. Some interesting conclusions were drawn.

- Modest change in hydrophobicity is observed after temperature cycling treatment, which can be attributed to the combined effect of silicone rubber matrix and small amounts of LMW silicone fluid on the surface of RTV silicone rubbers.
- After the temperature cycling treatment, the transfer ability of hydrophobicity is influenced remarkably. The remarkable change may result from the competition between the formation of more channels for transfer of LMW silicone fluid and the reduction of LMW silicone fluid in the bulk.
- Temperature cycling treatment results in the increase of hardness and the change in functional groups, which means that further cross-linking reaction may happen during the temperature cycling.
- Temperature cycling treatment alters the microscopic appearance of the surface of the samples, and the roughness of their surfaces changes.
- In the TG analysis, the increase of residues at 700 °C demonstrates the decrease of the content of the organic materials, which increases the weight ratio of inorganic filler materials in RTV silicone rubber samples.

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Author Contributions: Xishan Wen and Xiaoqing Yuan conceived and designed the experiments; Xiaoqing Yuan and Lu Hao performed the experiments; Lei Lan performed the hydrophobicity and its transfer ability measurements; Lu Hao performed the FTIR and the TG measurements; Shaodong Li performed the SEM measurements; Xishan Wen, Xiaoqing Yuan, and Lei Lan contributed to the interpretation of data; Zhenghong Bao contributed the RTV silicone rubber coatings; Xishan Wen and Xiaoqing Yuan wrote the paper; and Lei Lan, Yu Wang, and Hailiang Lu offered their modification suggestions for the manuscript.

Conflicts of Interest: The authors declare no conflict of interest.

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