Analysis of Physical–Chemical Properties and Space Environment Adaptability of Two-Component RTV Silicone Rubber

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ABSTRACT: Silicone rubber (SR) has the properties of organic−inorganic materials and good adaptability to the space environment, which can be used as an atomic oxygen (AO)-resistant layer on the spacecraft surface. In this study, SR coatings were used to spray on the polyimide surface to prevent atomic oxygen erosion. Then, the physical, chemical, and AO-resistant properties of the SR were investigated. By means of laser diffraction scattering particle size distribution tests, energy-dispersive spectroscopy elemental mapping, and X-ray photoelectron spectroscopy, the fillers and matrix were confirmed as precipitated silica and phenyl SR, respectively. Through analysis of swelling behavior, greater cross-linking density and less swelling ratio were observed at higher temperature and humidity. Furthermore, after AO was exposed with an accumulated fluence of 1.2 × 10\(^{21}\) atoms/cm\(^2\), cracks appeared on the coating surface with the change of element content. In addition, it was found that the degree of reaction between AO and SR should not be determined based on mass loss measurements. Our work showed the practical application and great potential of SR protective coating in spacecraft.

INTRODUCTION

Spacecraft will suffer from the harsh environment and synergistic effects in low earth orbit (LEO) at an altitude between 200 and 700 km\(^{1,2}\), such as high fluence of atomic oxygen (AO), changed particles, thermal cycling, and full spectrum of solar radiation, which can cause critical damages of polymers in spacecraft structures,\(^3\) so it is very necessary to protect the naked polymers from space flight. Because of the advantages of processability, chemical and thermal stability, and excellent adaptability for large-area complex surfaces, the protective coating is widely used for functional protection of the long-life spacecraft surface,\(^7\) e.g., thermal control coatings on satellite surfaces and AO protective coatings of solar array paddle.\(^5,6\) Polymide (PI) has been used on spacecraft for a wide variety of functions including solar array paddle, solar sail, satellite antenna, etc.\(^8\) However, as a prominent hazard in LEO, AO has an erosion effect on the PI film, which could reduce the reliability of the spacecraft.\(^9\) In order to enhance AO resistance of PI, various functional protective coatings, such as inorganic coating and organic coating, were developed.\(^9\) Among them, organic coating is seriously aging in the ultraviolet (UV) radiation environment and cannot meet the application requirements of long-life spacecraft.\(^9\) Despite the fact that inorganic coating has excellent antigen AO and UV radiation performance, there are several shortcomings as follows: (a) higher curing temperature to limit the application on various different substrate materials;\(^11\) (b) fragile character-
composition of organocsilicon, the two-component RTV SR combines the advantages of both inorganic and organic properties,\textsuperscript{16} which can achieve deep vulcanization without the limitation of the thickness of the adhesive layer.\textsuperscript{6}

The two-component RTV SR serves also as the interface adhesive between the solar cell and PI in solar arrays.\textsuperscript{20} Therefore, without changing the original bonding interface, coating the SR is a feasible technical means to protect the surface of the solar array paddle from AO erosion. At present, the internationally recognized space SR adhesives are DC 93500, RTV S691, and RTV S695.\textsuperscript{21} Ground simulation and space flight showed that they have good comprehensive performance, but their composition and preparation process have been less reported. This study focuses on the two-component RTV SR and describes its composition and physical—chemical and AO-resistant properties.

\section*{RESULTS AND DISCUSSION}

\textbf{Filler Elements and Particle Size Distribution.} The two-component RTV SR (film-forming material of the protective coating) contains components A and B: the component A is mainly composed of a matrix (hydroxyl capped polysiloxane) and reinforcing filler and the component B is mainly composed of a cross-linking agent.

As the dispersing phase and reinforcing body, the size and particle distribution can greatly affect the mechanical properties of SR composites.\textsuperscript{22} In order to gain further insights into the effects of the fillers, after solid—liquid separation of SR polymer solution, the fillers had been investigated by scanning electron microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDS), as can be seen in Figure 1.

The energy-dispersive X-ray spectroscopy (EDS) elemental maps have revealed the presence of Si and O elements. Therefore, it can be inferred that the filler is silica. Further, Figure 2 and Table 1 show the particle size distribution of silica by laser diffraction scattering particle size distribution tests. It can be concluded that the filler was precipitated silica.

Silica is often used as a reinforcing filler for SR,\textsuperscript{23} which can be divided into precipitated silica and fumed silica. By physical and chemical cross-linking (including covalent bonds, hydrogen bonds, van der Waals forces, electro-static interactions, etc.) between the filler and the SR, it could significantly improve the mechanical properties and thermostability of the SR composite. However, the residual Si—OH will cause uneven distribution and agglomeration of fillers in SR. In addition, researchers found that the hydroxyl groups at the polymer chain ends or on the filler surface initiate the thermal degradation of SR via a “back-bite” reaction.\textsuperscript{24} Therefore, it is necessary to passivate the particle surface while maintaining the chemical bonds to improve the compatibility of the filler with the SR composite. The usual use of a coupling agent between particles and the matrix was the approach to reduce agglomeration of the fillers and to even change the comprehensive performance of the SR.\textsuperscript{19}

\textbf{Resin Composite of Silicone Rubber.} X-ray photoelectron spectroscopy (XPS) was performed to investigate the composition and the chemical states of the silicone rubber. Figure 3 shows the survey XPS with insets presenting the high-resolution XPS of C 1s, O 1s, N 1s, and Si 2p. The peaks in C 1s XPS can be deconvoluted into two components centered at 283.8 and 291.4 eV, which were related to the phenyl. The O 1s peaks are centered at 530.4, 532.3, and 532.7 eV, which are related to the group of SiO2, Si-O-Si, and Si-O, respectively. The existence of the residual silica filler particle in the resin composite of RTV SR and the Si-O-Si group in the formed cross-linked network structure was verified. The peaks of Si 2p XPS at 102.6 eV, 100.6 eV and N 1s XPS at 398.5 eV, 399.8 eV were related to the Si-O, Si—N group and the —N=, —NH group, respectively. Therefore, it could be concluded that the type of cross-linking agent in the two-component SR was polysilazane.

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**Figure 1.** Scanning electron microscopy (SEM) photograph of solid fillers (left). Energy-dispersive X-ray spectroscopy (EDS) of filler elements (right).

**Figure 2.** Particle size distribution curve of silica.

**Table 1.** Test Results of Silica’s Particle Size Distribution

| Num | $S_g$ (m$^2$/cm$^3$) | RR-N | particle size ($\mu$m) | $\sigma_g$ | $\sigma_{50\%}$ ($\mu$m) |
|-----|---------------------|------|------------------------|----------|---------------------|
| 1#  | 0.719               | 2.556| 2.15—38.86             | 1.766    | 11.540              |
| 2#  | 0.688               | 2.184| 2.55—38.86             | 1.851    | 12.479              |

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Based on the residual small amount of nitrogen, it could be inferred that the cross-linking agents contained a “Si-N-Si” bond or “N-H” bond, and the type of cross-linking reaction of the vulcanized SR was deamination. It was confirmed that the structure of samples was phenyl SR through the analysis results.25 Through steric hindrance and conjugation effects of the phenyl group, it can prevent the cross-linking and degradation of the main chain caused by the oxidative decomposition. Phenyl SR has better thermal stability and excellent and higher residue after ablation than other SR and can maintain elasticity even when γ rays are as high as $1 \times 10^9$.

The group of phenyl would improve the low temperature elasticity of SR (the glass transition temperature $T_g$ down to $-120^\circ$ C).

Swelling Characteristics. The cross-link bond and cross-link density are intrinsic properties of high-molecular-weight polymers. Among them, with rising cross-link density, the cohesive strength of silicone rubber could be increased. Also, the effects of cross-link bonds are related to fatigue resistance and thermal oxidation aging properties.24 After cross-linking and curing, the physical properties of SR have been greatly changed. Therefore, it is necessary to study the curing reaction kinetics of SR by testing the cross-linking characteristics (swelling ratio, average molecular weight between cross-links, and cross-link density) under different curing conditions (component ratio, temperature, and humidity).

In order to obtain the actual environmental conditions and fully cured SR, a constant temperature and humidity box was used to control the curing conditions and environment (temperature: $22 \pm 1^\circ$ C; humidity: $50 \pm 3\%$; ratio of A:B components: 20:1; curing time: 20 days). The trend of swelling mass for the cured sample with time was determined in ethyl acetate solvent, and the corresponding formula parameter values (equilibrium swelling ratio, average molecular weight, and swelling density) are shown in Table 2 by carefully examining eqs 3–5.

Table 2. Swelling Date of the Silicone Rubber

| Processor | Equilibrium Swelling Ratio | Average Molecular Weight (g·mol$^{-1}$) | Swelling Density ($\times 10^{-5}$ mol·cm$^{-3}$) |
|-----------|-----------------------------|-----------------------------------------|---------------------------------------------|
|           | 2.47                        | 11,800                                   | 10.28                                       |

Swelling Ratio at Different Ratios of Two Components for RTV Silicone Rubber. The swelling curve and swelling ratio of SR at different proportions (the ratios of A and B components are 18:1, 19:1, 20:1, 21:1, and 22:1) are shown in Figure 4. It can be seen from the figure that with the increase in component A, the swelling ratio tends to increase.

Swelling Ratio at Different Temperatures and Humidities of Two Components for RTV SR. In addition to the ratio of two components, the curing conditions such as temperature and humidity of the SR also influence the swelling. Compared to a swelling ratio of 4.15 at 25 °C and 10% RH, the values of the swelling ratio were 3.64 and 2.97 at 35 °C and 80% RH, respectively (Figure 5). It has been previously shown that increasing the curing temperature and humidity can reduce the swelling ratio.

As can be seen in the above conclusion, when the matrix and fillers have been determined, the cross-linking density of SR can be changed by controlling the proportion of cross-
linking agents or changing the curing conditions so as to control key performance indicators such as mechanical strength, hardness, and elasticity.

**Mass Loss and Erosion Yield of Coating Samples.**

Mass loss measurements were carried out for measuring erosion effects of AO-exposed samples; the samples covered with SR coatings of different thicknesses (the coating thicknesses named 1#, 2#, 3#, and 4# were 20, 40, 60, and 80 μm) were exposed to AO for 1 and 240 h with cumulative fluence values of $4.9 \times 10^{18}$ atoms/cm² and $1.2 \times 10^{21}$ atoms/cm², respectively. The effective values of mass loss for ground-laboratory exposed samples are shown in Table 3 (the average mass loss values of vacuum reference samples were 1.46 and 2.18 mg after 1 and 240 h AO exposure, respectively).

As can be seen in Table 4, the samples numbered 2#, 3#, and 4# (the thicknesses of coating were 40, 60, and 80 μm, respectively) experienced greater mass loss during AO exposure after 1 h (cumulative fluence is $4.9 \times 10^{18}$ atoms/cm²); the mass loss of the coatings under AO exposure did not increase as the cumulative fluence increased. The sample 1# (the thickness of coating was 20 μm) obtained greater mass loss compared to 2#, 3#, and 4#, which could be due to the thinner SR coating that failed to form a dense protection for the base material; as the cumulative fluence increased, the AO caused further erosion for PI.

**Table 3. Mass Loss of the Samples**

| Num | pristine (mg) | 1 h ECR AO (mg) | 240 h ECR AO (mg) | mass loss 1 h | mass loss 240 h |
|-----|---------------|----------------|------------------|---------------|-----------------|
| 1#  | 165.71        | 162.31         | 161.37           | 1.94          | 2.16            |
| 2#  | 197.52        | 195.14         | 194.70           | 0.92          | 0.64            |
| 3#  | 231.92        | 229.32         | 229.06           | 1.14          | 0.68            |
| 4#  | 267.03        | 264.32         | 263.94           | 1.25          | 0.91            |

**Table 4. Surface Composition of SR Coatings**

| AO fluence               | element (at %) | C      | O      | Si     | N      |
|--------------------------|----------------|--------|--------|--------|--------|
| pristine sample          |                | 42.27  | 32.46  | 24.29  | 0.98   |
| $4.9 \times 10^{18}$ atoms/cm² |                | 34.82  | 41.91  | 22.08  | 1.19   |
| $1.2 \times 10^{21}$ atoms/cm² |                | 27.53  | 38.03  | 33.01  | 1.43   |

**Surface Morphologies.** The samples were exposed to directed ram AO from within the shuttle bay, which were 30 mm in diameter and were approximately 20–80 μm thick. After exposure to AO (fluence of about $1.2 \times 10^{21}$ atoms/cm²), the initially smooth paint surface cracked into well-distinguished damage. Pristine and post-exposure images (exterior and micrographs) of the sample surface were observed by unaided eye and electron microscope (magnification: 500X) in Figures 6 and 7.

In order to further distinguish the surface morphology characteristics of the coating after AO exposure, SEM investigation was conducted, as shown in Figure 8. Through observing the SEM image, the sizes of the cracks were approximately 6 μm in width and 5.5 μm in depth. The cracks of the coating did not penetrate into the surface of the base material, which indicated that the SR has resistance to the effect of AO. The SR coating with certain thickness can be used as an inert dissipative protective layer to effectively protect PI from AO.
XPS Analysis. The XPS analysis depth is approximately 30 atomic layers and about 10% signal from the outermost atoms on the surface of the test sample. The surface element composition of the SR coating for pristine and post-exposure samples in AO was detected by XPS, as shown in Table 4.

The XPS results showed that after 24 h AO exposure (equivalent AO fluence, $4.9 \times 10^{18}$ atoms/cm$^2$), the carbon content decreased slightly and the oxygen content increased significantly from Table 5. Due to the weak bond energy of carboxyl or methyl, the SR could be degraded under the action of high kinetic energy and oxidized AO, and then a chemical reaction occurred between carbon and AO to produce CO and CO$_2$. After 240 h AO exposure (equivalent AO fluence, $1.2 \times 10^{21}$ atoms/cm$^2$), the carbon content decreased from 42.27 to 27.53% and also the silicon content increased from 24.29 to 33.01%. It can be inferred that part of the oxygen and silicon elements changed from a low binding energy state to a high binding energy state, which was caused by the silicon oxide layer that has been formed. The generation of a silicon oxide layer could prevent AO from eroding the underlying substrate material. However, the mismatch in elastic moduli and thermal expansion coefficient between the two layers (silicon oxide layer and SR layer) could lead to further excessive residual stresses, which would cause delamination or cracks of the

### Table 5. Mass Loss of Exposed Kapton H

| Num | 1#  | 2#  | 3#  | 4#  | 5#  | 6#  | 7#  |
|-----|-----|-----|-----|-----|-----|-----|-----|
| $M_{1m}$ (mg) | 102.65 | 96.41 | 99.69 | 103.28 | 100.85 | 84.39 | 82.96 |
| $M_{2m}$ (mg) | 98.50 | 97.29 | 99.65 | 99.78 | 93.78 | 74.52 | 84.70 |
| $M_{3m}$ (mg) | 100.26 | 94.28 | 97.47 | 101.22 | 98.83 | 82.52 | 81.16 |
| $M_{4m}$ (mg) | 97.02 | 95.94 | 98.30 | 98.44 | 92.65 | 73.69 | 83.81 |
| $(M_{1m} - M_{3m}) - (M_{2m} - M_{4m})$ | 0.91 | 0.78 | 0.81 | 0.82 | 0.89 | 1.04 | 0.91 |
coating. In addition, a small amount of nitrogen was also detected, which may be due to the incomplete deamination reaction during the cross-linking of the two-component SR.

Through element content analysis, it can be shown that in the AO exposure test of SR coatings, both the loss of carbon content and the new product of silicon oxide occurred simultaneously. As the cumulative fluence of AO was increased and the exposure time was extended, it would cause degradation and re-cross-linking of the molecular chain. Therefore, it is not scientific to rely on measuring the mass loss of SR samples to judge their AO-resistant performance. In addition, the alternating high and low temperature causes stress and strain on the coating surface in LEO, which will further accelerate the aging and degradation of SR. Therefore, synergistic effects of the space environment should be carried out.

**CONCLUSIONS**

The two-component RTV SR could be used as a film-forming material of protective coating on PI surfaces. The filler composition, type of matrix, swelling behavior, and AO-resistant properties of the RTV SR were investigated. First, the solid and liquid contents of SR solution dissolved in ethyl acetate were separated by suction filtration. Through EDS and laser diffraction scattering particle size distribution tests, the filler of SR was confirmed as precipitated silica with a median particle size that ranged from 11.54 to 12.48 nm. Then, XPS analysis was further conducted, confirming that the type of matrix was phenyl silicone rubber. Through analyzing the swelling behavior, it showed that increasing the curing temperature and humidity or reducing the component A of SR could reduce its swelling ratio (related to cross-linking characteristics). Therefore, it is an effective means to regulate the cross-linking characteristics of SR by controlling the curing conditions such as component ratio, temperature, and humidity, thereby determining its bonding strength and mechanical properties.

The SR coating with a thickness 40 μm can prevent further erosion of the base material with a cumulative fluence of 1.2 × 10^{21} atoms/cm^{2} by AO radiation. However, cracks and the change of element contents appeared on the surface of the SR coating by analyzing the surface morphology. It was observed that the traditional methods for determining the degree of reaction between AO and SR in laboratory simulation techniques based on mass loss measurement should not be used.

**EXPERIMENTAL SECTION**

**Materials.** The two-component RTV SR was manufactured by Shanghai Resin Factory Co., and it was supplied as a two-part liquid component kit. The Kapton H film (PI, 25 μm thick) was purchased from Dupont Co.

**Ground-Based Simulation and AO Exposure Tests.** AO exposure tests were carried out in an electron cyclotron resonance (ECR) facility at the Shanghai Institute of Space Power Sources, which was controlled to an energy of ~5 eV by adjusting the bias plate. The AO fluence at the sample position was calibrated using a bare Kapton film based on mass loss measurements by eq 1

\[
\Phi = \Delta m/(\eta \rho t)
\]  

where \(\Phi\) is the AO fluence in atoms/cm^{2}; \(\Delta m\) is the exposed area of the sample in cm^{2} (the diameter of the samples were 30 mm); \(\eta\) is the erosion yield (3.0 × 10^{24} cm^{2}/atom); \(\rho\) is the density of the sample (1.42 g/cm^{3}); \(t\) is the exposure time in s; and \(\Delta m\) is the mass change of the exposed sample in mg. The calculation method of \(\Delta m\) is provided by eq 2, where \(M_{pr}\) is the pristine mass of the sample, \(M_{f} \) is the pristine mass of the vacuum reference sample, \(M_{te}\) is the mass of the sample after exposure tests, and \(M_{le}\) is the mass of the vacuum reference sample after exposure tests

\[
\Delta m = \sum [(M_{in} - M_{in}) - (M_{2n} - M_{le})]/n
\]  

The corresponding samples’ mass loss effective fluence values are shown in Table 5, the \(\Delta m\) was 0.88 mg, and it could be concluded that the AO fluence was 1.35 × 10^{15} atoms/cm^{2}.s.

**Coating Preparation.** The SR was chosen as the film-forming substance of the coating, while xylene and KH550 were chosen as the solvent and coupling agent. Briefly, the molar ratio of silicone rubber:xylene was selected as 1:2.5 to prepare a mixed solution. Then, these solutions were sprayed on Kapton H films and thermally treated in a furnace at 60 °C for 0.5 h. The thicknesses of the coating were 20, 40, 60, and 80 μm. The samples with a diameter of 30 mm were used in the AO exposure experiments, which were exposed 1 and 240 h, equivalent to effective fluence values 4.9 × 10^{16} and 1.2 × 10^{17} atoms/cm^{2}, respectively.

**Characterization of Physical–Chemical for SR.** The factors that affect the mechanical strength and durability of SR such as species and distribution of fillers, group type of component, and swelling characteristics determine the adaptability of nanocomposite materials in the space environment.

**Composition of Solid Fillers and Resin Composite.** The SR base dissolved in the ethyl acetate solution was suction-filtered with a filter membrane until there was no solid residue in the upper layer and the liquid resin was free of turbidity. By washing and drying, the mass of the filler and the liquid resin were weighed. A mixture of ethanol and water (molar ratio: 1:1) was used to ultrasonically disperse solid powder fillers, and laser diffraction scattering particle size distribution tests and energy-dispersive X-ray spectroscopy (EDS) were performed to measure the microscopic particle size and the element type of the fillers, respectively. High-resolution XPS were obtained from the fixed silicone rubber surface, which were separated into C 1s, O 1s, Si 2p, and N 1s peaks.

**Swelling Characteristics.** The cross-linking density of the SR was obtained by testing the swelling characteristics and calculating the equilibrium swelling ratio. These samples were approximately 15 × 15 mm in size and approximately 1 mm thick.

The swelling ratio (S) was measured in ethyl acetate. The obtained silicone rubber was weighed as \(w_{s}\) in g; the silicone rubber was subsequently immersed in ethyl acetate until equilibrium was reached and weighed as \(w_{e}\) in g; the swelling ratio (S) was calculated using eq 3

\[
S = (w_{e} - w_{s})/w_{0} \times 100\%
\]  

The average molecular weight between cross-links (\(M_{c}\)) was calculated by eq 4, where \(M_{w}\) is the number-average molecular weight of the initial uncross-linked polymer, \(\nu\) is the specific weight of the initial uncross-linked polymer, \(\nu\) is the specific
volume of the polymer, $V_1$ is the molar volume of solution, $V_2$ is the polymer volume fraction, and $x_1$ is the polymer solvent interaction parameter

$$\frac{1}{M_C} = \frac{1}{M_n} - \left(\frac{x_1}{V_1}\right)\ln(1 - V_2) + V_2 + \chi(V_2)^2 \left(\frac{V_2}{V_1}\right)^{1/3} - V_1^{1/3}$$

Cross-link density ($\rho_c$) can be calculated as follows (eq 5):

$$\rho_c = \frac{1}{\nu M_C}$$

Characterization of AO-Exposed Samples. The surface morphology effects of AO-exposed samples were studied by several complementary techniques including electron microscopy, scanning electron microscopy (SEM), and X-ray photoelectron spectroscopy (XPS). The microscopy image magnified 500X was observed using an LV150 electron microscope. SEM micrographs were obtained by using a FEI Quanta 200 microscope operating in low vacuum mode. The element contents and chemical bonding states in the near-surface region were assessed by XPS measurements including C 1s, O 1s, Si 2p, and N 1s.

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**Notes**

The authors declare no competing financial interest.

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