

Antiadhesion elastomer seal coatings for UV and atomic oxygen protection

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ABSTRACT: Radiation blocking sunscreen coatings have been developed for the protection of elastomer seals used in low-Earth-orbit (LEO). The coatings protect the seals from ultraviolet (UV) radiation and atomic oxygen (AO) damage. The coatings were developed for use on NASA docking seals. Docking seal damage from the UV and AO present in LEO can constrain mission time-line, flight mode options, and increases risk. A low level of adhesion is also required for docking seals so undocking push-off forces can be low. The coatings presented also mitigate this unwanted adhesion. Greases with low collected volatile condensable materials (CVCM) and low total mass loss (TML) were mixed with slippery and/or UV blocking powders to create the protective coatings. Coatings were applied at rates up to 2 mg/cm². Coated seals were exposed to AO and UV in the NUV (near-UV) and UV-C wavelength ranges (300 to 400 nm and 254 nm, respectively). Ground based ashers were used to simulate the AO of space. The Sun's UV energy was mimicked assuming a nose forward flight mode, resulting in an exposure rate of 2.5 MJ/m² day. Exposures between 0 and 147 MJ/m² (UV-C) and 245 MJ/m² (NUV) were accomplished. The protective coatings were durable, providing protection from UV after a simulated docking and undocking cycle. The level of protection begins to decline at coverage rates less than 0.9 mg/cm². The leakage of seals coated with Braycote + 20%Z-cote ZnO sunscreen increased by a factor of 40 after moderate AO exposure; indicating that this coating might not be suitable due to AO intolerance. Seals coated with DC-7-16.4%Z-cote ZnO sunscreen were not significantly affected by combined doses of 2×10^{21} atoms/cm² AO with 73 MJ/m² UV-C. Unprotected seals were significantly damaged at UV-C exposures of 0.3 MJ/m² and DC-7-16.4%Z-cote coated seals were undamaged at all exposures up to the limits tested thus far which were 147 MJ/m² UV-C and 245 MJ/m² NUV. The coatings decreased adhesion sufficiently for docking seals at temperatures equal to or greater than -8°C thus offer a simple and inexpensive way to mitigate adhesion. © 2014 Wiley Periodicals, Inc. *J. Appl. Polym. Sci.* **2015**, *132*, 41662.

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INTRODUCTION

The National Aeronautics and Space Administration (NASA) is currently developing docking mechanisms and seals for their next manned spacecraft.^{1,2} The docking mechanism and seal are expected to be located at the nose of a conical capsule similar in shape to the crew modules used during the Apollo era.³ The docking system is expected to use a set of two silicone-rubber seals, which are connected together by a web of the same material and held in place by a metal retaining ring. Silicone-based elastomers were chosen as the seal material primarily because silicone-rubbers meet the wide operating temperature range for space applications⁴ and do not lose mass due to atomic oxygen (AO) erosion. These seals are expected to be exposed to the space environment whenever the spacecraft is not docked. Several environmental hazards are present in space; these hazards depend on flight altitude and spacecraft orientation as well as

several other factors such as seasonal variations.^{5,6} Prior work has shown that the greatest environmental threats to the seal are ultraviolet (UV) radiation and AO.⁷⁻¹¹ Protection from the effects of AO and UV are needed, especially for longer missions.

Two UV protection methods were explored: (1) mixing a UV blocking compound into the elastomer prior to curing; (2) topical application of a UV blocking sunscreen coating. Sunscreen agents are generally classified as either organic (also referred to as chemical) or inorganic (also referred to as physical). The inorganic agents TiO₂ and ZnO were examined because of their availability, common use, low reactivity, and stability in vacuum.¹²⁻¹⁴ Parker, the manufacturer of the S0383-70 elastomer compound, chose TiO₂ as the additive to the proprietary elastomer. The sunscreen coating was made with ZnO because ZnO attenuates a wider swath of the UV spectrum. The objective of the research presented here was to determine the effectiveness

of UV blocking inhibitors added to the elastomer compound and UV blocking coatings to prevent damage from UV and AO.

A second problem that must also be addressed with the development of docking and hatch seals is related to adhesion. Silicone-based elastomers are naturally sticky. This stickiness helps to make a good seal; however, can cause problems when spacecraft undock.^{15,16} There are several options to mitigate this unwanted adhesion such as targeted use of AO,^{10,15} radiation in the vacuum-UV range <200 nm,^{17,18} and application of a suitable grease.¹⁹ The grease-based sunscreens developed to block UV were also designed to control adhesion; thus another objective of the research presented here was to sufficiently lower seal adhesive forces with the application of a UV blocking sunscreen.

There are several other uses that the sunscreens presented here might be beneficially applied to; here are some examples:

- An antiadhesion coating could consist of boron nitride added to the grease- this would be applied to a hatch seal, or the seal between the heat shield and crew module to ensure low adhesion forces when the heat shield is released after re-entry, and before parachute deployment.
- As a coating on seals used in motor vehicles, especially around the windshield and windows, or on tire side-walls.
- As a coating on the rubber seals on solar panels.

Space-qualified grease has been used in the past by NASA to lubricate gears and to ease the placement of elastomer seals.^{20,21} The greases examined in this study were Braycote Micronic 601 EF, a space rated perfluoroether grease²²; and Dow Corning 7 Release Compound (DC-7), which is a dimethyl silicone lubricant.²³

Vacuum leak rates of seals designed for spacecraft air locks are presented by Trout Jr.²⁴ Prior work on the effects of the space environment on silicone rubber and elastomer seals can be found in Refs. 7–10. Daniels *et al.* has presented the results of three different compounds of silicone rubber exposed in ground-based facilities and in orbit as part of NASA's Materials International Space Station Experiment (MISSE-6)²⁵; small #2-309 O-rings were exposed in space to an AO fluence up to 1.2×10^{22} atoms/cm² and 2620 ESH (equivalent Sun hours, ~ 1045 MJ/m²) of UV. These compounds were designed by their manufacturers to be UV durable. Daniels *et al.* found that damage incurred during orbit was less when the seals were given an AO pretreatment prior to exposure in space. This decline in damage was perhaps due to a change in UV absorbance caused by changes in reflectivity brought about by the AO pretreatments, which give silicone elastomers a glossy surface finish. The seals we are considering are expected to leak through bulk permeation as well as through the interface between the seal and mating counterface.²⁶ It is hoped that use of a protective sunscreen like those presented will both lower and stabilize leakage through the mating surface interface. Prior work by Gudimenko *et al.* presented a Si-rich coating (Photosil) for protection from AO and UV²⁷ wherein Photosil was placed on various polymer films and thermal control paints for space applications. Mass loss was prevented, however, exposure to

near-UV was not included. The lack of mass loss of silicone rubbers when exposed to AO is well known;²⁸ however, a solution to the deleterious combined effects of AO and UV in LEO was not found in the literature.

Silicone elastomer seals were made and tested before and after exposure to AO and UV [with wavelengths near 254 nm (UV-C) and 350 nm (near-UV also known as NUV)]. Seals were exposed and tested with and without sunscreen coatings. Seals of the base compound S0383-70 and with TiO₂ added were included. Seal adhesion and leak rate were measured before and after exposure; leakage was determined using a pressure decay method previously presented.²⁹

EXPERIMENTAL

Specimens

Specimens were made from the silicone elastomer S0383-70, a rust colored silicone elastomer with a Shore Hardness A durometer rating of 70 supplied by the Parker Hannifin Corporation, CSS-Division. In an effort to increase the compound's resistance to UV exposure, a second compound was created in which 6 wt % TiO₂ was added to the compound prior to molding and curing.

The full-scale docking seal this research applies to is ~ 50 inches (1.27 m) in diameter. Two types of sub-scale seals were used in this study for exposure and leakage testing:

- Medium-scale seals (Mss) consisting of a double bulb and web between the bulbs, held in place with a retainer ring, similar in design to those presented in Ref. 30. The centerline diameters of the two seals were about 10.2 inches (26 cm) and 11.5 inches (29.2 cm).
- Small-scale seals with an inner diameter of 0.41 inches (10.4 mm) and an outer diameter of 0.83 inches (21.1 mm). Small-scale seals were made from 0.21 inch (5.3 mm) thick sheets of S0383-70; these washer style seals were cut from the sheets using a drill press and custom-made core drill lubricated with soap. Figure 1 shows examples of as-received small-scale seals made of S0383-70 and with 6 wt % TiO₂ added.

Four different types of specimens were used for adhesion testing: flat topped buttons and #2–309 O-rings similar to those described previously,¹⁵ sections cut from Mss, and Mss. Figure 2 shows examples of these adhesion test specimens after being tested. It was believed that some of the adhesion of sunscreen coated seals was caused by a vacuum effect between the greased flat surfaces. To test this, #2-309 O-rings were glued to a 1 inch square block, which was vented to prevent any pressure effects from the inner volume; this vented block is shown in Figure 2.

Coatings

The general scheme in the development of UV protective coatings was to combine a slippery release agent with a physical UV blocking additive. Several issues need to be considered when choosing the grease and blocking additive such as the outgassing of the grease, and the effect of the UV blocking additive on seal leakage. The following 2 greases and 2 blocking agents were down-selected and used in this study:



Figure 1. Small-scale seals: baseline on left, with 6% TiO₂ on right, 0.21 inches thick.

- Braycote Micronic 601 EF, a low temperature, high vacuum perfluoroether grease made by Castrol. Braycote 601 is a flight qualified grease with low outgassing and an established history in NASA's flight program.
- Dow Corning 7 Release Compound (DC-7), is a very low outgassing silicone-based lubricant.
- UV blocking agent selected was ZnO in the form of uncoated Z-cote, a zinc oxide powder with a particle size less than 0.2 μm ; Z-cote is frequently used as an additive in sunscreens and cosmetics, and is made by BASF Corporation.
- Boron nitride (BN) powder was examined because of its favorable friction and release characteristics. The BN powder used was grade SHP-325 (-325 mesh, <50 μm diameter particles) supplied by Standard Oil Engineered Materials Company.

The sunscreen mixtures were made by hand-mixing the selected powder into the grease. The following recipes were examined:

- Braycote 601
- Braycote-12 wt % Z-cote
- Braycote-20 wt % Z-cote
- DC-7
- DC-7-16.4 wt % Z-cote
- DC-7-17.6 wt % BN

The mix of Braycote-20%Z-cote was used on small-scale seals and buttons, the Braycote-12%Z-cote was used on Mss. Sunscreen coating rates of 0, 0.5, 0.9, 1.5, and 2 mg/cm^2 were examined. The rate of coverage was measured by weighing the specimen before and after coating application and the amount of coverage adjusted by hand until the desired coverage was achieved. To put these application rates into perspective it may help the reader to know that the sun protection factor (SPF) application rate standard for sunscreen on a person's skin is 2 mg/cm^2 .

Atomic Oxygen Exposures

The facilities used to expose specimens to AO are discussed in Ref. 10 and located at the NASA Glenn Research Center (GRC). AO exposures were done using an isotropic air-based plasma created in SPI Plasma Prep II etchers; AO fluence was measured using Kapton H witness coupons prior to or during exposures.

When considering AO in low-Earth-orbit (LEO) the "facing forward" (also known as ram, or windward) flight orientation receives AO at a rate of $\sim 4.4 \times 10^{19}$ atoms/ cm^2 -day. The other flight modes of zenith, nadir, starboard and port are expected to receive about 1.76×10^{18} atom/ cm^2 -day. Groups of coated and uncoated small-scale seals were exposed to 2.2×10^{20} atoms/ cm^2 AO prior to near-UV (NUV) exposure. An exposure of 2.2×10^{20} atoms/ cm^2 is a level of AO expected during a period of about 5 days in LEO in the windward flight orientation, or about 125 days in the zenith, nadir, starboard, or port flight orientation. Small-scale seals exposed to UV-C received AO exposures up to 2.2×10^{21} atoms/ cm^2 .

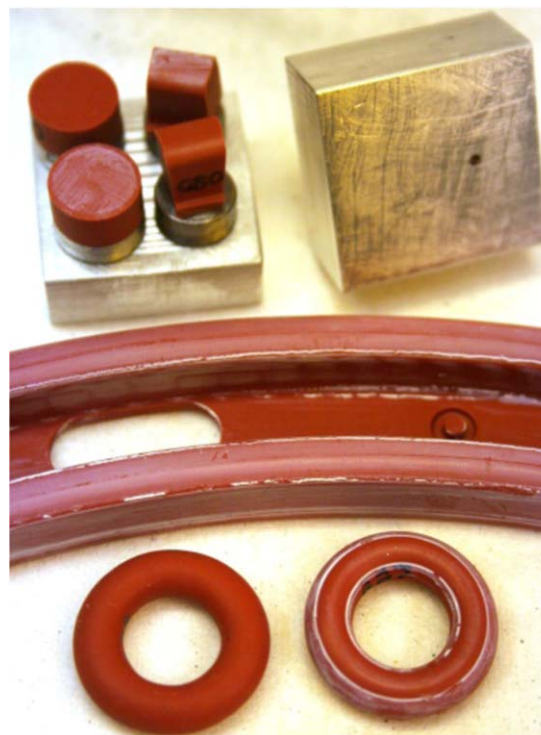


Figure 2. Adhesion specimens and 1 inch square vented block used with #2-309 O-rings. Top left: buttons and cut sections of Medium-scale seals (Mss) after adhesion testing. Center: Medium-scale seal after coating and adhesion testing. Bottom: #2-309 O-rings.

UV Exposures

The Sun delivers to spacecraft in LEO a UV irradiance (wavelengths <400 nm) of $\sim 110.9 \text{ W/m}^2$.⁶ A portion of this radiation will shine on a particular area of a spacecraft depending on the radiation view factor between the Sun and the surface being considered, as well as other factors such as the Sun being blocked by the Earth, or other parts of the spacecraft. The view factor of 0.26 used in this study is that expected for the forward facing windward, or “ram” flight mode. The UV dose for 1 day of windward flight is then calculated as follows:

$$\text{Sun's irradiance} \times 1 \text{ day} \times \text{radiation view factor} = 110.9 \text{ W/m}^2 \times 1 \text{ day} \times 0.26 = 2,490,000 \text{ J/m}^2.$$

This value can then be used to determine a UV dose characteristic of a number of days of LEO flight in the windward orientation where 1 day would be a dose of 2.49 MJ/m^2 , and 10 days would be 24.9 MJ/m^2 , and so on.

The following three different facilities were used to expose specimens to UV radiation:

- UV-C exposures at 254 nm were done at the NASA Glenn Research Center (GRC) in air at room temperature using an R-52G Mineralight UV lamp with the filter removed [manufactured by Ultra-Violet Products (UVP)]. This lamp has an intensity peak at a wavelength of 254 nm. At the 4.5 cm exposure distance used, the intensity of the lamp was $\sim 10 \text{ mW/cm}^2$. The intensity of the lamp was measured using a calibrated BLAK-RAY J-225 shortwave UV meter (manufactured by UVP). Coated and uncoated specimens were exposed to a variety of UV-C doses up to 147 MJ/m^2 . This maximum exposure is similar to the UV energy expected after about 60 days of windward LEO flight. However, these UV-C exposures delivered the energy in a narrow wavelength band (near 254 nm), where the Sun would be delivering the radiation over a much wider wavelength range with the longer (300–400 nm) UV-A wavelengths having the greatest intensity. While these UV-C exposures do not mimic on-orbit UV exposure accurately, they do demonstrate the ability of the coatings and compound additives to prevent damage due to radiation in the UV-C wavelength range.
- Near-UV exposures were done at GRC in a hard vacuum ($\sim 2 \times 10^{-4} \text{ Pa}$) using a 500 W mercury (xenon) arc source (Oriental Model 66141) with intensity peaks in the 300–360 nm range.¹⁰ The lamp's intensity (9 mW/cm^2) inside the facility was measured *in situ* using a calibrated BLAK-RAY J-221 long-wave UV meter. Coated and uncoated specimens were exposed to NUV doses up to 245 MJ/m^2 . This maximum exposure is similar to the UV energy expected after about 100 days of windward LEO flight. These NUV exposures mimic the Sun's radiation better (compared to UV-C) since NUV wavelengths are where the Sun's UV radiation is most intense. A vacuum was imposed during these GRC NUV exposures, thereby better simulating LEO and any combined effects of UV and vacuum.
- Near-UV exposures were also done at an outside vendor (Q-Lab Weathering Research Service). These Q-Lab NUV exposures were done near room temperature in air (not in a vacuum) using their Q-Sun Xenon test chamber, which delivered an irradiance of $\sim 87 \text{ W/m}^2$ (8.7 mW/cm^2) and a peak

intensity near 340 nm. Coated and uncoated specimens were exposed to NUV doses up to 245 MJ/m^2 . This group of exposures was done primarily to explore the effects of air and the absence of vacuum during UV exposure.

Adhesion Testing

Methods used to test adhesion have been presented in Refs. 10,15,19,30,31.

All button style and #2-309 O-ring specimens were: mated against a clean, anodized 6061 T651 aluminum alloy plate of average roughness of $13 \mu\text{m}$ ($0.33 \mu\text{m}$); brought together at a rate of 0.01 in/s (0.254 mm/s), compressed 25%, and held for 20 h at room temperature before unloading at a rate of 0.01 in/s . During unloading adhesion was measured using a calibrated force transducer.

Adhesion test conditions and results for the Medium-scale tests are provided in Table I. Mss were adhesion tested between two plates using an Instron material test system, Model 5584, encased in a thermal environmental chamber. Tests at a variety of constant temperatures between 65 and -50°C were accomplished. A laser extensometer was used to measure the displacement between plates. The dwell time for Medium-scale adhesion tests was 8 h except for the case of cyclic tests, which used a 1 min dwell time. Two Mss (-0282 and -0283) were subjected to cyclic adhesion tests which consisted of 25 room temperature 1 min dwell time adhesion tests.

The rate of sunscreen coverage is listed in Table I, however, frequently the same seal was adhesion tested more than once. In this way repeated docking/undocking cycles could be examined. When this occurred the coating was usually not replenished (and remeasured) between tests. The metal mating surface was cleaned after each test which used an 8 h dwell time. For example, seal -0306 was first tested uncoated at room temperature (RT), then coated with Braycote and tested again at RT; then -0306 was tested at -8°C without replenishing the coating. At this point, prior to this -8°C test, the rate of Braycote coverage is unknown because some of the coating was likely left behind on the metal mating surface after the previous test. This has been conveyed in Table I by listing the rate of coverage for the -8°C test of -0306 as “2nd cycle on 2.03”, with 2.03 mg/cm^2 being the original, 1st cycle, rate of coverage. If the coating was replenished between tests, the new rate of coverage appears in Table I. The coating was not doctored in any way during short cyclic tests on seals -0282 and -0283 , nor was the metal mating surface cleaned between cycles. The specific coating rate of coverage during these cyclic tests was not monitored; Table I lists the rate of coverage measured prior to the first cycle. The metal mating surface was thoroughly cleaned between all other (8 h dwell time) tests.

Leakage Testing

Leak rate test methods for small-scale^{9,18} and Medium-scale^{32,33} seals have been presented previously; however, a brief summary is provided here.

Small-scale leakage tests were done between flat anodized aluminum alloy plates, at room temperature, with 15% compression

Table I. Test Conditions and Adhesion of 12 inch Diameter Medium-Scale Seals; 1 lbf = 4.448 N

ID	Compound	Rate of coating coverage (mg/cm ²)	Adhesion test temp (°C)	Adhesion (lbf)
-0286	Uncoated	0	RT	60.6
-0287	Uncoated	0	RT	58.4
-0306	Uncoated	0	RT	50.3
-0306	Braycote only	1st cycle on 2.03	RT	1.1
-0286	Braycote only	1st cycle on 1.94	RT	1
-0306	Braycote only	2nd cycle on 2.03	-8.0	6.7
-0286	Braycote only	2nd cycle on 1.94	-8.0	4.5
-0306	Braycote only	3rd cycle on 2.03	65.0	0.8
-0286	Braycote only	3rd cycle on 1.94	65.0	0.7
-0287	Braycote-12%Z-cote	1st cycle on 1.69	RT	1.1
-0305	Braycote-12%Z-cote	1st cycle on 2.00	RT	1
-0305	Braycote-12%Z-cote	2nd cycle on 2.00	-8.0	4.2
-0287	Braycote-12%Z-cote	2nd cycle on 1.69	-8.0	5.3
-0305	Braycote-12%Z-cote	3rd cycle on 2.00	65.0	0.9
-0287	Braycote-12%Z-cote	3rd cycle on 1.69	65.0	0.9
-282	DC-7-16.4%Z-cote	2-26 cycles on 2.29	RT	1.9-12.8 increasing with each cycle
-0283	DC-7-16.4%Z-cote	2-26 cycles on 1.89	RT	0.7-7.6 increasing with each cycle
-0282	DC-7-16.4%Z-cote	1st cycle on 2.29	-8.0	6.5
-0283	DC-7-16.4%Z-cote	1st cycle on 1.89	-8.0	13.5
-0282	DC-7-16.4%Z-cote	1st cycle on 1.82	-50.0	322.3
-0902	6% TiO ₂ uncoated	0	61.0	72.6
-0903	6% TiO ₂ uncoated	0	-50.0	180.6

of the seals' height, and the volume inboard of the seal at a relative positive air pressure of 14.7 psig (0.1 MPa). The volume of the system was measured prior to testing. The pressure was measured using two pressure transducers, which were averaged and system temperature was measured using resistance temperature devices (RTD's).

The apparatus used to leak test the Mss consisted of two stainless steel plates of 16 μ in (0.41 μ m) or better surface finish, hermetic plumbing, and pressure and temperature sensors. The volume of the system was measured before testing. The top plate had a vent port located between the inner and outer bulbs which enabled isolation and testing of the inner seal. Dry air was supplied to the inner volume and a relative pressure of 14.7 psig (0.1 MPa) established and monitored by two pressure transducers. The apparatus was enclosed in a Tenney Benchmaster-BTRC environmental chamber with all leak tests done at 56°C. The leakage for small and Mss was determined using a pressure decay method²⁹ wherein the ideal gas law and definition of specific volume were used to determine air mass as a function of pressure, volume, and temperature. The mass of air in the system was thus measured over time; the slope of the mass versus time curve yielded leakage.

RESULTS

Adhesion

The average adhesion of the flat button, #2-309 O-ring, and cut Mss specimens are shown in Figure 3; the as-received condition is uncoated. The coating applied is noted with the specimen

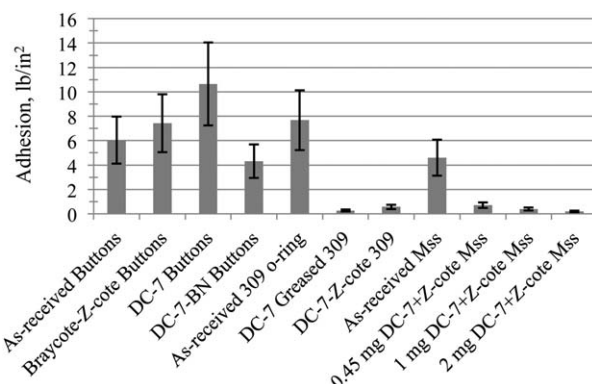


Figure 3. Average adhesion of buttons, 2-309 O-rings coated with 0 or 2 mg/cm², and cut sections from Medium-scale seals (Mss) coated with 0, 0.45, 1, and 2 mg/cm²; 1 lb/inch² = 6.9 kPa.

type. Each bar in Figure 3 is an average of two or more tests; the error bar is the average percent standard deviation of all tests. Figure 3 shows that the as-received adhesion of button, #2-309 O-ring, and specimens cut from a Mss is about the same as coated buttons; with the highest adhesion coming from the DC-7 coated buttons. Adhesion is due to molecular bonding between the surfaces.³⁴ Silicone elastomers are inorganic-organic polysiloxanes, with an inorganic Si—O—Si—O backbone and organic side groups attached, called radicals. The Si—O—Si bond angle and length are long compared to polymers with C—C backbones. These large bond lengths and angles allow for greater molecular movement and contribute to the flexibility and natural stickiness of silicone elastomers. Relatively mobile radicals at the surface are also available to contribute to adhesion.³⁵ Treatments that result in removal of radicals and/or limit backbone movement, can be expected to decrease adhesion.³⁶ Surface radicals can be removed by UV exposure in the presence of a vacuum; backbone movement can be limited by further crosslinking which can also be induced by UV exposure.¹⁸

The high adhesion of coated buttons is caused by a vacuum effect created between the flat greased button and the flat metal mating surface; when greased, the button acts like a suction cup. When the test article is crowned, or rounded, the edges release first thereby avoiding this vacuum effect. Figure 3 shows that the adhesion of nonflat test articles is lowered when coated. A crowned top prevents adhesion caused by vacuum when grease-based coatings are used. Figure 3 shows that the adhesion was lowered with the addition of boron nitride (BN); and that very small amounts (0.45 mg/cm²) of the coating are sufficient to lower adhesion. The physical presence of the coatings prevent the molecular bonding between the surfaces that result in adhesion.^{34,35}

Adhesion results for tests using full, uncut Mss seals are shown in Table I. The average 1st cycle room temperature adhesion of Mss was: 56.4 lb_f (251 N) for uncoated seals and 1.05 lb_f (4.76 N) for seals coated with either Braycote or the Braycote-12%Z-cote. The average 2nd cycle room temperature adhesion for Mss coated with DC-7-16%Z-cote was 1.3 lb_f (5.78 N). These data show that the coatings lower room temperature adhesion by a factor of about 50. For this elastomer compound prior work has shown that adhesion tends to increase at lower temperatures, with adhesion for an uncoated Mss of this type expected to be of the order of 300 lb_f (1.33 kN) at -50°C .³⁰ The adhesion of the DC-7 based sunscreen at -50°C (Table I) was 322 lb_f (1.43 kN), which indicates the coating to be dysfunctional at this temperature; however, -50°C is much lower than the current required use temperature specification for the docking seal. The minimum temperature specification for docking seal functionality is currently 4°C (40°F). There are future mission scenarios that are expected to require functionality at lower temperatures, thus improving the low temperature functionality of the sunscreens is an important facet of future work. Others have attributed the increase in adhesion at lower temperatures to increasing interfacial surface energy.^{30,31} Another cause is believed to be the hardening of the DC-7 at -50°C such that it acts more like a glue rather than a slippery lubricant. The service temperature range for DC-7 is -40 to 204°C .²³ The average

adhesion of Mss coated with sunscreen (Braycote12%Z-cote or DC-7-Z-cote) at -8°C was 7.4 lb_f (33 N). This adhesion value can be scaled-up to the full-scale docking seal by multiplying by 5: 7.4 lb_f \times 5 = 37 lb_f (164 N) (this approximate scale-up factor is the ratio of the full-scale seal diameter 50 inch to the Medium-scale's diameter 10.85 inch). The maximum full-scale adhesion allowed is expected to be 200 lb_f (890 N). The coatings thus appear to sufficiently lower adhesion at currently specified use temperatures.

Leakage

Coated and uncoated specimens were exposed to UV-C, and NUV radiations, and to AO in the form of an air plasma rich in AO. The effectiveness of the coating's ability to prevent damage to the seal was quantified by measuring the seal's leak rate.

Response to AO. A small group of small-scale seals were coated with 2 mg/cm² of either Braycote-20%Z-cote or DC-7-16.4%Z-cote, exposed to AO only, and leak tested. The results of these tests are presented in Figure 4. Leakage increased by a factor of about 40 for the Braycote-Z-cote coated seals after small doses of AO as compared to unexposed seals and to uncoated seals exposed to similar AO levels. It is clear this leakage increase is due to degradation of the coating and not the underlining elastomer because leakage for uncoated seals was unaffected by these low AO doses, as can be seen in Figure 4. The specimen coated with DC-7-Z-cote was unaffected by the AO. Dow Corning 7 is a silicone-based lubricant; as such, Si in the grease is expected to react with AO to form SiO_x-rich compounds at the surface, as has been found for the silicone-based elastomer itself.¹⁵ This prevents mass loss and may contribute to the durability of the DC-7-based sunscreens. Figure 5 shows images of three small-scale seals: one coated with Braycote-20%Z-cote and exposed to 1×10^{20} atoms/cm² AO; another coated with DC-7-16.4%Z-cote prior to AO exposure; and one coated with DC-7-16.4%Z-cote and exposed to 4.6×10^{20} atoms/cm² AO. All were coated at a rate near 2 mg/cm². The Braycote coated sample appears whiter because Braycote is thicker (more viscous) and less transparent than DC-7, and this particular recipe contained more Z-cote than the DC-7 coating. The whiter appearance of the Braycote coated sample might also be due to reaction with the AO. The important observation here is that after AO exposure the Braycote-20%Z-cote coating has cracked extensively in a mud-tile pattern; no such cracking was seen for the DC-7 sunscreen after AO exposure.

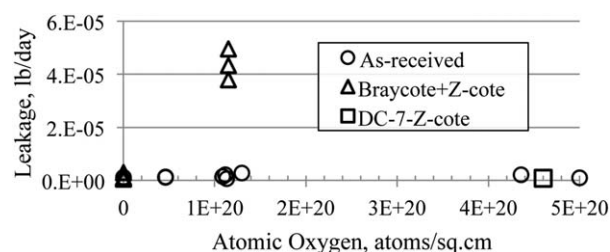


Figure 4. Leakage after AO exposure of small-scale seals as-received, coated with Braycote-20%Z-cote, or DC-7-16.4%Z-cote sunscreen; 1 lb/day = 0.454 kg/day.

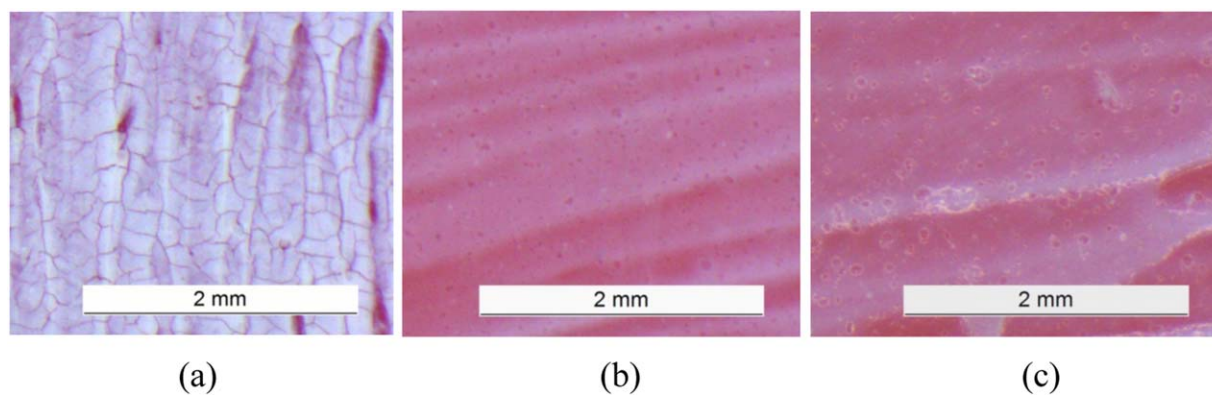


Figure 5. Optical images of small-scale seals: (a) coated with Braycote-20%Z-cote and exposed to 1×10^{20} atoms/cm² AO; (b) coated with DC-7-16.4%Z-cote prior to AO exposure; (c) coated with DC-7-16.4%Z-cote and exposed to 4.6×10^{20} atoms/cm² AO.

At first glance this susceptibility of Braycote to degrade when exposed to AO appears to be inconsistent with prior work presented by Christensen *et al.*⁸ who did not report a significant leakage increase for Braycote coated S0383 silicone seals exposed to AO. A small relative increase in leakage due to the presence of Braycote after AO exposure is however shown in their Figure 5 plot of leak rate versus equivalent hours of Sun exposure by comparing 11–12% compression nonlubricated and lubricated leakage results after AO exposure. We believe the reasons Christensen *et al.* did not see a large leakage increase for Braycote coated seals after AO exposure are because:

- Most of the Braycote was removed prior to AO exposure. In Ref. 8, seals were coated, then put through a set of 6 compression cycles with the grease being wiped off the mating surface after each cycle- thereby lowering the coverage on the seal with each cycle. Only after this set of compression and release cycles were the seals exposed to AO. The amount of grease actually on the seal was not measured; and
- AO exposure levels were low in Ref. 8, about 8×10^{19} atoms/cm² for the coated seals.

Response to UV-C and AO Exposure. UV-C. Figure 6 shows the response of small-scale seals made of the compound S0383-70 to exposure to UV-C radiation. For the as-received, unprotected seals, significant damage becomes apparent at UV-C exposure levels of 0.4 MJ/m^2 . With the Sun expected to deliver a dose of about 2.5 MJ/m^2 UV per day to a surface oriented in the windward flight mode, damage to the seal could be expected after less than 1 day in orbit. Addition of 6% TiO₂ to the compound appears to provide a moderate level of protection from UV-C at doses less than 100 MJ/m^2 but addition of TiO₂ appears to be ineffective at doses greater than 100 MJ/m^2 . Leakage for the seals coated with DC-7-16.4%Z-cote did not change as the level of UV-C exposure increased-indicating the seals were completely shielded from UV. The maximum exposure presented in Figure 6, 147 MJ/m^2 , is the approximate dose expected in space after 60 days of windward (ram) oriented LEO flight. The effectiveness of the coating to protect from UV is not unexpected since the transmittance of radiation through ZnO drops to near zero at wavelengths less than 380 nm ^{37,38}

and ZnO is a well-known additive to commercial UV protective sunscreens and thermal control paints.^{12–14,39}

UV attenuation. Rutile and wurtzite are the most common and stable forms of TiO₂ and ZnO, respectively. The attenuation of UV by these compounds is due to reflection, scattering, and absorption. The refractive index and reflectance of TiO₂ and ZnO are high, similar to those for diamond.⁴⁰ The very high refractive index for TiO₂ is partially responsible for its white appearance. Both compounds are semiconducting with electronic structures consisting of bands of orbitals separated by an energy band gap. Incoming UV radiation with energy equal to or greater than this energy band gap can excite an electron and move it from the valence band to the conduction band thereby absorbing the incoming radiation.⁴¹ The UV absorbance of TiO₂ and ZnO drop off at wavelengths greater than 380 nm because wavelengths greater than this are not energetic enough to overcome their band gap energies. Even though TiO₂ and ZnO have similar band gap energies (ca. 3.1 eV) TiO₂ absorbs more UV-B radiation ($200\text{--}300 \text{ nm}$) because it has more densely packed electron states that allow more absorption possibilities compared to ZnO¹³; ZnO however absorbs more UV-A. Thus a broader spectrum of UV protection should be able to be achieved through the inclusion of both TiO₂ and ZnO particles. The inability of the addition of 6% TiO₂ to the S0383-70

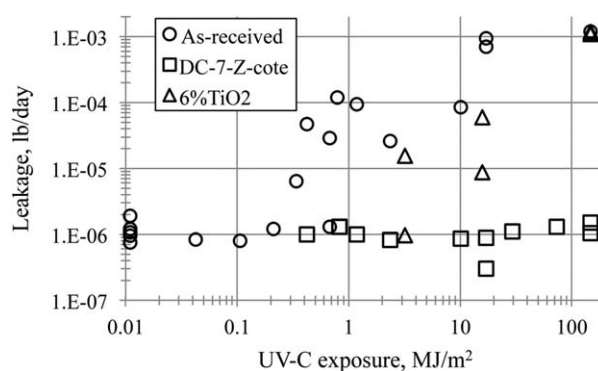


Figure 6. Small-scale seal leakage after UV-C exposure, as-received, coated with 2 mg/cm^2 DC-7-16.4%Z-cote sunscreen, and with 6% TiO₂ added to the compound; $1 \text{ lb/day} = 0.454 \text{ kg/day}$.

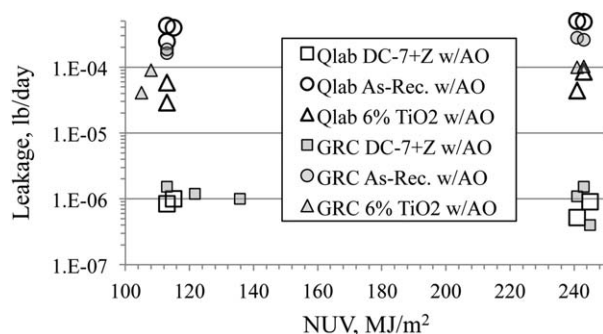
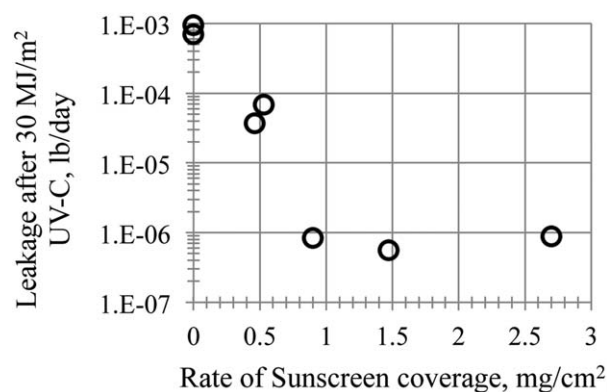
Table II. Leakage for Small-Scale Seals Coated with DC-7-Z-cote and Exposed to UV-C and AO; 1 lb/day = 0.454 kg/day

AO, atoms (cm ²)	UV-C (MJ/m ²)	Leakage (lb/day)
3.0E + 20	10.14	1.01E-06
4.6E + 20	15.29	9.81E-07
4.6E + 20	15.29	6.04E-07
4.6E + 20	15.29	4.20E-07
8.8E + 20	29.41	1.00E-06
2.2E + 21	73.53	4.00E-06

compound to protect it from UV is likely due to the low occurrence of TiO₂ particles at the surface. Because of the proprietary nature of the S0383-70 compound the TiO₂ particle size used is unknown; however, since absorbance is influenced by particle size it is believed improvements to the 6% TiO₂ compound are possible through the use of TiO₂ particles with diameters near 60–100 nm.¹⁴

Atomic oxygen. Table II presents leakage results for small-scale seals coated with 2 mg/cm² DC-7–16.4%Z-cote and exposed to UV-C and AO. Assuming an AO exposure of 4.4×10^{19} atoms/cm²-day, Table II AO exposures range from about 7–50 days of windward LEO exposure. Assuming 2.5 MJ/m²-day UV exposure, the UV-C exposures varied in energy from 4 to 30 days of windward UV exposure. Table II data indicates the DC-7-Z-cote sunscreen effectively protects the seals from UV and AO at these exposures, since all leakage rates after exposure were very close to as-received, unexposed levels (Figure 6, $\sim 1 \times 10^{-6}$ lb/day). The leakage of an uncoated seal at these exposure levels is of the order of 1×10^{-3} lb/day (Figure 6).

Response to NUV and AO. Figure 7 shows the leakage results for small-scale seals exposed to both NUV and AO combined. AO exposures of 2.2×10^{20} atoms/cm² were done first. Near-UV exposures were then done at Q-Lab Weathering Research Service (Qlab) in air; or at the NASA Glenn Research Center (GRC) in vacuum. Leakage differences between like exposures at Qlab and GRC were insignificant; thus the presence or absence of air had no noticeable effect on seal damage for these cases. The 113 and 240 MJ/m² NUV exposures caused leakage to increase markedly for the 6%TiO₂ compound, rising two orders

**Figure 7.** Leakage, after NUV + AO exposure, of small-scale seals as-received, coated with 2 mg/cm² DC-7–16.4%Z-cote sunscreen, and with 6% TiO₂ added to the compound prior to curing; 1 lb/day = 0.454 kg/day.**Figure 8.** Leakage, after 30 MJ/m² UV-C exposure, of small-scale seals coated with various amounts of DC-7–16.4%Z-cote sunscreen; 1 lb/day = 0.454 kg/day.

of magnitude above as-received leakage levels, indicating only moderate protection from UV because of the addition of TiO₂ to the compound. Leakage levels were unchanged compared to as-received leakage ($\sim 1 \times 10^{-6}$ lb/day, see Figure 6) for all seals coated with DC-7–16.4%Z-cote (DC-7+Z in the legend of Figure 7) indicating complete protection from NUV and no ill effects from AO at the exposure levels tested.

Leakage Response to Rate of Coverage. Figure 8 shows the leak rate of small-scale seals after being coated with various levels of DC-7–16.4%Z-cote and exposed to 30 MJ/m² UV-C. The characteristic of the lower coating levels (0.46 and 0.53 mg/cm²) is that “all” of the coating is wiped off. The coating level could not be lowered further by wiping it off with clean nitrile gloves. Figure 8 shows that some protection from UV-C is present at very low coverage rates, and that the seal appears to be fully protected at coverage rates greater than 0.9 mg/cm².

Coating Durability

Figure 2 shows various seals and test specimens after being adhesion tested. In Figure 2 the Mss, 1 button, 1 cut Medium-scale specimen, and 1 of the O-rings were coated prior to adhesion testing; the other specimens shown were not coated. The condition of the coating appears intact on the button, cut Medium-scale specimen, and Mss; however, the coating appears to have moved off the crown of the O-ring. Such behavior is important since we would like to know if the seal is still protected after undocking, and if significant levels of the coating can be expected to be left behind on the mating surface. Studies of aspects affecting coating durability are planned and include time between application and use; and prior AO and UV exposure. Our observations to date are included here.

The rate of coating coverage was measured before and after adhesion and leakage testing for a set of 4 buttons and 4 small-scale seals coated with Braycote-20%Z-cote; the coverage decreased 10% for the buttons, and 27% for the small-scale seals. Coverage decreased an average of 4.5% for 4 small-scale seals coated with DC-7–16.4% Z-cote. The optical micrographs of the DC-7–16.4%Z-cote coated small-scale seals before and after leak testing shown in Figure 9 indicate the coating to be

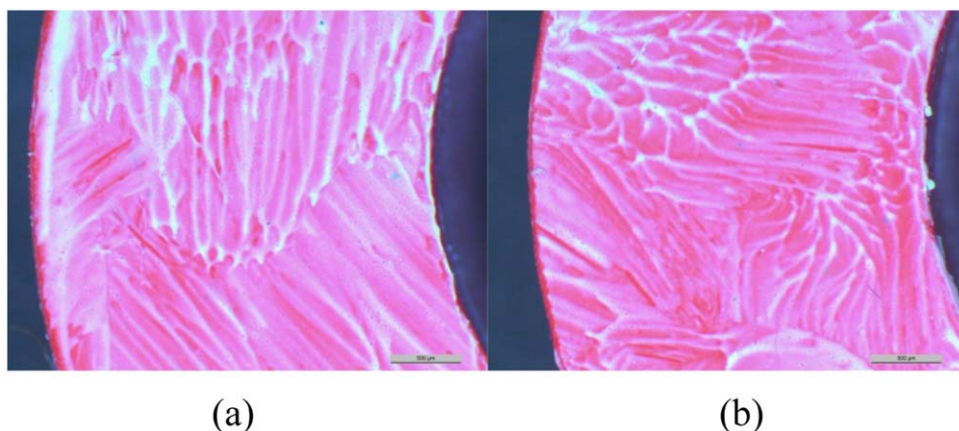


Figure 9. (a) Coated 5.4 mm wide small-scale seal after UV exposure; (b) similar seal after UV exposure and leak testing.

durable to a leak test, which is similar to a docking/undocking operation.

Figure 10(a) shows Mss -0282 after coating with 1.82 mg/cm^2 DC-7-16.4%Z-cote but before any testing; Figure 10(b) shows -0282 after adhesion testing at -50°C . Adhesion was very high for this test (Table I) and Figure 10(b) shows that much of the coating was removed during the adhesion test. However, the coating remained intact after adhesion tests at room temperature, -8°C , and 65°C , as shown in Figure 10(c) and after multiple tests at these temperatures. Figure 11 shows the Mss -0283 after being coated with 1.89 mg/cm^2 DC-7-16.4%Z-cote, adhesion tested at -8°C with an 8 h dwell time, and then subjected to 25 room temperature adhesion tests using 1 min dwell times. Figure 11 shows that after 26 adhesion tests some of the sunscreen coating has been worn off. Future work includes UV and AO exposures after adhesion and/or leak testing so the level of sunscreen protection after a simulated docking operation can be determined. One related test has been accomplished to date: in this series of tests a small scale seal was coated with 2.04 mg/cm^2 DC-7-Z-cote and exposed to 4.6×10^{20} atoms/ cm^2 AO, then tested with leakage found to be 8×10^{-7} lb/day; the seal was then exposed to 15.3 MJ/m^2 UV-C and leak tested again (resulting in 9.8×10^{-7} lb/day), which shows that the first leak test did not significantly damage the sunscreen coating. Since the coating appears to be intact after a

small number of compression cycles (1–3 adhesion and/or leak tests) and full protection is achieved at low mg/cm^2 levels of coverage, we currently believe the docking seal will remain protected from UV and AO after undocking in space.

There is a use limitation in the DC-7 product literature against using DC-7 on silicone rubber O-rings due to possible degradation of the rubber; and Parker S0383-70 product literature recommends against using this compound with silicone oil.⁴² Reactions between fluids and elastomer compounds are generally tested at elevated temperatures (302°F) with complete immersion. Reactions between silicone elastomers and Dow Corning 4 Compound (a grease similar to DC-7) at room temperature are reported to be moderate with a +5% volume change and -5 hardness point change after 1 day immersion.⁴³ We have been using sunscreens containing DC-7 on silicone O-rings for 2 years and have found no degradation. This absence of degradation is probably because our application does not require immersion, and our test temperatures are relatively low. A slight reaction with the silicone fluid may actually be beneficial in our applications wherein characteristic shrinking and hardening of S0383-70 caused by the vacuum and UV of LEO are mitigated by DC-7. If for whatever reasons this combination (S0383-70 and DC-7) is found to be undesirable, a fluorosilicone elastomer compound could be used instead of S0383-70

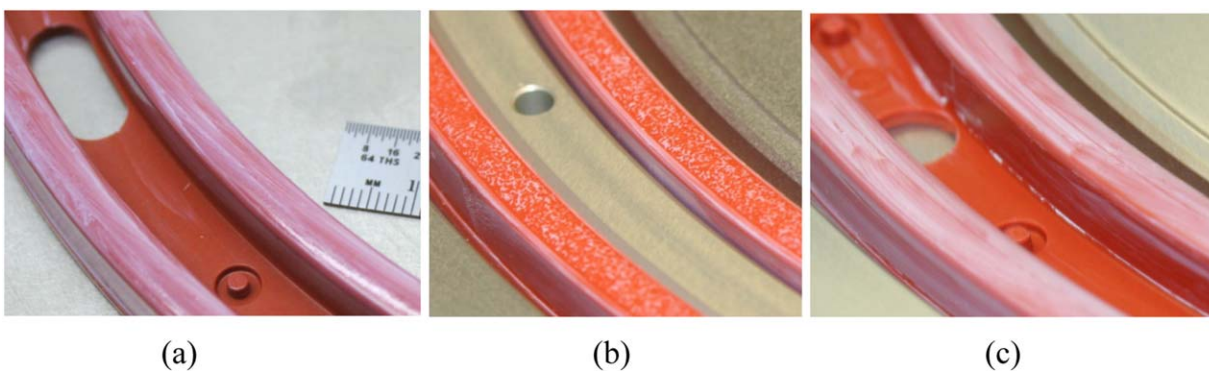


Figure 10. (a) Medium-scale seal -0282 after coating; (b) Coated -0282 after adhesion testing at -50°C ; (c) Coated Medium-scale seal -0283 after adhesion testing at -8°C .

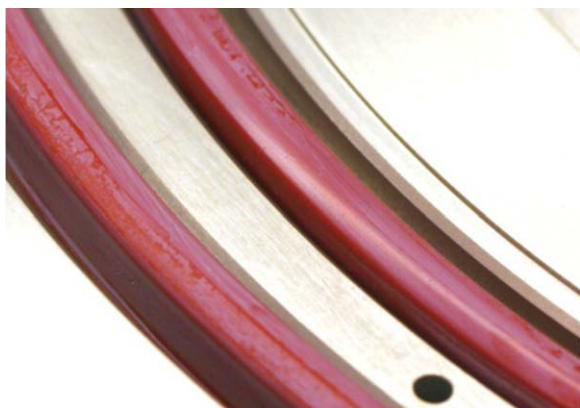


Figure 11. Coated medium-scale seal -0283 after adhesion testing at -8°C (8 h, dwell) and then 25 more times at room temperature (1 min dwell on each cycle).

since fluorosilicones are known to be more compatible with silicone fluids^{43,44} and also have a very low use temperature (-73°C). For reasons of AO durability, we believe it is important for both the elastomer compound and the sunscreen to be silicone-based. The presence of Si is a key to establishing a stable sunscreen coating and avoiding AO erosion through the creation of durable SiO_x -rich reaction products.

CONCLUSIONS

A class of grease-based coatings has been conceived and tested for use on elastomer docking seals used on spacecraft in LEO. The coatings use UV blocking additives to protect the coated article from UV radiation. The DC-7-16.4%Z-cote coating is silicone-based which may prevent its erosion by AO through the formation of nonvolatile SiO_x -rich compounds at the surface. Tests showed that the coatings successfully mitigate adhesion at temperatures greater than or equal to -8°C . To realize low adhesion with a grease-based sunscreen the seal must be constructed using a curved crown to prevent suction-cup like adhesion caused by the vacuum formed between greased flat surfaces. Adhesion was very high for the DC-7-16.4%Z-cote coated seal tested at -50°C indicating the coating's inability to prevent high adhesion at this low temperature. The sunscreen made with Braycote preformed badly when exposed to AO with leakage increasing by a factor of about 40 over base-line values. If further testing confirms Braycote's intolerance to AO, use of Braycote on docking seals exposed to AO will not be recommended. Seals coated with DC-7-16.4%Z-cote were not significantly damaged after being exposed to a variety of AO and UV levels up to 2.2×10^{21} atoms/cm² and 245 MJ/m². These exposure levels are characteristic to what might be expected after 50–100 days in LEO. UV radiation exposures included UV-C and NUV wavelengths. The DC-7-16.4%Z-cote coating proved difficult to wipe completely off, with ~ 0.4 mg/cm² remaining after repeated attempts to wipe it off with clean nitrile gloves. Seals coated with DC-7-16.4%Z-cote appeared protected from UV at coverage rates as low as 0.9 mg/cm². The DC-7-16.4%Z-cote coating proved to be durable to small numbers (1–3) of compression cycles, and to provide UV protection after a com-

pression cycle. The data presented indicate DC-7-16.4%Z-cote will protect docking seals from UV and AO and mitigate unwanted adhesion as long as use temperature is greater than or equal to -8°C and the seal's active surface is not flat.

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