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# Use of VUV Radiation to Control Elastomer Seal Adhesion

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This report is a formal draft or working paper, intended to solicit comments and ideas from a technical peer group.

This report contains preliminary findings, subject to revision as analysis proceeds.

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# **Use of VUV Radiation to Control Elastomer Seal Adhesion**

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#### Abstract

Due to their wide operating temperatures and low leakage rates, silicone elastomers are the only class of flight qualified elastomer materials that currently meet NASA's needs for various seal applications, which include docking and hatch seals for future space exploration vehicles. However, silicone elastomers are naturally sticky and exhibit sizeable adhesion when mated against metals and other silicone surfaces. This undesirable adhesion can make undocking spacecraft or opening a hatch problematic. Two approaches that can be used to reduce seal adhesion include use of grease or, application of low doses of atomic oxygen (AO). This paper investigates a third approach: the application of light doses of vacuum ultraviolet (VUV) radiation. Presented are the adhesion and leakage characteristics of S0383-70 silicone elastomer exposed to various VUV doses in the 115 to 200 nm wavelength range. The data indicate that adhesion is expected to be less than the target threshold maximum of 2 lb/in.<sup>2</sup> after about 1 J/cm<sup>2</sup> of VUV exposure for seal-to-metal configurations and after 2 J/cm<sup>2</sup> for seal-to-seal configurations with no significant damage, or increase in seal leakage. This paper shows that VUV, without AO or grease, can be an effective means to reduce adhesion to the desired levels necessary for space seals with minimal change in seal leak rates.

### I. Introduction

NASA has been developing a docking system called the international Low Impact Docking System (iLIDS).<sup>1,2</sup> This docking system and others being considered use a silicone elastomer seal to seal in cabin air while two spacecraft are docked and hatches between them are open. Silicone elastomers are the only known class of flight qualified elastomers that function properly over the temperature range expected in low-Earth-orbit (LEO).<sup>3</sup> Silicone elastomers are naturally sticky.<sup>4,5</sup> These adhesive properties help with sealing, however can also make separation or undocking problematic. In an effort to achieve low push-off forces during undocking, low adhesion between seal mating surfaces is desired. A method to decrease the adhesion of cured silicone elastomers which uses ultraviolet radiation in the vacuum (200 to 115 nm) wavelength range (VUV) has been developed. Vacuum UV attenuates rapidly,<sup>6</sup> thus does not penetrate the elastomer deeply. The goals of this work were to determine if seal adhesion could be sufficiently mitigated with VUV and to quantify the level of damage caused by such VUV treatments. Damage was quantified by measuring the leak rate of the seal.

Two other methods to lower the adhesion of silicone elastomers are currently being examined by NASA: atomic oxygen treatments,<sup>4,7,8</sup> and application of various greases.<sup>9,10</sup> Atomic oxygen treatments employ air based plasmas which produce atomic oxygen and VUV radiation. Atomic oxygen treatments are relatively expensive because they are done in a partial vacuum using specialized equipment to generate the plasma. Atomic oxygen treatments can also damage the silicone depending on the compound and the exposure being used.<sup>11</sup> Grease applications have certain merits, however use of grease may not be desirable due to outgassing, possible reactions with the LEO, or when the crown of the seal is flat due to a vacuum effect between greased, flat mating surfaces. Christensen and Underwood examined the effects of AO and plasma generated VUV (130 nm) on the leakage of both Braycote 601 greased and un-greased o-rings made of silicone (S383) and fluorocarbon (Viton®) V835.<sup>12</sup> These studies found AO increased leakage, in some cases considerably. Vacuum UV treatments are presented as an option for control of silicone elastomer adhesion.

## **II. Experimental Procedure**

#### A. Specimens

All tests were done using specimens cut from 0.21 in. (5.3 mm) thick sheets of the elastomer compound S0383-70 supplied by Parker Hannifin Corporation. Figure 1 shows examples of the seals used for leakage tests, and the buttons used for adhesion tests. Seals had an inner diameter of 0.41 in. (10.4 mm) and an outer diameter of 0.83 in. (21.1 mm). Button diameter was 0.365 in. (9.3 mm). The width and height of the specimens tested are similar to what is expected to be used for docking seals on NASA's future crew module.

#### **B.** Button Adhesion and Seal Leakage

Seal leakage and button adhesion test procedures have been presented previously.<sup>4,11,13</sup> Leakage tests were done between flat anodized aluminum alloy plates at room temperature with the inner space of the seal at a relative positive air pressure of 14.7 lb/in.<sup>2</sup> (0.1 MPa). The pressure was measured using two pressure transducers, which were averaged and the leakage determined using a pressure decay method.<sup>13</sup> Adhesion buttons were tested at room temperature against an anodized aluminum 6061 T651 plate which was flat with a surface roughness Ra =  $12 + 2 \mu in (0.3 \mu m + -0.05 \mu m);$ these tests are referred to as Seal-on-Plate, or SoP. Buttons were also mated against other like buttons in a Seal-on-Seal (SoS) arrangement. The SoP method simulates the seal mating with a metal flange such as what might be expected when docking to the International Space Station (ISS). Seal-on-Seal simulates the mating of two spacecraft, each equipped with an elastomer docking seal. Adhesion specimens were brought together, compressed 25 percent of their height (or thickness), and held for 20 hours before being pulled apart at a rate of 0.01 in./s (0.254 mm/s).



Figure 1.—Seals and buttons made from S0383-70 elastomer.

#### C. UV Exposures

Two methods were used to expose specimens to VUV: a 30-W deuterium lamp facility; and an AO facility which produces VUV radiation (with a peak intensity at 130 nm) with a MgF<sub>2</sub> lens to block AO. Vacuum UV doses in the lamp facility ranged between 0 and 2.11 J/cm<sup>2</sup> (54 equivalent Sun hours (ESH)). The ratio of UV lamp intensity compared to the Sun's zero air mass intensity in the same wavelength range is referred to as "equivalent Suns". The Sun's intensity at wavelengths less than 200 nm is approximately  $1.1 \times 10^{-5}$  W/cm<sup>2.14</sup> Thus the Sun delivers about 0.039 J/cm<sup>2</sup>-hr in the VUV range. Equivalent space exposure, referred to as "equivalent Sun hours," is obtained by multiplying the number of test hours by "equivalent Suns". The average intensity of the 30-W deuterium lamp was approximately 2.2 equivalent Suns (2.4×10<sup>-5</sup> W/cm<sup>2</sup>); the intensity of the lamp naturally drops off at 200 nm; and exposures were done at a vacuum pressure  $<10^{-5}$  torr, with 54 cm between the lamp and the center of the sample plate which was perpendicular to the beam. The VUV lamp radiation passed through a magnesium fluoride end-window which provided a lower cut-off wavelength of 115 nm. A cesium iodide (CsI) phototube was used to measure the lamp's intensity.<sup>11</sup> The dose of VUV radiation in the AO facility was qualitative, with more time in the facility defined as "more" radiation. The AO facility (referred to as Tank 9 and discussed in Ref. 11) uses a radio frequency power supply to create an oscillating electrical potential between two plates in the presence of a partial pressure of air, thereby generating oxygen-rich plasma. Molecules of O2 are broken to produce atomic oxygen, and the movement of electrons between energy levels in excited oxygen atoms produces VUV radiation. Specimens to receive only VUV in the AO facility were placed in a protective fixture with a MgF<sub>2</sub> lens covering the top mating surface of the specimen. These  $MgF_2$  lenses are transparent to VUV. By covering specimens with  $MgF_2$  and protecting them from AO, we determined if the VUV present affects the adhesive properties of the compound.

For comparison, some specimens were also exposed in a vacuum to near-UV (NUV, wavelengths between 200 and 400 nm) using a 500-W mercury (xenon) arc source.<sup>11</sup> The irradiance of this NUV source was measured after the exposure using a calibrated BLAK-RAY J-221 longwave UV meter; the sensor of the meter was place inside the exposure chamber at the same location the specimens were placed during the exposure. The average NUV irradiance was 6.55 mW/cm<sup>2</sup>. The Sun's NUV intensity in LEO<sup>15</sup> is 11.1 mW/cm<sup>2</sup> thus the irradiance ratio of our NUV lamp compared to the Sun (equivalent Suns) was approximately 0.6. NUV has the ability to affect bulk properties of materials beyond surface changes.

Radiation in the UV-C range (~254 nm) was examined since: the relatively short wavelength does not penetrate deeply thus may yield a low level of damage; lamps are commercially available; and exposures can be done in air. We used an R-52G Mineralight UV lamp from UVP LLC to produce radiation with peak intensity at 254 nm, at a distance that produced approximately 12 mW/cm<sup>2</sup>. Intensity was measured using a calibrated BLAK-RAY J-225 shortwave UV meter. UV-C exposures were done at room temperature in air.

## **III. Results and Discussion**

#### **D.** Adhesion

Figure 2 presents the average adhesion results of specimens exposed to VUV radiation in the AO exposure facility; each data point was the average of between 2 and 4 unique specimen tests. Specimens were protected from the AO in the plasma by a MgF<sub>2</sub> lens. Prior work has shown that the AO present in the plasma reacts with the silicone of the elastomer thereby enriching the surface with oxygen, creating a glassy SiO<sub>x</sub> rich layer at the surface which decreases adhesion.<sup>4</sup> It was thought this oxygen enrichment of the surface was responsible for the decrease in adhesion;<sup>4,7,11</sup> however, it is apparent that the material is also reacting to the radiation present. Exposures of 8 hours in the AO facility provided enough VUV radiation to sufficiently lower the unwanted adhesion for both SoP and SoS, decreasing average adhesion to 0.18 lb/in.<sup>2</sup> and 1.4 lb/in.<sup>2</sup>, respectively. The intensity of the VUV radiation in the AO facility is expected to be of the order of 100 to 200 times the Sun's in the neighborhood of 130 nm;<sup>11,12</sup> the portion of the Sun's solar constant (1366.1 W/m<sup>2</sup>) at wavelengths less than 170 nm is approximately 0.014 W/m<sup>2.15</sup> Thus the 8 hours in the AO facility is expected to impose a dose of the order of 6  $J/cm^2$ . The elimination of adhesion at this dose agrees with results achieved in the 30-W lamp facility shown in Figure 3. It is believed that the radiation breaks molecular bonds near the surface and that these broken bonds provide opportunities for further cross linking and hardening of the surface.<sup>16</sup>

Figure 3 shows the average adhesion of the S0383-70 elastomer buttons after various exposures to VUV radiation in the 30-W deuterium lamp facility; exposure to VUV decreased adhesion. The trends in the data indicate that adhesion can be expected to be less than 2 lb/in.<sup>2</sup> after about 1 J/cm<sup>2</sup> for SoP, and about 2 J/cm<sup>2</sup> for SoS. Seal adhesion of 2 lb/in.<sup>2</sup> is NASA's current threshold maximum for docking seals. The percent standard deviation among like tests at each exposure was determined, and then averaged with other exposures to yield an average percent standard deviation. The error bars shown in Figure 3 were set equal to +/- one average percent standard deviation.

Figure 4 shows the adhesion of S0383-70 after various exposures to UV-C radiation. The data indicate sufficient adhesion reduction is likely achieved after approximately 60 J/cm<sup>2</sup> exposure to UV-C. Note each data point in Figure 4 corresponds to a single test.

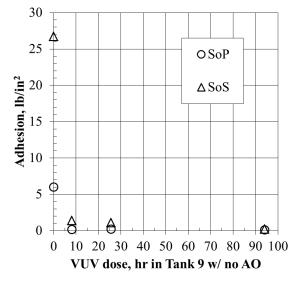


Figure 2.—Seal-on-Plate and Seal-on-Seal average adhesion of VUV exposed buttons, zero AO.

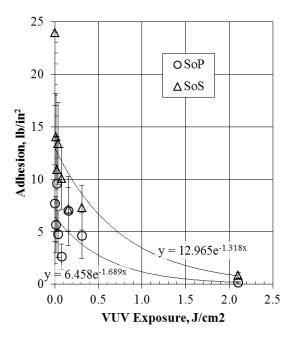


Figure 3.—Seal-on-Plate and Seal-on-Seal average adhesion of S0383-70 after VUV exposure in the 30-W lamp facility.

## E. Seal Leakage

The damage to the elastomer was quantified through air leak tests of small scale seals similar to those shown in Figure 1. Figure 5 shows the leakage results for seals exposed to VUV and NUV radiations where each data point corresponds to an individual test. There was no measureable damage or increase in Seal-on-Plate leakage in any of the VUV exposed specimens, with leakage not varying significantly from the as-received base-line of  $1 \times 10^{-6}$  lb/day. A slight, though measureable, Seal-on-Seal leakage increase (to  $1 \times 10^{-5}$  lb/day) was found after  $2 \text{ J/cm}^2$  of VUV. It is believed that, after optimization of VUV dose and wavelength, adhesion can be eliminated without measurable damage to the compound yielding an adhesion reduction treatment that is fast, inexpensive, and amenable to outside service suppliers when compared to AO treatments. An NUV dose of 230 J/cm<sup>2</sup> caused adhesion to drop to 1 lb/in.<sup>2</sup> and air leakage to increase to approximately  $5 \times 10^{-5}$  lb/day. Tests performed herein and by others in the authors' lab show exposing seals to combinations of AO and UV not only increase leakage but cause variability in leakage rates that sometimes can be significant. For instance, leakage at NUV 230 J/cm<sup>2</sup> (Fig. 5) varies by approximately an order of magnitude. It is noteworthy to mention that a NUV dose of 230 J/cm<sup>2</sup> is equivalent to about 5.5 ESH in the NUV wavelength range, and a VUV dose of 2 J/cm<sup>2</sup> is equivalent to about 52 ESH in the VUV range. This is because the intensity of the Sun is much greater in the NUV wavelengths.

Figure 4 shows that UV-C treatments decreased adhesion to below 2 lb/in.<sup>2</sup> at an exposure of about 60 J/cm<sup>2</sup>, however, as shown in Figure 6, leakage increased at this UV-C exposure to about  $2 \times 10^{-5}$  lb/day, with reliability losses beginning at about 40 J/cm<sup>2</sup>. Since it was clear NUV and UV-C damaged the seals, further testing was abandoned and these wavelengths judged not useful for adhesion reduction of docking seals. It may be possible to prevent elastomer damage during UV-C light sources typically have intensity peaks near 254 nm; filtering out wavelengths longer than this might sufficiently protect the elastomer from these more damaging wavelengths and enable UV-C's use for adhesion reduction.

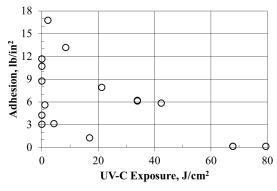


Figure 4.—Seal-on-Plate adhesion of buttons exposed to UV-C.

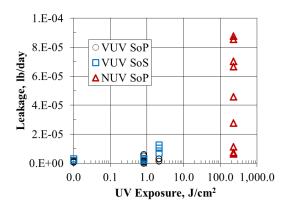


Figure 5.—Leakage of S0383-70 after exposure to VUV and NUV radiation.

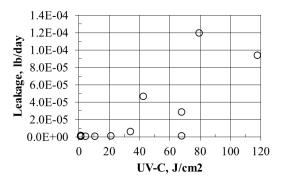


Figure 6.—Leakage of S0383-70 after exposure to UV-C radiation.

# **IV.** Conclusions

Vacuum UV radiation decreased the adhesion of the silicone elastomer S0383-70 and at the same time maintained low leakage levels. It was found that doses of NUV or UV-C radiation also decreased adhesion but also significantly damaged the elastomer. It was shown that VUV treatments can be used to lower the adhesion of silicone seals to levels below the general requirement of less than 2 lb/in.<sup>2</sup>; thus VUV offers another anti-adhesion treatment for future docking and hatch seals for space exploration vehicles.

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