



Adhesion of Silicone Elastomer Seals for NASA's Crew Exploration Vehicle

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Prepared for the
44th Joint Propulsion Conference and Exhibit
cosponsored by the AIAA, ASME, SAE, and ASEE
Hartford, Connecticut, July 21–23, 2008

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Acknowledgments

The authors would like to acknowledge and praise the efforts of our colleagues who contributed to this work. Daniel A. Scheiman for IR spectroscopy work and analysis, Richard Tashjian for mechanical support, Josh Finkbeiner for early work on adhesion rigs, Shawn Taylor for his assistance with the heat shield seal, Bruce Banks for his help with Tank 9 exposures, and Pat Dunlap, Marta Bastrzyk and Emily Owens for their comments and help with the manuscript.

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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Abstract

Silicone rubber seals are being considered for a number of interfaces on NASA's Crew Exploration Vehicle (CEV). Some of these joints include the docking system, hatches, and heat shield-to-back shell interface. A large diameter molded silicone seal is being developed for the Low Impact Docking System (LIDS) that forms an effective seal between the CEV and International Space Station (ISS) and other future Constellation Program spacecraft. Seals between the heat shield and back shell prevent high temperature reentry gases from leaking into the interface. Silicone rubber seals being considered for these locations have inherent adhesive tendencies that would result in excessive forces required to separate the joints if left unchecked. This paper summarizes adhesion assessments for both as-received and adhesion-mitigated seals for the docking system and the heat shield interface location. Three silicone elastomers were examined: Parker Hannifin S0899-50 and S0383-70 compounds, and Esterline ELA-SA-401 compound. For the docking system application various levels of exposure to atomic oxygen (AO) were evaluated. Moderate AO treatments did not lower the adhesive properties of S0899-50 sufficiently. However, AO pretreatments of approximately 10^{20} atoms/cm² did lower the adhesion of S0383-70 and ELA-SA-401 to acceptable levels. For the heat shield-to-back shell interface application, a fabric covering was also considered. Molding Nomex fabric into the heat shield pressure seal appreciably reduced seal adhesion for the heat shield-to-back shell interface application.

Acronyms

<i>AO</i>	= Atomic oxygen
<i>ATR</i>	= Attenuated total reflectance
<i>CBM</i>	= Common Berthing Mechanism
<i>CEV</i>	= Crew Exploration Vehicle
<i>EDU</i>	= Engineering Development Unit
<i>ESH</i>	= Equivalent sun hours
<i>GRC</i>	= Glenn Research Center
<i>ID</i>	= Identification
<i>IR</i>	= Infrared
<i>ISS</i>	= International Space Station
<i>LEO</i>	= Low Earth orbit
<i>LIDS</i>	= Low Impact Docking System
<i>LVDT</i>	= Linear variable displacement transformer
<i>NASA</i>	= National Aeronautics and Space Administration
<i>RF</i>	= Radio frequency
<i>SS</i>	= Stainless Steel

I. Introduction

IN current designs, silicone elastomer seals are being used on NASA's Crew Exploration Vehicle (CEV) for the docking seal, hatch seals, and the heat shield-to-back shell interface seals, amongst other locations. The function of the docking seal is to confine air within the habitable environment, while allowing crew and supplies to pass between two joined space vehicles. The hatch seals function similarly by confining breathable air within the vehicle around a passageway. The seals between the heat shield and back shell prevent high temperature reentry gases from leaking into the interface. For each of these locations, silicones are strong candidates due to their ability to be molded into seals, their high and low operating temperature, and their extensive use in NASA's Apollo vehicles.¹

The LIDS² is being developed to permit the CEV to dock to the ISS (Figs. 1 and 2) and future Constellation Program vehicles. The current LIDS interface design employs two functionally different versions of LIDS. One of the two LIDS will be an active docking system, while the other remains passive. The active half of a LIDS-to-LIDS interface includes a main interface seal (Fig. 2); the passive half of the interface is a flat metal surface for long-term durability considerations.

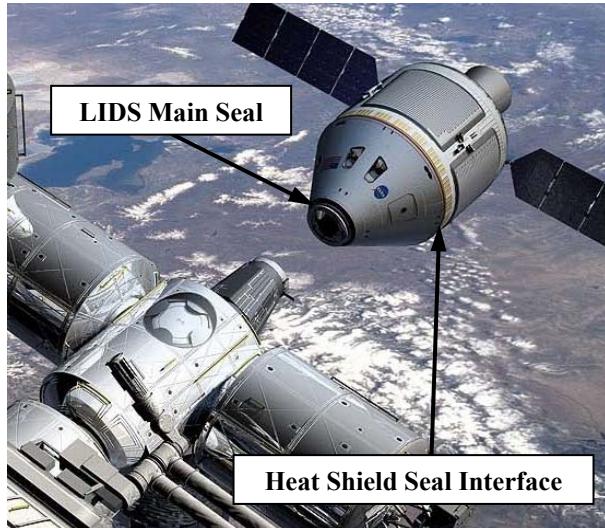


Figure 1. Illustration of the CEV docking with the ISS.

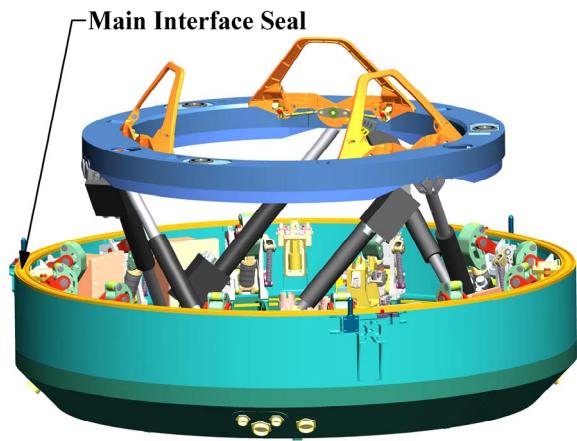


Figure 2. Schematic of an active LIDS and main interface seal.

The current design of the LIDS-to-LIDS interface is a departure from previous versions of LIDS in which both mating docking systems were identical and the main interface seal was to mate against a replicate seal. However, determining the feasibility of having a seal on both halves of a mating interface continues to be of interest. The capability of docking two identical systems would increase the subsystem redundancy and reduce risk for NASA Constellation missions.

The hatch seals are not included in this study. However, the design of the hatch seals have similar considerations, including leak rate through the seal interface, compression force required to seal the joint, and the force necessary to overcome the adhesion between the seal and its interfacing metal surface.

A combination of thermal and pressure seals is being developed to prevent high temperature reentry gases from leaking into the heat shield-to-back shell interface. The adhesion of this seal is important should the mission require the jettison of the heat shield prior to landing.

NASA's Glenn Research Center (GRC) is developing seals for both the LIDS docking interface and the heat shield-to-back shell interface and is assessing the durability of candidate seal materials for the space environment. Silicone rubber has an established history of success and flight qualification and is the primary material that can function over the expected operating temperature range. GRC is focusing on three silicone elastomers, two provided by Parker Hannifin (S0899-50 and S0383-70) and one from Esterline (ELA-SA-401), due to their suitable operating temperature capabilities and low outgassing characteristics.

In the as-received condition, these silicone elastomers have a considerable amount of adhesion. These adhesive forces, if left unchecked, could cause problems during undocking or separation of the heat shield from the vehicle. NASA's current goal for LIDS is to keep the release force during undocking to less than 300 lbf. To ensure that docking and release mechanisms and other LIDS subsystems are properly designed, the adhesion of candidate

rubbers must be characterized and, if possible, minimized. Daniels et al³ evaluated the compression set, adhesion, and leakage properties of small-scale coupons before and after exposure to simulated space environments. That study³ revealed both the high initial adhesion of the compounds in their as-received state and identified the potential benefits of AO exposure on adhesion reduction. A follow-up study by Daniels et al⁴ investigated the compression set, adhesion, and leakage of sub-scale docking seals (12 in. diameter). At the time of these investigations,^{3,4} the docking system was configured as an androgynous system, requiring that seal-on-seal performance be assessed. These studies evaluated the effects of hold time and material selection on adhesion. The adhesion of S0899-50 was found to be far greater than S0383-70 and longer hold times resulted in higher adhesion forces.

Banks et al^{5,6} showed that AO will oxidize the outer surface of silicone elastomers, causing a glassy layer to form. While AO is naturally occurring in low Earth orbit (LEO), it can be produced on Earth using oxygen ion beams or plasmas. Using this technique, a non-adherent glassy layer can be induced on specimens and used to reduce seal adhesion.

The current study builds on these previous works.³⁻⁶ The effects of seal and counter face materials on seal adhesion are assessed for both the docking system and the heat shield seal applications. Studies were performed to identify a minimum threshold of AO pretreatment needed to reduce adhesion to acceptable levels for the three candidate materials. Analytical tests were performed to assess surface changes due to AO exposure.

II. Experimental Procedures

Tests were conducted on three elastomers: two from Parker Hannifin with compound designations of S0383-70 and S0899-50, and one from Esterline made from XELA-SA-401 (now designated as ELA-SA-401). Specific dimensions, compound and processing details are proprietary; however, all are peroxide cured silicone-based elastomers.

A. Specimens

The forms of the different specimens and elastomers used in this study are summarized in Table 1.

1. "Button" Specimens

For both cost and efficiency considerations, many of the specimens used for these studies were made from sheet material of the corresponding silicone materials. Cylindrical "button" specimens, 0.91 cm (0.36 in.) in diameter, were cut from the 0.53 cm (0.21 in.) thick sheet material using a drill press and a custom-made core drill lubricated with soap. The specimens were thoroughly washed with water after fabrication. These specimens were used as surrogates for both docking seals (seal-on-seal and seal-on-plate configurations) and heat shield seals (seal-on-plate configuration) to evaluate adhesion characteristics.

2. Small-Scale Seal Specimens

For small-scale testing, materials were procured in the form of #2-309 size o-rings (inner diameter 1.05 cm (0.412 in.), cord diameter 0.53 cm (0.21 in.)). These o-rings were used to examine the release properties of the round-surfaced elastomers.

3. Medium-Scale Seal Specimens

Medium-scale adhesion tests were conducted on sub-scale candidate docking system seal designs. These included 31 cm (12 in.) outside diameter Gask-O-Seals™ by Parker, and a 31 cm (12 in.) outside diameter, "2-piece" molded seal made to GRC design specifications using the Esterline compound. The Gask-O-Seals consist of an aluminum ring with four seals molded into it: two elastomer seals on the top, or active side, and two elastomer seals on the bottom, which are permanently engaged when the ring is attached. The bulbs on the active side had major centerline diameters of 25 cm (10 in.) and 30 cm (12 in.). Additional details of the Gask-O-Seal and its testing are provided in a study of as-received material.⁴ The medium-scale NASA-Esterline 2-piece molded seal had two pads that were 0.91 cm (0.36 in.) wide by 0.84 cm (0.33 in.) tall, with the inner seal pad having a centerline major diameter of 26.0 cm (10.2 in.) and the outer pad with major diameter of 29.2 cm (11.5 in.). For reference purposes, the full-scale LIDS seal is expected to have an outer diameter of 58 in. (147.3 cm).

4. Heat Shield Seal Specimens

Two versions of this double bulb heat shield seal were tested. The first was an elastomer-only seal made of the ELA-SA-401 material. The second was made from the same material but with a heat resistant Nomex® fabric molded onto the surface. Cylindrical button specimens were taken from both the web section and the top of the seal bulb for adhesion tests.

5. Counter Face Materials

To simulate the metal docking seal interface, button, o-ring, and heat shield specimens were tested against counter face surfaces made of aluminum 6061-T651 in both the as-received (denoted Seal-on-Al) and anodized

(denoted Seal-on-Anodized) conditions. The anodized aluminum surface conformed to the MIL-A-8625 TYII specification. To simulate the heat shield interface where the seal would interact with the CEV internal flange material, specimens were tested against counter face surfaces made of titanium alloy Ti-6Al-4V and aluminum Al-6061-T651. Both counter face surfaces had mating surface areas of 6.45 cm^2 (1 in.²), which were machined to an average roughness, Ra, of $0.3 \mu\text{m} \pm 0.1 \mu\text{m}$ ($12 \mu\text{in.} \pm 4 \mu\text{in.}$).

The counter face material used to test medium-scale specimens was stainless steel machined to a roughness of $0.4 \mu\text{m}$ ($16 \mu\text{in.}$).

B. Atomic Oxygen Exposure of Button and Small-Scale Specimens

In an effort to reduce the adhesive tendency of the candidate elastomers, specimens were pre-treated with various levels of AO. Three facilities at GRC were used for exposing small-scale o-ring and button specimens to AO: (1) plasma ashers that use a radio frequency (RF) power supply in air to create a discharge between electrodes; (2) a directed beam facility that employs an Electron Cyclotron Resonance Plasma Source; and (3) the large scale facility known as Tank 9, which creates an RF generated plasma between two $1.5 \text{ m} \times 1.5 \text{ m}$ (5 ft x 5 ft) plates. A more detailed description of the AO exposure facilities can be found in Rutledge et al⁷ and Stidham et al.⁸

Elastomer buttons were exposed to various levels of AO, or AO fluence, ranging from $1 \times 10^{18} \text{ atoms/cm}^2$ to $1.5 \times 10^{22} \text{ atoms/cm}^2$. Project documents⁹ state that the expected AO fluence corresponding to one year of AO exposure in LEO is $5 \times 10^{21} \text{ atoms/cm}^2$. This conversion is influenced by the spacecraft's mission flight path and the current space environment.

At low temperatures, as shown by Daniels et al,³ the SiO_x -based layer that is created on the elastomers by the AO treatment does not significantly affect their ability to seal. Atomic oxygen exposure levels are provided in Tables 2, 3 and 4. Atomic oxygen fluence measurements were accurate within $\pm 10\%$.

C. Atomic Oxygen Exposure of Medium-Scale Seals

Medium-scale seals were exposed to AO in GRC's Tank 9 facility. Figure 3 shows a medium-scale and a precursor full-scale seal on the Tank 9 mounting plate. Kapton witness specimens 2.54 cm (1 in.) in diameter were positioned around the seals to measure Tank 9 AO fluence levels. Adhesion buttons were mounted next to the Kapton witnesses to get a direct correlation of adhesion to AO exposure level. This arrangement enabled the AO fluence levels to be determined at several locations around the test samples as measured by Kapton weight loss.

A calibration exposure in Tank 9 found that an uneven level of AO is distributed across the exposure plate during pretreatments. To even out the AO fluence levels, the specimen exposure plate was rotated 90° after approximately 10 of the 20 hours of exposure time to ensure an acceptable AO fluence on the specimens.

D. Surface Chemistry

To gain a better understanding of how AO exposures influence the adhesive properties of elastomers, surface chemistry changes resulting from AO exposures were measured using infrared (IR) spectroscopy. The surface



Figure 3. Full-scale seal on Tank 9 mounting plate. A full-scale and a medium-scale Gask-O-Seals are shown mounted to the exposure plate. Witness and button specimens are placed around the outer circumferences of the test specimens.

chemistry of the three candidate elastomers was analyzed before and after AO exposure. As-received sheet stock and #2-309 size o-rings were tested. O-rings that had been exposed to the following conditions were also tested: AO (AO fluence = 5.89×10^{21} atoms/cm²) and AO (5.6×10^{21} atoms/cm²). The exposed o-ring top surface and unexposed bottom and interior (examined by cutting out a small section of the o-ring) were assessed.

The samples were measured using a Nicolet 380 FTIR fitted with a SMART Omni sampler single pass ATR (Attenuated Total Reflectance) accessory using a Ge crystal. The technique measures the IR spectra of the surface of the samples to a depth of several microns (not the bulk properties). Comparing the spectra of as-received material to exposed material reveals chemical changes in the surface of the material. Knowledge of these chemical changes enable us to better explain adhesion changes in the elastomers.

E. Adhesion Testing of Small-Scale and Button Specimens

Adhesive forces for both the small-scale docking system seal specimens and the heat shield specimens were measured using the apparatus shown in Fig. 4. The force required to separate two specimens of similar elastomer compound was determined by compressing two specimens together by 25% of their combined height, holding for a period of time, and then separating them. For the majority of the tests, a dwell period of 24 hours was used. The elastomer specimens (buttons and o-rings) were attached to metallic holders using cyanoacrylate adhesive and allowed to cure for 24 hours before testing. The bottom surface of the specimens (buttons and o-rings) were roughened with sandpaper and cleaned before using LOCTITE® 4502 instant adhesive to bond them to the metal holder. For “seal-on-seal” tests, one test specimen and its holder were attached to a stationary load cell. The other specimen and holder were attached to a movable platform (see Fig. 4). A servomotor was used to (1) move the platform to compress the specimens, (2) hold during the dwell period, and (3) move the platform to decompress the specimens. A linear variable displacement transformer (LVDT) was used to measure relative positions of the two elastomers. For all adhesion tests, the rate at which the specimen pairs were compressed together followed a

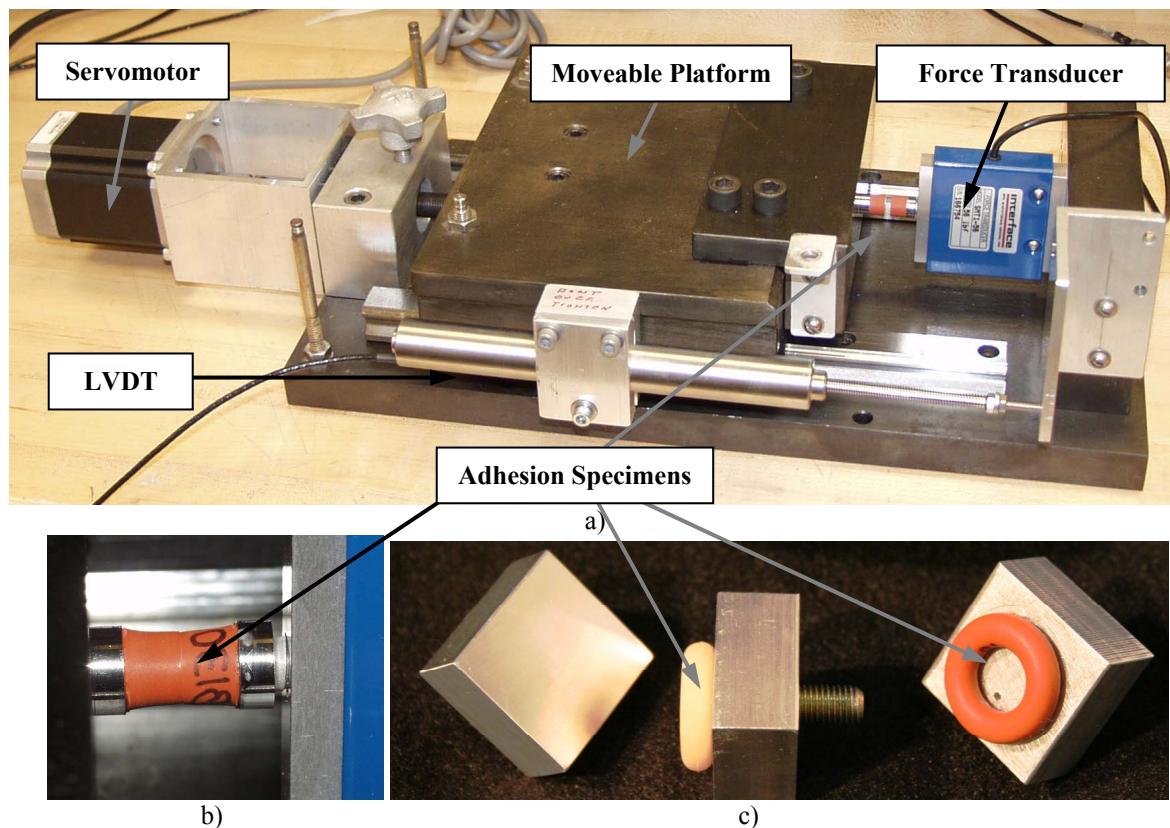


Figure 4. Photographs of the apparatus used to quantify adhesion between specimens. a) Overall Adhesion Test Apparatus: The stage on the left moves compressing the two elastomer buttons 25% while force is measured by the stationary load cell. b) Close-up photo of two elastomer buttons: 0.21 in. diameter test specimens are pulled apart and any tension is measured by the force transducer. c) 1 in. square Al holders, with and without o-rings mounted. A small hole for venting can be seen in the block on the right.

prescribed function of the distance between the two specimen surfaces (see Fig. 5). In the figure, the dwell period occurs when position equals zero and corresponds to 25% compression of combined specimen height. The force transducer has an accuracy of about ± 0.3 psi, thus force measurements between +0.3 and -0.3 psi are recorded as 0.3 psi tension for conservatism.

1. Docking System Seal Specimens

Tests were conducted in three configurations. To simulate a “seal-on-seal” docking configuration, tests with two elastomers were conducted. Tests were conducted compressing elastomer against a finely machined plate to simulate a “seal-on-plate” docking configuration. Lastly, select tests were performed using #2-309 o-rings pressed against the plate to assess the adhesion of seals with a “crown” rather than the flat top of the buttons. In tests using buttons or o-rings pressed against a plate (seal-on-plate) the elastomer was compressed 25%, as in the seal-on-seal tests. Figure 4 shows button and o-ring samples bonded to metal holders.

2. Heat Shield Seal Specimens

Figure 6 shows sections of two heat shield seals being considered. Button specimens were cut from the heat shield seals and adhesion tested in the same manner as the button docking seal specimens, as described above. For early exploration of the effects of Nomex in reducing seal adhesion prior to receiving the seals, tests were done using a loose sheet of Nomex cloth between an as-received ELA-SA-401 o-ring and the Al plate. In the tests with the Al plate, the Nomex cloth was held in place by compression, so the cloth was not bonded to the o-ring or the Al plate.

F. Adhesion Testing of Medium-Scale Seals

An Instron material test system, Model 5584, was used to determine the adhesive force generated by the 12 in. diameter medium-scale seals during separation. The test specimens were mounted to a stainless steel platen attached to the actuator rod via a threaded stud connection. An identical platen was attached to the opposing rod to provide a metal surface against which to seal. This elastomer seal-on-metal plate configuration can be seen in Fig. 7.

To better simulate the actual LIDS docking operation, the actuator was programmed to follow the motion of the LIDS latch mechanisms, rather than an arbitrary loading rate. The compression/decompression path followed was similar to that shown in Fig. 5.

Prior to compression, as-received seals were cleaned with isopropyl alcohol, while specimens exposed to AO were lightly dusted with compressed air only. The seals were compressed and held together for 70 hours before being pulled apart. During separation, the adhesion force was measured using an Instron 2525-171 150 kN (33,720 lb) load cell with an accuracy of $\pm 0.25\%$ of the reading.

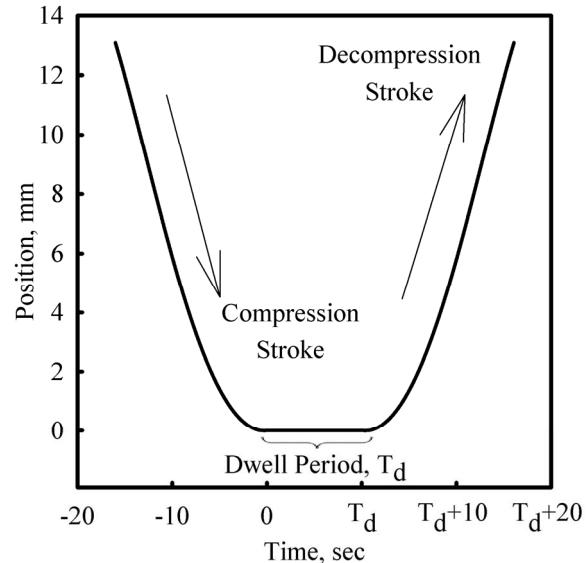


Figure 5. Graph showing the rate of compression and decompression during an adhesion test.



Figure 6. Image of hollow heat shield seals being considered for CEV applications. The seal on the left was covered with a Nomex heat resistant cloth during manufacturing. Buttons cut from the seal were adhesion tested.

III. Results and Discussion

A. Adhesion

Docking Seals

1. As-Received and AO Pretreated Button and Small-Scale O-rings

Adhesion results for Parker S0383-70, S0899-50, and Esterline ELA-SA-401 in their as-received and AO pretreated states (various levels) for both buttons and small o-rings (where available) are provided in Tables 2, 3, and 4. The seal-on-seal adhesion of as-received material was highest in Parker's S0899-50 at approximately 82 lbf per square inch of seal contact area. The lowest level of as-received adhesion was achieved by ELA-SA-401 at approximately 22 psi. As-received seal-on-seal adhesion for S0383-70 was approximately 38 psi. These levels of adhesion are prohibitively high for applications such as the LIDS docking system. The seal-on-aluminum plate adhesion levels were much less than seal-on-seal adhesion. Adhesion for as-received material against the aluminum plate was only about 45%, 19% and 18% of seal-on-seal values for S0899-50, S0383-70 and ELS-SA-401, respectively. However, it should be noted that there was a very high level of scatter in the as-received S0383-70-on-Al plate adhesion, ranging from 4 to 22 psi.

Figure 8 shows how exposure to AO lowers the level of adhesion for both seal-on-seal and seal-on-plate configurations, and that an AO dose of $\sim 10^{19}$ atoms/cm² effectively decreases their adhesion. After AO treatments of $>10^{20}$ atoms/cm², the adhesion of Parker S0383-70 and Esterline ELA-SA-401 are nearly negligible at ~ 1 psi, while the adhesion of S0899-50 remains fairly high, ~ 10 psi. Figure 9 shows the as-received o-ring-Al plate adhesion was higher than button-Al plate adhesion. This is surprising since the round cross-section of the o-ring was expected to assist in releasing from the plate.

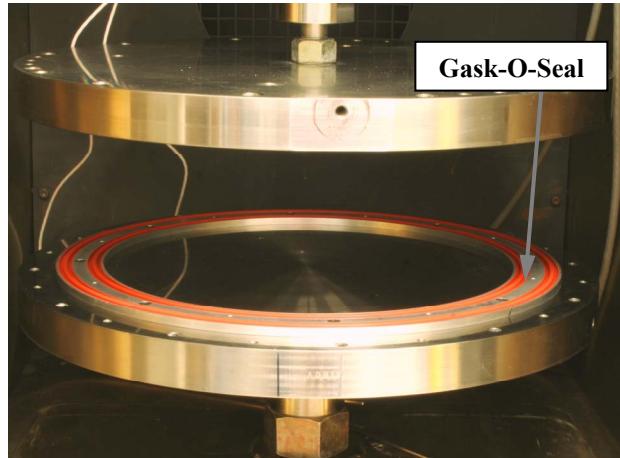


Figure 7. Medium-Scale load testing setup. The test specimen was mounted to the bottom fixture and then compressed by the top plate.

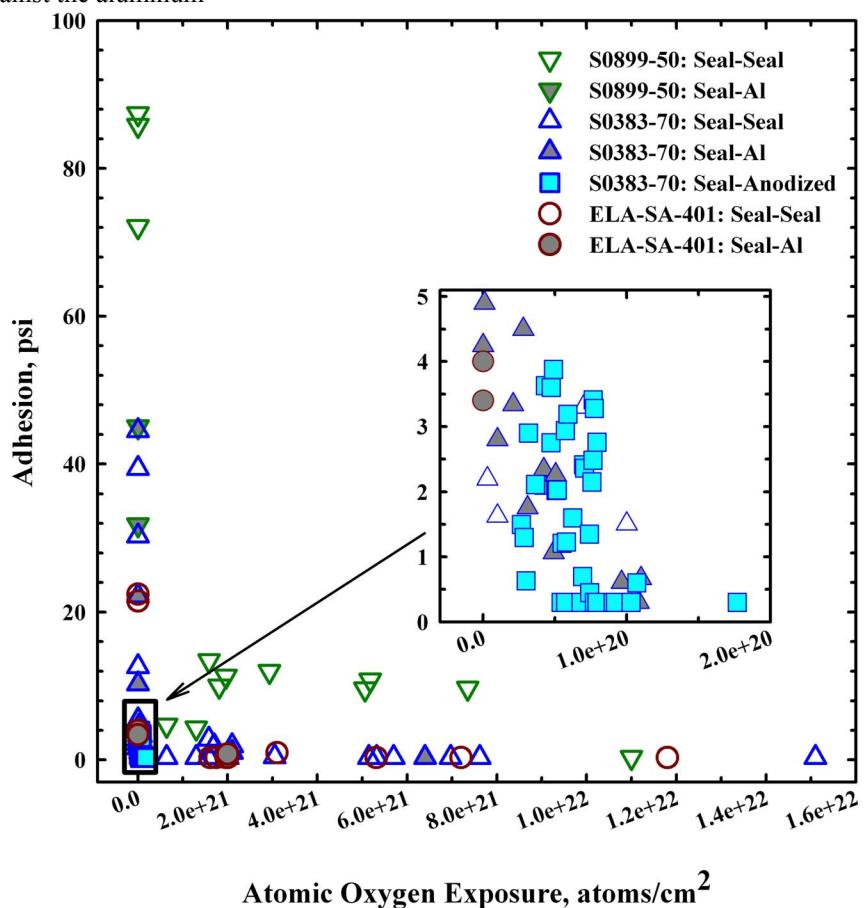


Figure 8. Adhesion of Seal Materials with Exposure to Atomic Oxygen. Adhesion and AO exposure data from Tables 2, 3, and 4 plotted with a broken abscissa, so as-received (zero exposure) adhesion can be shown.

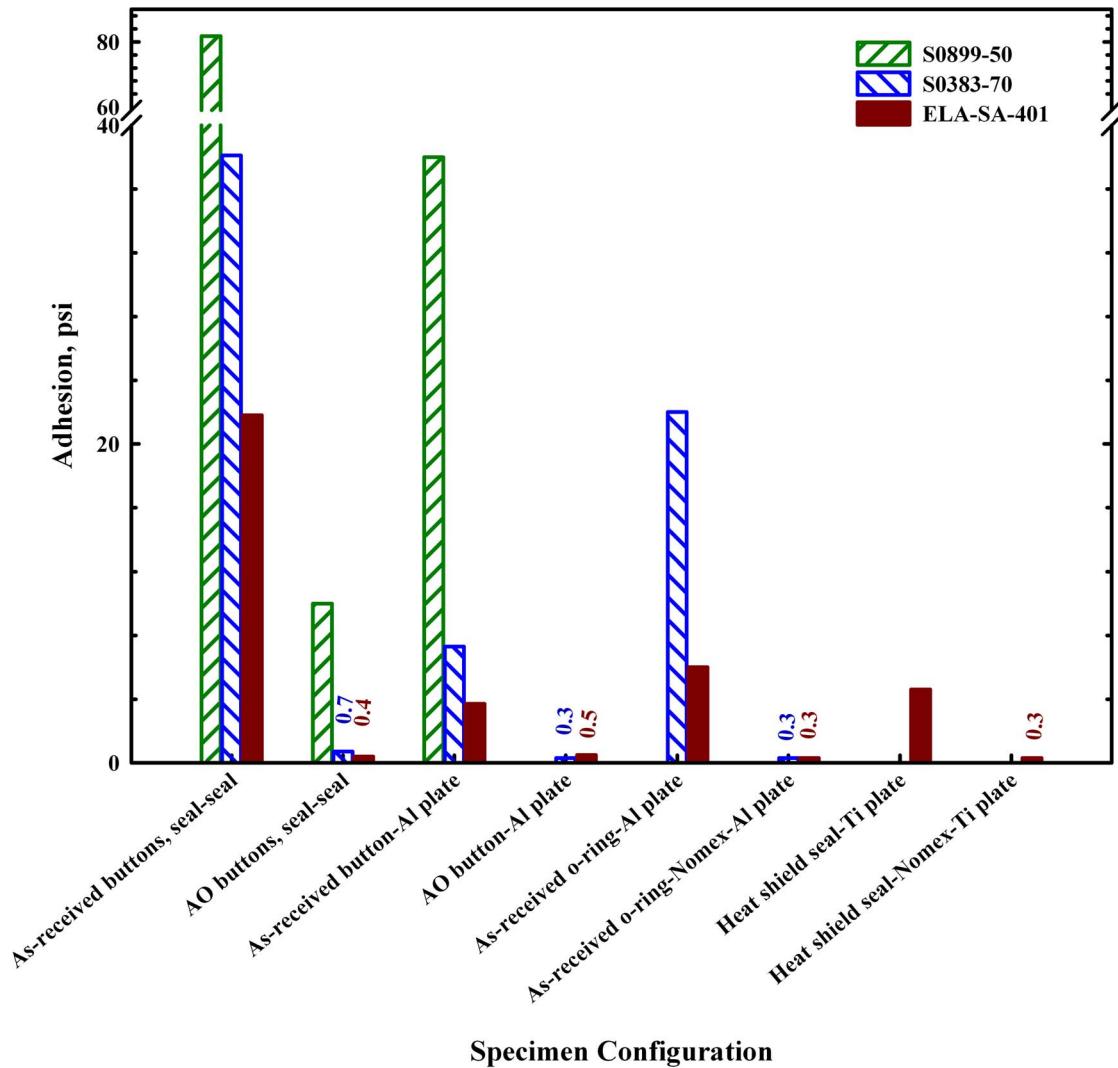


Figure 9. Summary of Button and #2-309 o-ring Adhesion Results. Bar chart shows the average adhesion between seal materials from Parker (S0899-50, S0383-70) and Esterline (ELA-SA-401) in the as-received condition, and after exposure to AO ($>6.3 \times 10^{20}$ atoms/cm² fluence). Test modes were a button seal pressed against another button seal, a button pressed against an aluminum plate, a #2-309 o-ring pressed against an aluminum plate, o-ring and Al plate with a layer of Nomex in between, and button specimens cut from the heat shield (Esterline compound) double bulb seal pressed against a Ti-6Al-4V plate. Data drawn from Tables 2, 3 and 4.

As can be seen from the data in Tables 2, 3, and 4, a significant level of variability exists in the adhesion results. Although instruments used to make the measurements have a relatively high level of accuracy, the stochastic nature of release dynamics of imperfect specimens and batch to batch material variability result in scattered data. Adhesion results for as-received S0899-50 and S0383-70 buttons had a standard deviation of about 7 psi. However, ELA-SA-401 adhesion tests were generally more consistent. The standard deviation of adhesion results for as-received ELA-SA-401 buttons was only about 0.4 psi. Standard deviation was based on the entire population, or “n” method.

2. As-Received and AO Pretreated Medium-Scale Seals

Figure 10 and Table 5 show results for the medium-scale 12 in. seal in both the as-received (S0383-70 and ELA-SA-401 compounds) and AO pretreated (S0383-70 compound only) conditions. These tests revealed the adhesion benefits of AO treating on medium-scale seals. For instance, on average, the as-received medium-scale S0383-70 seals had adhesion values of about 11 psi and after AO pretreatments adhesion values of about 2 psi. There was some variability in the results as the seal receiving the slightly higher AO fluence level (Table 5, specimen 206200-

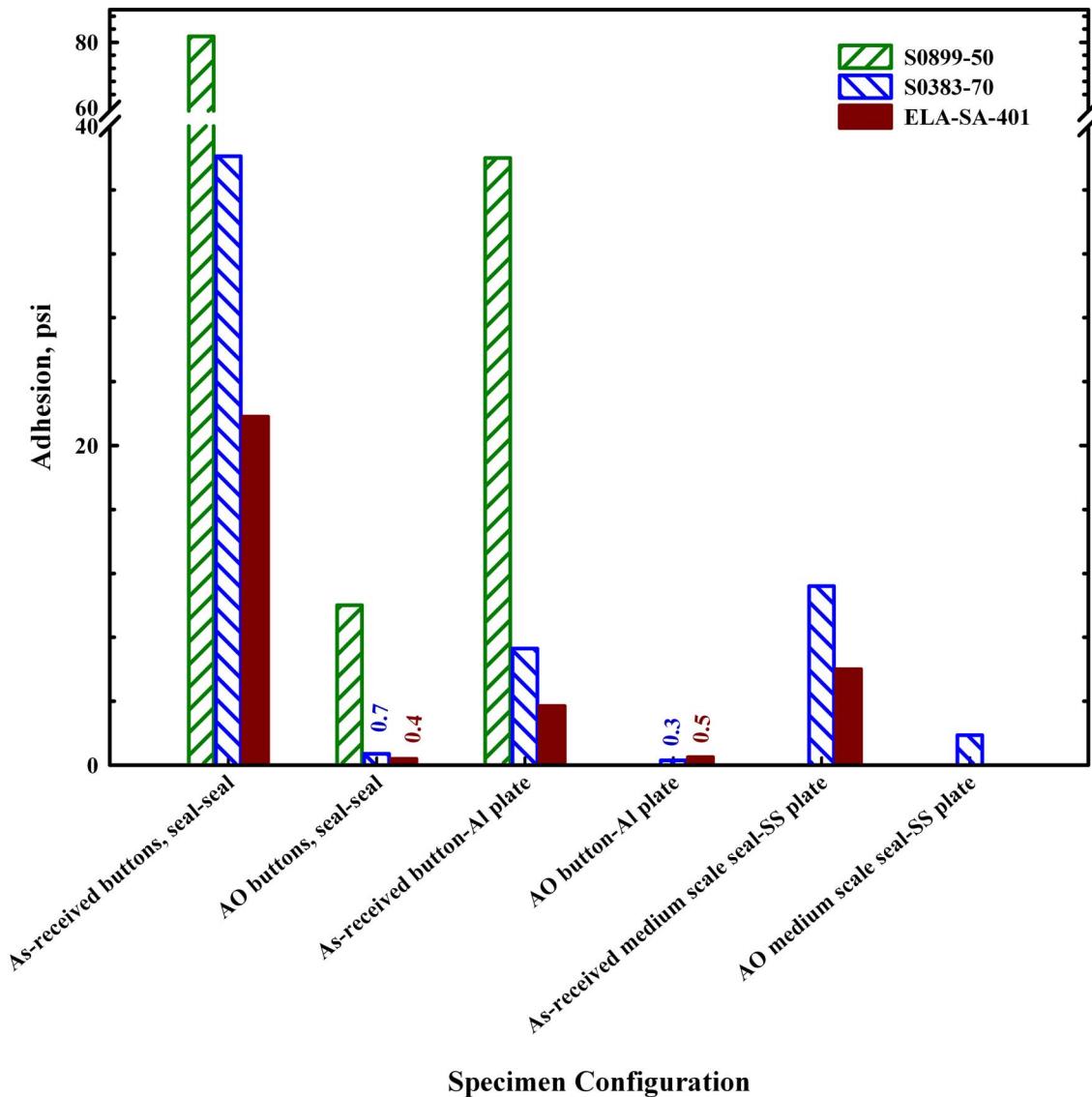


Figure 10. Summary of Button and 12 in. Medium-scale LIDS Seal Adhesion Results. Bar chart shows the average adhesion between seal materials from Parker (S0899-50, S0383-70) and Esterline (ELA-SA-401) in the as-received condition and after exposure to AO ($>6.3 \times 10^{20}$ atoms/cm² fluence). Test modes were a button seal pressed against another button seal, a button pressed against an aluminum plate, and as-received and AO exposed ($>4.3 \times 10^{19}$ atoms/cm²) 12 in. diameter medium-scale LIDS seals pressed against a stainless steel (SS) plate. Data drawn from Tables 2, 3, 4, and 5.

1-0004) exhibited slightly higher adhesion. However, when comparing as-received and AO pretreated button and medium-scale adhesion results, it is clear the AO pretreatment reduced adhesion considerably (Fig. 10).

NASA-Esterline 2-piece seal design: Figure 10 and Table 5 also show the medium-scale adhesion results for the as-received NASA-Esterline 2-piece seal design. As seen in the button tests in the as-received state, the medium-scale seal made of the ELA-SA-401 compound exhibited lower unit adhesion than the average of the medium-scale seals made of the S0383-70 compound. Note that test results for AO pretreated medium-scale seals made of the Esterline compound were unavailable at the time of writing this paper.

3. Projection of Full-Scale Seal Adhesion

With medium-scale seal adhesion results in both as-received and AO pretreated conditions, predictions can be made for full-scale seal adhesion levels. The rightmost column of Table 5 presents an estimate of the force required to release a full-scale 58 in. outer diameter docking seal from an aluminum flange mounted to the ISS. The estimate was obtained by first determining the amount of force generated per linear inch of the 12 in. seal. Since the full scale seal was an identical cross section, this value can be scaled up using the total linear length of the full-scale seal.

The docking system design requires the full-scale seal separating loads to be ≤ 300 lbf to allow the separating mechanism to undock the two vehicles. Table 5 shows that if left unchecked, the adhesion forces of full-scale seals exceed the design requirement. For the CBM seals, separating loads upward of 618 lbf were projected. The release load for the narrower EDU58 seal design was much lower (301 lbf) due to its smaller footprint. The EDU58's narrow design lowers both the force required to compress it when docking and the force required to undock. The results shown in Table 5 should be considered preliminary, since only one test at each condition has been completed thus far, and the counter face used was stainless steel rather than an aluminum alloy expected to be used on ISS.

4. Minimum Required AO Pretreatment for Low Adhesion

Table 5 also shows the merits of AO pretreatment to a level of approximately 10^{20} atoms/cm² for lowering overall adhesion to acceptable levels. For instance, the expected release load of the full scale CBM design seal would drop from 618 lbf to 68 lbf by applying the AO pretreatment. Similarly low full-scale loads are shown for the EDU54 design. As shown in Table 5, the as-received medium-scale Esterline 2-piece seal has the highest "expected full-scale release force" even though the Esterline material exhibits less adhesion per square inch compared to the as-received Parker compounds. The wider Esterline 2-piece seal has a larger footprint, which results in this larger release force. If similar decreases in adhesion are realized post AO treatment – the release force for the full-scale Esterline 2-piece seal should drop to approximately 192 lbf assuming a post AO treatment adhesion of 1.5 psi (taken from the adhesion of the AO exposed S0383-70, Mod. CBM seal in Table 5).

It is desirable to achieve the adhesion load reduction without increasing leakage rates. Daniels et al³ showed in small-scale seal leakage tests that these low levels of AO pretreatment do not result in appreciable change in overall leakage. The data presented herein is forming the basis for an AO pretreatment exposure for future full-scale seal evaluations.

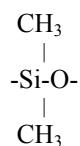
Heat Shield Seals

Figure 9 and Table 4 provide adhesion results for the heat shield seals. The heat shield seal (elastomer compound only) exhibited adhesion unit stress of on average 4.7 psi. The heat shield seal with Nomex molded as an outer cover exhibited adhesion unit loads at the lower threshold (0.3 psi) of the measurement technique. Originally, NASA was considering a separable heat shield that would have required very low adhesion from the sealing elements, making the Nomex covering highly desirable. NASA is now considering a retained heat shield where adhesive loads may be less important.

B. Chemistry

So that the mechanism responsible for changes in adhesion could be better understood, the chemical composition of the surface of the elastomers in the as received and pretreated conditions was examined using IR spectroscopy. In IR spectroscopy, the spectrum of absorbed IR reveals atomic vibration frequencies, which are characteristic of the molecules and compounds present. Figure 11 shows the results from tests on ELA-SA-401 using ATR. Results for S0383-70 and S0899-50 were similar.

Once the wavenumbers of absorbance peaks were located, various literature sources were used to identify the molecular components present in the samples.¹⁰ This information was used to determine changes in the surface chemistry. The peaks located at wavenumbers 1260 cm⁻¹, 796 cm⁻¹, and the weak peak near 860 cm⁻¹, taken as a group, are characteristic of the Si-CH₃ group present in polysiloxanes, particularly dimethyl units:



These two primary Si-CH₃ peaks (1260 cm⁻¹ and 796 cm⁻¹) are relatively large in the as-received material and smaller after AO exposure, indicating a relative decrease in the Si-CH₃ group present at the surface.

The other peaks, near 1020 cm^{-1} and 1070 cm^{-1} , are characteristic of siloxanes (Si-O-Si). Disiloxanes and small-ring cyclosiloxanes show a single peak. As chains become longer or branched, the Si-O-Si absorption becomes broader and more complex. Absorption shows two or more overlapping bands, as shown in Fig. 11. The peaks at 1020 cm^{-1} and 1070 cm^{-1} are characteristic of Si-O groups present in poly(dymethylsiloxane) $[(\text{CH}_3)_2\text{SiO}]_x$, and are thus consistent with the determination of dymethyl groups based on the other peaks. After exposure to AO, the Si-O-Si peaks increase relative to the Si-CH₃ peaks, indicating methyl groups are being replaced by oxygen.

Tests on the interior of exposed o-rings indicate that the reactions were limited to near the surface only. The lines in Fig. 11 labeled “Bottom exposed o-ring” show the data resulting from the unexposed underside of the exposed o-ring. The absorbance profiles of these two tests and the as-received sheet and o-ring were all similar. Thus exposure to AO resulted in a SiO_x rich layer on the surface of the elastomers. This SiO_x rich layer effectively lowered the adhesion of the elastomers tested. Since chemistry changes were similar in the three elastomers, only results for XELA-SA-401 are presented.

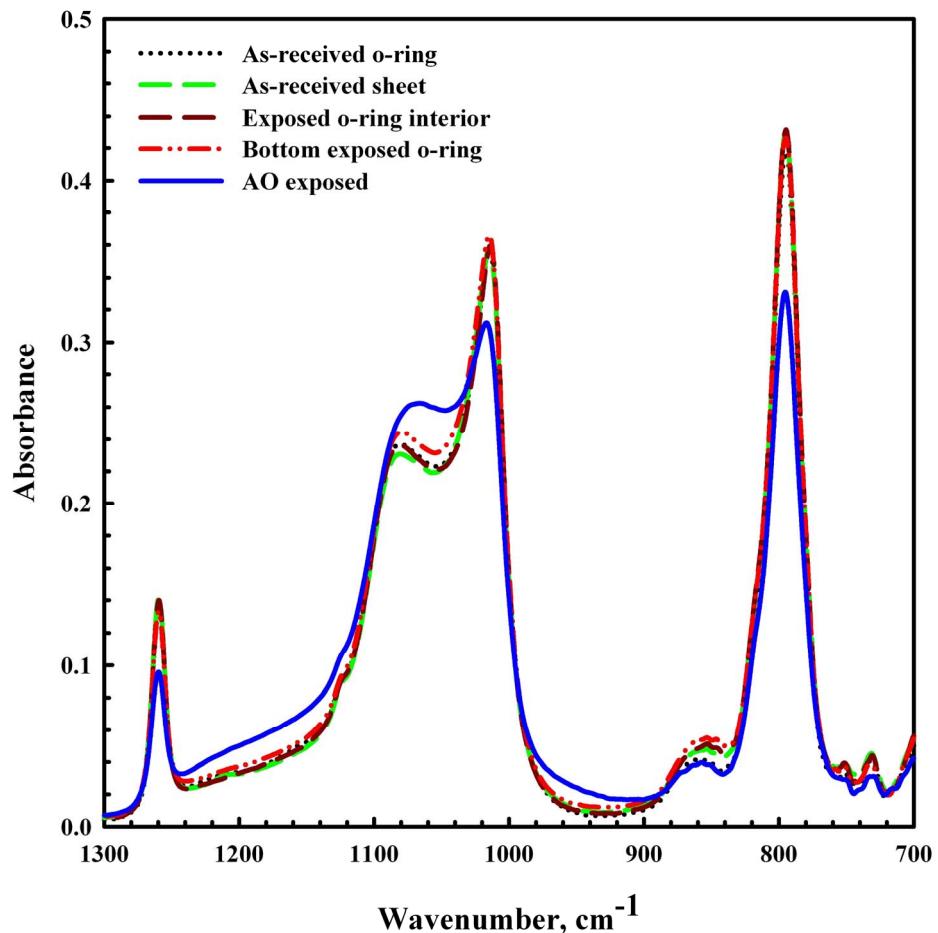


Figure 11. ATR IR spectroscopy of Esterline ELA-SA-401 in the as-received condition, and after AO exposure. Due to the simulated space exposure, CH₃ related peaks ($1260, 796, 860\text{ cm}^{-1}$) decrease relative to Si-O related peaks ($1020, 1070\text{ cm}^{-1}$). Peaks and trends were similar for the Parker S0383-70 and S0899-50 materials.

IV. Conclusions

Silicone-based seals are being considered for a number of locations on NASA’s Crew Exploration Vehicle, including the docking interface, hatches, heat shield seals, amongst others. In the as-received condition, silicone seals exhibit considerable adhesion and could lead to excessive separating loads if left unchecked. This paper presents adhesion properties for seals mated against candidate counter face materials in both the as-received and adhesion-mitigated conditions.

Docking System Seals: In the as-received condition, candidate elastomers being considered for the main docking (LIDS) interface seal have estimated adhesion forces in excess of 300 lbf, which is greater than allowable limits for CEV. Therefore, countermeasures are required to decrease the adhesion of the rubber used in the LIDS main interface seal. Exposure to atomic oxygen (AO) has proven effective at decreasing seal adhesion. AO reacts with the surface of the seal and forms a thin SiO_x rich layer, dramatically decreasing adhesive properties in the three elastomers examined (Parker Hannifin S0899-50 and S0383-70, and Esterline ELA-SA-401). The benefits of AO on reducing adhesion were examined in small-scale idealized tests and in medium-scale engineering tests. AO exposure of approximately 10²⁰ atoms/cm² fluence lowered adhesion in S0383-70 and ELA-SA-401 to acceptable levels (release force <300 lbf) when extrapolated to full-scale seals. Adhesion for the S0899-50 compound remained unacceptably high even after 10²¹ atoms/cm² AO exposure. IR spectroscopy supported the contention that decreases in adhesion are due to the formation of a glassy, non-sticky, SiO_x rich layer on the AO exposed surface of the rubber.

Heat Shield Seals: Adhesion tests were performed on specimens cut from candidate heat shield pressure seals. Nomex covered ELA-SA-401 heat shield seals showed reduced unit adhesion from 4.7 psi to the threshold of the measurement system (0.3 psi).

Table 1. Specimens and Materials used in Adhesion tests.

Specimen	Outer Diameter (in.)	Contact Area (in ²)	Materials		
			S0383-70	S0899-50	ELA-SA-401
Cylindrical Buttons from Sheet	0.37	0.107	✓	✓	✓
Cylindrical Buttons from Heat Shield seal	0.37	0.107			✓
Small scale #2-309 o-rings	0.83	0.25	✓		✓
12 in. Medium-Scale Parker Gask-O-Seals					
Mod. CBM	12	8.54	✓		
EDU58	12	6.83	✓		
EDU54	12	8.13	✓		
12 in. Medium-Scale Esterline 2-piece					
LIDS6016	12	24.6			✓

Table 2. Adhesion of Parker S0899-50 Button Specimens: ID numbers for specimens tested are included for traceability of data. Al was a bare aluminum block with a surface finish of $Ra = 8 \times 10^{-6}$ in. Compression dwell time was 24 hours, except for the pair 526/527, which used a dwell time of one hour. Maximum accuracy of adhesion measurements was ± 0.3 psi, thus measurements of 0.3 psi or less are listed as 0.3 psi. Standard deviation was 6.9 psi and 6.6 psi for as-received seal-on-seal and seal-on-Al, respectively.

Seal-on-Seal			Seal-on-Al		
ID	Average AO Exposure (atom/cm ²)	Adhesion, (psi)	ID	Average AO Exposure (atom/cm ²)	Adhesion, (psi)
26/27	0	85.8	533/Al	0	31.8
22/23	0	72.1	532/Al	0	45
24/25	0	87.4			
524/525	6.40E+20	4.7			
526/527	1.30E+21	4.3			
145/144	1.59E+21	13.41			
138/141	1.81E+21	9.97			
142/149	1.96E+21	11.3			
197/198	2.93E+21	11.94			
192/201	5.06E+21	9.6			
90/93	5.18E+21	10.8			
88/91	7.35E+21	9.7			
273/274	1.10E+22	0.3			

Table 3. Adhesion of Parker S0383-70 Button and Small Diameter O-ring Specimens: ID numbers for specimen pairs are included for traceability of data; specimens were 0.94 cm (0.38 in.) diameter buttons unless otherwise noted. When testing Seal-on-Al, Al was a bare aluminum block with a surface finish of $Ra = 8 \times 10^{-6}$ in. When testing Seal-on-Anodized, a similar aluminum block that had been anodized was used. The average atomic oxygen fluence for the specimen pair is provided. Compression dwell time was 24 hours except for the pair 130/131 which used a dwell time of one hour. Maximum accuracy of adhesion measurements was ± 0.3 psi, thus measurements of 0.3 psi or less are listed as 0.3 psi. Standard deviation was 5.9 psi and 7.5 psi for as-received seal-on-seal, and seal-on-Al, respectively.

Seal-on-Seal			Seal-on-Al			Seal-on-Anodized		
ID	Average AO Exposure (atom/cm ²)	Adhesion, (psi)	ID	Average AO Exposure (atom/cm ²)	Adhesion, (psi)	ID	Average AO Exposure (atom/cm ²)	Adhesion, (psi)
16/17	0	30.3	530/Al	0	4.25	768/AlAn	2.67E+19	1.5
18/19	0	44.5	531/Al	0	10.27	782/AlAn	2.87E+19	1.3
20/21	0	39.4	o-ring 535/Al	0	22.2	779/AlAn	2.99E+19	0.63
A12/A13	1.00E+18	12.6	A12/Al	1.00E+18	4.9	761/AlAn	3.16E+19	2.9
B22/B23	3.00E+18	2.2	B22/Al	3.00E+18	5.4	762/AlAn	3.66E+19	2.11
C32/C33	1.00E+19	1.6	C32/Al	1.00E+19	2.8	756/AlAn	4.33E+19	3.63
748/749	7.00E+19	3.3	753/Al	2.10E+19	3.34	771/AlAn	4.73E+19	2.75
750/751	1.00E+20	1.5	754/Al	2.80E+19	4.5	812/AlAn	4.75E+19	3.6
520/521	6.40E+20	0.3	784/Al	3.10E+19	1.76	808/AlAn	4.90E+19	3.88
522/523	1.30E+21	0.3	789/Al	4.23E+19	2.34	807/AlAn	5.07E+19	2.02
130/131	1.58E+21	2.8	791/Al	4.34E+19	2.06	763/AlAn	5.17E+19	2.03
130/131	1.58E+21	0.3	785/Al	4.91E+19	1.07	770/AlAn	5.42E+19	0.3
124/128	1.70E+21	1.9	787/Al	5.05E+19	2.26	772/AlAn	5.51E+19	1.21
123/129	2.00E+21	0.3	769/Al	7.30E+19	0.3	811/AlAn	5.73E+19	2.94
124/125	2.10E+21	1.0	764/Al	9.65E+19	0.61	757/AlAn	5.75E+19	0.3
126/127	2.10E+21	1.9	767/Al	1.08E+20	0.3	755/AlAn	5.80E+19	1.23
181/184	3.05E+21	0.4	758/Al	1.10E+20	0.67	778/AlAn	5.91E+19	3.19
178/179	5.15E+21	0.3	520/Al	6.40E+21	0.3	806/AlAn	6.25E+19	1.6
84/187	5.32E+21	0.3				810/AlAn	6.82E+19	0.3
65/69	5.70E+21	0.3				819/AlAn	6.92E+19	0.7
64/85	6.97E+21	0.3				818/AlAn	6.99E+19	2.41
68/87	7.62E+21	0.3				817/AlAn	7.07E+19	2.36
261/262	1.51E+22	0.3				805/AlAn	7.40E+19	1.35
						760/AlAn	7.42E+19	0.45
						815/AlAn	7.58E+19	2.15
						816/AlAn	7.65E+19	2.48
						814/AlAn	7.66E+19	3.41
						804/AlAn	7.74E+19	0.3
						813/AlAn	7.75E+19	3.28
						809/AlAn	7.92E+19	2.76
						809/AlAn	7.92E+19	0.3
						775/AlAn	9.06E+19	0.3
						776/AlAn	9.21E+19	0.3
						765/AlAn	1.03E+20	0.3
						766/AlAn	1.07E+20	0.6
						759/AlAn	1.77E+20	0.3

Table 4. Adhesion of Esterline ELA-SA-401 Button and Small Diameter O-ring Specimens. ID numbers for specimen pairs are included for traceability of data; specimens were 0.94 cm (0.38 in.) diameter buttons unless otherwise noted. When testing Seal-on-Al, Al was a bare aluminum block with a surface finish of $R_a = 8 \times 10^{-6}$ in. When testing Seal-on-Anodized, a similar aluminum block that had been anodized was used. Seal-on-Ti used a Ti-6Al-4V block. The average atomic oxygen fluence for the specimen pair is provided. Compression dwell time was 24 hours except for the pair 158/160 and 94/96 which used a dwell time of 8 hours. Maximum accuracy of adhesion measurements was ± 0.3 psi, thus measurements of 0.3 psi or less are listed as 0.3 psi. Standard deviation was 0.42 psi and 0.3 psi for as-received seal-on-seal and seal-on-Al respectively.

Seal-on-Seal			Seal-on-Al			Heat shield seal-on-Ti		
ID	Average AO Exposure (atom/cm ²)	Adhesion (psi)	ID	Average AO Exposure (atom/cm ²)	Adhesion (psi)	ID	AO	Adhesion (psi)
28/29	0	21.5	528/Al	0	4	745/Ti	0	3.7
30/31	0	22.4	529/Al	0	3.4	744/Ti	0	5.6
32/33	0	21.5	o-ring 534/Al	0	6.51	747/Nomex/Ti	0	0.3
153/159	1.61E+21	0.3	534/Nomex/Al	0	0.3	746/Nomex/Ti	0	0.3
158/160	1.74E+21	0.3	151/Al	2.00E+21	0.3			
150/151	1.96E+21	0.3	151/Al	2.00E+21	0.8			
206/213	3.10E+21	0.96						
208/208	5.31E+21	0.3						
94/96	7.20E+21	0.3						
286/285	1.18E+22	0.3						

Table 5. Adhesion of 12 in. outer diameter Medium-Scale Docking Seals. Adhesion data as a function of exposure to atomic oxygen for Parker S0383-70 and Esterline ELA-SA-401. Adhesion (in pounds) was the force required to pull the seal off a stainless steel plate. This force depends on the surface area contacting the plate. The adhesion in units of lbf/in² was calculated using a width characteristic of the seal's footprint. An average centerline diameter of 56.5 in.(143.5 cm) was used to estimate the force required to separate the full scale seal. Compression dwell time was 70 hours.

Material	Specimen	Design	Average AO Exposure (atom/cm ²)	Adhesion (lbf)	Average Centerline Seal dia. (in.)	Adhesion (psi)	Expected Full Scale Release Force (lbf)
S0383-70	206147-1-0003	Mod. CBM	0	119	10.88	13.9	618
S0383-70	207253-3-0003	EDU58	0	58	10.88	8.5	301
S0383-70	206147-1-0003	Mod. CBM	4.38E+19	13	10.88	1.5	68
S0383-70	206200-1-0003	EDU54	4.90E+19	13	10.88	1.6	68
S0383-70	206200-1-0004	EDU54	1.18E+20	20	10.88	2.5	104
ELA-SA-401	Esterline 2-piece	LIDS6016	0	147	10.88	6.0	789

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1. REPORT DATE (DD-MM-YYYY) 01-10-2008			2. REPORT TYPE Technical Memorandum			3. DATES COVERED (From - To)	
4. TITLE AND SUBTITLE Adhesion of Silicone Elastomer Seals for NASA's Crew Exploration Vehicle			5a. CONTRACT NUMBER				
			5b. GRANT NUMBER				
			5c. PROGRAM ELEMENT NUMBER				
6. AUTHOR(S) de Groh, Henry, C., III; Miller, Sharon, K., R.; Smith, Ian, M.; Daniels, Christopher, C.; Steinetz, Bruce, M.			5d. PROJECT NUMBER				
			5e. TASK NUMBER				
			5f. WORK UNIT NUMBER WBS 644423.06.31.04.01.03.22				
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) National Aeronautics and Space Administration John H. Glenn Research Center at Lewis Field Cleveland, Ohio 44135-3191			8. PERFORMING ORGANIZATION REPORT NUMBER E-16598				
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES) National Aeronautics and Space Administration Washington, DC 20546-0001			10. SPONSORING/MONITORS ACRONYM(S) NASA				
			11. SPONSORING/MONITORING REPORT NUMBER NASA/TM-2008-215433				
12. DISTRIBUTION/AVAILABILITY STATEMENT Unclassified-Unlimited Subject Categories: 27 and 18 Available electronically at http://gltrs.grc.nasa.gov This publication is available from the NASA Center for AeroSpace Information, 301-621-0390							
13. SUPPLEMENTARY NOTES							
14. ABSTRACT Silicone rubber seals are being considered for a number of interfaces on NASA's Crew Exploration Vehicle (CEV). Some of these joints include the docking system, hatches, and heat shield-to-back shell interface. A large diameter molded silicone seal is being developed for the Low Impact Docking System (LIDS) that forms an effective seal between the CEV and International Space Station (ISS) and other future Constellation Program spacecraft. Seals between the heat shield and back shell prevent high temperature reentry gases from leaking into the interface. Silicone rubber seals being considered for these locations have inherent adhesive tendencies that would result in excessive forces required to separate the joints if left unchecked. This paper summarizes adhesion assessments for both as-received and adhesion-mitigated seals for the docking system and the heat shield interface location. Three silicone elastomers were examined: Parker Hannifin S0899-50 and S0383-70 compounds, and Esterline ELA-SA-401 compound. For the docking system application various levels of exposure to atomic oxygen (AO) were evaluated. Moderate AO treatments did not lower the adhesive properties of S0899-50 sufficiently. However, AO pretreatments of approximately 10^{20} atoms/cm ² did lower the adhesion of S0383-70 and ELA-SA-401 to acceptable levels. For the heat shield-to-back shell interface application, a fabric covering was also considered. Molding Nomex fabric into the heat shield pressure seal appreciably reduced seal adhesion for the heat shield-to-back shell interface application.							
15. SUBJECT TERMS Adhesion; Rubber; Elastomers; Atomic; Oxygen; Exposure; Docking; Silicones; Spacecraft docking							
16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT	18. NUMBER OF PAGES	19a. NAME OF RESPONSIBLE PERSON STI Help Desk (email:help@sti.nasa.gov)		
a. REPORT U	b. ABSTRACT U	c. THIS PAGE U	UU	22	19b. TELEPHONE NUMBER (include area code) 301-621-0390		

