

## EVALUATIONS OF CANDIDATE MATERIALS FOR ADVANCED SPACE-RATED VACUUM SEALS TO EXPLORE SPACE ENVIRONMENT EXPOSURE LIMITS

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### ABSTRACT

For many materials used in space hardware, the environment in which they need to operate is harsher than the environment on earth. Exposure to vacuum conditions, atomic oxygen, and ultraviolet radiation can be detrimental, so testing of space hardware in simulated space environments is required. This is especially true for elastomeric components such as seals. NASA is developing advanced space-rated vacuum seals in support of future space exploration missions. These seals must exhibit extremely low leak rates to ensure that astronauts have sufficient breathable air during extended-duration missions. In some applications the seals are not mated during portions of the mission and are left uncovered and exposed to the conditions in space for prolonged periods of time prior to mating. Space-rated vacuum seals are often made of silicone because of the material's wide operating temperature range and ability to be molded or extruded into various shapes and cross sections. One approach being considered to achieve improved performance is to add titanium dioxide to the silicone material to make it more resistant to damage from ultraviolet radiation. In this study, seals made of the baseline material with and without titanium dioxide additive were exposed to atomic oxygen and increasing levels of ultraviolet radiation and then leak tested. Test results revealed that seals made of the new material could withstand longer exposures while still satisfying the leak rate requirement even under worst-case conditions of partial compression at the extremes of the anticipated operating temperature range.

### INTRODUCTION

The National Aeronautics and Space Administration (NASA) is developing advanced space-rated vacuum seals in support of future space exploration missions to low Earth orbit (LEO) and deep space. This includes seals for future docking systems and vehicle hatches as shown in Figure 1. These seals can be relatively large with diameters on the order of 127 cm (50 in.), and they must exhibit extremely low leak rates to ensure that astronauts have sufficient breathable air during extended-duration missions. In some applications the seals are not mated during portions of the mission and are left uncovered and exposed to the conditions in space for prolonged periods of time prior to mating. This includes exposure to vacuum conditions, atomic oxygen (AO), and ultraviolet (UV) radiation. Space-rated vacuum seals are often made of silicone because of the material's wide operating temperature range and ability to be molded or extruded into various shapes and cross sections. While seals made of silicone have some capacity to withstand exposure to these conditions, testing has shown that prolonged exposure results in a degradation of seal performance including higher leak rates (ref. 1).

One approach being considered to achieve improved performance in space-rated seals is to add titanium dioxide ( $\text{TiO}_2$ ) to the silicone material to make it more resistant to damage from UV radiation. Titanium dioxide is a naturally occurring material that is often added to sunscreens and cosmetics because of its high refractive index and strong UV light absorbing capabilities. In this study, seals made of the baseline silicone material with and without  $\text{TiO}_2$  additive were exposed to AO and increasing levels of UV radiation and then leak tested. The objectives were twofold: (1) to determine if the addition of  $\text{TiO}_2$  to the baseline silicone material provided protection to the seals from damage caused by UV radiation exposure and (2) to evaluate how much UV radiation exposure, in terms of equivalent sun hours (ESH), the seals could tolerate and still satisfy leak rate requirements.

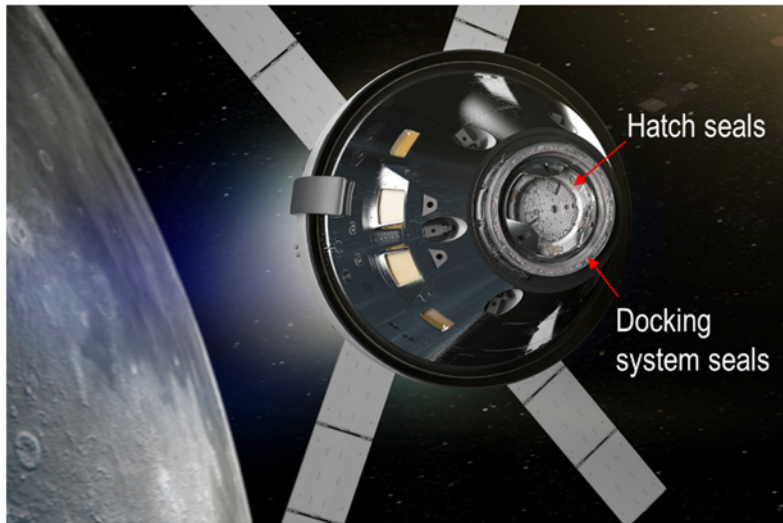


Figure 1. Potential locations for advanced space-rated vacuum seals on future spacecraft such as Orion.

### TEST SPECIMENS

The test specimens evaluated in this study were all subscale versions of a multi-piece seal design that consisted of an elastomer element and a separate metal retainer (Figure 2). The elastomer element had two seal bulbs connected by a web, and the metal retainer was installed between the seal bulbs. Periodic raised bosses on the bottom of the retainer passed through openings in the elastomer web, and fasteners installed in the retainer on a 27.622 cm (10.875 in.) bolt circle diameter secured the seal assembly to the base of the seal groove in which it was installed. Additional details on this seal design can be found in reference 2. In this study, the seal bulbs were the sections of the elastomer element exposed to AO and UV radiation.

The baseline silicone material evaluated in this study was S0383-70 silicone, a 70 durometer material offered by Parker Hannifin Corporation's Composite Sealing Systems Division (San Diego, CA). This formulation of silicone has been used successfully in a variety of space flight applications including seals for multiple locations on the International Space Station. Test specimens were made of either the baseline silicone compound or the baseline compound with TiO<sub>2</sub> added.

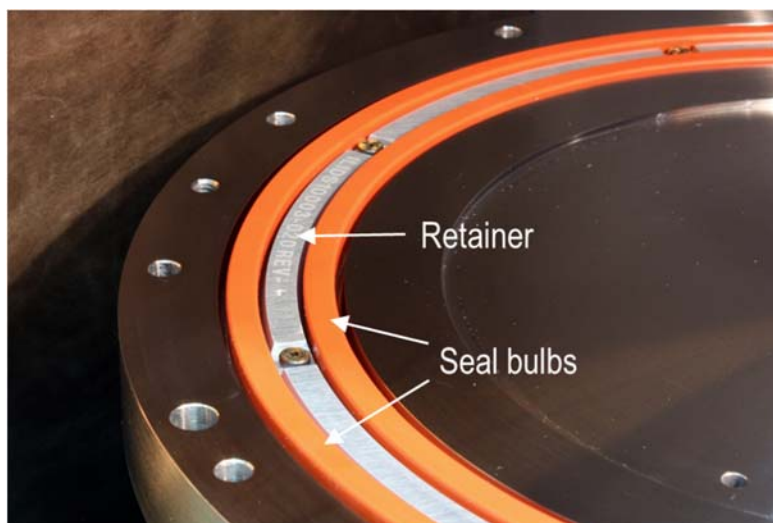


Figure 2. Representative photograph of multi-piece seal test specimen.

## TEST APPARATUS AND PROCEDURES

### AO Exposures

Seal test specimens were exposed to AO in the Large Area Atomic Oxygen Exposure Facility at the NASA Glenn Research Center (GRC) (Figure 3). This facility exposed the seals to AO by creating a radio frequency-generated plasma between two 1.5 m by 1.5 m (5 ft by 5 ft) plates. The AO fluence that the seals were exposed to in each run was calculated by weighing Kapton H and Kapton HN witness samples that were exposed at the same time as the seals. Kapton erodes at a known rate when exposed to AO, so the weight loss during an exposure was used to estimate the AO fluence. The test specimens evaluated in this study were exposed to a nominal AO fluence of  $8.8 \times 10^{19}$  atoms/cm<sup>2</sup> which corresponded to about two days of exposure in LEO for ram-facing (i.e., forward facing) surfaces. This duration was chosen based on the assumption that a vehicle would spend a short amount of time in LEO before travelling to a destination beyond LEO where AO is no longer present. Additional details on this facility can be found in references 3 and 4.

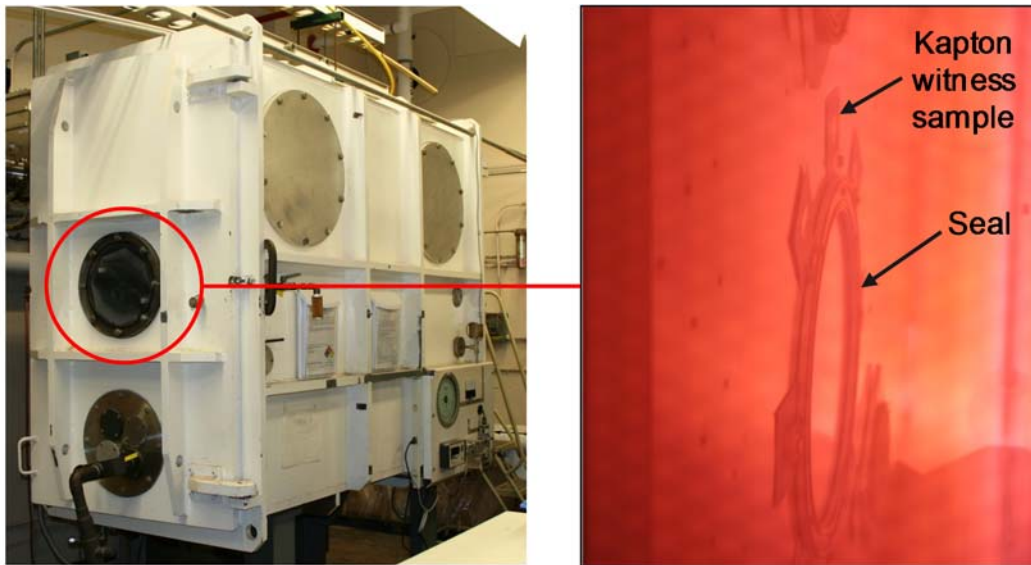


Figure 3. Large Area Atomic Oxygen Exposure Facility at NASA GRC (left) and seal test specimens being exposed to AO plasma (right).

### UV Radiation Exposures

UV radiation exposures were performed in the X-25 Solar Simulator Facility at NASA Marshall Space Flight Center (MSFC). During the exposures, the seals were simultaneously exposed to UV radiation in both the near UV (NUV) wavelength range of 250 to 400 nm and the vacuum UV (VUV) range with wavelengths up to about 200 nm. The exposures were performed under vacuum. This approach was followed to ensure that the test specimens were exposed to the full spectrum of UV radiation experienced during a mission. Figure 4 shows the separate ports on the vacuum chamber that permitted this to occur, while Figure 5 shows a seal test specimen mounted inside the facility for a UV radiation exposure.

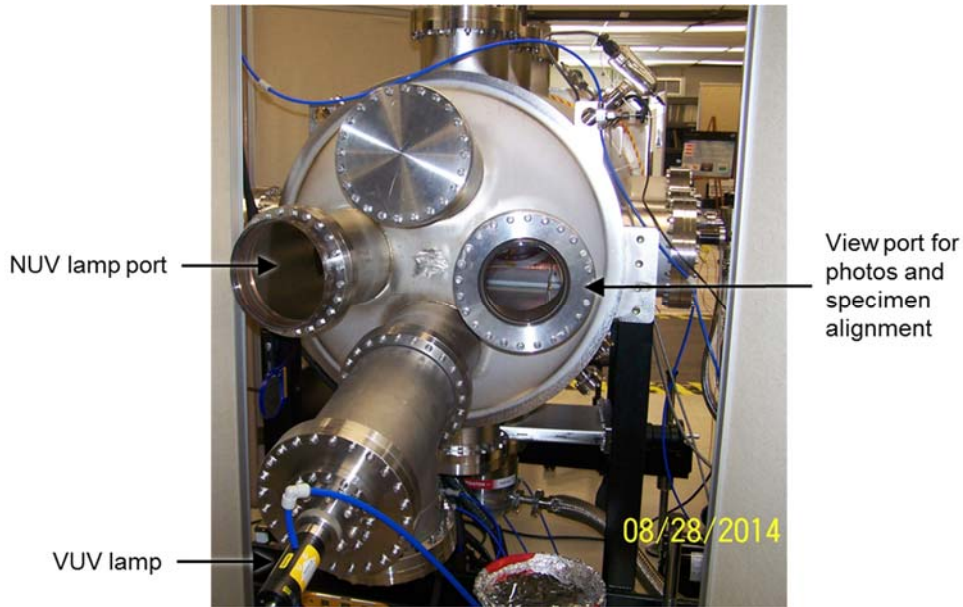


Figure 4. MSFC X-25 Solar Simulator Facility used for seal UV radiation exposures.

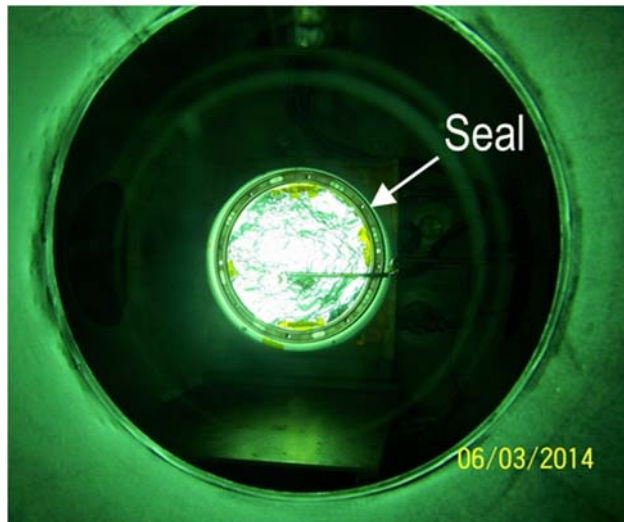


Figure 5. Seal test specimen in X-25 Solar Simulator Facility at NASA MSFC during UV radiation exposure.

### Leak Tests

Leak tests were performed on the seals using a test apparatus consisting of a pair of clear anodized 6061-T6 aluminum test plates, near-hermetic plumbing and valves, and the necessary measurement instruments (Figure 6). This setup was installed into a Tenney Benchmaster BTCR environmental control chamber capable of cooling or heating the test article from -150 to 600°C (-238 to 1122°F) with an accuracy of  $\pm 0.1^\circ\text{C}$ . The seals were installed in a groove and were compressed against a flat plate with a surface finish of  $0.41\ \mu\text{m}$  (16  $\mu\text{in.}$ ). Tests were performed on fully compressed seals as well as on partially compressed seals to evaluate their capability to tolerate potential joint separation at the sealing interface. Most leak tests were performed at a nominal, regulated temperature of  $23^\circ\text{C}$  ( $73^\circ\text{F}$ ), but some tests were also performed at the extremes of the anticipated operating temperature range ( $-7^\circ\text{C}$  ( $19^\circ\text{F}$ ) and  $56^\circ\text{C}$  ( $133^\circ\text{F}$ )) to evaluate the effects of temperature on seal leakage.



The test fixture was designed so that either seal bulb could be tested with a nominal pressure differential of 101 kPa (14.7 psid) across it with the pressure source inboard of the seal bulb and low pressure conditions on the outboard side. The nominal upstream and downstream pressures were 126 and 25 kPa, respectively. Leak tests were performed using a pressure decay methodology, and the leak rate of each test specimen was quantified using the mass point leak rate technique with comprehensive error analysis (references 5 and 6). All leak test results presented for this study were for the inner seal bulb of each test specimen.



Figure 6. Seal leak test fixture installed in environmental control chamber.  
(Note: Top plate of test fixture not installed.)

## TEST RESULTS

### Effects of Test Duration

Figure 7 presents results for long duration leak tests performed at 23°C on fully compressed seals made of the TiO<sub>2</sub> material after exposure to a nominal AO fluence of  $8.8 \times 10^{19}$  atoms/cm<sup>2</sup> and various amounts of UV radiation. In each case as a leak test progressed, the leak rates for exposed seals decreased while the seal remained in a compressed state. For UV radiation exposures up to 1000 ESH, steady state leak rates were two to three times lower than those measured early in the test with the majority of the decrease occurring within the first 24 hrs of testing. However, the decrease in leak rate occurred more gradually for the seals exposed to 1772 or 2500 ESH of UV radiation, and the amount of the decrease was less. For UV radiation exposure levels up to 1000 ESH, steady state leak rates were about 35-60% of what they were at the start of a leak test, whereas steady state leak rates for the seals exposed to 1772 or 2500 ESH of UV radiation were about 60-70% of initial measured values. Overall, this behavior may be beneficial for long-term sealing applications as the seals seem to “recover” over time from the AO and UV radiation exposure damage. However, the amount of recovery appears to diminish as the seals are exposed to UV radiation for longer periods of time. Note that this behavior has not been observed for unexposed seals where leak rates typically remain fairly constant throughout a test.

Figure 8 presents results for long duration leak tests performed on seals made of the baseline material after exposure to AO and 250, 500, 750, 1000, or 1250 ESH of UV radiation. While leak rates for these seals decreased as the tests progressed, the magnitude of the decrease during each test was generally less on a percentage basis than what was observed for the seals made of the TiO<sub>2</sub> material.

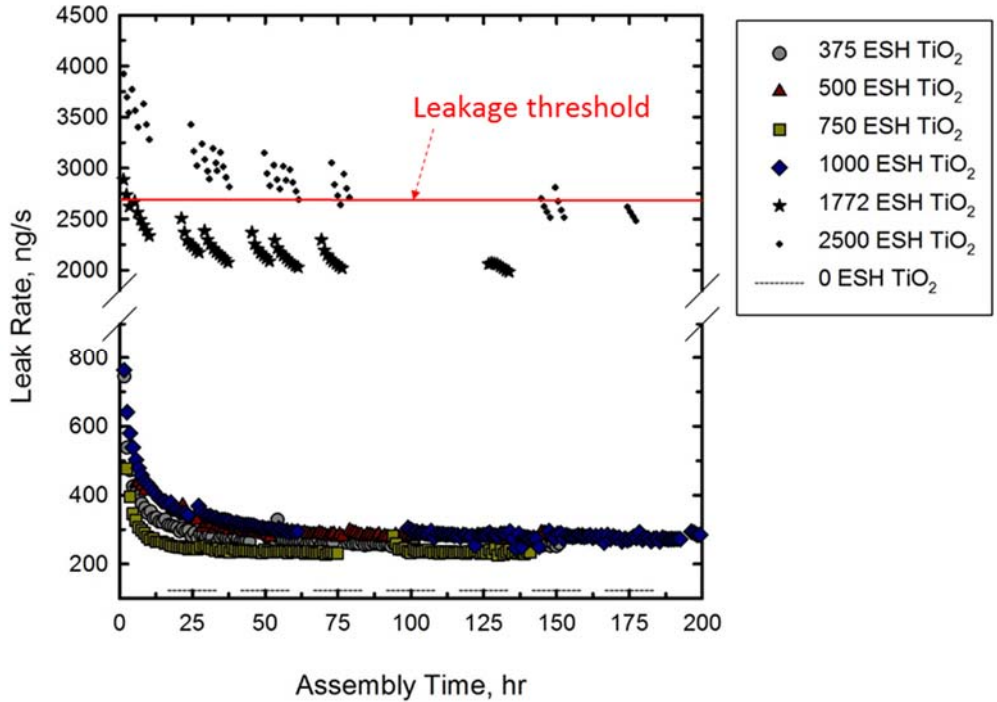


Figure 7. Leak rates at 23°C versus time for inner bulb of fully compressed seals made of baseline material with TiO<sub>2</sub> after exposure to nominal AO fluence of 8.8x10<sup>19</sup> atoms/cm<sup>2</sup> and increasing amounts of UV radiation.

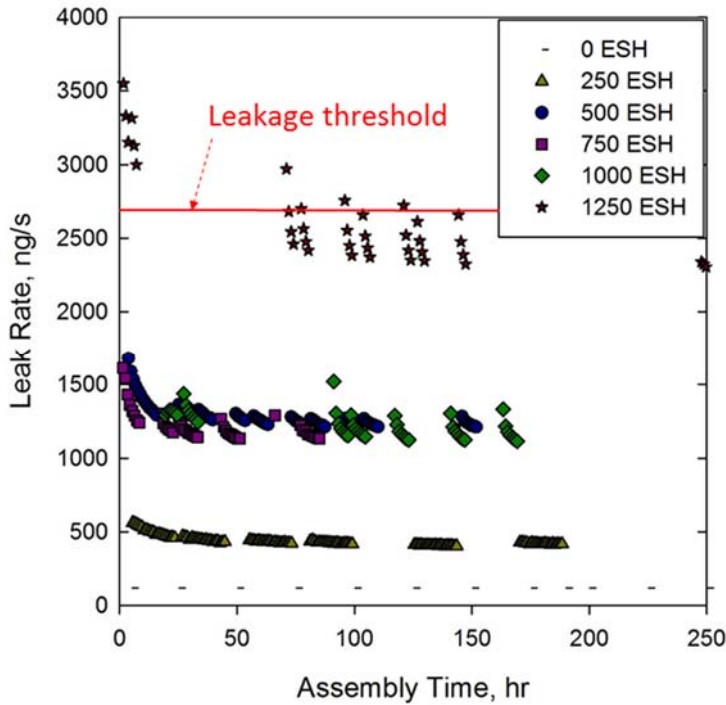


Figure 8. Leak rates at 23°C versus time for inner bulb of fully compressed seals made of baseline material after exposure to nominal AO fluence of 8.8x10<sup>19</sup> atoms/cm<sup>2</sup> and increasing amounts of UV radiation.

## Effects of AO/UV Radiation Exposure

The seal made of the TiO<sub>2</sub> material that was exposed to AO and 2500 ESH of UV radiation was shinier and considerably darker than an unexposed seal indicating that the exposures had caused a change in the seal material. Areas of the seal that were covered by mounting hardware during the exposure remained lighter in color indicating that they were protected by the mounting hardware.

Figure 9 shows the results for leak tests performed at 23°C on fully compressed seals made of the baseline material and the TiO<sub>2</sub> material after exposure to AO and various levels of UV radiation. The results are presented as seal inner bulb leak rates based on steady state leak rates measured for the seals as shown in Figures 7 and 8. All exposed seals were subjected to a nominal AO fluence of  $8.8 \times 10^{19}$  atoms/cm<sup>2</sup> followed by exposure to increasing amounts of UV radiation. Leak rates for unexposed seals are also shown for comparison.

For the seals made of the TiO<sub>2</sub> material, steady state leak rates for UV radiation exposure levels up to 1000 ESH plateaued at a level about two times higher than what was measured for unexposed TiO<sub>2</sub> seals. Leak rates increased at higher UV radiation exposure levels, though. After a UV radiation exposure of 1772 ESH, the steady state leak rate at 23°C for the inner bulb of the seal was 2000 ng/s. After exposure to 2500 ESH of UV radiation, the leak rate increased to 2450 ng/s. Both of these steady state leak rates were below the leakage threshold. However, the leak rates measured at the beginning of each test were above the leakage threshold and decreased to below the threshold by the end of the test as shown in Figure 7. For reference, the scaled leak rate requirement shown in Figures 7 and 8 for these seals is 2690 ng/s.

Leak rates for the seals made of the baseline compound also increased after exposure to AO and UV radiation (Figure 9). An increase in steady state leak rate versus UV radiation exposure level was observed between 0 and 500 ESH. However, like the TiO<sub>2</sub> seals, leak rates for the seals made of the baseline material plateaued after exposure to between 500 and 1000 ESH of UV radiation. At that point, leak rates were about 10 times what they were for unexposed seals and about 40% of the leakage threshold. After a UV radiation exposure of 1250 ESH, the leak rate increased to 2300 ng/s, a value just below the leakage threshold. Figure 8 shows that leak rates for that seal started out above the leakage threshold at the start of leak testing but decreased to below the threshold by the end of the test.

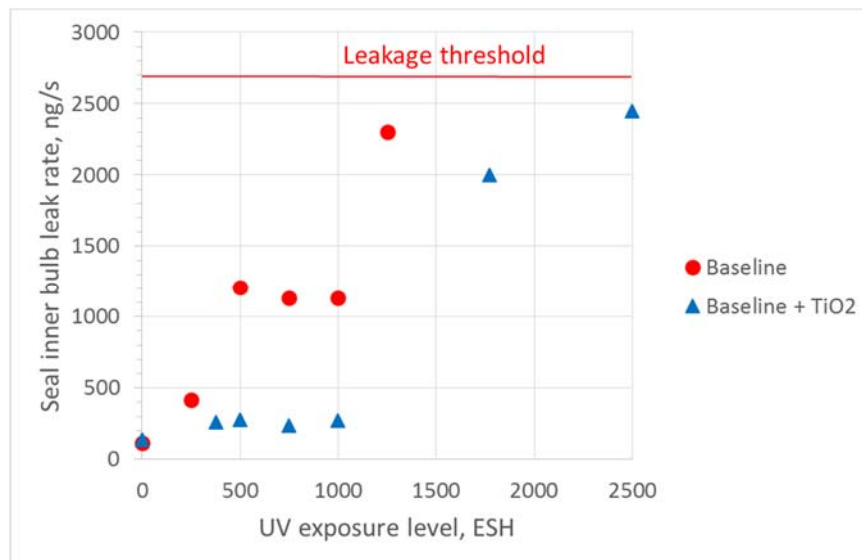


Figure 9. Steady state leak rates at 23°C for inner bulb of fully compressed seals made of baseline material and baseline material with TiO<sub>2</sub> after exposure to nominal AO fluence of  $8.8 \times 10^{19}$  atoms/cm<sup>2</sup> and increasing amounts of UV radiation.

For UV radiation exposure levels up to 1000 ESH, leak rates were generally four to five times higher for seals made of the baseline compound than for seals made of the TiO<sub>2</sub> material, indicating that the addition of TiO<sub>2</sub> to the baseline compound provided protection from damage caused by UV radiation exposure. Although direct

comparisons cannot be made above 1000 ESH based on the available data, there is evidence that the TiO<sub>2</sub> material continued to perform better at higher levels of UV radiation exposure. As noted previously, the steady state leak rate for the seal made of the TiO<sub>2</sub> material after exposure to 2500 ESH of UV radiation was 2450 ng/s. A similar leak rate, 2300 ng/s, was measured for a seal made of the baseline compound after a UV radiation exposure that was only half as long (1250 versus 2500 ESH). The seal made of the TiO<sub>2</sub> material was able to tolerate about twice as much UV radiation exposure as a seal made of the baseline compound while exhibiting similar leakage performance afterward.

### Effects of Test Temperature

Tests were also performed to evaluate the effects of test temperature on seal leak rates after exposure to AO and UV radiation. Figure 10 summarizes the leak rates measured at various temperatures for seals made of the baseline material and the TiO<sub>2</sub> material in an unexposed condition and after exposure to a nominal AO fluence of  $8.8 \times 10^{19}$  atoms/cm<sup>2</sup> and 750 or 1000 ESH of UV radiation. Each test specimen was tested at three different temperatures in the following order: 23°C, 56°C, and -7°C. The test order is noted in the figure by the numbered boxes above the test results for each temperature. For the test specimens exposed to 750 and 1000 ESH of UV radiation, the long duration tests shown in Figures 7 and 8 were performed at 23°C prior to testing at 56 and -7°C. Note that the “warm” leak test for the unexposed (i.e., 0 ESH) baseline seal was performed at 61°C instead of 56°C.

For the unexposed seals, the leak rate increased as the temperature increased. This was consistent with previous test results (ref. 7). However, a different trend was observed for the seals that were exposed to AO and UV radiation. After leak rates were measured at 23°C, tests were performed at 56°C. For each test performed on an exposed seal, the leak rate decreased at the warmer temperature. Then, when the test temperature was reduced to -7°C, the leak rate increased to a level higher than previously measured at 23°C for three of the four seals. For the TiO<sub>2</sub> seal exposed to 1000 ESH of UV radiation, though, the leak rate at -7°C was comparable to what was measured at 56°C. It is unclear why this seal performed differently than the others. This general trend of lower leak rates at higher temperatures is inconsistent with what was observed for the unexposed seals, and it is unclear why this occurred. However, the leak rates for all the seals shown in Figure 10 were below the leakage threshold (2690 ng/s). For example, the highest leak rate for the TiO<sub>2</sub> seal that was exposed to 1000 ESH of UV radiation was still only about 30% of the requirement.

Comparing the results for the seals that were exposed to 1000 ESH of UV radiation, leak rates at the warm and cold temperatures for the seals made of the baseline material were three to five times higher than those for the seals made of the TiO<sub>2</sub> material providing further evidence that the addition of TiO<sub>2</sub> to the baseline compound protects the seals from damage caused by UV radiation exposure.

As noted previously, the seal made of the baseline material that was exposed to 1250 ESH of UV radiation and the TiO<sub>2</sub> seal that was exposed to 2500 ESH of UV radiation exhibited comparable steady state leak rates at 23°C even though the TiO<sub>2</sub> seal was exposed to twice as much UV radiation. Figure 11 compares the leak rates for those two seals for the same test conditions and sequences shown in Figure 10. At each temperature the leak rates for the two seals were fairly similar and within approximately 3 to 10% of each other. The lowest leak rates were measured at the warmest temperature (56°C) with leak rates below the leakage threshold. However, the leak rates for both seals exceeded the leakage threshold at -7 and 23°C.



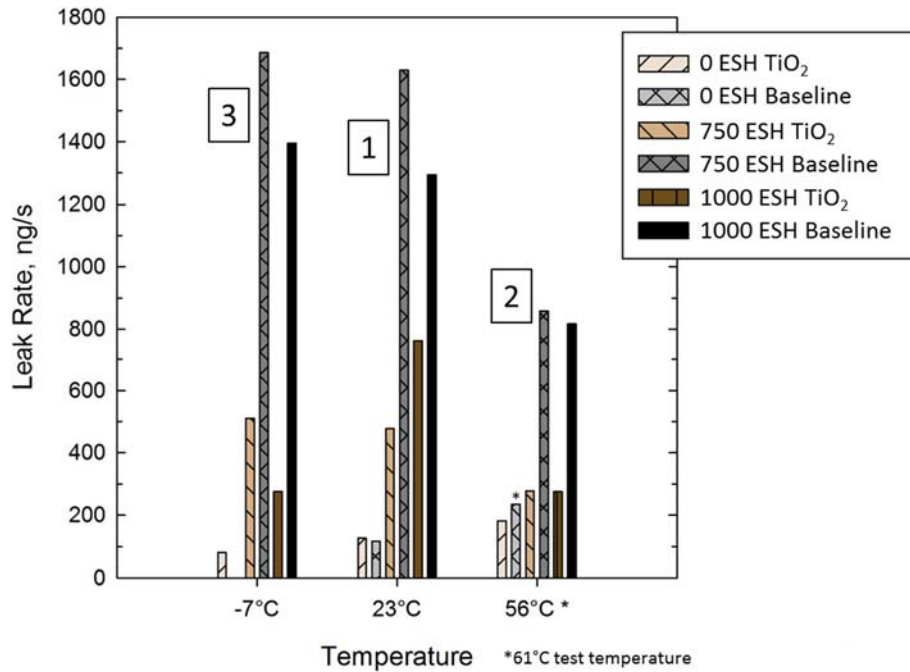


Figure 10. Leak rates at various temperatures for inner bulb of fully compressed seals made of baseline material and baseline material with TiO<sub>2</sub> in an unexposed condition and after exposure to nominal AO fluence of  $8.8 \times 10^{19}$  atoms/cm<sup>2</sup> and 750 or 1000 ESH of UV radiation.

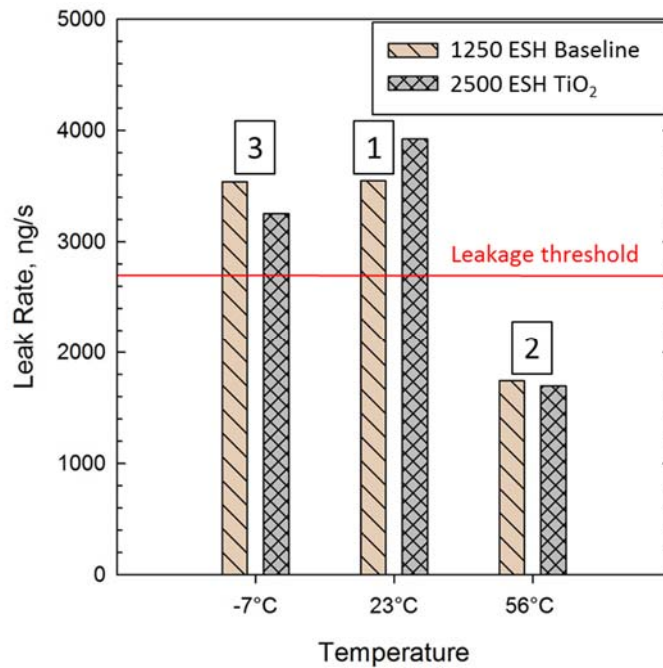


Figure 11. Leak rates at various temperatures for inner bulb of fully compressed seals made of baseline material after exposure to 1250 ESH of UV radiation and baseline material with TiO<sub>2</sub> after exposure to 2500 ESH of UV radiation. Both seals were exposed to nominal AO fluence of  $8.8 \times 10^{19}$  atoms/cm<sup>2</sup>.

## Effects of Partial Compression

As noted earlier, leak tests were performed on partially compressed seals to evaluate their ability to tolerate potential joint separation at the sealing interface. In this study, a uniform gap size of 0.066 cm (0.026 in.) was used to simulate the maximum anticipated steady state gap at the sealing surface. Figure 12 summarizes the leak rates measured at various temperatures for partially compressed seals made of the baseline material and the TiO<sub>2</sub> material after exposure to a nominal AO fluence of  $8.8 \times 10^{19}$  atoms/cm<sup>2</sup> and 500, 750, or 1000 ESH of UV radiation. Each test specimen was tested at four different temperatures in the following order: 23°C, 56°C, -7°C, and back to 23°C. As with Figures 10 and 11, the test order is noted in the figure by the numbered boxes above the test results for each temperature. Each test specimen was subjected to the long duration tests shown in Figures 7 and 8 and the series of tests described in the previous section while under full compression prior to being tested in a partially compressed state.

As with the tests on the fully compressed seals (Figures 10 and 11), leak rates generally decreased as the temperature increased. After leak rates were measured at 23°C, tests were performed at 56°C where lower leak rates were measured for each seal. Then, when the test temperature was reduced to -7°C, the leak rate increased for each seal to a level higher than what was measured at 23°C. Finally, when the test specimen was warmed back to 23°C, the leak rate decreased again. However, in five of the six cases the final leak rate measured at 23°C (f) was greater than what was measured initially (i). It is not yet known why leak rates at 23°C for the partially compressed seals typically increased after the temperature excursion.

The test conditions shown in Figure 12 represent worst case mission conditions where a seal would be exposed to AO and UV radiation prior to having to seal at the extremes of the operating temperature range while only partially compressed. Under these conditions, leak rates for the partially compressed seals made of the TiO<sub>2</sub> material were below the leakage threshold in all cases, whereas the leak rates measured for seals made of the baseline material were at or above the threshold in many cases.

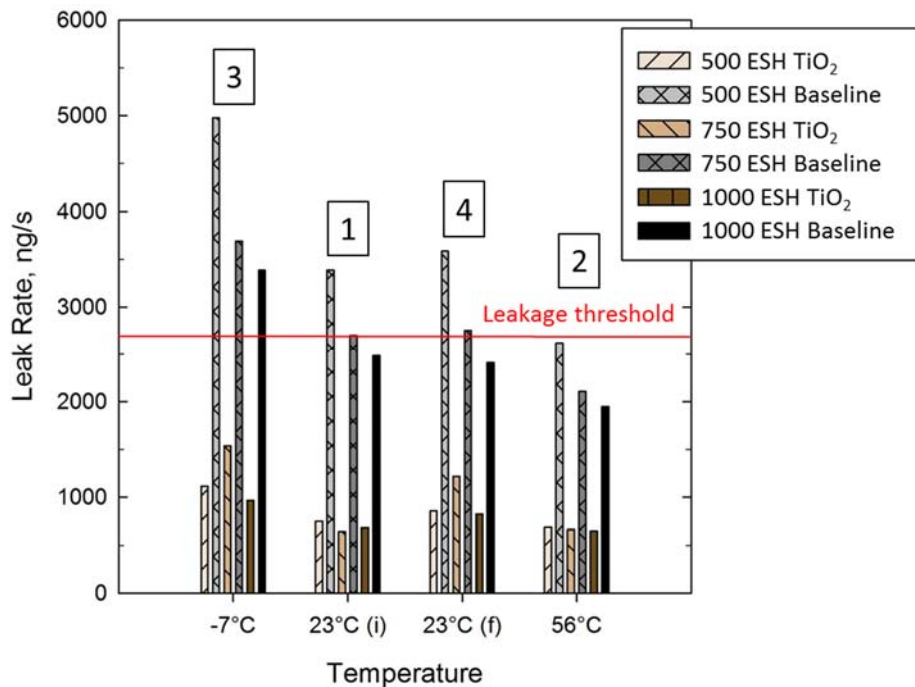


Figure 12. Leak rates at various temperatures for inner bulb of partially compressed (0.066 cm gap) seals made of baseline material and baseline material with TiO<sub>2</sub> after exposure to nominal AO fluence of  $8.8 \times 10^{19}$  atoms/cm<sup>2</sup> and 500, 750, or 1000 ESH of UV radiation.

Figure 13 compares the leak rates for the seal made of the baseline material that was exposed to 1250 ESH of UV radiation and the TiO<sub>2</sub> seal that was exposed to 2500 ESH of UV radiation for the same test conditions and

sequence shown in Figure 12. These two seals behaved similarly to those shown in Figure 12 with leak rates decreasing as the temperature increased. Both seals continued to exhibit similar leak rates, although the seal made of the baseline material that was exposed to 1250 ESH of UV radiation had lower leak rates for each test condition. At these worst-case conditions for partially compressed seals at the extremes of the operating temperature range, both seals exhibited leak rates above the leakage threshold for each test.

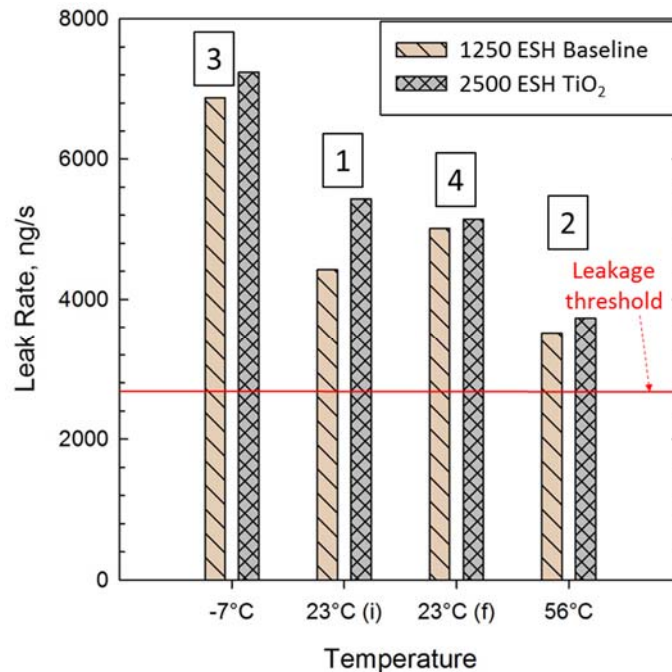


Figure 13. Leak rates at various temperatures for inner bulb of partially compressed (0.066 cm gap) seals made of baseline material after exposure to 1250 ESH of UV radiation and baseline material with TiO<sub>2</sub> after exposure to 2500 ESH of UV radiation. Both seals were exposed to nominal AO fluence of  $8.8 \times 10^{19}$  atoms/cm<sup>2</sup>.

## SUMMARY AND CONCLUSIONS

Seals for future space exploration missions must exhibit extremely low leak rates to ensure that astronauts have sufficient breathable air during extended-duration missions. In some applications, the seals may be left uncovered and exposed to the conditions in space for prolonged periods of time prior to mating. Because of this, there is interest in developing seals capable of withstanding longer exposures to space environments while still satisfying stringent leak rate requirements after mating. One approach to achieve better performance is to modify the baseline seal material by adding TiO<sub>2</sub> to improve resistance to damage from UV radiation. In this study, seals made of the baseline silicone material with and without TiO<sub>2</sub> additive were exposed to AO and increasing levels of UV radiation and then leak tested. The following findings were observed:

- Leak rates for seals exposed to AO and UV radiation decreased as leak tests progressed. Steady state leak rates for seals made of the TiO<sub>2</sub> material after exposure to a nominal AO fluence of  $8.8 \times 10^{19}$  atoms/cm<sup>2</sup> and up to 2500 ESH of UV radiation were lower after several days in a compressed state than they were early in the test. However, the decrease in leak rate occurred more gradually for the specimens exposed to 1772 or 2500 ESH of UV radiation. The amount of the decrease was less for these seals (30-40%) than for those exposed to 1000 ESH of UV radiation or less (40-65%). Overall, this behavior may be beneficial for long-term sealing applications as the seals seem to “recover” over time from damage caused by exposure to AO and UV radiation. However, the amount of recovery appears to diminish after the seals are subjected to longer UV radiation exposures.
- Leak rates generally decreased as the test temperature increased for both fully and partially compressed seals exposed to AO and UV radiation. This is the opposite effect of what occurs for unexposed seals where leak rates generally increase as the temperature increases.

- Leak rates for seals made of the baseline material were below the leakage threshold when fully compressed at 23°C and at the extremes of the operating temperature range (-7 and 56°C) after exposure to a nominal AO fluence of  $8.8 \times 10^{19}$  atoms/cm<sup>2</sup> and up to 1000 ESH of UV radiation. However, when those seals were tested in a partially compressed state (0.066 cm gap), they exhibited leak rates above the leakage threshold after UV radiation exposures as low as 500 ESH. Leak rates for a fully compressed seal made of the baseline material exceeded the leakage threshold at -7 and 23°C after exposure to 1250 ESH of UV radiation.
- Leak rates for a seal made of the TiO<sub>2</sub> material after exposure to a nominal AO fluence of  $8.8 \times 10^{19}$  atoms/cm<sup>2</sup> and 1000 ESH of UV radiation were below the leakage threshold for all test conditions that were evaluated in this study. This included anticipated worst-case conditions of partial compression (0.066 cm gap) at the extremes of the operating temperature range. However, leak rates for a similar seal exposed to AO and 2500 ESH of UV radiation exceeded the leakage threshold for most test conditions. Tests were also performed on a seal made of the TiO<sub>2</sub> material after exposure to 1772 ESH of UV radiation to investigate seal performance between 1000 and 2500 ESH. During long duration leak testing of the fully compressed seal at 23°C, leak rates were slightly above the leakage threshold at the beginning of the test but decreased to below the threshold by the end of the test indicating that the maximum UV radiation exposure that the seal can withstand while still satisfying the leak rate requirement may be in that range.
- Leak rates were similar for a seal made of the baseline compound after 1250 ESH of UV radiation exposure and a seal made of the TiO<sub>2</sub> material after exposure to 2500 ESH of UV radiation reinforcing the idea that addition of TiO<sub>2</sub> to the baseline compound provided protection to the seals from damage due to UV radiation exposure.

Based on the results of these tests, seals made of the baseline S0383-70 silicone material with TiO<sub>2</sub> additive show promise of being able to withstand increased exposure to AO and UV radiation for future seal applications beyond LEO. Test results presented herein indicate that seals made of the TiO<sub>2</sub> material could withstand 1000 ESH of UV radiation exposure and still satisfy leak rate requirements even under worst-case conditions of partial compression at the extremes of the operating temperature range. Tests performed after longer durations of UV radiation exposure revealed that seal leak rates were above the leakage threshold after exposure to 2500 ESH of UV radiation but may still be acceptable after exposure to 1772 ESH of UV radiation based on initial findings.

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