

# Temperature and Atomic Oxygen Effects on Helium Leak Rates of a Candidate Main Interface Seal

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### Abstract

Helium leak tests were completed to characterize the leak rate of a 54 in. diameter composite space docking seal design in support of the National Aeronautics and Space Administration's (NASA's) Low Impact Docking System (LIDS). The evaluated seal design was a candidate for the main interface seal on the LIDS, which would be compressed between two vehicles, while docked, to prevent the escape of breathable air from the vehicles and into the vacuum of space. Leak tests completed at nominal temperatures of -30, 20, and 50 °C on untreated and atomic oxygen (AO) exposed test samples were examined to determine the influence of both test temperature and AO exposure on the performance of the composite seal assembly. Results obtained for untreated seal samples showed leak rates which increased with increased test temperature. This general trend was not observed in tests of the AO exposed specimens. Initial examination of collected test data suggested that AO exposure resulted in higher helium leak rates, however, further analysis showed that the differences observed in the 20 and 50 °C tests between the untreated and AO exposed samples were within the experimental error of the test method. Lack of discernable trends in the test data prevented concrete conclusions about the effects of test temperature and AO exposure on helium leak rates of the candidate seal design from being drawn. To facilitate a comparison of the current test data with results from previous leak tests using air as the test fluid, helium leak rates were converted to air leak rates using standard conversion factors for viscous and molecular flow. Flow rates calculated using the viscous flow conversion factor were significantly higher than the experimental air leakage values, whereas values calculated using the molecular flow conversion factor were significantly lower than the experimentally obtained air leak rates. The difference in these sets of converted flow rates and their deviation from the experimentally obtained air leak rate data suggest that neither conversion factor can be used alone to accurately convert helium leak rates to equivalent air leak rates for the test seals evaluated in this study; other leak phenomena, including permeation, must also be considered.

# Nomenclature

 $t_0$  time constant

#### I. Introduction

To successfully accomplish the Constellation Mission, the National Aeronautics and Space Administration (NASA) required a new docking system with advanced capabilities. The Low Impact Docking System (LIDS) was designed to dock with a variety of manned and autonomous vehicles with minimal forces imparted upon either vehicle. The system was intended to be lighter weight, and to reduce the risks associated with docking.

The docking system required a seal at the interface between the two vehicles to maintain the gas pressure inside the adjoining vehicles and minimize the leakage of breathable air from the cabin to the vacuum of space. This seal, referred to as the main interface seal, was 54 in. in diameter and was designed to withstand the harsh environments of Low Earth Orbit (LEO) and outer space. The operating environment of the docking system was expected to subject the main interface seal to temperatures between -75 and 125 °C. The seal was designed to function mechanically (i.e., be compressed) at temperatures between -50 and 75 °C. The requirements of the docking system stipulated that the seal maintain low leak rates at temperatures of 4 to 40 °C.

Considering the docking system's exposure temperature range, silicone elastomers were chosen as seal system candidates based upon prior successful use in space applications, a capability to survive exposure to harsh

temperatures, and an ability to be formed into seals. Select silicone elastomers could also meet other program requirements, including being low outgassing (Ref. 1).

One candidate seal design consisted of a composite of a metal retainer and four elastomer seal bulbs that were vacuum molded into the retainer. The metal retainer provided a means to fasten the one piece assembly to the docking system, and limited the compression of the elastomer during docking. Additionally, the retainer minimized the ability of the elastomer to be removed undesirably during undocking (Ref. 2).

Although silicone elastomers offer a wide operating temperature range, they are more permeable to gases than many other elastomer compounds. Additionally, the permeation rate of silicone elastomers varies with temperature and gas type (Ref. 3). This combination of characteristics necessitates that the leak rate behavior of silicone elastomers be thoroughly investigated prior to application on flight vehicles.

Elastomers tend to adhere to contact surfaces (Ref. 4). Adhesion levels depend upon many factors, including the elastomer compound, temperature, amount and duration of contact, amongst others. Adhesion mitigation techniques include lubrication and controlling the temperature of the interface during separation. Another effective technique for minimizing adhesion is the pretreatment of the seal's elastomer surfaces with atomic oxygen (AO). Studies have shown that moderate exposure to AO can reduce or eliminate surface adhesion (Ref. 5). A minor drawback to the AO pretreatment technique is a modest increase in the leak rate of the seal (Ref. 6).

Previous investigations to characterize leak rates have been conducted on seals of similar designs using dry air as the test gas (Ref. 7). While useful in understanding the behavior in application, dry air is rarely utilized during qualification, acceptance, and verification testing processes. Tracer gases (usually helium) are used because of their small size and tendency to readily permeate through porous media. Using proper techniques and procedures, tracer gas methods can accurately predict very small leak rates.

In this work, a helium leak detector was used to quantify the leak rate of four 54 in. diameter candidate composite seal assembly test specimens. A full factorial matrix of tests was conducted using test temperatures of -30, 20, and 50 °C on test specimens with and without AO surface pretreatment. The effects of test temperature and surface pretreatment on the seal leak rates, as well as a comparison of air leak rates to helium leak rates are discussed.

#### **II.** Description of Experiments

#### A. Test Specimens

Each test specimen consisted of four individual elastomer seals vacuum molded into a metal retainer, as shown in Figure 1. There were two back seals and two front seals. The two front seals had identical cross-sections, and the two back seals had identical cross-sections, though the front and back seals were different. The dimensions of the seals are proprietary and therefore are not reported. The front and back seals closest to the inner diameter are referred to as the primary seals, and the front and back seals closest to the outer diameter are referred to as the primary seals, and the front and back seals closest to the outer diameter are referred to as the redundant seals. The seal assemblies were manufactured per a design referred to as EDU54 using Parker-Hannifin S0383-70 silicone elastomer compound. The metal retainer was manufactured from aluminum 7075-T651 with a chemical conversion coating applied per Mil-C-5541 (Ref. 8) to minimize corrosion. The test specimen's silicone elastomer met mission outgas requirements of total mass loss (TML) less than 1.0% and collected volatile condensable materials (CVCM) less than 0.1% when tested per ASTM E595 (Ref. 9).

The specimens were tested in one of two conditions. Two specimens were tested as received from the manufacturer, without surface pretreatment. These specimens' sealing surfaces were cleaned prior to testing with

lint free wipes moistened with isopropyl alcohol to remove any accumulated debris, and then allowed to dry for a minimum of 15 min. Two additional test specimens were pretreated with an AO fluence of approximately 10<sup>20</sup> atoms/cm<sup>2</sup>. These AO exposures were conducted at NASA GRC according to the ASTM E 2089 specification (Ref. 10). Prior to pretreatment, these specimens were cleaned as described above. After pretreatment, cleaning was limited to lightly blowing the sealing surfaces with canned compressed air to remove any accumulated debris.



Figure 1.—Representative illustration of a composite seal assembly.



Figure 2.—Photograph of non-actuated full-scale flow fixture.

#### B. Leak Test System

Helium leak rates of the candidate main interface seal specimens were determined using the non-actuated fullscale flow fixture, as shown in Figure 2 (Ref. 11). The flow fixture was comprised of two primary assemblies, an upper assembly and a lower assembly, which were nearly identical. The foundation of each assembly was a 2 in. thick aluminum "strongback" plate to which the other components of the assembly were attached with mechanical fasteners. These components, moving from the strong back to the test interface, included a phenolic thermal insulator plate, a heat exchanger plate, and a seal cartridge holder. Those components were identical on both the upper and lower assemblies.

The test seal assembly was attached to the lower seal cartridge holder with 72 fasteners. These fasteners applied the preload required to fully compress the test sample's back seals against the anodized surface (32 µin. surface finish) of the seal cartridge holder, resulting in metal-to-metal contact between the test seal retainer and the seal cartridge holder. During testing, the test specimen's front seals mated against an anodized aluminum mating counter-face plate (7075-T6 Al, 16 µin. surface finish) that was fastened to the upper seal cartridge holder. To minimize leakage between the mating counter-face plate and the upper seal cartridge holder, two silicone O-rings were installed in grooves in the back side of the mating counter-face plate. The upper assembly was then positioned over the lower assembly such that the test seal aligned and mated with the mating counter-face plate. Twenty-four external fasteners were installed through the two halves of the flow fixture. The fasteners were then tightened to simulate latching loads that would be generated across the sealing interface by the LIDS on orbit. The assembled flow fixture isolated the test section from the ambient environment with the inner and outer isolation o-rings, as shown in Figure 3. The test section encompassed the test specimen, mating counter-face plate, and the outer annulus.

Once the flow fixture was assembled, the outer annulus was evacuated through a vacuum port. This was accomplished with a Varian Model 979 series Helium Mass Spectrometer leak detector, with a manufacturer stated accuracy of 2%, and a Varian Triscroll 600 rough vacuum pump. During testing, the rough pump was isolated from the test section, so that none of the test gas was diverted from the leak detector.

A helium leak standard was used to calibrate the helium leak detector at the beginning or end of each leak test, depending on the test. The leak standard was either a VIC Leak Detection glass orifice, with an uncertainty of  $\pm 10\%$ , or a LACO Technologies Micro-tube capillary orifice, with an accuracy of  $\pm 3.8\%$ . Both had a leak rate in the range of the test specimens and were mounted to the non-actuated full-scale flow fixture at a port opposite the vacuum port. Helium from the leak standard circulated around the outer annulus and not through the test specimen. During the calibration process, the time constant,  $t_0$ , which was the time required for the helium leak detector to indicate a steady-state leak was determined.



Figure 3.—Illustration of assembled test section with magnification of test specimen and mating counter-face.



Figure 4.—Illustration of helium flow path.

During testing, high purity helium (99.997%) was supplied between the inner and outer test specimen seals at 0.2 psig, as shown in Figure 4. With a constant helium supply, helium permeated through both the primary and redundant seals, but only the helium that passed through the redundant seals to the outer annulus was measured. The duration of time that helium was supplied to the test specimen included the time required for the test specimen leak rate to reach steady-state and three time constants.

#### C. Thermal Control System

The temperature of the test specimens was controlled using a Mydax 2VLH30W Chiller/Heater unit, which circulated Dow Syltherm HF heat transfer fluid through passages in heat exchanger plates attached to the backside of the upper and lower seal cartridge holders (Fig. 2). The temperature control unit maintained the test section temperature to  $\pm 0.5$  °C. The test section temperature was monitored with 24 evenly spaced circumferentially mounted type-T thermocouples (Fig. 2), distributed equally on the upper and lower seal cartridge holders. Each thermocouple had an accuracy of  $\pm 0.036$  °C.

#### **D.** Data Acquisition System

A PC based data acquisition system was used to display and record the test data. National Instruments hardware and a custom written LabVIEW (National Instruments) code collected the interbulb pressure, the outer annulus vacuum pressure, the test section temperatures, and the helium leak rate. Unfiltered data was sampled at 25 Hz and recorded every 10 sec with each recorded value being an average of 25 sampled data points.

#### **III.** Experimental Results and Discussion

As shown in Figure 5, the leak detector data from a typical leak test contained five distinct regions. Regions I and II represented the data from the leak standard. Region I represented the time it took the leak detector to indicate a steady-state leak rate,  $t_0$ . During the analysis of the test data,  $t_0$  was used in the determination of the test specimen's leak rates. Region II represented the steady-state leak rate of the leak standard. Region III represented the system clean-up where the helium introduced by the leak standard into the test section, and subsequently the leak detector, was removed from the system. The next regions of the graph characterized the performance of the test specimen. Region IV represented the rise to steady-state, and Region V showed the steady-state leak rate of the test specimen.

For each test, the helium leak rate for the test specimen was calculated from the averaged steady-state measurements in Region V. The results are summarized in Table 1. The reported leak rates represented the arithmetic average of two tests conducted at each temperature for both the untreated and AO exposed test specimens. These leak rates were normalized per unit length based on the circumferential length of the redundant seal bulb centerline. This allowed comparison between the tests conducted with helium, which measured the leak rate of the redundant seal bulbs, and previously reported tests conducted with air (Ref. 7), which measured the leak rate of the primary seal bulbs. The experimental error for the results shown in Table 1 was  $\pm 33\%$  of the reading.



**Time, hrs** Figure 5.—Typical leak rate test data.

Temperature, °C	Helium Leak Rate,					
	atm-cc/s-in.					
	Untreated	AO exposed				
-30	2.09×10 <sup>-6</sup>	$1.24 \times 10^{-5}$				
20	$5.91 \times 10^{-6}$	6.63×10 <sup>-6</sup>				
50	$1.33 \times 10^{-5}$	$1.51 \times 10^{-5}$				

TABLE 1.—HELIUM LEAK RATE RESULTS

#### A. Temperature Effects on Leak Rate

The helium leak rates for the test specimens were measured at three test temperatures: -30, 20, and 50 °C. The leak rates for the untreated test specimens increased as the temperature increased, as shown in Figure 6. This result followed the trend of previously reported air leak rates for the same test specimens at the same test conditions (Ref. 7). The temperature effect on the test specimens exposed to AO was not as straight forward. As shown in Figure 6 the leak rate increased as the temperature increased from 20 to 50 °C, however, the leak rate also appeared to have increased as the temperature decreased from 20 to -30 °C. This did not correspond to the trend observed for the air leak rate results; the air leak rate results showed an increase in leak rate with temperature similar to the leak rates of the untreated test specimens. A contributing factor to the differences could be the associated test equipment. It has been shown that measurement error can occur when the nontemperature compensated leak detector was used for test conditions other than room temperature (Ref. 12). Although this was not apparent in the untreated seal test results, it is a factor that requires further investigation.



Figure 6.—Comparison of untreated and AO exposed test specimens.

#### B. AO Exposure Effects on the Leak Rate

When the leak rates of the untreated test specimens were compared to the leak rates of AO exposed test specimens, the leak rates for the AO exposed specimens appeared greater than the leak rates of the untreated specimens, as shown in Figure 6. However, this observed increase for the 20 and 50 °C tests was within the experimental error of the tests and the increase was not considered significant. At -30 °C, the average leak rate of the AO exposed test specimens was approximately 5.9 times greater than that of the untreated specimens.

#### C. Comparison of Helium Leak Rates to Air Leak Rates

To compare the helium leak rates reported here with the previously reported air leak rates (Ref. 7), differences in the type of measurement (volume versus mass) and the type of gas (helium versus air) needed to be addressed. The conversion from a volumetric leak rate to a mass leak rate was accomplished using the density of the gas. However, the conversion from helium to air was not straight forward. Conversion factors typically used in industry for converting helium leak rates to air are 1.08 and 0.374 for viscous flow and molecular flow respectively. The viscous flow conversion factor is the ratio of the dynamic viscosity of the two gases, and the molecular flow conversion factor is the ratio of the average molecular velocity of the two gases (Ref. 13). In both the viscous and molecular flow cases, the conversion factors are based on flow through an orifice of finite dimensions, however, based on previous work, it was shown that the flow through these test specimens was dominated by permeation (Ref. 6). Permeation does not rely on physical defects in the material to provide a leak path as required for viscous and molecular flow leaks. Therefore, applying viscous or molecular flow conversion factors to permeation dominated leak rates can produce erroneous results.

As an example, the helium leak rates reported for the untreated test specimens were converted to equivalent air leak rates using both the viscous flow and molecular flow conversion factors. The resulting converted air leak rates were then compared to the previously reported experimental air leak rates for the same test specimens at the same temperature and test conditions. Using a NIST traceable leak standard, the pressure decay mass loss technique has been shown to have less uncertainty associated with the results when compared to the helium leak detector method (Ref. 12). Therefore, the experimentally determined air leak rates were considered the most accurate. A comparison of the converted air leak rates and the experimental air leak rates is summarized in Table 2. As shown in Table 2, when viscous flow was assumed, the converted air leak rates were 35 to 73% greater than the experimental air leak rates, however, when molecular flow was assumed, the converted air leak rates were 40 to 53% less than the

Temperature,	Helium leak	Converted air leak rate		Experimental air	Experimental to	
°C	rate			leak rate (Ref. 7)	converted leak rate ratio	
	atm-cc/s-in.	Viscous flow	Molecular flow	lbm/s-in.	Viscous	Molecular
		lbm/s-in.	lbm/s-in.		flow	flow
-30	2.09×10 <sup>-6</sup>	$7.24 \times 10^{-12}$	$2.51 \times 10^{-12}$	5.15×10 <sup>-12</sup>	0.71	2.05
20	5.91×10 <sup>-6</sup>	$1.70 \times 10^{-11}$	$5.88 \times 10^{-12}$	$1.26 \times 10^{-11}$	0.74	2.14
50	$1.33 \times 10^{-5}$	$3.47 \times 10^{-11}$	$1.20 \times 10^{-11}$	$2.00 \times 10^{-11}$	0.57	1.67

TABLE 2.—COMPARISON OF HELIUM AND AIR LEAK RATES

experimental air leak rates. In both cases, when the helium leak rates were converted to air leak rates, the results were significantly different than the experimental air leak rate values. This example highlights the importance of understanding the type of flow and test conditions when leak testing with helium and then converting the results to leak rates for other gases.

### IV. Summary

A near-full-scale candidate composite seal assembly to be used as the main interface seal of the LIDS has been helium leak tested. From the experimental results, the following observations were noted:

- 1. The helium leak rate for the untreated test specimens increased as the temperature increased over the temperature range of -30 to 50 °C, however, the helium leak rate for the AO exposed test specimens decreased over the temperature range of -30 to 20 °C then increased as the temperature increased over the temperature range of 20 to 50 °C. The inconsistent trend requires further investigation.
- 2. There was an apparent increase in the helium leak rate of test specimens exposed to AO when compared to untreated test specimens over the temperature range of 20 to 50 °C. However, the differences were within the experimental error of the tests and the increase was not deemed significant. There was a significant increase in the helium leak rate of test specimens exposed to AO when compared to untreated test specimens at -30 °C.
- 3. Using standard conversion factors for viscous or molecular flow can produce inaccurate results when applied to this study's test specimens. The test specimen seal leakage was dominated by permeation, which exhibits different flow behavior than either viscous or molecular flow.

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