EFFECTS OF ATOMIC OXYGEN ON OUTGASSING OF SILICONE MATERIALS

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ABSTRACT

Effects of Atomic Oxygen on Outgassing of Silicone Materials

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An important consideration for spacecraft material selection is the space environment that the spacecraft will be operating in. Two features of the space environment that drive material selection are material outgassing and the presence of atomic oxygen in low Earth orbit (LEO). Materials that are considered for use in space are tested to be able to understand how they’ll outgas on orbit and how they’ll respond to interactions with atomic oxygen. However, testing to understand how atomic oxygen interaction with a material will affect how the material will outgas is rare and not standardized. This thesis used a vacuum chamber intended to determine the outgassing properties of materials using ASTM E595 and another vacuum chamber intended to determine how materials are affected by atomic oxygen using ASTM E2089 to determine how atomic oxygen affects outgassing of silicones, which are of interest as atomic oxygen can alter the chemical composition of the surface of silicones. CV-2500, CV2-2289-1, and SCV2-2590, three silicone elastomers that are products of NuSil Technology LLC, were tested. Significant trends in atomic oxygen reducing the amount of matter outgassed from these three materials were observed. This can be explained by the conversion of the surface of silicone to silica, which was confirmed using Fourier Transform Infrared (FTIR) spectroscopy. Retesting of these three materials in a chamber designed for ASTM E595 with a temperature measurement system capable of adhering to ASTM E595 to confirm the results of this thesis with more confidence in uniform temperature exposure is recommended.
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Chapter 1

INTRODUCTION

1.1 The Space Environment

The environment beyond Earth’s atmosphere has a myriad of effects that need to be understood so a system can reliably satisfy its mission requirements. While space is often considered a vacuum, it has many constituents such as photons, particle radiation, meteoroids, neutral and ionized molecular and atomic interactions, variable thermal environments, and orbital debris that can adversely affect spacecraft [42]. A better understanding of the interactions of the space environment with spacecraft subsystems contributes to the steady progress in spacecraft performance and reliability. Out of the many aspects of the space environment, two are considered in this work.

1.1.1 Outgassing

The lack of pressure in space compared to the pressure on Earth at sea level can result in gases that were withheld in a material due to the pressure on Earth to be released over time once the material is in orbit through diffusion, where atoms and molecules will move from regions of high concentration to regions of low concentration [42, 14, 24]. This matter that diffuses will migrate towards the surface of the material that is in contact with the vacuum of space and can desorb from the material. This is known as material outgassing, and the released material is considered a contaminant [42, 23, 11]. The outgassing of a material can result in particulates depositing on a surface of a spacecraft, and absorption (the contaminant assimilates into the surface)
or adsorption (the contaminant adheres to the surface) may occur [42, 55]. This is known as spacecraft contamination, and surface properties of contaminated surfaces may change in a manner that adversely affects the spacecraft mission [42, 41, 6].

Thermal properties such as absorptance can be altered, and contamination of a thermal control system could result in a space system being unable to maintain its proper temperature [42, 11]. Contamination can change optical properties such as transmittance and reflectance, and contamination of an optical system could result in poor imaging [42, 6, 11]. Even contamination in the vicinity of optical systems can cause scattering and distortion of images. Contamination from outgassed materials that are deposited on the exterior of a spacecraft can result in unpredictable interactions with the system exterior and the space environment, such as reactions between plasma present in the ionosphere and the contaminant [42]. Contaminants can even create a localized increase in pressure and induce arcing between two surfaces of a spacecraft.

There is a positive exponential relationship between the amount that a material outgasses and the temperature of the material, and contaminants are more likely to condense on cold surfaces such as optics or thermal control surfaces that are often at lower temperatures [29, 24, 19]. The national aeronautics and space administration (NASA) maintains a database of outgassing properties of materials that have been considered for aerospace use [10, 42]. These materials are divided into eighteen categories depending on their primary use, such as adhesives, coatings, tapes, etc.

1.1.2 Atomic Oxygen

Ultraviolet (UV) radiation from the sun dissociates diatomic oxygen found in the low-density atmosphere of LEO into atomic oxygen (AO) [41, 42]. Photons with wavelengths less than 243nm have energy greater than the dissociation energy of
diatomic oxygen (5.12 eV). The mean free path (approximately 10^8 m) makes the re-association of atomic oxygen into diatomic oxygen or ozone unlikely [41, 24]. Figure 1.1 shows that of all of the molecules and atoms that the LEO atmosphere is composed of, atomic oxygen is the most prevalent. The density of atomic oxygen is dependent on the solar cycle (figure 1.2), with increased UV radiation during solar maxima causing higher atomic oxygen densities in LEO and decreased UV radiation during solar minima causing lower atomic oxygen densities in LEO [41, 14]. Atomic oxygen density also varies by altitude, and mostly resides in lower LEO orbits as can be seen in figure 1.1. Solar heating of Earth’s atmosphere from UV radiation combined with the rotation of the thermosphere with Earth results in daily maximum atomic oxygen number densities occurring in the late afternoon at a given longitude. This results in anti-solar facing surfaces such as the back sides of solar arrays to receive more atomic oxygen per area (fluence) than solar facing surfaces.

Figure 1.1: Density of Atmospheric Species as a Function of Altitude [41]

Atomic oxygen is highly reactive, and the combination of AO’s high reactivity as well as the impact energy associated with AO colliding with a material will cause oxidative erosion of the material [42, 41]. The unfilled valence shell of oxygen allows it to form bonds with atoms on the surface of polymers and many metals, resulting in surface oxidation. Polymers are especially susceptible to atomic oxygen erosion. Atomic
oxygen reactions with polymers can result in the oxygen abstracting hydrogen from the polymer and forming H2O or OH. Oxygen addition or insertion may also occur where the oxygen bonds with the molecule and removes other molecules in the process or forms unstable radicals that can be removed in further reactions[17]. All of these reactions will result in volatile contaminants in the form of oxidation products with continued atomic oxygen exposure, and the polymer will erode, losing hydrocarbon or halocarbon content. This erosion of polymers will reduce their useful lifetime on orbit.

The impact energy associated with atomic oxygen colliding with a spacecraft surface is dependant on the magnitudes and directions of the orbital spacecraft velocity, Earth’s atmospheric co-rotation velocity, and the thermal velocity of the oxygen atoms themselves. For example, the average impact energy for spacecraft in circular orbits at a 400km altitude with an inclination of 28.5° and thermal velocities associated with a temperature of 1000K at LEO is 4.5 ±1eV[41]. Figure 1.3 shows how atomic oxygen impact energy for this orbit varies by altitude. It can be observed that impact energy increases slightly at lower altitudes, due to the increase in spacecraft velocity at lower orbits.
For spacecraft with elliptical orbits, the increased velocity of the spacecraft at its periapsis will result in greater impact energies upon collision with atomic oxygen. Figure 1.4 provides a reference for the maximum atomic oxygen impact energy that a spacecraft will experience depending on the eccentricity of the spacecraft. For more elliptical orbits, it can be observed that the impact energy increases. To see how these impact energies may affect polymers, table 1.1 provides a list of common bonds in polymers and the dissociation energy required to break their bonds. C, H, Cl, and F are carbon, hydrogen, chlorine, and fluorine, respectively, and - and = represent single and double bonds between atoms. These values will vary depending on the molecule that the bonds are a part of, such as if a hydrogen with a single bond with a carbon (C-H) is a part of a Methyl (CH3) or an Ethyl (CH2CH3).
Due to the high velocity of spacecraft in LEO (7 to 9 km/sec, depending on the eccentricity of the orbit), most atomic oxygen will collide with the surfaces of the spacecraft whose normal vectors point in the spacecraft’s direction of travel (the ram surfaces). If the spacecraft has zero inclination, the average atomic oxygen angle of attack is perpendicular to ram direction, but inclined orbits result in a sinusoidal variation of the angle of attack as the spacecraft travels in its orbit. The thermal velocities of atomic oxygen follows a Maxwell-Boltzman distribution, and a small fraction of the atoms with higher velocities at the end of the distribution are capable of traveling faster than a spacecraft and colliding into the trailing surfaces [41]. Figure 1.5 shows a polar plot of the relative atomic oxygen flux on a spacecraft compared to the ram direction, where $0^\circ$ is the ram direction. The flux of atomic oxygen the
ram surfaces receive is several orders of magnitude higher than the flux the trailing surfaces receive.

![Polar Plot of Atomic Oxygen Flux](image)

**Figure 1.5: Polar Plot of Atomic Oxygen Flux Relative to Normal of Spacecraft Arrival Surface for a 400-km Orbit at 28.5° Inclination and 1000-K Thermosphere [41]**

Polymers on a spacecraft will eventually develop cones that point in the ram direction where the majority of the atomic oxygen arrives as they erode over time, as shown in figure 1.6. This evolution of peaks and valleys will increase the surface roughness of the polymer, and the change in texture from a smooth to more rough surface will increase the diffuse reflectance and decrease the transmittance of the polymer, which can result in undesired changes in optical and thermal properties. As the location that atomic oxygen arrives on a surface is random, the roughness of the surface will follow Poisson statistics and as the atomic oxygen fluence increases the surface roughness will increase as the square root of the fluence [41].

If texturing does not affect the purpose of the polymer, the thickness can be chosen such that the polymer will survive the duration the mission or spacecraft lifetime without eroding away to the point where the material can no longer fulfill its purpose.
Figure 1.6: Scanning Electron Microscope Images of Directed LEO Atomic Oxygen Textured Materials [41]

Often though materials that are susceptible to atomic oxygen erosion are coated with a thin film of another material that is resistant to atomic oxygen erosion, such as a metal, metal oxide, or fluoropolymer-filled metal oxide protective coating [42, 25, 6]. Thin films with thicknesses ranging from a few hundred angstroms to 100nm can be applied using sputter deposition or vapor deposition.

1.2 Silicones and the Space Environment

Years of analysis has shown that silicone materials are troublesome outgassing species, and several missions have suffered from outgassed products condensing on sensitive components [11, 16]. Commonly, small volatile siloxanes can be released from the parent silicone material and can condense on other surfaces [54, 55]. Hydrocarbons such as methyls and phenyls attached to the siloxane backbones of silicones can also be outgassed [54]. Factors that may affect the outgassing property of silicones include the cure history, operation temperature, and the degree of crosslinking between siloxane chains [40, 37, 16]. Research at NASA Goddard Research Center from 1970 to 1979 that involved testing an extensive list of materials revealed that methyl silicones accounted for 16% of outgassing issues during thermal vacuum testing and aromatic
silicones accounted for 5.7% of outgassing issues, with outgassing issues being defined
as outgassing more than ten milligrams of material weighing 1 gram[11]. The only
other compounds with more outgassing issues were Esters and aliphatic hydrocarbons.
Goddard spaceflight center has since developed the outgassing database previously
mentioned for the thousands of spacecraft materials they had tested, and search
options allow for search results to be categorized by specific material and values for
TML and CVCM. Searching through this database will show that as of the year 2022
nearly 1600 silicone materials have been tested. Out of these materials, approximately
60% of these silicones do not pass the screening level set by ASTM E595. Out of these
silicones with outgassing characteristics deemed higher than acceptable, 99% had a
CVCM greater than 0.1% while 30% had a TML greater than 1.0%.

While most polymers experience noticeable mass loss due to atomic oxygen erosion,
many silicones do not experience a significant change in mass [41, 24]. Atomic oxygen
interactions with silicones results in the volatizing of the surface hydrocarbon chains
and the formation of a silica (SiO2) based layer [41, 6, 25]. Infrared Spectroscopy
of polysiloxanes before and after atomic oxygen exposure have shown reduction in
hydrocarbons such as methyl groups and the replacements of oxygen [15]. Visually,
silicone rubbers exposed to atomic oxygen will appear glossier [16]. This silica surface
layer is resistant to atomic oxygen and can prevent further erosion of the silicone
[41, 23, 24]. Silica coatings are applied to polymers as a protective coating, and
studies have found that a protective coating of SiO2 as thick as 130nm can reduce the
rate of erosion of Kapton® H, a polymer commonly used for aerospace applications,
to less than 1% the rate of erosion of unprotected Kapton® H.
1.3 Motivation

While there has been extensive research and design considerations for the outgassing of silicones in spacecraft as well as their AO resistive properties, there have been few studies into how the combination of atomic oxygen exposure and the low pressure environment affects the mass loss of silicones and how much they outgas. How the change in surface chemistry of silicones affects the amount of volatile content released and how much the silicone outgasses after being exposed to atomic oxygen can reveal to designers if a material will contaminate more or less than determined from the outgassing database. Previous studies from Henry de Groh III et al. from NASA Glenn Research Center and Tyler Harty from California Polytechnic State University (Cal Poly) have observed a trend in reduction of the amount silicones outgas after being exposed to atomic oxygen to varying degrees depending on the type of silicone [24, 13].

With variability between silicones on the amount outgassed, specific silicone materials that have not been subjected to synergistic atomic oxygen and outgas testing should be subjected to such tests to see how drastically atomic oxygen affects their outgassing. NuSil Technology LLC, a company that specializes in low-outgassing silicones for space applications, donated three different silicone elastomers (CV-2500, CV2-2289-1, and SCV2-2590) to Cal Poly. These three materials were tested to further the study of synergistic effects between atomic oxygen and vacuum on silicones and to quantify how atomic oxygen exposure specifically affects the outgassing of these three silicones. CV2-2289-1 and SCV2-2590 are two silicones that have not been studied extensively, while CV-2500 is a widely used silicone that has been well studied in literature. [49]. CV-2500 can be considered a benchmark to compare with the results of the other two materials.
Chapter 2

BACKGROUND

2.1 Test Standards

The American Society for Testing and Materials (ASTM) has thousands of standards for materials, products, systems, and services. A few of these standardize how materials considered for use in space should be tested and how to determine if the material is acceptable for use.

2.1.1 Atomic Oxygen Test Standard

ASTM E2089 is a test standard for characterizing atomic oxygen erosion for materials [3, 41]. The standard is intended to minimize the variability of results for a single material within any specific atomic oxygen exposure facility rather than a specific type of atomic oxygen exposure apparatus, and can be used to highlight differences in material response when tested in different facilities.

Four standard witness materials (Kapton® H or HN, tetrafluoroethylene (TFE)-fluorocarbon fluorinated ethylene propylene (FEP), low density polyethylene (PE), and pyrolytic graphite (PG)) should be exposed to atomic oxygen in the atomic oxygen apparatus using the same operating conditions and duration of exposure within a factor of three as samples being tested. The atomic oxygen flux and fluence for Kapton® H or HN should be reported along with the mass or thickness loss relative to Kapton® H or HN of the material being tested, TFE-fluorocarbon FEP, PE, and PG. TFE-fluorocarbon FEP, PE, and PG do not need to be tested if previous data
for these three materials exists for the same exposure conditions and fluence within a factor of three of the sample materials being tested.

The test samples should be chemically representative of the material used in space, so chemicals, cleaning solutions, or washing and wiping procedures that would not be used to prepare the materials for space should be avoided to prevent altering the surface chemistry of the samples [3]. Samples should be handled such as to minimize contamination, abrasions, and flexure, such as using soft fluoropolymer tweezers and handling with latex or nitrile gloves.

Portions of samples may be wrapped in a metal foil, covered in glass, or sprayed in salt to limit the exposure of atomic oxygen to a specific area of the material. This is known as masking, and if samples are to be masked then it is recommended that the protective covering be in intimate contact with the samples so that no atomic oxygen can reach the masked region. Use of metal foils to mask samples may also create electromagnetic effects between the produced oxygen plasma and the foil and affect the flux of atomic oxygen; in these situations the four standard materials should be tested in the same masking configuration to determine the effect of the masking on the flux.

If the change in mass due to atomic oxygen exposure is being measured, then non-metal and nonceramic test samples, such as organic polymers, should be vacuum dehydrated to eliminate errors in mass measurement due to water or other volatiles stored within the material. Before the mass of the samples before atomic oxygen exposure is recorded, samples should be dehydrated in a vacuum with a pressure of less than 200 mTorr for 48 hours. If samples are thicker than 0.127mm, this dehydration procedure should be repeated multiple times and the mass of the samples should be recorded between dehydration periods until the mass measurements indicate that no further water vapor is being lost. As hydration occurs quickly after being removed
from vacuum, samples should be weighed within five minutes of being removed from vacuum. The scale needs to be sensitive enough to capture mass loss of the witness sample (Kapton® H or HN) after AO exposure, which for a fluence of at least $10^{19} \text{atoms cm}^{-2}$ is a readability of $\pm 1 \text{mg}$ [3].

To limit error due to spatial distribution of the AO plasma in the apparatus, test samples should be placed as close to the Kapton® H or HN samples as possible. After the samples have been exposed to atomic oxygen, the witness samples should be visually compared to witness samples from previous accepted tests to ensure that no contamination had occurred during the test. Test samples and witness samples should be weighed within five minutes of being removed from the atomic oxygen apparatus.

The effect of atomic oxygen on a material is determined by the erosion yield, which is the volume of a material that is eroded by atomic oxygen per incident oxygen atom. This quantity can be calculated by equation 2.1.

$$E_y = \frac{dm}{A \cdot \rho \cdot F_k}$$  \hspace{1cm} (2.1)

$E_y$ is the erosion yield, $A$ is the surface area of the test sample exposed to atomic oxygen, $F_k$ is the effective atomic oxygen fluence, $dm$ is the mass loss of the test sample, and $\rho$ is the density of the test sample. The effective atomic oxygen fluence is calculated using the Kapton® H or HN witness sample, with equation 2.2.
\[ F_k = \frac{dm_k}{A_k \times \rho_k \times E_k} \] (2.2)

\( dm_k, A_k, \) and \( \rho_k \) are the mass loss, exposed area, and density of the witness sample, and \( E_k \) is the erosion yield of Kapton\textsuperscript{®} H or HN, which has been determined to be \( 3 \times 10^{-24} \) and \( 2.81 \times 10^{-24} \text{cm}^3/\text{atom} \), respectively for LEO.

ASTM E2089 does not standardize a duration of time that samples are exposed to atomic oxygen, nor a fluence of atomic oxygen that samples should receive. It is therefore necessary to compare duration of exposure and atomic oxygen fluence when comparing results of samples exposed to atomic oxygen from different facilities.

### 2.1.2 Outgassing Test Standard

To reduce the negative outcomes of outgassing materials in spacecraft applications, the test standard ASTM E595 was developed and is used to determine if materials are approved for use in space [4, 42]. Samples being tested are exposed to \( 125 \pm 1 \degree C \) at less than \( 5 \times 10^{-5} \text{Torr} \) for 24 hours. Condensable matter outgassed from the samples will outgas onto a collector maintained at \( 25 \pm 1 \degree C \). The two parameters measured from this test are the total mass loss (TML) of the sample and the collected volatile condensable materials (CVCM), which are obtained from equations 2.3 and 2.4.

\[ TML = \frac{S_i - S_f}{S_i} \times 100 \] (2.3)

\[ CVCM = \frac{C_i - C_f}{S_i} \times 100 \] (2.4)
$S_i$ and $S_f$ are the initial and final mass of the sample respectively, while $C_i$ and $C_f$ are the initial and final mass of the collector. TML is the mass the sample outgassed as a percentage of the sample’s initial mass while CVCM is the percentage of the sample’s initial mass that condensed onto the collector. Historically, a TML of 1.00% and a CVCM of 0.10% have been used as recommendations for rejection of spacecraft materials, with materials that have values larger than this being considered too great of a contamination threat [4, 37, 13]. The test standard is mainly a screening technique for materials and is not a method to calculate actual contamination of a system or component of a system. Differences in configuration of outgassing sources and condensable surfaces, temperatures, and material processing may alter the amount outgassed from a material as well as the amount of contaminants condensed on a surface.

Test specimen are exposed to $23 \pm 2^\circ C$ and $50 \pm 5\%$ relative humidity for 24 hours before being tested. After this exposure, the samples and the collectors that the condensed material will gather on are weighed and placed into the apparatus, where they are exposed to the elevated temperature and low pressure for 24 hours. After this period is over, the chamber is vented with a dry, inert gas and the test specimen are weighed again along with the collectors. The percentage TML and CVCM are obtained from the mass measurements, and are typically reported as an average of the values from three samples of the same material. The precision, which is a guideline for the magnitude of variability that can be expected from a competent facility, of the test method is defined as standard deviation being $\pm 10\%$ the average TML value for TML, and $\pm 20\%$ the average CVCM value for CVCM [2, 4].

Before a test is performed, all portions of the microvolatile apparatus must be cleaned to prevent contamination from previous tests. The standard recommends using a solvent blend of acetone and ethanol in a 1:1 volume ratio for this purpose. Empty
specimen chambers in the apparatus can have collectors and may be used as controls to ensure that uniform cleaning procedures have been performed after each test.

As the 24 hour test duration does not represent actual outgassing from years of operation, the high temperature of 125°C is used as it is assumed to be above the operating temperature of materials used in spacecraft [4]. The high temperature and short time duration allows for material comparison with no intention of predicting actual outgassing when the material is in orbit.

2.2 Atomic Oxygen Apparatuses

One way to quantify the effects of atomic oxygen is to perform ground-based tests following ASTM E2089 where materials are exposed to atomic oxygen to better understand the phenomenon, and different types of facilities can be used to create atomic oxygen [3, 42]. Correlation between in-space degradation and ground-testing degradation needs to be determined for individual AO apparatuses as there are differences in atomic and molecular species, energies, thermal exposures, and radiation exposures that can result in different reactions and exposure rates [41]. It should be restated that all of these apparatuses described below, as different as they are, follow ASTM E2089 as described in the previous section.

Plasma ashers use RF energy to create an isotropic thermal energy plasma at around 0.01-0.1eV [22, 16]. This energy is much less than the energy of atomic oxygen in LEO (4.5eV), and the erosion of samples in a plasma asher does not result in the pits and cones that are present on the surface of materials in LEO as the atomic oxygen does not come from one direction. Samples in plasma ashers also receive an intense amount of UV radiation while a spacecraft in LEO will receive electron and proton radiation as well as broadband UV radiation, and solar flare x-rays. As
plasma ashers use air that is bled into the system to dissociate diatomic oxygen into AO, the created plasma that samples are exposed to is actually a blend of diatomic and mono-atomic nitrogen and oxygen in excited and ground energy states, while LEO primarily consists of ground-state atomic oxygen [5]. All of these differences can affect the erosion of material, and it is for this reason that ASTM E2089 uses the effective fluence of Kapton® H or HN calculated using the known erosion yield values from LEO exposures. This effective fluence is able to correlate how much atomic oxygen in LEO would have caused the erosion of the samples observed in the plasma asher.

Other methods to produce atomic oxygen include continuous or pulsed lasers. Lasers can use thermal heat to create a blast wave that dissociates diatomic oxygen. This method is capable of producing conditions similar to LEO with a high flux of atomic oxygen at 5eV [22]. The European Space Research and Technology Centre (ESTEC) in the Netherlands has a facility that simulates the effect of atomic oxygen on materials in LEO using the pulsed laser detonation method [39]. A CO2 laser with high pulse energy (7J) is used to create a hypersonic beam of neutral atomic oxygen atoms at 5eV with a flux of $10^{14} - 10^{15} \text{ atoms/cm}^2\text{sec}$. O2 that is pulsed into the chamber using a pulse valve is disassociated by the incoming CO2 laser detonation and is accelerated by the created blast wave.

Microwave electron cyclotron resonance can gyrate electrons at the same phase of an induced magnetic field to dissociate diatomic oxygen as it collides with electrons [22, 16]. An electron cyclotron resonance plasma source at NASA Lewis center is capable of dissociating, ionizing, and exciting O2 to create a beam where atomic oxygen accounts for 10% of the species in the plasma [7, 45]. The facility can be adjusted to produce either a directed beam with an average energy of 13eV at $9 \times 10^{14} \text{ ions/cm}^2\text{sec}$ or an isotropic distribution of oxygen at 0.04eV and $3 \times 10^{14} \text{ ions/cm}^2\text{sec}$ [50].
Gridded or gridless (also known as end Hall) ion sources can produce high energy atomic oxygen, but the flux of atomic oxygen is small as the primary constituent produced from these ion sources are diatomic oxygen ions [22]. The monoatomic oxygen produced from these beams are mainly positively or negatively ionic, and are neutralized by methods such as gas phase charge exchange, interactions with solid surfaces, or photo-detachment [9]. An end Hall ion source at NASA Glenn Research Center can emit hyperthermal energy atomic oxygen at 70eV, but as previously mentioned the beam largely consists of diatomic oxygen [8].

2.3 CVCM Apparatuses

The test method ASTM E595 is applicable for a specific type of microvolatile condensable system. These apparatuses typically have two resistance-heated copper bars, each with twelve compartments that samples can be stored in, and open sections in each compartment that allows outgassed matter to travel to collectors that are removable chromium-plated collector disks [4]. Power to the heating element in the copper bars is generally controlled by variable transformers, and the plate that the collectors reside on is typically maintained at 25°C via a heat exchanger using a suitable fluid. An example of such a system is shown in figure 2.1, which is an outgassing chamber from JPL used for ASTM E595 tests [24].

Other vacuum systems that measure outgassing and contamination of materials can characterize time and temperature dependence of material outgassing and deposition [1]. These systems follow a different test standard, ASTM E1559, which has separate temperature, time, and pressure requirements. The layout of such a system is shown in figure 2.2. Typically, samples are held in an effusion cell that is heated to an elevated temperature while the chamber is pumped down. Three or four quartz crystal
microbalances (QCMs) that are thermally isolated from each other and thermally grounded to a cryogenic heat sink collect volatile condensed matter (VCM) from the sample. Each QCM has two crystals, one for mass collection and one for reference, and all QCMs are individually controlled to be at different set temperatures, collecting different amounts of outgassed matter. A shroud, such as liquid nitrogen, is cooled by the cryogenic cooler and surrounds the effusion cell and QCMs to limit contaminant flux to the line of sight from the effusion cell to the QCMs.

After twelve hours of the sample outgassing and the QCMs collecting VCM, the temperatures of the QCMs are increased at a constant rate to determine the evaporation characteristics of the species in the deposit, and a residual gas analyzer (RGA) can be used to identify the released species. The total mass loss of the sample can be determined the same way as samples tested in apparatuses made for ASTM E595.
The VCM is calculated taking into account both the mass gain of each QCM and the view factor between each QCM and the sample.

2.4 FTIR Spectroscopy

Fourier transform infrared (FTIR) spectroscopy can be used to aid in determination of chemical composition of materials. An infrared photon of a specific frequency will change the dipole moment of a molecule that corresponds to a specific vibrational energy [28]. The vibrational energy is dependant on the reduced mass of a bond $\mu$ and the spring constant of a bond $k$. The wavenumber, which is the inverse of the wavelength, associated with the vibrational energy can be calculated as equation 2.5.

$$\tilde{v} = \frac{1}{2\pi} \sqrt{\frac{k}{\mu}} \quad (2.5)$$
Vibrational energies are unique to functional groups as functional groups are composed of unique bonds. An FTIR spectrometer will expose a sample and a detector to a broadband IR beam, typically in the mid-infrared range (4000-400cm\(^{-1}\)) but sometimes in the near-infrared (14000-4000cm\(^{-1}\)) and far-infrared (400-4cm\(^{-1}\)), depending on the spectrometer and/or settings [47]. The signal, which is in a time domain, is converted into a frequency domain via a Fast Fourier Transform. The recorded spectrum from this can be transmittance, reflectance, or absorbance by wavenumber. Vibrational energy will be shown as a peak in the spectrum at a wavenumber that can be used to identify functional groups present in the sample. There are many IR functional group tables available to identify functional groups based on their wavenumbers. Specific bonds that are not a functional group will have unique peak(s), though it is difficult to identify these bonds in an FTIR spectrum without prior knowledge of the chemical composition of the sample. FTIR libraries can be used to compare a collected spectrum to a database and peaks not associated with functional groups maybe identified in this manner if no prior knowledge of the chemical composition is known.

An advantage with the use of an absorbance spectrum is that absorbance is linearly related to concentration. Equation 2.6 is known as Beer’s Law.

\[
A = \epsilon lc
\]

\((2.6)\)

\(A\) is the absorbance, \(\epsilon\) is the molar absorbtivity, \(l\) is the pathlength of light, and \(c\) is the concentration. With this relation, the height or area of a peak in an absorbance spectrum is proportional to the concentration, which means multiple spectra can be compared to determine the difference in concentration of specific functional group or
bond, such as the difference in concentration of a functional group before and after atomic oxygen exposure.

2.5 Silicons in Spacecraft Technology

Silicone has been a material used on spacecraft as potting compounds, seals, gaskets, adhesives, hydrophobic surfaces and atomic oxygen protective coatings [6, 15, 37]. Generally, the material has a wide operating temperature range and a low leakage rate, which makes them ideal for seals [13, 40, 43]. Silicone elastomers have been investigated for seals between two modules of a spacecraft such as docking and habitat systems for space stations [13, 15]. Pretreated silicone exposed to Atomic oxygen can be used as a protective coating for components on a spacecraft [6, 25]. As an example, use of silicone as an atomic oxygen treatment coating could be applied to wire insulation for spacecraft [25].

Silicone adhesives (such as Sylgard 182/184 and RTV 511/560/580) used for solar cell adhesives have historically been one of the largest sources of condensable outgassed material for spacecraft, requiring multiple thermal bakeouts before the materials were ready for flight [11]. Thermal vacuum tests of entire spacecraft have also shown silicone as a primary outgassing material, with large amounts of outgassed residue from tests on spacecraft IMP-H being from methyl silicones from the solar cell adhesive and RTV-560 for both spacecraft RAE-B and NRL [11]. Incorporation of materials engineering practices emphasizing using low outgassing materials in flight hardware has resulted in the reduction of excessive outgassing in silicone materials [11, 37]. Techniques to reduce outgassing include reducing the amount of low weight molecular species in silicone compounds and having a filler of a different material within the silicone, resulting in less silicone to be outgassed [43]. A process known as post curing
involves removing volatiles from silicone via diffusion and evaporation while exposing the material to a temperature greater than the materials service temperature [43, 29]. The process of removing volatiles from silicone in a vacuum is also known as vacuum stripping [41]. Many elastomeric-type silicones achieve acceptable TML and CVCM values after baking material in a thermal vacuum chamber post-cure 400-480°F [20]. Pre-baking silicones will not completely remove all volatiles though [43, 54]. In fact, baking of silicones after they have been cured can even lower mechanical properties [37]. Degradation of the material at elevated temperatures will result in the continual outgassing of low molecular weight siloxanes [40]. Studies into the failure of systems using a silicone adhesive known to be a low-outgassing silicone revealed that while post cure removed volatiles from the silicone, thermal degradation that the adhesive experienced caused continuous outgassing of hydrogen, silanes, and hydrocarbons with molecular masses less than Pentane [54].

Even with the current outgassing requirements established by ASTM E595, any outgassing and contamination is an issue, and there are applications for which TML and CVCM values much lower than the values required by ASTM E595 may be desired. In response there have been silicones deemed as ultra-low outgassing silicone systems that have TML values of less than 0.1% and CVCM values of less than 0.01% [37, 16]. These systems are typically elastomers reinforced with resin or silica, and do not require the added process of baking in vacuum chambers to be used in space [37]. silica reinforced silicones generally have improved physical properties, but the silica contains volatile components that are readily outgassed [37]. Common fillers put in silica reinforced silicone to reduce the amount of silica and silicone is iron oxide, which aids in thermal stability, and micro balloons, which offsets the specific gravity of the material [37]
While the silica that forms on the surface of siloxanes when bombarded with atomic oxygen protects the underlying polymer from further erosion, the elastic modulus and tensile strain of the surface increases [6]. As a result, the silica tends to be under tension relative to the underlying silicone, and this can cause cracks to form on the surface [41, 6, 25]. Cracks can cause underlying silicone to be exposed and converted to silica, which can cause even more cracking [6]. In a test where silicone samples were exposed to atomic oxygen to determine the usefulness of the material as a wire insulator, it was found that silicone samples that were cured at high pressures formed a compressive stress on the surface, and when exposed atomic oxygen the samples developed a wavy surface texture rather than cracking, but more studies need to be conducted to confirm this [25].

Roughening the surface of silicone can also prevent cracking in an atomic oxygen environment [25]. A smooth silica surface will shrink relative to the underlying silicone material, causing it to pull apart and form cracks, but with a rough surface the shrinkage will only cause the valleys and peaks of the material to flatten out [25]. The ideal rough surface would have peak heights taller than the atomic oxygen penetration depth [25]. The roughness of polymer surfaces exposed to atomic oxygen following Poisson statistics as previously discussed could contribute to these peaks and valleys.

As previously mentioned, the aerospace industry has used this atomic oxygen-induced silica surface to create atomic oxygen resistant coatings [41]. A thin film coating of silica can be applied to a surface via sputter deposition or vapor deposition [41]. silica coatings around 100 nm thick can be used to prevent erosion of the underlying material, but coating thicknesses that are factors bigger can cause cracking and spall due to the difference between the flexure and compression or expansion of the coating and the underlying material [41]. Silica based protective coatings can become non-protective at locations of cracking [6]. This scenario occurred with silicone-coated
acrylic retroreflectors on the Long Duration Exposure Facility (LDEF) spacecraft as the retroreflectors were exposed to atomic oxygen [6]. Pretreating silicone with atomic oxygen can reduce the material’s adhesion, which could be desirable for applications such as seals that need to repeatably attach and detach from a surface without sticking [13, 15]. Moderate atomic oxygen exposure will not significantly degrade the sealing properties of the rubber, but care must be taken though as long term atomic oxygen exposure can increase the leakage rate of silicone seals [13, 15].

Silicone contamination that has been exposed to atomic oxygen can become dark in color with UV exposure [41, 6]. Oxidized silicone contaminants will get fixed to a surface and will not re-evaporate like non-oxidized silicone contaminants can [41, 55, 14]. This was observed on the LDEF experiment AO171 (figure 2.3), where silicone contaminants that landed on a portion of a sample holder exposed to atomic oxygen oxidized and formed a dark region, and silicone contaminants that landed on a portion of the sample holder not exposed to atomic oxygen re-evaporated, leaving a lighter colored region [41, 6].

Figure 2.3: Post-Flight Photograph of Solar Array Materials Passive LDEF Experiment AO171 [41]
A foldable solar panel array from the MIR space station that had silicone in the sutures that cladded the solar array structure together was brought back to Earth for analysis and had silicone contaminant from the sutures on the surface of the array [6, 41]. The volatile silicone had oxidized, and although the performance of the solar panel was not negatively affected, the solar reflectors and neighboring white thermal control surfaces had darkened because of the contamination [41]. Figure 2.4 shows a component of the retrieved solar array where tape had removed the contamination in the top right corner, providing visual evidence of how much the oxidized silicone had reduced the transparency of the array surface.

Figure 2.4: Oxidized Silicone Contamination on Solar Cell Components from a Mir Solar Array Retrieved After 10.4 Years in LEO [41]

Atomic Oxygen interaction with silicone contaminants can cause a reduction in the optical transmittance of the surface that the contaminants arrive on [6]. On an Israeli Ofseq-3 spacecraft silicone volatiles had landed on the sun sensors that were being bombarded with atomic oxygen [6]. The development of darkened silica contaminants on the sun sensors caused a 40-60% attenuation of solar radiation that the sun sensors received resulting in a 40-60% reduction in sun sensor output in less than 150 days [6]. Other properties of a surface material that can be affected by darkened silica are
an increase in diffuse reflectance and an increase in absorptance [6]. Occurrences such as the OffSeq-3 sun sensors and the MIR solar array highlight the importance of why testing atomic oxygen interaction with silicone outgassing is important.

CV-2500, CV2-2289-1, and SCV2-2590 are all silicone elastomers intended to be used as adhesives or sealants. CV-2500 is a two-part optically clear silicone system that has applications as an adhesive for parts of a spacecraft such as solar cell arrays where clarity is important [34]. It can also be used as a potting compound to protect electronic components or assemblies, and can provide protection from radiation, thermal stress, and mechanical stress. CV2-2289-1 is a two-part white silicone that can be used to protect electronic components from shock, vibration, and contaminants [35]. SCV2-2590 is another two-part clear silicone but is an ultra-low outgassing silicone (ultra-low outgassing being defined as having a TML less than 0.1% and a CVCM less than 0.01%) that can be used as a potting compound to protect electronic components that are especially sensitive to volatile contamination [36]. It is used as an adhesive between the cover glass and solar cell for the IMM4 solar cells from SolAero Technologies [21]. Table 2.1 has the TML and CVCM data provided by NuSil Technology LLC for these three materials [34, 35, 36].

Table 2.1: Reported TML and CVCM from NuSil Technology LLC [34, 35, 36]

<table>
<thead>
<tr>
<th>Silicone</th>
<th>TML</th>
<th>CVCM</th>
</tr>
</thead>
<tbody>
<tr>
<td>CV-2500</td>
<td>0.05%</td>
<td>0.01%</td>
</tr>
<tr>
<td>CV2-2289-1</td>
<td>0.31%</td>
<td>0.07%</td>
</tr>
<tr>
<td>SCV2-2590</td>
<td>0.05%</td>
<td>0.008%</td>
</tr>
</tbody>
</table>

2.6 Atomic Oxygen Effects on Outgassing of Silicones

Few studies have investigated how atomic oxygen interactions with silicones affect the outgassing of silicones. Two silicone elastomers (S0383-70 from Parker-Hannifin
Corporation and ELA-SA-401 from Esterline Corporation) underwent a series of tests at NASA Glenn Research Center to determine their viability as a seal for a docking module between the Crew Exploration Vehicle and the International Space Station [16]. One of the tests included determining how outgassing was affected by atomic oxygen [16].

All samples went under an initial outgassing test in accordance with ASTM E595 [16]. Samples that did not pass the standards had been determined to have been cured improperly during manufacturing [16]. Three O-rings of each material went through a four day bake under vacuum at $8 \times 10^{-6}$ Torr and 205$^\circ$C to get rid of volatiles, then treated with atomic oxygen at a fluence of $4.9 \times 10^{20}$ atoms/cm$^2$ to seal the surface of the O-rings with silica [16]. Another test was performed where O-rings were only exposed to atomic oxygen without the four-day vacuum bake [13].

The results of the four-day bakeout and atomic oxygen treatment is presented in table 2.2. The four-day bake out and atomic oxygen treatment had little effect on the S0383-70 O-rings (there was a slight increase in TML and CVCM), but the TML and CVCM of the ELA-SA-401 decreased by nearly half [16]. On the other hand, just exposing the O-rings to atomic oxygen without the four-day bake out (figure 2.5) resulted in a decrease in the TML and CVCM for S0383-70 and negligible change for ELA-SA-401 [16]. These results are shown in figure 2.5.

<table>
<thead>
<tr>
<th>Condition</th>
<th>Parker S0383-70</th>
<th>Esterline ELA-SA-401</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>TML,%</td>
<td>CVCM,%</td>
</tr>
<tr>
<td>As Received</td>
<td>0.105</td>
<td>0.047</td>
</tr>
<tr>
<td>Post-Treatment</td>
<td>0.118</td>
<td>0.074</td>
</tr>
</tbody>
</table>

IR Spectroscopy was used to examine the chemical composition of the O-rings before and after atomic oxygen exposure [15]. After comparing the spectra, it was observed...
that there was a decrease in Si-CH3 bonds after atomic oxygen exposure and little decrease in (Si-O-Si). It was concluded that as Si-O-Si peaks in the spectrum increased relative to the Si-CH3 peaks, methyl groups were being replaced by oxygen. Figure 2.6 shows the IR spectrum, with the peaks at 1260cm\(^{-1}\), 769cm\(^{-1}\), and 860cm\(^{-1}\) indicative of Si-CH3 and the peaks at 1070cm\(^{-1}\) and 1020cm\(^{-1}\) indicative of Si-O-Si.

Research at Cal Poly has been done to better understand the synergistic effects between outgassing and atomic oxygen with silicone materials. In their work to validate the CVCM chamber and the atomic oxygen chamber at the Space Environments Laboratory, Eli Gurnee had tested three materials from Sheldahl to see if there were any changes in outgassing properties after atomic oxygen exposure, with one of the materials being aluminum-coated 1.0 mil Kapton tape with a silicone adhesive [23]. Samples that were exposed to atomic oxygen and then tested for TML and CVCM were compared to samples that were tested for TML and CVCM without being exposed to atomic oxygen [23]. The initial test sequence for the samples exposed to atomic oxygen entailed dehydrating the samples in a desiccator for 48 hours, exposing the samples to atomic oxygen for 24 hours, and then placing the samples in the micro-volatile condensable mass (micro-VCM) chamber for 24 hours [23]. This procedure
Figure 2.6: ATR IR Spectroscopy of Esterline ELA-SA-401 [15]
was changed to dehydrate the samples in a dessicator for 48 hours, place the samples in micro-VCM for 24 hours, expose the samples to atomic oxygen for 24 hours, and then place the samples back in micro-VCM for another 24 hours [23]. The reasoning for this was that as atomic oxygen erodes layers of the material, more trapped gas is allowed to outgas during the second micro-VCM test that could not outgas during the first test [23].

There was no trend between control samples and the samples exposed to atomic oxygen [23]. The aluminum Kapton tape had no change in TML, and reduction in CVCM when exposed to atomic oxygen was within the error bounds [23]. A semi microbalance was used to record the masses of the samples, and the use of a full scale microbalance could have reduced the error bars and made the CVCM results statistically significant [23]. Another issue with testing is that ASTM E595 requires that samples be in a desiccator at a constant temperature of 23°C and 50% relative humidity for 24 hours before being placed in the micro-VCM chamber [4]. A Tupperware® container with humidity control packs was used to keep the samples at a relative humidity of 50%, but the temperature was uncontrolled [23].

To further study the effects of atomic oxygen on outgassing properties, Tyler Harty tested four silicone materials (NuSil R-2141, T flex, RTV 566, CV-2566) using the second procedure used by Eli [24]. A full microbalance was used this time instead of a semi microbalance. Each test consisted of 12 samples (6 samples per material, 2 materials pert test). For each material, 3 of the samples were exposed to atomic oxygen while 3 were kept under vacuum as a control. There are 12 sample slots in the micro-VCM chamber, meaning that there as no witness disk. This was deemed acceptable as preliminary outgassing tests with the same silicone materials to validate that the micro-VCM chamber showed no significant gain in witness disk mass.
Three of the four silicone samples showed a statistically significant smaller TML for the samples exposed to atomic oxygen compared to the control samples [24]. Statistically significant was defined with a p-value $\leq 0.05$ indicating strong evidence that there was a difference in mean TML between the AO-exposed groups and the control groups and $\leq 0.10$ indicating some evidence. Independent samples T-tests resulted in a p-value for CV-2566 that was too large too conclude moderate evidence of a lower TML after being exposed to atomic oxygen, but the TML value for the samples exposed to atomic oxygen was still lower than the control samples [24]. Both the T flex and the CV-2566 showed strong evidence that atomic oxygen exposure resulted in a decrease in mean CVCM. There was no apparent correlation between TML trends and CVCM trends as low p-value TML differences did not correspond with low p-value CVCM values. Comparing the initial TML and CVCM values of the control samples to their final values for the second micro-VCM test indicates that most outgassing occurs early on when a material is put into vacuum [24]. The average TML and CVCM results of the four silicones are shown in figure 2.7, where the results of AO exposure on outgassing can be observed.

Fourier transform infrared spectroscopy (FTIR) was performed on all of the silicone samples except for the NuSil R-2141 samples as the adhesive was cured onto aluminum boats that would interfere with the tests and be difficult to remove. The purpose was to see if the difference in chemical composition between the atomic oxygen and control samples would provide an explanation for the decrease in TML values observed in the samples. The peaks of the transmittance plots for RTV 566 and CV-2566 signified a reduction in methyl groups when the material was exposed to atomic oxygen [24]. It was concluded that while there may be synergistic effects between atomic oxygen and outgassing of these silicones, the effects may not warrant design revisions as the effects were small, and after a few weeks in orbit the outgassing rate becomes so small that there is no significant concern.
Figure 2.7: TML and CVCM of AO-Exposed and Control Silicones Tested by Tyler Harty [24]
3.1 ELI

The Environmental mass Loss Investigation chamber (ELI), shown in figure 3.1 was used to outgas samples in accordance with ASTM-E595. ELI is a bell jar chamber with two copper sample bars that hold samples and collector disks. The second sample bar is not operational and therefore only the operational sample bar will be referred to. Figure 3.2 shows the sample bar that the samples rest in within the chamber while Figure 3.3 is a drawing of a portion of the sample bar. The sample bar has 12 0.625” diameter circular compartments that samples are held in, and smaller 0.250” diameter through holes located in the center of each compartment. Circular copper plates attached the sample bar with set screws are used to cover the compartments.

A 26Ω resistor is welded around the perimeter of the sample bar. Two wires, connected on either end of this resistor goes through an electrical feedthrough and are connected to a Variac® Model NO:TDGC-2KM AC power supply. Current running through the resistor-bar dissipates heat and increases the temperature of the copper sample bar, in turn increasing the temperature of the samples to encourage outgassing. Type-T thermocouples that are fed into the chamber through another feedthrough can be attached to the bar at each sample compartment location by either using Kapton® tape or by tightening the cover plate down with the thermocouple between the cover plate and the sample bar.

Parallel to the sample bar is a cooling plate that the copper collector disks are attached to. Each collector disk rests on two pins attached to the copper plate and secured
with a spring-loaded screw. A waterline welded to the other side of the plate exits the chamber through a feedthrough and is connected to a Brinkmann Lauda water cooling unit. The water cooling unit holds decalcified water and maintains the water at a specified temperature. The water either conducts heat from or deposits heat to the plate as it moves past it and is adjusted to its original temperature once it is back in the water cooling unit. This helps keep the temperature of the plate and collector disks at $25\pm1^\circ C$. Three bolts connect the sample bar and the collector disk plate
together, with nylon spacers to prevent heat traveling from the sample tower to the collector plate.

Between the sample tower and the collector plate is a metal piece that acts as a spacer between the tower and the plate and isolates each collector disk as well. This is to prevent cross contamination, so outgassed matter that condenses on one plate from one sample does not condense on another plate. This separator plate has holes
aligned with the through holes in the sample tower so that each sample compartment has access to one collector disk that the outgassed matter can travel to.
Beneath the sample tower and collector bar is the pump feed through that both the roughing pump and turbo pump are connected to. The roughing pump is a Cacejen CVP 36 two stage rotary vane pump that evacuates air from the chamber initially at 0.65 m$^3$ min$^{-1}$. Once the pressure is low enough (50 mTorr or lower) the Turbovac 450 iX turbo pump, connected between the chamber and the roughing pump, is activated and runs in tandem with the roughing pump to reduce the chamber pressure below 5x10$^{-5}$ Torr with a pump speed of 25.8 m$^3$ min$^{-1}$ [31]. Two Granville-Phillips 275 convectron gauges are used to measure the pressure of both the roughing pump and the pressure of the chamber while it is above one mTorr. Once the chamber pressure reaches a magnitude of 10$^{-4}$ Torr, the convectron gauge is no longer able to read the pressure of the chamber and a Granville-Phillips 274 ion gauge is used to measure the pressure. The pressures of both the chamber and the roughing pump are displayed on a Granville-Phillips 307 vacuum gauge controller.

### 3.2 MAX

The Minimum Atmospheric eXperimentation chamber (MAX) was the chamber used to expose samples to atomic oxygen. Within the chamber is a 25.4x25.4 cm aluminum plate that is grounded. This plate is known as the two-slot plate, and has two small slots to place witness samples (Kapton® HN) and two 20.6x4.5cm slots to place silicone samples in. The bottom of the sample slots are covered in aluminum tape and have platforms made of aluminum tape to elevate samples so the top face of the samples are level with the top face of the witness samples. This is to ensure that the fluence of atomic oxygen that both the samples and the witness samples receive are the same. Figure 3.5 is a picture of the grounded two-slot sample plate. Another plate that can be used as the grounded plate is known as the four hole plate, which has four 2.540 ± 0.003cm diameter holes that are equidistant from the center of the
plate. A cover plate secures the samples to the ground plate with an exposed area of 5.06 ± 0.02 cm². The two slot plate was used for this thesis as it allows the samples to be closer to the center of the beam.

Figure 3.5: Slotted Sample Plate in MAX

In between the two witness sample slots is a pipe where air from outside of the chamber is bled into the chamber. This is to provide diatomic oxygen that will be dissociated into atomic oxygen. The portion of the pipe outside of the chamber has a
Nupro gas valve with a hand-adjustable knob that controls the flow of air that enters the chamber.

An Adixen ACP 28 mechanical pump from Pfeiffer Vacuum is attached to the bottom of the chamber to reduce the pressure. The combination of inserting air into the system with the NuPro valve and removing air from the system with the roughing pump ensures that there are enough di-oxygen molecules (chamber pressure of 175 ± 10 mTorr) to produce atomic oxygen. Adjusting the NuPro valve ensures that
the chamber pressure is within 10 mTorr of 175 mTorr. A Granville-Phillips 275 convectron gauge is attached through a feed through to monitor the chamber pressure. Another convectron gauge of the same make is connected to the line between the chamber and the roughing pump right next to the pump to monitor the pump pressure (also known as base pressure).

7.62cm above the ground plate is an aluminum electrode powered by an RF generator. A Seren R301 MKII RF power supply is used to power the electrode with 125W at a frequency of 13.6 MHz, which dissociates the di-oxygen into atomic oxygen, forming a capacitively coupled plasma (seen in figure 3.6) between the RF-powered electrode and the ground electrode (the slotted sample plate). A Seren IPS AT3 matching network matches the impedance load of the power supply to protect the system from reflected RF power. A Seren MC3 controller is used to adjust variable capacitors in the AT3 matchbox to automatically find the matching impedance required[22]. A dark space shield keeps the generated plasma contained between the electrodes. The
produced atomic oxygen is omnidirectional with energies ranging from 0.04-0.1eV [22]. While this energy is low compared to the atomic oxygen in LEO, the high fluence of atomic oxygen that MAX produces (on the order of $1 \times 10^{21} \text{atoms cm}^{-2} \text{in 24 hours}$) leads to similar erosion levels, albeit without the pits and cones observed on the surface of samples exposed to atomic oxygen in LEO. A copper strap is attached to the bottom aluminum plate to ground it. Type-T thermocouples fed in through a different feedthrough can be attached to the ground plate with Kapton® tape to monitor the temperature of the samples as the plasma will heat up the plate.

### 3.3 Microbalance Scale

A Mettler Toledo XS3DU microbalance scale (Shown in figure 3.8) was used to measure the mass of samples, before, in between, and after test sequences. The microbalance scale has a reading of $\pm 10 \mu g$ but can be adjusted in the settings to display a reading of $\pm 1 \mu g$ [52]. The scale has a draft shield to prevent outside forces (such as a draft of air) from affecting the measurements.

The maximum capacity of the scale is 3.1g with a repeatability (measured as standard deviation) of 3$\mu g$ at a low load (low load being defined as 0.2g) [52]. The scale also has a typical linearity deviation of 3$\mu g$.

A microbalance scale with a readability of $\pm 10 \mu g$ was desirable as the samples for the three silicone materials had masses between 360 and 390 milligrams, and with SCV2-2590 having a reported CVCM of 0.008% of the total mass, the expected mass of condensed matter on collector plates from this material would be around 300 micrograms or less. Also, the ASTM E595 standard requires a readability of $\pm 10 \mu g$ as mass gains of the witness collector disk as small as 20 and 50$\mu g$ are used to indicate if there were contamination issues within the chamber [4].
The microbalance scale has an issue measuring the mass of dielectric material as the charge the material stores due to static electricity will cause the scale reading to drift. A solution to this is to use a Milty Zerostat 3 anti-static gun when measuring the mass of dielectrics. Squeezing the trigger of the gun while pointing at a dielectric sample will bombard the dielectric with positively ionized air while relaxing the air will then emit negatively ionized air [48]. The combination of positive and negative ions dissipates the charge of the dielectric, and eliminates the scale drift.
3.4 FTIR Spectrometer

A JASCO FT/IR-4600 Spectrometer from the Materials Engineering Department at Cal Poly was used to obtain sample absorbance in the infrared spectrum. The spectrometer uses attenuated total reflectance (ATR), where the Infrared (IR) light is steered towards an IR transparent diamond crystal, known as an ATR crystal, at a 45° angle [27]. If a sample is resting on top of this crystal, the IR light will penetrate the sample and get reflected back down towards a detector, as seen in figure 3.9 from [26].

![Figure 3.9: IR Beam Traveling Through an ATR Crystal and Sample][1]

The key difference between ATR spectroscopy and traditional transmittance spectroscopy is the path length. The path length of the light in transmittance spectroscopy is greater than ten times the path length the light travels in ATR spectroscopy [26]. As a result, solids typically have to be diluted with potassium bromide with transmittance spectroscopy, which is a laborious process while ATR spectroscopy requires no sample preparation to gather a spectrum.
The major components of the FTIR spectrometer are the infrared source, the interferometer, and the detector [28]. The light source is a high intensity ceramic source. A nichrome wire increases in temperature when current is passed through it and heat, or infrared light, is emitted [47]. The ceramic is in thermal contact with the wire to allow the wire to operate at higher temperatures.

The interferometer is a Michelson interferometer, which is shown in figure 3.10. It consists of aluminum coated mirrors and a beamsplitter. A collimating mirror collects the photons from the infrared source and aligns them in a uniform direction headed for the beamsplitter. The beamsplitter is a germanium film that is sandwiched between two potassium bromide windows that are IR-transparent for protection [27, 47]. The germanium film both reflects some of the IR irradiation from the collimating mirror and transmits some, acting as a two-way mirror. The reflected photons travel towards a mirror at a fixed position away from the beamsplitter and are reflected back towards the beamsplitter. The transmitted photons travel towards a mirror that moves in position relative to the beamsplitter and are also reflected back towards the beamsplitter. The two IR beams recombine at the beamsplitter and the combined beam is redirected towards the ATR crystal and sample. There are differences in path length that the IR photons have to travel between the fixed mirror and the moving mirror as the moving mirror travels to its maximum position away from the beamsplitter then back to its initial position. This creates constructive and destructive interference in the combined beam over the duration of a scan. The signal of this recombined beam over time or mirror position is called an interferogram [28].

The recombined IR beam is then sent through the ATR crystal and sample as shown in figure 3.9. The intensity of the beam is attenuated by the sample and then steered to a deuterated L-alanine doped triglycine sulphate (DLaTGS) detector where the intensity vs time spectrum is converted to an intensity vs frequency or wavenumber.
spectrum via a Fast Fourier Transform [27, 47]. The depth that the IR beam penetrates into the sample is variable, but is on the order of microns. Equation 3.1 can be used to calculate the penetration depth of the IR beam.

\[
d_p = \frac{\lambda}{2\pi n_{atr} \sqrt{\sin^2 \theta - \left( \frac{n_{sample}}{n_{atr}} \right)^2}} \quad (3.1)
\]
\( \lambda \) is the wavelength of the IR photon, \( \theta \) is the angle of incidence between the IR beam and the sample, and \( n_{\text{atr}} \) and \( n_{\text{sample}} \) is the refractive index of the crystal and the sample respectively [26]. The penetration depth will affect the intensity of the peaks observed in the spectrum. This means that bonds with vibrational energies associated with lower frequency peaks or higher wave numbers will naturally have lower intensity peaks on the spectrum [26].

The spectrometer was connected to a computer with Spectra Manager™ software in order to view and export spectra, with the capability to record reflectance, transmittance, or absorbance spectra. With the relation of absorbance and concentration from Beer’s law, The software and spectrometer were configured to collect absorbance data in order to compare spectra of samples affected by different aspects such as outgassing or atomic oxygen.

### 3.5 Junior

Junior (figure 3.11) is the desiccant chamber used to rid the Kapton®HN witness samples of their water vapor content and hold them at a pressure similar to the pressure the silicone and witness samples will be exposed to in MAX. The desiccator is a Kartell DYNCR 243065, and is attached to a Cacejen CVP 24 two stage rotary vane pump with a pump speed of 15.2 \( \text{ft}^3/\text{min} \) [33]. The chamber pressure is estimated to be under 200 mTorr [53]. A valve on the lid of the desiccator is used to vent the chamber.
Figure 3.11: Junior
4.1 ASTM E595

The TML and CVCM of samples were determined chiefly following the procedures of ASTM E595, although not all of the standard’s requirements were able to be fully met. For full details of ASTM E595 please see the previous section describing the test standards. ASTM E595 has an optional method to estimate the water vapor regained in samples that involves remeasuring the mass of the samples after being stored in a container at $23 \pm 2^\circ C$ and $50\% \pm 5\%$ relative humidity 24 hours after the mass was measured post-vacuum. This was not calculated as there was no means to control the temperature of the samples and the samples were measured as soon as possible after being pulled out of vacuum. This prevents the possibility of subtracting the predicted mass the silicone samples regain in atmosphere while being removed by ELI and measured with the microbalance scale and calculating a more accurate TML. However, the omission of this portion of the test sequence does not prevent comparison of control samples to samples that have been exposed to atomic oxygen.

The temperatures measured with a Perfectprime Datalogger thermometer TC0520 thermocouple reader connected to the T thermocouples had a range of $\pm 20.9^\circ C$, and with a mean temperature of $122.5^\circ C$. Portions of this wide range of temperatures recorded could be due to the accuracy of the thermocouple reader of $\pm (0.1\% T + 0.7^\circ C)$, where $T$ is the reading. The error could also be associated with the accuracy of the thermocouples themselves ($\pm 1^\circ C$ or $\pm 0.75\%$ of the reading, whichever is greater[24]), or non-uniform distribution of heat transfer in the sample bar. One of the the ther-
mocouples measured temperature values on average $7.6^\circ C$ lower than the rest of the thermocouples, regardless of where on the sample bar the thermocouple was located, and another thermocouple read extremely low values until it was repaired. Disregarding the temperatures due to faulty thermocouples resulted in a mean temperature measurement of $124.3^\circ C$ with a range of $\pm 11.6^\circ C$ and a standard deviation of $3.1^\circ C$. This meant that it could not be confirmed that all of the samples for each test were at the same temperature. However, the Variac® was set to 30V for each test, meaning that the same power was supplied to the sample bar for all tests.

Samples were stored in separate compartments of a pill box in a Tupperware® container along with a D’Addario Humidpak acting as a two-way humidity control system. An AcuRite model 00619 digital indoor temperature and humidity monitor was also in the Tupperware® container to record the temperature and relative humidity of the samples before samples were moved to ELI. The average humidity throughout all tests was 51%, with a minimum recorded humidity of 46% and a maximum recorded humidity of 53%. There was no method or equipment within the laboratory to produce a temperature controlled container. The average temperature recorded within the Tupperware® container was $20^\circ C$, with a minimum recorded temperature of $15^\circ C$ and a maximum recorded temperature of $25^\circ C$. The temperatures the samples were exposed to were not compliant with the standard and had to be accepted. This variation in temperature could result in a variation in water vapor content in the samples between each test that could not be quantified, but this is considered minimal as the humidity the samples were exposed to were in acceptable ranges.

Using cleaned containers (boats) to keep samples in during the test was disregarded for multiple reasons. All three material were solid elastomers rather than liquids or epoxies that would need to be contained. The sample bar was cleaned before each test sequence, so the samples would not be exposed to previous outgassed matter in
the sample compartment. The time added to measure the mass of the boats would also negatively impact the test results, allowing the samples to gain more water vapor content. This deviation from the standard does not impact the test.

Rather than chromium, the collector disks used are copper, but wrapped in ultra-high vacuum (UHV) aluminum foil. UHV aluminum foil is clean and contamination free, and is what the National Aeronautics and Space Association (NASA) Jet Propulsion Laboratory (JPL) uses for ASTM E595 tests[24]. They are advantageous as they have a small enough mass to use with the micro balance scale, as the mass of the copper disks far exceeds the maximum mass allowed on the scale (3.1 grams). Besides the mass limit that copper disks exceed, it was also previously determined that plating the disks in chromium would be a tedious process, placing a tremendous amount of stress on the malleable copper[24]. Since UHV aluminum foil is disposable, new foil was used for each test to mitigate contamination. An empty sample compartment in the sample bar had an associated copper disk wrapped in UHV aluminum foil on the collector plate for each test to measure contamination and determine the adequacy of the cleaning and bake out procedure before each test. This deviation from the standard does not impact the test either.

With the exception of two tests, six samples were in the sample bar per test. As previous theses speculated that the upper and lower sample compartments experienced temperature deviations, samples were placed in the center six sample compartments of the bar[23, 24].

4.2 ASTM E2089

Samples were exposed to Atomic Oxygen following ASTM E2089. Only Kapton® HN was used as the other three witness materials (TFE-FEP, PE, and PG) were not
available for use. The Kapton® HN samples were cleaned with dry Kimwipes® while the samples were handled with nitrile gloves and stored in the clean pill box before testing. Chemical cleaning agents such as acetone and methanol were not used to clean the silicones or the witness samples. These cleaning products could alter the chemical composition of the samples in unknown ways, affecting how they erode from atomic oxygen and how they outgas. No masking techniques were used as there was no need to control the exposure area of the samples. Samples were not clad together either.

Each test had two Kapton® HN samples exposed simultaneously with the test samples to determine the effective atomic oxygen fluence, assuming that Kapton® HN has an erosion yield of $2.81 \times 10^{-24} \text{cm}^3/\text{atom}$ [3]. Samples were elevated on aluminum shims to they were level with the witness samples, and located as close to the center of the ground plate as possible so they could receive the most uniform and maximum AO fluence possible.

MAX was pumped down to a pressure of $175 \pm 10\text{mTorr}$ before the RF Generator, the system controller, and the load/matching network were turned on and the plasma of atomic oxygen was generated. Each test exposed the samples to atomic oxygen for 24 hours before turning off the RF Generator and venting the chamber. 24 hours of exposure in this chamber equates to four to six months in LEO at a 400km orbit with a 28.5 degree inclination in the ram direction, assuming an average fluence of $2 \times 10^{21} \text{atoms/cm}^2$ for a year. This comes from data collected from the Materials International Space Station Expirement (MISSE) for an entire duration of a solar cycle (2004-2016) [41]. For some tests, after leaving the laboratory and returning, the pressure would be outside of this $175 \pm 10\text{mTorr}$ range and would need to be corrected via the NuPro valve. The average pressure in MAX during these tests was 165mTorr with a standard deviation of 11mTorr. This is a result of one test with an
excessively low pressure observed after returning to the laboratory. The rest of the tests had an average pressure of 168mTorr with a standard deviation of 6mTorr.

4.3 FTIR

When analyzing samples using the FTIR Spectrometer, the surface that the samples rest on when being analyzed was first cleaned with a Kimwipe® and acetone to remove any contamination from previous tests. A dry Kimwipe® was then used to remove any residual acetone from the surface to prevent acetone from contributing to the spectra measured.

A spectrum of nothing in the spectrometer, known as a background spectrum would be collected. This would be used to calibrate the spectrometer and eliminate any peaks associated with the background spectrum from contributing to the spectrum of the sample to be measured. The sample would be placed in the spectrometer, and a spectrum would be recorded using the software.

If the spectra of multiple samples needed to be measured, the same cleaning procedure described in the previous paragraph would be performed between each sample measurement. A background spectrum was also collected after cleaning each time. Table 4.1 describes all sample conditions for each material that FTIR spectra was collected, as well as the number of samples for each condition. FTIR spectra of samples of all materials as received by NuSil Technology LLC were collected without any exposure to atomic oxygen or outgassing for comparison. Samples exposed to just outgassing were inserted into ELI while other samples of the same material were undergoing an ASTM E595 test and removed immediately after the test was complete to be analyzed with FTIR Spectroscopy. Samples exposed to solely atomic oxygen were likewise inserted into MAX while other samples were undergoing an ASTM E2089 test
and removed immediately afterwards to be analyzed. Samples that went through the test sequence described in the next section were analyzed after the test sequence for each material to record the spectra of samples that were both outgassed and expose to atomic oxygen.

Table 4.1: FTIR Tests for each Material, Number of Samples Used

<table>
<thead>
<tr>
<th>Condition</th>
<th>Number of Samples</th>
</tr>
</thead>
<tbody>
<tr>
<td>Outgassing</td>
<td>2</td>
</tr>
<tr>
<td>Atomic Oxygen</td>
<td>2</td>
</tr>
<tr>
<td>Outgassing and Atomic Oxygen</td>
<td>3</td>
</tr>
</tbody>
</table>

The Purpose of collecting spectra of samples at different phases was to examine how outgassing and atomic oxygen individually affected the samples. It should be emphasized that the overall purpose of the FTIR spectroscopy was not to determine the material composition of the materials before or after atomic oxygen and outgassing. FTIR spectroscopy was used as a method of verification to determine that samples exposed to atomic oxygen had alterations to it’s composition compared to samples not exposed to atomic oxygen.

4.4 Test Sequence

Figure 4.1 shows a diagram of the test sequence that would be performed for one batch of samples. This test sequence is the same sequence used by Harty in their work [24]. Each test sequence took a week to perform. For all testing, nitrile gloves were worn and Teflon-tipped forceps were used to handle the samples and UHV aluminum foils.

Before every test sequence, ELI was thoroughly cleaned using 1:1 volume mixture of acetone and denatured ethanol, as recommended by ASTM E595[4]. The sample bar, collector plate, the spacer between the two, and the UHV aluminum foils that would collect condensable matter from samples in ELI were thoroughly wiped with
Figure 4.1: Test Sequence for One Batch of Samples

this mixture and Kimwipes®. The chamber, with the UHV aluminum foils to be used for the entire test sequence inside, was pumped down to < 1mTorr and the sample bar temperature was elevated to 125°C for a minimum of 16 hours. This bake out would remove contaminants in ELI from previous tests that could affect the mass measurements of both the samples and the foils.

All silicone samples and foils being tested were placed in pill boxes that were stored in the Tupperware® container along with the digital temperature and humidity monitor and Humidpak for at least 24 hours. The Tupperware® container, Humidpak, and digital temperature and humidity monitor acted together as a desiccator to keep samples from being affected from environmental humidity, and the three in conjunction will be referred to as a desiccator for the remainder of this thesis. At the same time, two Kapton® HN witness samples would be placed in Junior. Junior would pump down to remove the water content from the witness samples, and the witness samples would remain in Junior at < 200mTorr for at least 48 hours.

While the Kapton® HN samples remained in Junior, All samples (those that would be exposed to atomic oxygen and those that would not) as well as the appropriate
number of foils (one foil for each sample plus an extra serving as a witness foil) were moved to an office with the micro balance scale after 24 hours and their masses were measured. Each sample and foil was measured three times for repeatability, and the average of the three measurements was the mass value to be used for analysis. After a sample or a foil was finished being measured it was immediately returned to the desiccator to limit the amount of water vapor regained. This procedure of measuring samples and foils three times and immediately returning them to the desiccator after measurement was followed for all micro balance scale measurements throughout the test sequence.

Afterwards the desiccator with the samples and foils was returned to the lab. The samples and foils were removed from the desiccator and placed in ELI. An initial outgassing test following ASTM E595 was performed. The purpose of the initial outgassing test was to eliminate uncertainty in the mass loss of samples that would be exposed to atomic oxygen. If the samples were exposed to atomic oxygen without the initial outgassing test, it would be impossible to determine what portion of the sample mass loss was due to atomic oxygen erosion and what portion was due to outgassing.

After the initial outgassing test was performed, all samples and the foils tested were removed from ELI and placed back in the pill boxes in the desiccator. The two Kapton® HN witness samples were also removed from Junior and placed inside the desiccator as well. The samples were relocated back to the micro balance scale and the mass of each sample, foil, and witness sample was measured. The Kapton® HN samples, which are dielectrics, would gain static electricity and result in a drift in the micro balance scale reading. The anti static gun was used whenever a witness sample was measured to discharge the samples and eliminate drift and errors due to the static electricity. This process of using an anti-static gun was followed anytime
the witness sample masses were recorded with the micro balance scale throughout the
test sequence.

Once masses were measured, the desiccator was brought back to the laboratory and
the samples that would not be exposed to atomic oxygen (the control samples) were
placed back in ELI. The samples that were to be exposed to atomic oxygen were placed
in MAX along with the two Kapton® HN witness samples, and were irradiated with
atomic oxygen following ASTM E2089. While ASTM E2089 requires samples to be
stored at a pressure of less than 200mTorr for at least 48 hours before atomic oxygen
exposure, it was considered okay for the silicone samples to have only been in vacuum
for 24 hours as they were at pressure < 5x10^{-5}Torr and at temperatures of 125°C, so
majority of water vapor content in the samples would no longer be present.

Eli was also pumped down to 175 ± 10mTorr and the Variac power supply was ad-
justed to 20-21V so that the temperature of the sample bar would be equivalent to
the temperature of the ground plate in MAX, which was on average 64.5°C with a
standard deviation of 5.8°C. The purpose of exposing the control samples to temper-
atures and pressures similar to those of the samples being exposed to atomic oxygen
for the same duration of time is to ensure that the only factor that could explain a
difference in TML and CVCM between samples exposed to atomic oxygen and control
samples is the presence of atomic oxygen.

After 24 hours of AO production in MAX, the RF generator, the system controller,
and load/matching network are turned off and MAX is vented. The Variac power
supply connected to ELI is turned off and ELI is vented as well. All samples and
witness samples are placed in the desiccator and returned to the micro balance scale,
where their masses are measured once again. The remaining foils that were not used
in the initial outgassing test that have been stored in the desiccator have their initial
masses measured at this time. The desiccator is returned to the laboratory and the
control samples, the samples exposed to atomic oxygen, and the remaining foils are placed in ELI and a final ASTM E595 test is run. After 24 hours in ELI the chamber is vented, the samples and foils are placed in the desiccator, and the mass of the samples and foils is measured using the micro balance scale one last time.

While ASTM E2089 states that samples should be measured within five minutes of being removed from vacuum to prevent hydration of the samples, this would be impossible to achieve as the samples all had to be removed from vacuum at the same time. They also had to be moved to a separate location and measured multiple times for repeatability. The time between removing the samples from the chamber, measuring the masses, and returning them to vacuum took on average 53 minutes, with the longest time to return the samples back to vacuum being 1 hour and 8 minutes. Although this duration was 10 times greater than the recommended time, the use of a desiccator was a mitigating step taken to reduce the water vapor regained, and samples were only outside of this controlled environment for the brief moments they were removed from the vacuum chamber and when they were removed to be measured.

For both of the ASTM E595 tests in a test sequence, samples were randomly assigned to the middle sample compartments. regardless if they were designated atomic oxygen samples or control samples. Thermocouples were also randomly assigned to record the temperature of each sample compartment used. The random assignment was intended to eliminate bias in results between samples exposed to atomic oxygen and control samples if there was an uneven temperature distribution in the sample bar. The random assignment of the thermocouples would also eliminate bias from variation in thermocouple readings.
5.1 TML and CVCM

In total, 11 week-long test sequences were performed to quantify how atomic oxygen affected the outgassing properties of CV-2500, CV2-2289-1, and SCV2-2590. Table 5.1 describes when all these tests were performed and how many samples were used in each test. "AO" indicates number of atomic oxygen exposed samples while "Control" indicates number of control samples.

<table>
<thead>
<tr>
<th>Date</th>
<th>Material</th>
<th>AO</th>
<th>Control</th>
</tr>
</thead>
<tbody>
<tr>
<td>12/6/2021-12/10/2021</td>
<td>CV-2500</td>
<td>4</td>
<td>4</td>
</tr>
<tr>
<td>12/18/2021-12/22/2021</td>
<td>CV-2500</td>
<td>4</td>
<td>4</td>
</tr>
<tr>
<td>1/28/2022-2/1/2022</td>
<td>CV2-2289-1</td>
<td>2</td>
<td>2</td>
</tr>
<tr>
<td>2/17/2022-2/21/2022</td>
<td>CV2-2289-1</td>
<td>3</td>
<td>3</td>
</tr>
<tr>
<td>2/24/2022-2/28/2022</td>
<td>SCV2-2590</td>
<td>3</td>
<td>3</td>
</tr>
<tr>
<td>3/3/2022-3/7/2022</td>
<td>CV-2500</td>
<td>3</td>
<td>3</td>
</tr>
<tr>
<td>3/10/2022-3/14/2022</td>
<td>CV2-2289-1</td>
<td>3</td>
<td>3</td>
</tr>
<tr>
<td>3/31/2022-4/3/2022</td>
<td>SCV2-2590</td>
<td>3</td>
<td>3</td>
</tr>
<tr>
<td>4/6/2022-4/10/2022</td>
<td>CV-2500</td>
<td>3</td>
<td>3</td>
</tr>
<tr>
<td>4/14/2022-4/18/2022</td>
<td>SCV2-2590</td>
<td>3</td>
<td>3</td>
</tr>
<tr>
<td>4/20/2022-4/24/2022</td>
<td>SCV2-2590</td>
<td>3</td>
<td>3</td>
</tr>
</tbody>
</table>

It was not until the first two test sequences were completed that an error in the method of recording sample temperatures in ELI was realized. The thermocouple reader used has four ports to collect readings from four individual thermocouples simultaneously. The input of the thermocouples into the thermocouple reader is a voltage that is dependant on the temperature gradient between the two metal wires that the thermocouple consists of. The source of this temperature gradient is the temperature of
the object being measured, and the assembly of the object being measured acting as a voltage source, the thermocouple acting as wires, and the thermocouple reader forms a circuit. While all of the thermocouples are attached to the copper sample bar at different locations, they are still connected to the same conductive piece of copper, so if multiple thermocouples are plugged into the thermocouple reader, a circuit that includes all of the thermocouples is created and the voltages produced by the thermocouples is altered, drastically affecting the temperature value displayed on the thermocouple reader. Experimentation verified this, and it was determined that connecting one thermocouple into the reader at a time produced the temperature value that the thermocouple was reading.

Without knowing this, the first test sequence using CV-2500 samples had thermocouple readings much lower than 125°C while the Variac® power supply was set to 30V, which was the voltage of the power supply that had been predetermined through previous use of the chamber to raise the temperature of the sample bar to 125°C. As a response the power supply voltage was increased until temperatures displayed were reading in the order of 100°C, although there was variation in the thermocouple readings from 100°C to 130°C. This meant that in actuality the samples were experiencing temperatures much greater than 125°C, and as a result the average TML of these samples were 6 times greater than the average TML of the other test sequences involving CV-2500, and as a result the data from 12/6/2021 to 12/10/2021 was not used in the reported results for CV-2500.

The second test sequence with CV-2500 also had the issue of inaccurate temperature display without knowing the cause, but rather than adjusting the voltage of the power supply to produce temperature readings closer to 125°C the power supply was left at 30V. The resulting mass loss of the samples were of the same order of magnitude of the CV-2500 tests that did not have temperature display issues, so the data from
12/18/2021 to 12/22/2021 was used in the reported results for CV-2500. It had to be accepted that the temperatures of the sample locations in the sample bar were unknown, but assumed similar enough to the other tests as the power supply voltage was the same for these tests.

After this was resolved, thermocouples were plugged one by one into the thermocouple reader while attached to the same location of the sample bar while ELI was under vacuum and the Variac® was on. There was a resulting variation in thermocouple readings on the range of 20°C due to a few thermocouples reading lower temperatures than the rest. The number of samples to be tested was reduced to four (two samples to be exposed to atomic oxygen and two control samples) for the next test sequence, using four thermocouples that were not outliers contributing to the 20°C variation in reading. Afterwards 6 samples in total were used for test sequences, and only one of the six thermocouples used had a low reading, but typically read 10°C lower than the other thermocouples rather than 20°C.

CV-2500 data from the ASTM E595 test after samples were exposed to atomic oxygen for all of the test sequences besides the first one is shown in figure 5.1. The mean TML of the samples exposed to atomic oxygen was 0.0198% with a standard deviation of 0.0075% while the mean TML of the control samples was 0.0290% with a standard deviation of 0.0051%. The mean CVCM of the samples exposed to atomic oxygen was 0.0108% with a standard deviation of 0.0047% while the CVCM of the control samples was 0.0148% with a standard deviation of 0.0043%. A noticeable trend in decreased TML and CVCM of samples that are exposed to atomic oxygen can be observed.

During the test sequence from 12/18/2021-12/22/2021, The recorded CVCM from one the samples that had been exposed to atomic oxygen was greater than TML of the sample itself. Since it is impossible for the collector foil to gain more mass
than the sample lost without condensable matter coming from another source, it was
determined that contamination had occurred, and as a result both the TML and
CVCM from this sample was not included in the analysis.

CV2-2289-1 data from the ASTM E595 test after samples were exposed to atomic oxygen for all of the test sequences is shown in figure 5.2. The mean TML of the samples exposed to atomic oxygen was 0.0342% with a standard deviation of 0.0041% while the mean TML of the control samples was 0.0528% with a standard deviation of 0.0095%. The mean CVCM of the samples exposed to atomic oxygen was 0.0187% with a standard deviation of 0.0035% while the CVCM of the control samples was 0.0185% with a standard deviation of 0.0038%. While the plot shows a noticeable

Figure 5.1: CV-2500 TML and CVCM of AO Exposed Samples Vs Control Samples Post AO Exposure
decrease in TML for CV2-2289-1 samples that were exposed to atomic oxygen. There is no apparent difference in the CVCM.

**Figure 5.2: CV2-2289-1 TML and CVCM of AO Exposed Samples Vs Control Samples Post AO Exposure**

During the first test sequence involving CV2-2289-1, the RF generator had stopped producing atomic oxygen. This was discovered only after returning back to the laboratory, and it is unknown what time the system had stopped functioning. By calculating the effective fluence using the Kapton® HN witness samples and comparing it to the average effective fluence of the other witness samples from different test sequences, the samples in this test were only exposed to atomic oxygen for 76% of the time that samples from other test sequences were exposed to atomic oxygen, or 18 hours rather than 24 hours. This did have an effect on the mass lost by samples due to atomic oxygen erosion, as observed in figure 5.3, where the TML was calculated by using
the mass measurements before the samples were placed in MAX and after they were removed from MAX. Not only this, but the water cooling unit was not operating during both of the ASTM E595 tests during this test sequence, so there was no control of the temperature of the UHV foils. For these reasons, the data from this test are not included in these results, but can be found in the appendices of this document.

![Figure 5.3: Average TML due to AO Erosion of CV2-2289-1](image)

SCV2-2590 data from the ASTM E595 test after samples were exposed to atomic oxygen for all of the test sequences is shown in figure 5.4. The mean TML of the samples exposed to atomic oxygen was 0.0143% with a standard deviation of 0.0070% while the mean TML of the control samples was 0.0207% with a standard deviation of 0.0077%. The mean CVCM of the samples exposed to atomic oxygen was 0.0064% with a standard deviation of 0.0039% while the CVCM of the control samples was 0.0083% with a standard deviation of 0.0040%. Like CV-2500, the plot shows that
there is a noticeable trend where samples exposed to atomic oxygen have a decreased TML and CVCM.

For the test sequence from 3/31/2022-4/3/2022, a sample exposed to atomic oxygen had a TML value less than the associated CVCM that was measured, and as a result the TML and CVCM values were not included in the analysis. The TML and CVCM from two AO-exposed samples from 4/20/2022-4/24/2022 as well as the TML and CVCM values of a control sample from the test sequence 2/24/2022-2/28/2022 were not included for the same reason.

Another control sample from 4/14/2022-4/18/2022 was dropped on the ground after removing the sample from ELI when the final ASTM E595 test was complete. Due to the contamination of the sample from collecting matter from the floor in a non-clean room environment, the TML of this sample was not included in analysis, but the CVCM associated with this sample was kept as the mass gained by the UHV aluminum foil was not affected by this accident.

The standard deviations for all three materials did not meet the precision defined by ASTM E595. Tables 5.2-5.5 shows the average TML and CVCM of all of the tests performed (with the exception of the first test sequences involving CV-2500 and CV2-2289-1) with the readability of the microbalance scale incorporated into the uncertainty of the values. Included is the standard deviation observed as well as the precision, defined as the standard deviation divided by the mean value. Note that the standard deviation and precision is unavailable for the AO-exposed samples in the final SCV2-2590 test sequence as only one sample had a recorded TML higher than its CVCM. A majority of the test sequences produced TMLs and CVCMs with deviations well above what ASTM E595 considers precise (10% the mean TML and 20% the mean CVCM).
Figure 5.4: SCV2-2590 TML and CVCM of AO Exposed Samples Vs Control Samples Post AO Exposure

<table>
<thead>
<tr>
<th>Test</th>
<th>TML [%]</th>
<th>Standard Deviation [%]</th>
<th>Precision [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>CV-2500 #2</td>
<td>0.0171 ± 0.0041</td>
<td>0.0043</td>
<td>25.1</td>
</tr>
<tr>
<td>CV2-2289-1 #2</td>
<td>0.0337 ± 0.0039</td>
<td>0.0050</td>
<td>14.8</td>
</tr>
<tr>
<td>SCV2-2590 #1</td>
<td>0.0143 ± 0.0041</td>
<td>0.0046</td>
<td>32.2</td>
</tr>
<tr>
<td>CV-2500 #3</td>
<td>0.0158 ± 0.0041</td>
<td>0.0087</td>
<td>55.1</td>
</tr>
<tr>
<td>CV2-2289-1 #3</td>
<td>0.0346 ± 0.0040</td>
<td>0.0040</td>
<td>11.6</td>
</tr>
<tr>
<td>SCV2-2590 #2</td>
<td>0.0149 ± 0.0041</td>
<td>0.0067</td>
<td>45.0</td>
</tr>
<tr>
<td>CV-2500 #4</td>
<td>0.0266 ± 0.0040</td>
<td>0.0050</td>
<td>18.8</td>
</tr>
<tr>
<td>SCV2-2590 #3</td>
<td>0.0182 ± 0.0040</td>
<td>0.0097</td>
<td>53.3</td>
</tr>
<tr>
<td>SCV2-2590 #4</td>
<td>0.0119 ± 0.0041</td>
<td>N/A</td>
<td>N/A</td>
</tr>
</tbody>
</table>

A major source of error in the experimentation is the variation in temperature measurements, as previously mentioned. Comparing all the measurements recorded for each thermocouple during both the initial and final outgassing tests to each other, as
shown in figure 5.5, it is noticeable that thermocouple T11 is lower than the rest of the thermocouples by an average of 8.4°C. The error bars on the plot were calculated by taking the maximum and minimum values recorded from each thermocouple and adding the errors due to the accuracy of the both the thermocouple and the thermo-
couple reader, and adding or subtracting that value to the maximum or minimum value, respectively. The error bars had an average range of 14.4°C.

![Temperature Graph](image)

**Figure 5.5: Mean Temperature of Sample Bar in ELI by Thermocouple**

Besides thermocouple T11, the average recorded value of the remaining thermocouples were within 2.6°C of each other and were within ±1.4°C of 125°C, but the range of recorded temperatures for each of these thermocouples is still a source of uncertainty. Due to the variation in the standard deviation of the recorded temperatures between the thermocouples (mainly, the standard deviation of recorded temperatures from thermocouple T8 being more than double the standard deviation from thermocouple T14), no meaningful tests such as a one way analysis of variance (ANOVA) F-test could be done to provide evidence as to whether or not these five thermocouples are recording the same temperatures [32].
The temperature recorded at each sample position in the sample bar is displayed in figure 5.6. Using the middle six sample compartments of the sample bar, the numbers on the y axis are associated with the location of the sample (position 1 is the top compartment of the middle six, position 6 is the bottom). As it was previously determined that thermocouple T11 records lower temperatures than the rest of the thermocouples, the data from T11 was not included. The error bars were determined with the same process as the ones in figure 5.5.

![Figure 5.6: Mean Temperature of Sample Bar in ELI by Sample Position](image-url)

With the exception of position 1 having an average recorded temperature 2.3° below 125°C, each position had an average recorded temperature within ±0.6°C of 125°C. The range of temperature measurements at each position is once again a source of uncertainty, but experimentation involving recording temperature with all of the thermocouple measurements at the same location on the sample bar still resulted in
ranges that indicate that it is the thermocouples responsible for the variation recorded
temperatures, not the position on the sample bar. This also supported by the fact
that the variation in recorded temperature at each position is roughly equal, with
"roughly equal" defined as the largest standard deviation of recorded temperatures is
no more than double the smallest standard deviation of recorded temperatures. The
position with the largest standard deviation, position 1, is less than double of the
smallest standard deviation, which is the temperatures recorded at position four.

The distribution of recorded temperatures at all of the positions resembled normal
distributions, so a one way ANOVA F test was able to be done to determine if there
is a true difference in temperature of the sample bar depending on the location. The
treatment sum of square (SSTr) and error sum of squares (SSE) are calculated.

\[ SSTr = \sum n_i (x_i - \bar{x})^2 \]  \hspace{1cm} (5.1)

\[ SSE = \sum (n_i - 1)s_i^2 \]  \hspace{1cm} (5.2)

In this case \( n_i \) is the number of temperature recordings from sample location \( i \), \( x_i \)
is the average recorded temperature from sample location \( i \), \( s_i^2 \) is the variance of
the recorded temperatures from sample location \( i \), and \( \bar{x} \) is the weighted average
temperature measurements at all six sample locations, with \( n \) representing the total
number of measurements recorded from all of the sample locations being analyzed.

\[ \bar{x} = \sum \left( \frac{n_i}{n} \right) \bar{x}_i \]  \hspace{1cm} (5.3)
Using the SSTr, the SSE, and the degrees of freedom between the sample locations and within each sample location, the mean square for treatments (MSTr) and the mean square error (MSE) can be computed. The F-statistic is a ratio of these mean squares.

\[ MSTr = \frac{SSTr}{k - 1} \quad (5.4) \]

\[ MSE = \frac{SSE}{n - 1} \quad (5.5) \]

\[ F = \frac{MSTr}{MSE} \quad (5.6) \]

\( k \) is the number of groups being compared (six sample slots). With the degrees of freedom in the treatments \((k - 1)\) and the degrees of freedom in the error \((n - k)\), an F distribution curve can be generated. If using a significance level of \( \alpha = 0.05 \), a critical F statistic can be determined where the area under the curve from F statistics equal to the critical F statistic and greater is 5% of the total area under the F distribution curve. An F statistic greater than or equal to this critical value would indicate that there is evidence that at least one of the sample slot positions on ELI used in the test sequences has a different temperature than the others while ASTM E595 tests are performed.

The F distribution curve as a result of the degrees of freedom from these tests is shown in figure 5.7. The critical F statistic value is calculated to be 2.33, while the F statistic from the thermocouple measurements at the center six sample location is 1.8.
While the ANOVA test indicates that there is not enough evidence to support that there is a difference in temperature on the sample bar depending on the location, the variation in the recorded temperature due to the thermocouples themselves is large and contributes to the low F-statistic. This conclusion was also made in previous work by Tyler Harty [24]. Each thermocouple was either not randomly assigned to all six positions throughout all of the test sequences performed or one of the thermocouples was only at a certain position a singular time throughout all of the test sequences. Therefore, the recorded temperature based on location on the sample bar could not be analyzed for statistical significance based on the recordings of one thermocouple, which would reduce the uncertainty due to the variability of measurements from different thermocouples.
To further quantify the variation of the thermocouples an experiment was set up where Thermocouples T9, T11, T14, and T15 were secured with Kapton® tape to the inside of a PASCO Scientific TD-8554A thermal radiation cube. A light bulb inside the cube causes the cube to increase in temperature, and plugging a multimeter into the cube with banana connectors allows for reading of the temperature of the cube using the resistance. A laser thermometer was also used as another source of temperature measurement. Table 5.6 includes the resulting thermocouple measurements compared to the temperatures recorded by the multimeter and the laser thermometer. The thermocouple reader read values as K-type rather than T-type, but the variation of the thermocouple measurements compared to the consistency of the multimeter and thermometer further supports that the thermocouples themselves are the reason for the wide variation in the recorded temperatures in ELI.

Table 5.6: Comparison of Thermocouple Readings to Multimeter and Thermometer Readings [°C]

<table>
<thead>
<tr>
<th>Thermocouple</th>
<th>Thermocouple</th>
<th>Multimeter</th>
<th>Thermometer</th>
</tr>
</thead>
<tbody>
<tr>
<td>9</td>
<td>70.7</td>
<td>85.0</td>
<td>85.3</td>
</tr>
<tr>
<td>11</td>
<td>79.3</td>
<td>84.0</td>
<td>83.6</td>
</tr>
<tr>
<td>14</td>
<td>73.5</td>
<td>84.0</td>
<td>84.1</td>
</tr>
<tr>
<td>15</td>
<td>70.5</td>
<td>84.0</td>
<td>84.2</td>
</tr>
</tbody>
</table>

The TML of a sample is dependant on the temperature of the sample, and since the CVCM is dependant on the TML, CVCM is also inherently dependant on the temperature of the sample. Comparing TML and CVCM from the initial ASTM E595 tests in the test sequences for each material (shown in figure 5.8), there was no correlation between temperature and outgassing except between the recorded temperature and CVCM of CV-2500, which had a moderate correlation ($r = 0.64$).

A factor that could influence the recorded TML and CVCM of samples is the total time samples were in vacuum with the Variac on throughout the test sequence. Variation in this time was a result in the variation of time it took for the sample
bar to reach 125°C. The greatest difference in time that samples were in vacuum at elevated temperatures was between the first and last test of SCV2-2590. The samples in the last test sequence of SCV2-2590 were in vacuum at elevated temperature for 24 minutes longer than the samples in the first test, but this was only a 0.55% increase in duration. Seeing as the average TML for both the control samples and samples exposed to atomic oxygen in the first test sequence was slightly greater than the final test sequence, the amount of time in vacuum was disregarded as a source of variation.

It had been known that the fluence of atomic oxygen received by the ground plate in MAX for a certain duration of time is not spatially uniform, namely the fluence steadily decreases as a function of distance away from the center of the electrode [24]. This had never been quantified. An experiment was performed where 32 2cmx2cm
Kapton® HN samples were laid out in the slotted ground plate as shown in figure 5.9 and exposed to atomic oxygen in the same conditions that the silicone samples were in (ASTM E2089).

Figure 5.9: Layout of Kapton® HN Samples in MAX to Quantify a Beam Map

After calculating the effective fluence that each Kapton® HN sample received, another test where only 16 samples were used were combined with the results from this test to produce the beam map observed in figure 5.10. The center samples (rows four and five and columns two and three) were assumed to be 7.62cm away from
the center of the AO source (the gap distance between the RF plate and the ground plate). With the samples laid out side by side, the samples farthest from the center (the corners of the beam map) were calculated to be 9.90cm away from the center of the AO source.

Figure 5.10: Map of Effective Fluence Values as a Result of ASTM E2089 Tests in MAX [atoms/cm$^2$]

The Silicone samples were as close to the center of the electrode as possible in each of the slots, which would be where the Kapton® HN samples contributing to rows

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four and five and columns two and three on the beam map were located. The witness samples in each test sequence were located farther away from the center of the center of the electrode, with one of the witness samples being between rows six and seven and columns two and three and the other witness sample between rows two and three and columns two and three. Comparing the average of the fluence values in these regions, it is predicted that the witness samples received approximately 85% of the atomic oxygen fluence that the samples were bombarded with. The predicted effective fluence the samples witnessed for each test sequence, as well as the effective fluence recorded from the witness samples, are displayed in table 5.7.

<table>
<thead>
<tr>
<th>Test</th>
<th>Witness Sample 1</th>
<th>Witness Sample 2</th>
<th>Predicted Fluence</th>
</tr>
</thead>
<tbody>
<tr>
<td>CV-2500</td>
<td>1.27 * 10^{21}</td>
<td>1.19 * 10^{21}</td>
<td>1.47 ± 0.13 * 10^{21}</td>
</tr>
<tr>
<td>CV-2289-1</td>
<td>1.01 * 10^{21}</td>
<td>8.89 * 10^{20}</td>
<td>1.13 ± 0.13 * 10^{21}</td>
</tr>
<tr>
<td>CV-2289-1</td>
<td>1.26 * 10^{21}</td>
<td>1.27 * 10^{21}</td>
<td>1.51 ± 0.08 * 10^{21}</td>
</tr>
<tr>
<td>SCV2-2590</td>
<td>1.33 * 10^{24}</td>
<td>1.28 * 10^{24}</td>
<td>1.55 ± 0.11 * 10^{21}</td>
</tr>
<tr>
<td>CV-2500</td>
<td>1.21 * 10^{24}</td>
<td>1.30 * 10^{24}</td>
<td>1.49 ± 0.03 * 10^{21}</td>
</tr>
<tr>
<td>CV-2289-1</td>
<td>1.23 * 10^{24}</td>
<td>1.28 * 10^{24}</td>
<td>1.49 ± 0.05 * 10^{21}</td>
</tr>
<tr>
<td>SCV2-2590</td>
<td>1.24 * 10^{24}</td>
<td>1.20 * 10^{24}</td>
<td>1.48 ± 0.13 * 10^{21}</td>
</tr>
<tr>
<td>CV-2500</td>
<td>1.29 * 10^{24}</td>
<td>1.30 * 10^{24}</td>
<td>1.54 ± 0.08 * 10^{21}</td>
</tr>
<tr>
<td>SCV2-2590</td>
<td>1.25 * 10^{24}</td>
<td>1.18 * 10^{24}</td>
<td>1.45 ± 0.12 * 10^{21}</td>
</tr>
<tr>
<td>SCV2-2590</td>
<td>1.27 * 10^{24}</td>
<td>1.24 * 10^{24}</td>
<td>1.49 ± 0.10 * 10^{21}</td>
</tr>
</tbody>
</table>

With the exception of CV-2500, there was no correlation between the predicted atomic oxygen fluence the samples received and the TML and CVCM of the samples afterwards (figure 5.11). Even with the correlation observed in CV-2500 ($r = 0.63$ for TML and $r = 0.68$ for CVCM), the measure of uncertainty in one of the tests ($1.49 ± 0.03 * 10^{21} \text{atoms/cm}^2$) is completely within the bounds of uncertainty of another test ($1.47±0.13*10^{21} \text{atoms/cm}^2$). The magnitude of uncertainty in the predicted atomic oxygen fluence allows for no conclusions to be made about how the variation in the amount of atomic oxygen received by the samples affects their outgassing afterwards.
Besides assessing the precision of the measurements, p-values with a significance value of $\alpha = 0.05$ was used to determine the statistical significance of the difference in mean TML and CVCM for each material. For clarification, the statistic being considered is the difference means between the TML and CVCM of the samples exposed to atomic oxygen and the control samples for each material ($\bar{\mu}_{AO} - \bar{\mu}_C$).

The null hypothesis is that the effect of atomic oxygen on the TML and CVCM of these three silicone materials is $\geq 0$ ($\bar{\mu}_{AO} - \bar{\mu}_C = 0$). The alternate hypothesis is that the effect of atomic oxygen on the TML and CVCM of these three silicone materials is $\leq 0$, or $\bar{\mu}_{AO} - \bar{\mu}_C \leq 0$.

To calculate a p-value, an unpooled T-statistic is first calculated using equation 5.7.
\[ t = \frac{\bar{\mu}_{AO} - \bar{\mu}_C}{\sqrt{\frac{s^2_{AO}}{n_{AO}} + \frac{s^2_C}{n_C}}} \]  

(5.7)

\( S \) is the standard deviation of either the samples exposed to atomic oxygen or the control samples and \( n \) is the number of samples for either AO exposed or control. This T-statistic considers the random variation in the test statistic being analyzed \((\bar{\mu}_{AO} - \bar{\mu}_C)\) arising from randomness in the data collection process. It depends on the variation in the data \((S)\) and sample sizes \((n)\), and is advantageous as it reflects the sample size of the study.

From this T-statistic a p-value can be determined by approximating a null distribution of T-statistics. In the case of this work, a single tailed p-value is to be calculated, meaning that rather than the p-value indicating evidence that atomic oxygen has any effect on the TML and CVCM of these materials after exposure (either increase or decrease in TML and CVCM), the p-value will indicate if there is evidence that atomic oxygen has an effect on the TML and CVCM of these materials in one direction after exposure (decrease in TML and CVCM). The number of T-statistics from the null distribution less than the unpooled T-statistic originally counted is calculated as a percentage of the total number of T-statistics in the null distribution, and if that number is less than the significance value \((\alpha = 0.05)\) then it is said that there is enough evidence to reject the null hypothesis. In this work, that would mean there would be enough evidence to support that atomic oxygen exposure reduces future TML and/or the CVCM of these silicone materials.

The null distribution of T-statistics can be approximated from a mathematical model if three validity conditions are met. The first condition is that the samples are independent of each other. The second condition is that sample standard deviations are roughly equal. In this sense ”roughly equal” means the standard deviation of the
group with the greatest standard deviation is not greater than twice the standard deviation of the other group. The third condition is that the distribution of the samples for each group is roughly symmetric or that both groups have at least 20 samples without a strong skew or outliers in the distribution.

All of the samples in the test sequences were randomly assigned to be exposed to atomic oxygen or to be used as a control. Also, all of the samples and all of the UHV aluminum foils were randomly assigned to positions on the sample bar and collector disk plate. As such the samples are independent of each other due to random assignment. The greatest difference in standard deviation of either TML or CVCM of all materials was the TML of the AO exposed samples and control samples of CV2-2289-1, with the standard deviation of the control samples being 1.5 times greater than the standard deviation of the samples exposed to atomic oxygen. Therefore the second validity condition is met.

Not all of the TML or CVCM data for each material had a symmetric distribution, and none of the sample sizes were greater than or equal to twenty. It was determined that the third validity condition was not met and that the single tailed p-value could not be calculated as described above.

Instead, a simulation was run to approximate the null distribution of T-statistics [51]. To do this it is assumed that the null hypothesis is true. If exposure to atomic oxygen does not reduce the TML or CVCM of these materials and the variation in data collected was due to randomness alone, then the data from the samples exposed to atomic oxygen and the data from the control samples could be shuffled between each other. Figure 5.12 shows an example of this shuffling.

With this shuffled data, a new T-statistic is calculated. This process is iterated many times, and eventually a distribution of T-statistics is created assuming the null
hypothesis is true. The center of this null distribution in this case should be zero as the null would assume no effect ($\bar{\mu}_{AO} - \bar{\mu}_{C} = 0$). With this null distribution a simulated single tailed p-value can be calculated rather than a theoretical p-value.

For each difference of means, a null distribution of ten thousand T-statistics was created to create the simulated p-values. Figure 5.13 shows an example of a null distribution created using the TML of the AO exposed and control SCV2-2590 samples.

Table 5.8 contains both the unpooled T-statistics and p-values for the effect of atomic oxygen on both the TML and CVCM of the three materials.
The p-values indicate that there is evidence with a 95% confidence level that the effect of atomic oxygen on CV-2500 and CV2-2289-1 is a reduction in the total mass loss from outgassing after the material has been exposed to atomic oxygen. However, in terms of CVCM, only the simulated p-value for CV-2500 indicated that there was evidence that the effect of atomic oxygen reduces the CVCM from outgassing after the material has been exposed to atomic oxygen.
5.2 FTIR

The FTIR results of control samples vs samples that were exposed to atomic oxygen after a test sequence was complete are shown in figures 5.14, 5.15, and 5.16. While the chemical composition of the material was not provided and is unknown, the fact that all of these materials were silicones allowed for identification for a majority of the peaks. For reference, polydimethylsiloxane (PDMS), the most extensively used polysiloxane, consists of an Si-O-Si backbone and CH3 sidechains [38]. For functional groups, peaks in the range of 2840-3000 cm\(^{-1}\) are indicators of C-H stretching of alkanes (hydrocarbon compounds where the carbon bonds are single bonds), and peaks at 2960 cm\(^{-1}\) are indicative of aliphatic hydrocarbon compounds, specifically methyl (CH3) [46]. Considering the three materials being tested, it can be safely concluded that the peak at 2961 cm\(^{-1}\) for all three is attributed to methyls.

For the other peaks that are not associated with functional groups, results of IR analysis of organosilicon compounds that have been compiled was referenced for peak identification in CV-2500, CV2-2289-1, and SCV2-2590 [30]. Table 5.9 has some wavenumber to peak associations from this work. Si-CH3 groups are easily recognizable from a strong sharp peak at about 1260 cm\(^{-1}\) with one or more strong peaks in the range of 865-750 cm\(^{-1}\) [30]. Blocks of dimethyl units (siloxane chain with two methyls bonded to each silicon) will show peaks at 860 cm\(^{-1}\), and if the dimethyl units are randomized and not in a uniform chain, the peak will shift to 845 cm\(^{-1}\). Siloxanes will show one or more very strong peaks in the region of 1130-1000 cm\(^{-1}\), and as siloxane chains become longer or branched the peaks become broader and overlap.

<table>
<thead>
<tr>
<th>Group</th>
<th>Wavenumber(s) [cm(^{-1})]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Si-CH(_3)</td>
<td>1275-1245, 865-750</td>
</tr>
<tr>
<td>Si-O-Si</td>
<td>1130-1000</td>
</tr>
</tbody>
</table>
Figure 5.14: FTIR Spectrum of CV-2500 Control Samples and Samples Exposed to Atomic Oxygen

With this information, the peaks at 1257 cm\(^{-1}\) and 786 cm\(^{-1}\) for the three silicones tested are indicative of Si-CH\(_3\) bonds, and the smaller peaks observed by 865 and 845 cm\(^{-1}\) are indicative of Si-(CH\(_3\))\(_2\). The indication of methyls from the peak at 2961 cm\(^{-1}\) further supports this. The strong peak at 1010 cm\(^{-1}\) and the peak in the 1070-1060 cm\(^{-1}\) range that overlaps and appears as shoulder is representative of Si-O-Si. Comparing the spectra from CV-2500, CV2-2289-1, and SCV2-2590 to the spectra of Esterline ELA-SA-401 recorded by Henry deGroh et al. (where the peaks at 1260, 796, and near 860 cm\(^{-1}\) were identified as Si-CH\(_3\), particularly dimethyl units, and the peaks near 1020 and 1070 cm\(^{-1}\) were identified as Si-O-Si) supports that these bonds are present in the samples tested in this thesis. The solid blue line in figure
Figure 5.15: FTIR Spectrum of CV2-2289-1 Control Samples and Samples Exposed to Atomic Oxygen

5.17 represents the control group spectrum while the dotted orange line represents the spectrum of the group exposed to atomic oxygen.

The conclusion that methyl groups were being replaced with oxygen in Esterline ELEA-SA-401 due to atomic oxygen interactions was made as the Si-O-Si peaks increased relative to the Si-CH3 peaks when the o-rings were exposed to atomic oxygen [15]. Comparison of Si-O-Si peaks to Si-CH3 peaks in the three materials was done by taking advantage of Beers law and calculating the ratio of Si-O-Si and Si-CH3 peaks for the control and AO exposed samples. The same conclusion that methyl groups are being replaced by oxygen is made as the absorption, and therefore concentration of the Si-CH3 peaks decreased with atomic oxygen exposure more than the Si-O-
Si peaks. The IR spectra successfully verifies that atomic oxygen, the only factor different between the two groups of samples for each material, created a change in the chemical composition of the materials. Table 5.10 is the percent reduction of peaks for Si-CH3 and Si-O-Si due to atomic oxygen exposure, calculated as \(1 - \frac{A_{AO}}{A_C}\), where \(A_{AO}\) is the absorbance value of the peak for groups exposed to atomic oxygen and \(A_C\) is the absorbance value of the peak for the control groups. Occasionally there is a slight difference in wavenumbers for a specific peak between the AO-exposed and control samples for a material, on the order of one to two wavenumbers. As such the maximum absorbance value of a peak for the control groups and the maximum absorbance value of the same peak for the AO-exposed groups were used to calculate the percent reduction, rather than the absorbance values at the same exact wavenumber. The
other Si-O-Si peak from 1070-1060 cm\(^{-1}\) was left out as the maximum value of the peak is difficult to determine due to the overlapping of the Si-O-Si peaks, but by looking at the spectra it can be concluded that there is no percent reduction in the peak when exposed to atomic oxygen and that for CV-2500 there is even a slight increase in Si-O-Si concentration. Comparing the difference in peaks 786 cm\(^{-1}\) and 1010 cm\(^{-1}\), FTIR for control samples showed a 19.7% difference in absorbance for CV-2500, a 13.3% difference in absorbance for CV2-2289-1, and a 15.2% difference in absorbance for SCV2-2590. FTIR for AO-exposed samples showed a 6.8% difference in absorbance for CV-2500 and a 6.4% difference in absorbance for both CV2-2289-1 and SCV2-2590. The relative peak difference between 786 cm\(^{-1}\) and 1010 cm\(^{-1}\) decreased by 12.9%, 6.9%, and 8.8% for CV-2500, CV2-2289-1, and SCV2-2590 respectively.

Figure 5.17: FTIR Spectra Comparison of CV-2500, CV2-2289-1, SCV2-2590, and Esterline ELA-SA-401
which is increase in Si-O-Si relative to Si-CH3 as the peak at 786 cm$^{-1}$ is greater than the peak at 1010 cm$^{-1}$.

**Table 5.10: Peak Reduction in Silicone IR Spectrum due to Atomic Oxygen**

<table>
<thead>
<tr>
<th>Wavenumber [cm$^{-1}$]</th>
<th>CV-2500</th>
<th>CV2-2289-1</th>
<th>SCV2-2590</th>
</tr>
</thead>
<tbody>
<tr>
<td>1010 (Si-O-Si)</td>
<td>8.2%</td>
<td>11.1%</td>
<td>14.0%</td>
</tr>
<tr>
<td>1257 (Si-CH3)</td>
<td>43.3%</td>
<td>41.3%</td>
<td>42.7%</td>
</tr>
<tr>
<td>786 (Si-CH3)</td>
<td>18.2%</td>
<td>21.7%</td>
<td>19.8%</td>
</tr>
</tbody>
</table>
Chapter 6

CONCLUSION

Understanding how a material will react to the space environment is vital in order to protect spacecraft and payloads from unwanted anomalies. This means considering individual as well as combined aspects of the space environment and their effects on material properties is critical.

Samples of silicones CV-2500, CV2-2289-1, and SCV2-2590 were exposed to atomic oxygen in a vacuum chamber that produces low-energy isotropic atomic oxygen. They were then relocated to another vacuum chamber along with control samples that had not been exposed to atomic oxygen. These samples were then outgassed at elevated temperatures. The total mass loss of each sample and volatile condensable mass collected from each sample was recorded, and for all samples there was an observed trend supporting that samples exposed to atomic oxygen outgassed less.

The outgassing tests performed could not adhere to all of the requirements of ASTM E595, and the variation in recorded TMLs and CVCMs for each test did not satisfy the precision defined in the standard. The microbalance scale was not calibrated with a set of standard weights, so the true TML and CVCM of these samples is unknown. The current facilities available at Cal Poly and methodologies used are not capable of producing test results with variability as low as would be expected from a facility competently testing materials with ASTM E595.

Even with error in the outgassing method and the uncalibrated scale, the data is still useful and provides insight to outgassing performance of these three silicones after they have been exposed to atomic oxygen. For each test the samples exposed to atomic
oxygen and the control samples, which were all randomly assigned, were exposed to the same pressures for the same duration of time in and out of the vacuum chamber, experienced the same temperatures, and were weighed using the same uncalibrated scale, allowing the recorded mass loss to be comparable between the control and AO-exposed groups. This leads to the conclusion that the exposure of atomic oxygen resulted in a trend of decreasing TML in the three silicones. A trend in decreasing CVCM is observed for CV-2500 and SCV2-2590, but not CV2-2289-1.

The observed decrease can be explained by the conversion of the silicone surface to silica as the atomic oxygen removes hydrocarbon content and forms bonds with the siloxane chain. This change in chemical composition on the surface results in reduction of contaminants escaping and outgassing from the silicones. This conversion to silica was confirmed with FTIR which showed an increase in Si-O-Si relative to Si-CH3.

These observed trends in decreased outgassing could be useful for preparation of these silicone materials before use in space. The atomic oxygen chamber simulates forty weeks in low Earth orbit while the outgassing chamber simulates one day, so the gradual and uneven conversion of silicone to silica on orbit would not aid in reducing the silicone’s outgassing. However, these silicones can be pretreated with atomic oxygen while being integrated into the spacecraft to reduce the amount of contaminants from exposed surfaces of the silicone outgassing and condensing on other surfaces. This would be dependant on the application of the silicone, as the increased brittleness of silica might be undesirable for the silicone’s purpose. While these silicone materials are well below the normal screening level of 1.0% TML and 0.1% CVCM, any amount of outgassing and the possibility of contamination is unwanted and should be avoided. This thesis shows a trend supporting that exposing CV-2500, CV2-2289-1,
and SCV2-2590 to atomic oxygen before use in space can reduce the TML of these low-outgassing silicones further.
For the ELI chamber at Cal Poly to be able to run ASTM E595 tests with competency, the most important change that needs to be made is to replace the thermocouples that are currently used in the chamber. The test with the radiation cube has shown that the current thermocouples read values at least 5°C lower than two different temperature measurement sources that were consistent with each other, and the variation between thermocouple readings is greater than the ±1°C that is required by the standard. The wires are visually degraded, and half of the thermocouples that are present in ELI are not functional.

Fourteen high accuracy T-type thermocouples with an accuracy of ±0.5°C have been purchased from TIP Temperature Products and are currently in a container labeled "Harty" on top of shelf in the northeast corner of the space environments laboratory. The current thermocouples are fed into ELI through a feedthrough that has the standard dimensions of neither CF nor KF flanges. This feedthrough is filled with a potting compound of an unknown cured substance to form a seal and prevent leaks.

In order to remove the current thermocouples from ELI, two options are considered. One would be to find a way to dissolve the cured potting compound without damaging the feedthrough and without using chemicals that the university would deem too hazardous to for students to use. Heating up the cured sealants with a heat gun and applying a solvent such as acetone or denatured alcohol removes cured epoxy and could have success with this feedthrough. Proper safety precautions should be taken. Since the substance is unknown a respirator is recommended if it is decided
to use heat to loosen the potting compound as unknown vapors might be released. The second option would be to purchase a custom sized feedthrough, although this option will require funding that may not be available for the laboratory. For either option, the new thermocouples would need to be resealed in the feedthrough once the old thermocouples are removed. Two recommended sealants to use would be Hysol 1C high vacuum epoxy solvent or PRO™ Epoxy model EQ-FH-EPOXY, which are intended for gas and electrical feedthroughs.

Reducing the variation in temperature readings with these new thermocouples will help ensure that the sample bar in ELI is at 125°C when testing samples. These new thermocouples should also be tested with the radiation cube in the same manner that the current thermocouples were to validate that the thermocouple readings agree with each other and other temperature measurement sources. With confidence in the performance of thermocouples, the variation in TML and CVCM of samples of the same material can be further explored. Potential sources of variation, such as a true uneven distribution of temperature along the sample bar or collector plates, or variation in Variac output between tests, can be further investigated.

Another major repair that needs to be made to ELI is the installation of a new hoist motor. Early in this thesis the limit switches failed and the previous motor was destroyed. Current operation of the hoist involves using a handheld drill to rotate the motor shaft in order to raise or lower the hoist. A new motor has been purchased, and the last step is to install new limit switches into the hoist in order to use the new motor.

The scale should be calibrated before every test or test sequence with a set of standard weights. These should either be purchased or borrowed from another department at the university.
With improvements made to ELI that would allow tests to adhere to ASTM E595 more closely and reduce variation in TML and CVCM between samples of the same condition, test sequences with CV-2500, CV2-2289-1, and SCV2-2590 should be repeated. This will allow for lower p-values and a higher level of confidence in the difference of means between the AO-exposed samples and samples exposed to atomic oxygen. Another effort that should be pursued is to test silicones that are known to have TML > 1.00% and CVCM > 0.10%. The restrictions on materials spacecraft designers can use, depending on the application, can be eased if samples of these silicones exhibit TML < 1.00% or CVCM > 0.010% after being exposed to atomic oxygen.

While the fluence map for MAX was useful to approximate the fluence of atomic oxygen that each sample received, the current fluence map is the result of only two tests. More tests using Kapton® HN squares should be performed and compiled with the current fluence map to obtain a better estimation of the fluence samples in the center of MAX’s ground plate will receive in 24 hours. If exposing samples to the highest fluence of atomic oxygen in 24 hours is not a concern, placing samples as close to the witness sample slots as possible would provide the most accurate fluence the samples have received. Another characterization of the atomic oxygen in MAX besides the spatial distribution of fluence that should be investigated is the spatial distribution of atomic oxygen energy. It is currently unknown if there are regions of the ground plate that receive higher energy atomic oxygen than other regions.

The task of ensuring the camber pressure in MAX is 175±10mTorr can be automated if an active control system was implemented. This controller could adjust how much a valve is open or shut depending on the inputted chamber pressure it is reading.

More testing on Silicone response to AO exposure in MAX could be studied, such as the roughness of the silicone with isotropic AO rather than unidirectional AO. A
profilometer can be used to measure the peaks and troughs created in the material, and this could be compared to literature on silicones exposed to unidirectional AO at 4.5eV.

Additional testing with extra parameters could be applied to better understand the outgassing nature of silicones with combined space environment effects. MAX has a UV lamp that can be installed inside the chamber. As discussed earlier in this thesis the combination of UV and AO is known to create darkened silica, and this combination could be tested to see if the change in coloration affects how the samples outgas. A great test that could be added to the test sequences performed in this thesis would be to obtain FTIR spectra of the foils that outgassed matter has condensed on to determine the species of contaminant that had condensed. The magnitude of the peaks observed from this spectra would be very small compared to peaks of the spectra presented in this thesis, and the spectra of aluminum foil with no contamination would need to be collected. This spectra could be subtracted from the contaminated foil’s spectra in order to observe the peaks due to the contaminants. This could also be another method to verify if matter that condensed on the foils was from the sample or from contamination due to a poorly cleaned and baked out chamber.

Knowing that atomic oxygen has shown a trend in reducing the outgassing of CV-2500, which is used as an adhesive for coverglass and solar arrays, work should be done to determine if the exposing CV-2500 to AO would effect the power solar arrays would produce. Both CV-2500 and SCV2-2590 had a glossy finish after being exposed to atomic oxygen, and with visual observation it is clear that samples are slightly less transparent. It would be useful to study how much the transmittance of CV-2500 in the visible light range is reduced and how much the power generation of a solar panel with a cover glass and AO-exposed CV-2500 as an adhesive affected.
BIBLIOGRAPHY


[27] JASCO. Ftir specifications, Jul 2022.


[31] Lesker. Turbovac i(x) operating instructions.


[34] N. T. LLC. Cv-2500 controlled volatility silicone elastomer, 2022.


FTIR Spectra of samples that had been outgassed, samples that had been exposed to atomic oxygen, and samples that had been exposed to atomic oxygen and outgassed through the test sequence were collected. Spectra of samples that had never been in vacuum nor exposed to atomic oxygen, as well as the control samples in the test sequences, were also collected. Upon comparison, it was determined that outgassing did not alter the spectra, and only atomic oxygen resulted in a change to the peaks. It was planned to have the spectra of three samples recorded after a test sequence where samples were both outgassed and exposed to atomic oxygen. However, for CV2-2289-1, the test sequence that the spectra was recorded from was the test sequence with two samples exposed to atomic oxygen.
Figure A.1: CV-2500 Outgassed, No AO Exposure 4/11/22
Figure A.2: CV-2500 Not Outgassed, No AO Exposure 4/11/22
Figure A.3: CV-2500 AO Exposed, Not Outgassed 2/27/22
Figure A.4: CV-2500 Not Outgassed, No AO Exposure 2/27/22
Figure A.5: CV-2500 Outgassed and AO Exposed 3/10/22
Figure A.6: CV-2500 Not Outgassed, AO Exposed 3/10/22
Figure A.7: CV2-2289-1 Outgassed, No AO Exposure 4/11/22
Figure A.8: CV2-2289-1 Not Outgassed, No AO Exposure 4/11/22
Figure A.9: CV2-2289-1 AO Exposed, Not Outgassed 2/27/22
Figure A.10: CV2-2289-1 Not Outgassed, No AO Exposure 2/27/22
Figure A.11: CV2-2289-1 Outgassed and AO Exposed 2/4/22
Figure A.12: CV2-2289-1 Not Outgassed, No AO Exposure 1/14/22
Figure A.13: SCV2-2590 Outgassed, No AO Exposure 4/11/22
Figure A.14: SCV2-2590 Not Outgassed, No AO Exposure 1/14/22
Figure A.15: SCV2-2590 AO Exposed, Not Outgassed 2/27/22
Figure A.16: SCV2-2590 Not Outgassed, No AO Exposure 2/27/22
Figure A.17: SCV2-2590 Outgassed and AO Exposed 3/4/2022
Figure A.18: SCV2-2590 Not Outgassed, No AO Exposure 3/4/2022
Appendix B

INITIAL OUTGASSING RESULTS

The following plots contain the TML and CVCM values recorded from the initial ASTM E595 test in each test sequence. The CVCM recorded for one of the samples during the CV-2500 test from 12/18/21-12/22/21 was negative and not included in this data. The most probable reason is that a piece of the UHV foil broke off during the test or while measuring the mass. Similar to the outgassing results from the second ASTM E595 test in each test sequence, the precision for both TML and CVCM for all three materials does not meet the standard.

Figure B.1: CV-2500 Initial ASTM E595 Results
Table B.1: Initial Outgassing Values and Precision

<table>
<thead>
<tr>
<th>Material</th>
<th>Mean [%]</th>
<th>Standard Deviation [%]</th>
<th>Precision [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>CV-2500 TML</td>
<td>0.0885</td>
<td>0.0190</td>
<td>21.5</td>
</tr>
<tr>
<td>CV-2500 CVCM</td>
<td>0.0215</td>
<td>0.0101</td>
<td>47.0</td>
</tr>
<tr>
<td>CV2-2289-1 TML</td>
<td>0.4613</td>
<td>0.0561</td>
<td>12.2</td>
</tr>
<tr>
<td>CV2-2289-1 CVCM</td>
<td>0.0523</td>
<td>0.0156</td>
<td>29.8</td>
</tr>
<tr>
<td>SCV2-2590 TML</td>
<td>0.0547</td>
<td>0.0074</td>
<td>13.5</td>
</tr>
<tr>
<td>SCV2-2590 CVCM</td>
<td>0.0072</td>
<td>0.0034</td>
<td>47.2</td>
</tr>
</tbody>
</table>
Figure B.3: SCV2-2590 Initial ASTM E595 Results
Appendix C

ATOMIC OXYGEN EROSION YIELD DATA

The following are the erosion yields, or reaction efficiencies, calculated for all of the samples exposed to atomic oxygen. This was done using the predicted fluence the samples received which was calculated from the the effective fluence of the Kapton®HN samples assuming an erosion yield in LEO for Kapton®HN of $2.81 \times 10^{-24} \text{cm}^3/\text{atom}$. One of the samples exposed to atomic oxygen in the final test sequence for CV-2500 lost no recordable mass due to AO erosion, so that data point was not included. An SCV2-2590 sample that was exposed to atomic oxygen actually gained mass. This data point was not included either as the rest of the SCV2-2590 samples lost mass due to atomic oxygen erosion.

Table C.1: Erosion Yield of CV-2500 Samples

<table>
<thead>
<tr>
<th>Samples</th>
<th>Erosion Yield [$\text{cm}^3/\text{atom}$]</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>$1.29 \times 10^{-26}$</td>
</tr>
<tr>
<td>2</td>
<td>$1.94 \times 10^{-26}$</td>
</tr>
<tr>
<td>3</td>
<td>$1.12 \times 10^{-26}$</td>
</tr>
<tr>
<td>4</td>
<td>$8.34 \times 10^{-27}$</td>
</tr>
<tr>
<td>5</td>
<td>$7.30 \times 10^{-27}$</td>
</tr>
<tr>
<td>6</td>
<td>$4.35 \times 10^{-27}$</td>
</tr>
<tr>
<td>7</td>
<td>$5.44 \times 10^{-27}$</td>
</tr>
<tr>
<td>8</td>
<td>$5.24 \times 10^{-27}$</td>
</tr>
<tr>
<td>9</td>
<td>$2.10 \times 10^{-26}$</td>
</tr>
<tr>
<td>Average</td>
<td>$1.06 \times 10^{-26}$</td>
</tr>
</tbody>
</table>
Table C.2: Erosion Yield of C2-2289-1 Samples

<table>
<thead>
<tr>
<th>Samples</th>
<th>Erosion Yield [cm$^3$ atom$^{-1}$]</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>2.41 \times 10^{-26}</td>
</tr>
<tr>
<td>2</td>
<td>5.79 \times 10^{-27}</td>
</tr>
<tr>
<td>3</td>
<td>3.88 \times 10^{-26}</td>
</tr>
<tr>
<td>4</td>
<td>2.50 \times 10^{-26}</td>
</tr>
<tr>
<td>5</td>
<td>3.05 \times 10^{-26}</td>
</tr>
<tr>
<td>6</td>
<td>2.18 \times 10^{-26}</td>
</tr>
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<td>7</td>
<td>2.52 \times 10^{-26}</td>
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<tr>
<td>8</td>
<td>1.83 \times 10^{-26}</td>
</tr>
<tr>
<td>Average</td>
<td>2.37 \times 10^{-26}</td>
</tr>
</tbody>
</table>

Table C.3: Erosion Yield of SCV2-2590 Samples

<table>
<thead>
<tr>
<th>Samples</th>
<th>Erosion Yield [cm$^3$ atom$^{-1}$]</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>5.27 \times 10^{-27}</td>
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<td>2</td>
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</tr>
<tr>
<td>3</td>
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</tr>
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<td>8</td>
<td>2.78 \times 10^{-27}</td>
</tr>
<tr>
<td>9</td>
<td>9.94 \times 10^{-26}</td>
</tr>
<tr>
<td>10</td>
<td>8.21 \times 10^{-27}</td>
</tr>
<tr>
<td>11</td>
<td>1.08 \times 10^{-26}</td>
</tr>
<tr>
<td>Average</td>
<td>1.58 \times 10^{-26}</td>
</tr>
</tbody>
</table>
Appendix D

CV-2500 12/6/21-12/10/21 AND CV2-2289-1 1/28/22-2/1/22

The results of the two test sequences that were not included in the results due to differentiation in methodology are included here. No trends can be observed for the CV-2500 test sequence where the Variac was adjusted inappropriately. The CV2-2289-1 test sequence where the time samples were exposed to atomic oxygen was reduced still shows a trend in reduction of outgassing after the surface of the silicone is converted to silica.

Figure D.1: CV-2500 12/6/21-12/10/21
Figure D.2: CV2-2289-1 1/28/22-2/1/22
Appendix E

PROCEDURES

E.1 ELI Sample Preparation

1. Use nitrile gloves when handling samples and UHV aluminum foil.

2. Place the samples and baked out UHV foils (see ELI bakeout procedure) into separate compartments in the pill boxes and store them in the Tupperware® desiccator assembly with the HumidPak®, maintaining a relative humidity of 50 ± 5% for a minimum of 24 hours.

3. Measure the samples and UHV foils using the microbalance scale (see microbalance scale and antistatic gun procedure).

4. Wrap the collector disks in the UHV foil and ensure that the foil-wrapped collector disks are secured to the cold plate.

5. Insert samples into the appropriate sample compartments on the sample bar.

6. Screw the cover plates over the sample compartments being used while pinching a thermocouple between each cover plate and the sample bar.

E.2 ELI Pump Down

1. Check the interior of the chamber and ensure that the chamber is clear of debris and dust that may fall into the inlet feeding to the pumps. Clean the chamber if necessary.
2. Ensure that no cables or other components will fall into the inlet.

3. Using a handheld drill with a hex-socket attached to the shaft of the hoist motor, rotate the shaft counterclockwise to lower the bell jar and seal the chamber.

4. Visually check that no debris or wires are between the O-ring of the bell jar and the base of the chamber to ensure a proper seal.

5. Ensure that the vent valve and the valve for N2 gas insertion are shut.

6. Turn on the gauge controller by pressing the ON button on the front panel.

7. Check the oil level on the side of the Cacejun CVP 36 pump. If it is below the minimum level, inform Professor Saucier, Dr. Abercromby, or Cody, or if trained replace the oil yourself. Never operate the pump when it is below the minimum oil level.

8. Use the switch on the Cacejun CVP 36 pump to turn it on.

9. Monitor the chamber pressure as the chamber pumps down. Note the base pressure in the ELI logbook.

10. Once the chamber is below 50mTorr, check the lights on the side of the turbo pump to see if the turbo pump is ready for operation.

   (a) Top (green) light should be off.

   (b) Middle (yellow) light should be on and not flashing.

   (c) Bottom (red) light should be off.

11. If the red light is on, or if the yellow light is not on, do not attempt to operate the turbo pump and inform Dr. Abercromby or Professor Saucier.
12. If the green light is flashing, it maybe residual motion from the flow of air evacuating the chamber to the roughing pump. Wait until the green light is no longer flashing and is off.

13. Once the chamber is below 50 mTorr and the lights on the turbo pump indicate that the turbo pump is ready, turn on the turbo pump by moving the switch to the start position (down). The green light on the turbo pump will flash as the blades inside the pump are spinning up.

14. When the chamber pressure goes below $1 \times 10^{-3}$ Torr, the convectron gauge will no longer display the chamber pressure. Turn on the ion gauge to continue monitoring the chamber pressure by pressing the IG1 button on the front panel of the gauge controller.

15. Wait for the turbopump to reach its full speed and the chamber to reach the desired chamber pressure. The green light on the turbopump will be on and no longer flashing once the full operational speed of the turbopump has been reached.

- Note that the base pressure at the roughing pump will increase as the air is forced from the chamber into the roughing pump. This is normal. However, if the pressure at the roughing pump goes above 100 mTorr, turn off the turbo pump and inform Dr. A or professor Saucier.

**E.3 ELI Operation**

1. Once the chamber is below $5 \times 10^{-5}$ Torr, turn on the Variac® power supply and turn the knob to 30V.
2. Turn on the main switch (green) and the cooling switch (yellow) of the Brinkmann MGW Lauda RM water cooling unit.

3. Press and hold the button with the "v" symbol on the water cooling unit and turn the knob with the "v" symbol above it to adjust the desired temperature to 25°C. Release the button.

4. Monitor the temperature of the sample bar and the collector plate, as well as the pressure in the chamber, as increasing the temperature of the sample bar will increase the chamber pressure.

5. Once the sample bar is at 125°C, the cold plate at 25°C, and the chamber pressure is below $5 \times 10^{-5}$Torr, maintain these conditions for 24 hours. When recording temperatures with a thermocouple reader, ensure that one thermocouple is plugged into the thermocouple reader at a time.

### E.4 ELI Shutdown

1. If either the Variac® power supply or the water cooling unit are on, turn them off.

2. Turn the turbo pump off by moving the switch on the turbo pump to the stop position (up).

3. Turn the ion gauge off by pressing the IG1 button before the pressure in the chamber rises to 1mTorr.

4. The green light on the turbo pump will begin to flash as the blades slow down. When the green light is off the blades have fully stopped.

5. Once the turbo pump has completely stopped spinning (the green light is off), turn off the roughing pump by using the switch on the pump.
6. Slowly open the vent valve and let the chamber return to atmospheric pressure. Letting air in too fast will dry out O-rings and cause leaks. Once at atmospheric pressure, close the vent valve so that excess water vapor does not enter the chamber.

7. If samples or collector disks were inside the chamber, use the handheld drill and rotate the shaft of the hoist motor clockwise to lift up the bell jar and remove the samples and collector disks.

8. Complete the ELI logbook with the number of hours the roughing pump was in operation and the number of hours the oil was in use since its last replacement.

9. Ensure the bell jar is lowered if it was raised and that it is flush with the base of the chamber.

E.5 ELI Bakeout

1. Clean the collector disks, UHV foils, copper sample bar, separator plate, and cold plate with a 1:1 volume ratio mixture of acetone and ethanol using Kimwipes. Use nitrile gloves.

2. Dry out the collector disks and foils along with the sample bar, separator plate, and cold plate at 125 ± 5°C and less than 1mTorr for a minimum of sixteen hours. Follow the ELI pump down procedure.

3. Vent the chamber afterwards following the ELI shutdown procedure. Store the UHV aluminum foils in the Tupperware® desiccator assembly before the next test, and keep the copper collector disks in the test stand assembly in ELI.
E.6 MAX Sample Preparation

1. Use nitrile gloves when handling samples.

2. Cut two Kapton® HN witness samples into 5x1cm rectangles.

3. outgas witness samples in Junior for a minimum of 48 hours (see Junior procedure).

4. Once witness samples have been outgassed in Junior and samples removed from ELI, measure the mass of the samples and witness samples using the microbalance scale (see microbalance scale and antistatic gun procedure).

5. Raise the lid of MAX by moving the hoist switch on the front panel to the up position. Ensure that the coaxial caple connecting the RF power supply to the electrode is disconnected from the lid before moving the hoist.

6. Relax the lever that applies pressure to the glass wall by raising its handle. Remove the glass wall.

7. Unscrew the backing plates from the center witness slots and remove them. Place the witness samples on the backing plates and reattach the backing plates to the center slots.

8. Insert the samples into the main slots. Use aluminum shims to ensure the exposed faces of the samples are level with the witness samples.
E.7 MAX Pump Down

1. With the samples already in the slotted plate, place the glass wall back around the ground plate. Ensure that the two O-rings on the glass wall are properly fitted onto the wall.

2. Lower the handle of the lever to reapply pressure on the glass wall to ensure proper orientation of the glass wall.

3. Lower the lid using the hoist switch.

4. Attach the coaxial cable to the lid.

5. Ensure that all ports are closed, including the black nupro valve on the gas insertion line, the vent valve, and the valve to the roughing line.

6. On the front panel, flip the mechanical pump power and the pump power switches to the "on" position (up).

7. On the pump, press the red switch to the "on" position.

8. Once the base pressure at the pump has reached 150mTorr, open the roughing valve to the chamber by switching the vent valve switch on the front panel to the "on" position (up).

9. Monitor the roughing line and chamber pressures. Once the chamber pressure reaches 165mTorr open the gas insertion line by turning the nupro valve counterclockwise and induce a leak to the chamber.

10. Adjust the nupro valve until an equilibrium chamber pressure of $175 \pm 10\text{mTorr}$ is reached. Monitor the chamber for several minutes to ensure stability.
E.8 MAX Operation

1. Once MAX is pumped down and is at a stable equilibrium of 175 ± 10mTorr, turn on the R301 generator.

2. Set the power on the generator to 125 Watts.

3. Turn on the Mc2 controller.

4. Switch to manual adjustment mode and adjust the load and tune capacitors each to 50%.

5. Switch the adjustment mode back to auto for both tune and load. This is necessary or else the controller will not be able to produce the reflected power.

6. Check the chamber pressure to ensure it is still at 175 ± 10mTorr.

7. Turn on the RF power on the R301 generator.

8. The Mc2 will automatically adjust to find a stable point where the reflected power is 1 or 0 Watts. If at anytime the reflected power is greater than 0 Watts, manually adjust the tune and/or load until the reflected power is zero Watts. If it is not possible to obtain 0 or 1 Watt of reflected power, turn off the system and report to Dr. Abercromby.

9. Adjust the phase and magnitude to be 0 ± 25mV each. These can be adjusted by turning the potentiometers on the side of the AT3 unit manually with a flat-head screwdriver.

10. Maintain the system at these settings for 24 hours.
E.9 MAX Shutdown

1. After samples have been exposed to atomic oxygen for 24 hours, turn off the RF power using the button of the R301 generator.

2. Turn off the Mc2 controller.

3. Turn off the R301 generator.

4. The system will be at an elevated temperature, so either wait for the system to cool down or use protective gloves.

5. Disconnect the coaxial cable from the lid.

6. Close the nupro valve to the gas insertion line.

7. Close the chamber roughing valve using the switch on the front panel.

8. Turn off the roughing pump using the red switch on the pump.

9. Flip the pump power and mechanical pump power switches to the off position on the front panel.

10. Flip the vent valve switch to the "on" position (up) and fully open the nupro valve to the gas insertion line, while holding the chamber O-ring.

11. Once the chamber is vented to 700 Torr, flip the vent valve switch to the "off" position. Failing to do so will result in the lid to suddenly lift off the chamber with a loud popping sound.

12. Continue to vent the chamber through the gas insertion line until the chamber reaches atmospheric pressure.

13. Once the chamber has reached equilibrium, use the hoist switch to raise the lid, holding the coaxial cable out of the way in order to reduce strain on it.
14. Remove the glass wall and remove the samples and witness samples from the slotted plate using nitrile gloves.

**E.10 Junior**

1. Secure the witness samples inside Junior’s desiccator.

2. Place the lid on the desiccator.

3. Ensure that the ball valve for the airline between the desiccator and the roughing pump is in the open position.

4. Ensure the venting plug on the desiccator lid is closed.

5. Turn on the roughing pump using the switch on the pump.

6. Ensure that the desiccator is under vacuum by gently lifting the desiccator by the lid.

7. Maintain this condition for at least 48 hours.

8. After 48 hours, turn off the roughing pump using the switch on the pump.

9. Twist and pull up on the vent plug until you start to hear air entering the desiccator. Avoid pulling the vent plug too far as excess air will cause samples inside Junior to fly around.

10. Once the pressure inside is in equilibrium with the atmosphere, the lid should lift off the desiccator easily.

**E.11 FTIR**

1. Use nitrile gloves when handling samples.
2. Ensure the switch on the back of the spectrometer is in the "on" position and the BioRad USB is plugged into the back of the computer monitor.

   - If the switch on the back of the spectrometer is not in the "on" position, flip it on. The spectrometer will take approximately fifteen minutes to warm up afterwards.

3. Turn on the computer tower and computer monitor.

4. The green LED next to the "resume" option on top of the spectrometer should be illuminated. If so, turn the power switch on top of the spectrometer to the "on" position. The green LED next to "bot" should illuminate. Wait for approximately fifteen seconds after for the green LED next to "laser" to illuminate (listen for beeps).

5. Open the Spectra Management program on the computer.

   - Spectra Management will not open if the spectrometer has been on and idle for an extended period of time. If this is the case, turn the switch on top of the spectrometer to the "off" position and repeat step three.

6. Open the spectra measurement option on the left side menu.

7. Open the lid of the spectrometer. twist the black nob counterclockwise to rotate the arm away from the sample surface.

8. Using acetone and Kimwipes, lightly wipe down the center of the sample surface and bottom tip of the arm.

9. with the arm rotated away from the sample surface and no samples on the sample surface, close the lid of the spectrometer.
10. Click on the red "B" icon on the computer screen to conduct the background scan. The background scan is only for calibration, and data from the background scan does not need to be recorded.

11. Once the background scan is complete open the lid of the spectrometer and place the sample on the center of the sample surface.

12. position the arm over the sample and rotate the black knob clockwise to until the arm makes contact with the sample.

13. Close the lid of the spectrometer.

14. Click on the "S" icon with the red arrow under it on the computer screen to run an IR scan of the sample. Once the scan is complete, a graph of the spectra will pop up.

15. Save the spectra as a csv file.

16. remove the sample from the spectrometer and wipe down the sample surface and bottom tip of the arm with Kimwipes and actone.

17. Exit out of the software program, and then turn the switch on top of the spectrometer to the "off" position (do not turn this switch off before exiting out of the software).

E.12 ImageJ

1. Take a picture of the witness samples irradiated with atomic oxygen with a ruler in the picture.

2. On a computer, open the ImageJ.exe software.
3. In the ImageJ window that pops up, select File → Open and select the photo of the witness samples with a ruler. The photo should pop up.

4. In the ImageJ window select the straight line icon and create a straight line on the picture that goes from one millimeter tick on the ruler to an adjacent millimeter tick.

5. Select Analyze → Set Scale. In the new window that appears, insert “1” in the known distance box and “mm” in the unit of length box, then select “okay”. This gives ImageJ pixel to mm conversion to use with the photo.

6. Select Image → Color → split channels to produce three images of the picture that are solely the red channel, the green channel, and the blue channel, respectively.

7. Select a channel to use to approximate the irradiated area of the witness samples
   - Generally, the red channel results in the best distinction between the irradiated and pristine portions of the witness samples, though if the lighting of the picture results in difficulty distinguishing between the two, the other two channels may provide a more stark contrast.

8. Select the polygon selections icon and on the single color channel image of choice, outline the perimeter of the irradiated area of a witness sample.

9. Select Image → Adjust → Threshold. In the new window that appears, drag the bottom slider until all of the pixels within the perimeter of the irradiated area are highlighted red.

10. Select Analyze → Analyze Particles. In the new window that appears, ensure that the ”display results” box is checked, then select ”ok”. Another window will appear with the resulting area of the witness samples that was exposed
to atomic oxygen in mm\(^2\) that can be used to calculate effective fluence once converted to cm\(^2\).

11. Repeat steps 8-10 for any other witness samples in the image.

E.13 Microbalance Scale and Antistatic Gun

1. Use nitrile gloves when handling samples and UHV foils

2. Ensure that the microbalance is on. If it is not on, turn the microbalance on by pressing the power button on the front panel. The scale will take 24 hours to calibrate before measurements can be made.

3. Read the display and check the the display reads zero without any samples on the scale. If the scale does not read zero, press the tare button on the front panel and wait for the display to read zero.

4. Open the draft shield and, using Teflon-tipped forceps, gently place the sample or foil on the scale.

5. Close the draft shield.

6. Wait for the scale to read a consistent value, then record the displayed mass.

   • If the sample is a dielectric, then use the anti-static gun to neutralize the sample.

     (a) Remove the glass top from the draft shield.

     (b) Remove the cap from the anti-static gun and aim it down at the sample about four to six inches away.

     (c) Slowly pull the trigger all the way down to the handle and then slowly release the trigger while still pointing the gun at the sample. A clicking
sound will indicate that the trigger was pulled or released too fast and that the process needs to start over.

(d) Repeat this process until the scale drift is minimized.

7. Open the draft shield and remove the sample or foil with the forceps, then repeat the measurement process two more times. Average the three mass readings.

8. Once the mass of all samples and foils are recorded, the process is complete.

Do not turn off the microbalance scale unless directed so by Dr. Abercromby.